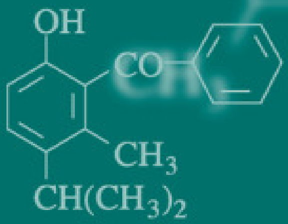
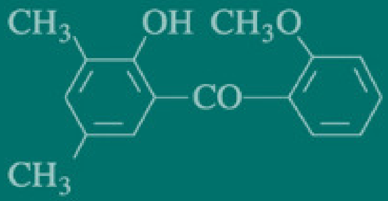
Aromatic Hydroxyketones:
Preparation and Physical Properties 1

3rd Edition





Robert Martin

Hydroxybenzophenones

Springer

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This book is dedicated to Canon Joseph Clerc priest at Montmorot (Jura, France, 1899-1985) who in 1944 gave a home to the Martin brothers, young Parisians refugees.

Robert Martin

Foreword

It is my great pleasure to write a foreword for Dr. R. Martin's handbook on Fries rearrangement and their aromatic hydroxyketones products. The present work is the result of numerous years of experience in compiling and editing data.

I have known Dr. Martin since my arrival at the Curie Institute in the 1990s and his enthusiasm was most encouraging, setting a good example of passion for chemistry. Although being retired from industry, he deliberately came to work in our team, not only for bibliographical work but also for working at the bench.

In this work, special effort has been made to select material suitable to meet the needs of chemists who do not benefit from unlimited time for specialized research in the field of hydroxyacetophenones and hydroxybenzophenones. These compounds are precursors of substituted aromatic derivatives which are often not straightforwardly obtained but have many potential applications in fine and medicinal chemistry.

I recognize that it represented a huge effort for Dr. Martin to produce such an exhaustive compilation within the limits set to him by the economy of space. This work will primarily be of great value to professional chemists, from physicists to pharmacists, who are often called upon to solve problems about the synthesis of this kind of aromatic compounds.

In this handbook set, the reader will find four independent parts which successively cover the topics of monoaroyl and polyaroylphenols in Volume 1, hydroxyacetophenones in Volume 2, substituted hydroxyacetophenones in Volume 3 and hydroxypropiophenones, hydroxyisobutyrophenones and hydroxypivalophenones in Volume 4.

For such needs as described above, this compilation will supply helpful and easy-to-read information, even in our computerized era.

Dr. Jean-Claude Florent
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Synthesis and Targeting of Biomolecules"
Institut Curie, Paris

Preface

Aromatic hydroxyketones are widely used as starting materials in organic synthesis to obtain medicines, dyes, perfumes, plastics, explosives, preservatives, etc. and, what is more, many of them also offer very specific uses.

This Handbook contains information on Hydroxybenzophenones, Hydroxyacetophenones, Hydroxypropiophenones, Hydroxyisobutyrophenones, Hydroxypivalophenones and numerous methylethers, collecting more than 6,000 ketones of which an indicated preparation as well as their physico-chemical and spectrochemical data are presented. This updated study now also covers the literature from 1890 until December 2008, including *circa* 8,500 references.

Aromatic Hydroxyketones: Preparation and Physical Properties is presented in dictionary style, with a logical classification of the ketones, which makes the information easily available for consultation. Ketones are classified methodically. As a result, they are easily accessible to the reader from three tables provided at the end of Volume 4: the molecular formula index, the Chemical Abstracts Registry Numbers and the usual names index.

This is the reason why it seemed interesting to update the contents of the four previous volumes and regroup them into one big handbook set. In this format, it will be more convenient for its main users, academics and industrial chemists.

Robert Martin
Institute Curie, France

Acknowledgements

I wish to express my heartfelt thanks to Dr. Pierre Demerseman who accepted me in his Laboratory at Institut Curie in 1987, and kindly revised my manuscript.

I am also grateful to Dr. Jean-Pierre Buisson, always so amiable and efficient, whose knowledge of word-processing largely contributed to the final page-setting of this work.

My thanks are also directed to Prof. Claude Monneret, formerly Head of the Chemical Department at Institut Curie, who has always been so benevolent to me.

I acknowledge as well his successor to the management of the laboratory, Dr. Jean-Claude Florent, who maintains the tradition and always welcomes me with much kindness, and all his collaborators for their warm welcome at each of my visits. The foreword of this Handbook was also written by Dr. Jean-Claude Florent. I most appreciate this mark of kindness.

I thank my son Serge Martin for friendly advice on the English edition of this book. Moreover, Mr. Serge Martin was a constant aid to me as regards data processing.

Various friends who readily agreed to translate foreign publications are also to be acknowledged here, in particular Dr. Jean Burkhard who has been of invaluable help for translating German papers over the last 30 years. The diverse abbreviations used in ancient reviews – particularly *Chemisches Zentralblatt* – had no secrets for him. Unfortunately, he left us in 2001 at the age of 91.

In this connection, thanks are due to Mrs. Feiga Weisbuch for her precious assistance as regards Rumanian and Russian texts, Mrs. Elisabeth Matarasso-Tchiroukhine as regards German and Russian texts as well as to Miss Marie-Françoise Liachenko, Drs. Daniel Dauzonne and Frédéric Schmidt. I wish to express my thanks to Mrs. Mireille Guyonneau, Mrs. Françoise Boucheron and my grandson Julien Martin for their contribution to my bibliographic research.

Before closing, I would like to remember my dear departed. My affectionate thoughts are turned towards Prof. Léon Denivelle who transmitted to me his passion for aromatic organic chemistry in 1945, and Prof. Albert Kirrmann who accepted me among his students in 1961 and was always so amiable and well-disposed whenever I went to him. I cannot mention without emotion Prof. Albert Saint-Maixen who largely communicated to me his knowledge of analytical chemistry.

I also have a personal thought towards my friends from the industry who left us too soon. I am particularly thankful to Drs. Henri Barbier, Félix Lepors and Henri Ruelleux (SPCA, Ltd.) who gave me the practical means to carry out my work on aromatic hydroxyketones. In this firm, I started my research on the Fries reaction. I also wish to acknowledge the late Dr. François Krausz who, at that time, made me benefit from his precious advice.

Last but not least, I am in debt beyond words to Angèle, my life for almost 60 years. Without her unfailing affection, support and understanding, nothing had been possible.

Short Biography

Robert Martin graduated as engineer from CNAM, then as doctor-engineer and doctor es sciences (Ph.D.) from Paris University. He studied with professors Léon Denivelle and Albert Kirrmann.

After having worked in the pharmaceutical industry, Robert Martin completed his career of organic chemist at a Research Laboratory of the French CNRS, located in the Curie Institute in Paris.

He has been studying the Fries reaction since 1956 without interruption. He has prepared a considerable number of aromatic hydroxyketones. A large part of these are included in the reference NMR and IR spectra collection of Sadtler (Philadelphia, USA).

His research on aromatic hydroxyketones gave rise to about 40 publications between 1963 and 1992, some of them in collaboration with Mainz University (Germany) and others with Institut Curie (Paris).

In 1992, he published a review on the Fries reaction in *Organic preparations and Procedures International*. This was followed by two books dealing with aromatic hydroxyketones, published by Kluwer in 1997 and 2000, then a third book by Springer in 2005.

For his various works concerning aromatic hydroxyketones he received the silver gilt medal from the Société d'Encouragement à l'Industrie Nationale in 1985.

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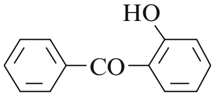
Part I
Monoaroylphenols

Chapter 1
Unsubstituted Hydroxybenzophenones *(Class of METHANONES)*

1.1 Monohydroxybenzophenones

(2-Hydroxyphenyl)phenylmethanone

[117-99-7] $C_{13}H_{10}O_2$ mol.wt. 198.22



Syntheses

- Preparation by Friedel–Crafts acylation of benzene in the presence of aluminium chloride,
  - with 2-hydroxybenzoyl chloride (salicylic acid chloride) [1], (52%) [2], (39%) [3], at temperature <60° [4];
  - with o-anisoyl chloride [5–12], (65%) [13], (57%) [14], (53%) [15], (37%) [16], (30%) [17]. Demethylation occurred during the Friedel–Crafts acylation, especially in the presence of ferric chloride at 130–140° [5];
  - with 2-ethoxybenzoyl chloride in a boiling water bath (42%) [13].
- Preparation by dealkylation,
  - of 2-methoxybenzophenone,
    with aluminium bromide in refluxing benzene for 4 h (96%) [18];
    with 48% hydrobromic acid in boiling acetic acid for 2 h [19];
    with boron tribromide in methylene chloride at r.t. for 12 h [20], according to [21];
  - of 5-tert-butyl-2-methoxybenzophenone with aluminium chloride in benzene at 65–70° for 45 h (79%) [9,22]. The starting keto ether was obtained by reaction of benzoyl chloride with 4-tert-butylanisole in the presence of zinc chloride in refluxing tetrachloroethane for 40 h [22];
  - of 5-tert-butyl-2-hydroxybenzophenone with aluminium chloride in benzene at 65–70° for 45 h (63%) [9].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_1,

- Preparation by Fries rearrangement of phenyl benzoate,
  - with aluminium chloride [23],

    without solvent

    at 200° for 20 min [20] or for 15 min (26%) [24], at 190° for 7 h (26%) [25], at 140° (24%) [26] or at 140° for 15 min (27%) [27], at 130° for 1 h, then at 160° for 1 h (30%) [28], between 100° and 150° for 3 h (minor product) [29], or at 63° for 4 h (<2%) [30],

    with solvent

    in nitrobenzene at 98° for 5 h (<9%) or at 120° for 2 h (15%) [31];
  - with an aluminium chloride and sodium chloride mixture at 140–200° (37%) [26];
  - with aluminium bromide in chlorobenzene at 110° for 15 min (2%) or for 4 h (20%) [32];
  - with aluminium iodide in refluxing acetonitrile (82°) for 10 h (23%) [33];
  - with boron trifluoride-etherate in the presence of phenol in refluxing benzene for 3 h (68%) [34];
  - with ferric chloride in a boiling water bath for 6 h [35];
  - with titanium tetrachloride in nitrobenzene at 60° for 18 h (7%) [27] or in refluxing nitromethane for 30 min (15%) [36];
  - with TFMS (trifluoromethanesulfonic acid) in tetrachloroethane at 170° for 24 h in a sealed tube (39%) [37];
  - with Nafion-H, a polymeric perfluorinated resin sulfonic acid, in refluxing nitrobenzene for 12 h (24%) [38];
  - with Nafion-XR, a H^+-form ion exchange resin, at 150° for 4 h (23%) [39];
  - with polyphosphoric acid at 100° (1%) [40];
  - with K10 montmorillonite, in refluxing N,N-dimethylformamide for 8 h (17%) [41] or in the presence of Na-exchanged montmorillonite clay as catalyst for 5 h at 140° (24%) [42].
- Preparation by saponification of 2-(benzoyloxy)benzophenone [43], with sodium hydroxide in refluxing dilute ethanol for 1 h [44] or for 10–15 min (45%) [45] or with potassium hydroxide [46] in methanol at 15° for 24 h (70%) [47]. The starting keto ester was obtained by oxidation of 2-benzylphenyl benzoate with chromium trioxide in boiling acetic acid [43], of 2,3-diphenyl-benzofuran [44,45] and of 2-(4-methylphenyl)-3-phenylbenzofuran or 2-(4-methoxyphenyl)-3-phenylbenzofuran [46]. The starting keto ester can be also obtained by photooxidation of 2,3-diphenylbenzofuran in chloroform in the presence of methylene blue as initiator [47].
- Also obtained by reaction of benzotrichloride with phenol in the presence of aqueous sodium hydroxide in a water bath (14%) [48], (1%) [49].
- Preparation by reaction between phenyloxymagnesium bromide complexed with HMPT and benzaldehyde in refluxing benzene for 48 h (30%) [50].
- Also obtained by reaction of salicylaldehyde with iodobenzene by using a catalyst system of palladium chloride/lithium chloride in the presence of sodium carbonate in N,N-dimethyl-formamide at 100° for 3.5 h (91%) [51].

- Also obtained by reduction of o-hydroxybenzophenone 2,4-dinitrophenylhydrazone with stannous chloride dihydrate in the presence of concentrated hydrochloric acid in boiling dilute acetic acid for 1 h (73%) [52].
- Also obtained (poor yields) by action of benzoic acid with phenol,
  - in the presence of polyphosphoric acid at 100° (1%) [40];
  - in the presence of Amberlyst-15 in refluxing chlorobenzene for 48 h (10%) [53].
- Also obtained by action of benzoyl chloride,
  - with phenol in the presence of aluminium chloride or titanium tetrachloride in nitrobenzene at 60° for 18 h (6–7%) [54];
  - with phenyl borate in the presence of aluminium chloride in refluxing carbon disulfide for 2 h (6%) [55].
- Also obtained (poor yield) by diazotization of 2-aminobenzophenone, followed by hydrolysis of the diazonium salt formed^{T} [7,56], (6%) [57] or by thermal decomposition of 2-benzoyl-benzenediazonium fluoborate in dilute sulfuric acid between 25° and 50° (37%) [17]; ^{T}In these conditions, the fluorenone was the major compound formed [7].
- Also obtained (poor yield) from o-bromophenyl benzoate on treatment with n-butyllithium in a mixture of ethyl ether, hexane and tetrahydrofuran at −70° for 2 h, followed by treatment with saturated aqueous ammonium chloride (7%) [58,59].
- Also obtained (poor yield) by pyrolysis of o-(allyloxy)benzophenone at 750°, under pressure of 1×10^{-3} Torr, for 2 h (8%) [60].
- Also obtained by cleavage of phenylindoxazene with fuming hydriodic acid (d = 1.70) in the presence of red phosphorous at 140–160° for 6–7 h. The intermediate compound formed (2-hydroxybenzophenone imine), unstable, gave by hydrolysis the expected ketone [61].
- Preparation from thioxanthen-9-one 10,10-dioxide (SM) by a three-steps synthesis: refluxing SM in 2% sodium hydroxide-65% dioxane-water solution for 4 h gave the 2-(2-hydroxybenzoyl)-phenylsulfinic acid (25%). The former, by reaction with mercuric chloride in refluxing aqueous acetic acid for 4 h, led to the 2-chloromercuri-2′-hydroxybenzophenone (69%). Removal of the chloromercury group was achieved with concentrated hydrochloric acid in refluxing ethanol for 2 h (91%) [62].
- Also obtained by heating xanthone with lead monoxide [7].
- Also obtained by action of an alkaline solution of sulfur on 4-nitrodiphenylmethane [63].
- Also obtained by photo-Fries rearrangement of phenyl benzoate,
  - in methanol (3%) [64], (48%) [65], using 3000 Å lamp (35%) or using 2537 Å lamp (41%) [66];
  - in isopropanol (35%) [66], (<4%) [64];
  - in butanol (48%) [66];
  - in ethanol at 30° for 3 days (20%) [67] or at r.t. for 70 h (18%) [68];
  - in water (50%) [69]. Adding amylose, α-cyclodextrin or β-cyclodextrin does not improve the yield (45%, 34% and 18% yields, respectively) [69], although in the presence of β-cyclodextrin, a 99% yield has been obtained [65]; in water

in the presence of soluble starch (67%) [69] or in the presence of SDS (sodium dodecyl sulfate), a micelle, was obtained a 74% yield [70];

- in hexane in the presence of zeolites for 1 h (92–98%) [71];
- in benzene for 12–24 h (55%) [65] or at 52° for 8 h (13%) [72];
- in cyclohexane at 55° for 24 h (11%) [72];
- in pentane (14%) [64]. Adding silica gel does not improve the yield (12%);
- in dioxane at 61° for 24 h (13%) [72];
- in ethyl ether (11%) [66];
- without solvent on K10 montmorillonite using microwave radiations (640 W, 10 min) (25%) [41].

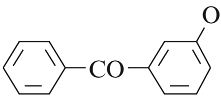
– Also obtained by photo-Fries rearrangement of phenyl salicylate in ethanol for 76 h (19%) [68].
– Also refer to: [58,60,73–87].

N.B.: Na salt [88].

oil [18];
m.p. 41° [19,25,44,49], 40–41° [14,57], 39–41° [2],
39–40° [7,10,47], 39° [6,13,15,27,52,89],
38°8 [90], 38°5–39° [17,91], 38–39° [9,22,41,62,70],
38° [26,46,50], 37°5–38° [3], 37–39° [92], 37° [24,93–95],
36°5–37° [72], 36°5 [96], 36–38° [4], 36° [61], 35–36° [97];
b.p.$_{0.2}$ 124–126° [60], b.p.$_{1.5}$ 127–133° [12], b.p.$_{1.9}$ 136–138° [62],
b.p.$_{12}$ 170–185° [9], b.p.$_{14}$ 175° [16], b.p.$_{15}$ 177° [48],
b.p.$_{10}$ 200° [35], b.p.$_{560}$ 250° [61];
^{1}H NMR [41,47,50,60,94,95,97–102];
^{13}C NMR [60,97,101];
IR [10,41,44,47,50,91,94,95,97,100,103–108]; UV [10,91,93,99,109–113]; MS [50,114];
pK_a [93,96,104,115]; TLC [116];
polarographic study [117]; thermal behaviour [94,95].

(3-Hydroxyphenyl)phenylmethanone

[13020-57-0] $C_{13}H_{10}O_2$ mol.wt. 198.22



Syntheses

– Preparation by aromatization of 5-benzoyl-2-cyclohexenone,

- in the presence of 10% Pd/C in refluxing xylene for 6 h (60%) [118];
- in the presence of lithium bromide and cupric bromide in refluxing acetonitrile for 1 h (75%) [118].

– Preparation by reaction of m-anisoyl chloride with benzene in the presence of excess aluminium chloride first at 20°, then at reflux for 2 h (95%) [119].
– Preparation by demethylation of m-methoxybenzophenone (SM),

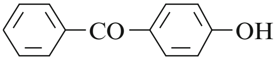
- with boron tribromide in methylene chloride (55–60%). SM was obtained by condensation of m-methoxybenzoyl chloride with benzene in the presence of aluminium chloride [3];
- with aluminium bromide in refluxing benzene for 2 h (89%) [18];
- with 48% hydrobromic acid [13] in refluxing acetic acid [19], for 6 h (60%) [120]. SM was prepared by a two-steps synthesis: first, formation of 3-methoxybenzhydrol (SM1) by condensation of a Grignard reagent (prepared from m-iodoanisole) with benzaldehyde. Then, oxidation of SM1 by adding a solution of potassium dichromate in dilute sulfuric acid in an hot solution of SM1 in acetic acid and heating for 15 min in a water bath (60%) [120].

– Preparation by reductive deamination of 2-amino-5-hydroxybenzophenone [121].
– Preparation by diazotization of m-aminobenzophenone, itself obtained by reduction of m-nitro-benzophenone [11,122].
– Also obtained by treatment of m-bromophenol in tetrahydrofuran with tert-butyllithium in pentane for 15 min at −78° under argon, after which a solution of phenyl-N,O-dimethylhydroxamide in tetrahydrofuran was slowly added (68%). – Refer to: Chem. Abstr., **119**, 49010t (1993)^{T}.
– Also refer to: [123].

m.p. 118° [119], 117° [120], 116° [13,18,19,121,122], 115° [3], 114–116°^{T};
b.p.$_{20-30}$ 285–295° [120];
IR [124].

(4-Hydroxyphenyl)phenylmethanone

[1137-42-4] $C_{13}H_{10}O_2$ mol.wt. 198.22



Syntheses

– Preparation by condensation of benzotrichloride,

- with phenol in the presence of aluminium chloride in carbon disulfide at 0° (90%) [125] or in the presence of zinc oxide in a water bath [126];
- with p-bromophenol in 30% sodium hydroxide solution at 80–85° (81%) [127]. There is substitution of the bromine atom by the benzoyl group.

– Preparation by Fries rearrangement of phenyl benzoate,

- with aluminium chloride [128], (55%) [129],

  without solvent

  at 140° (usually for 15 min), (quantitative yield) [130], 88–91% [131,132], (69%) [26], (65%) [24], at 63° for 4 h (19%) [30], at 130° for 1 h and at 160° for 1 h (9%) [28],

  with solvent

  in nitrobenzene at 98° for 5 h (64%) [31], at 120° for 2 h (54%) [31] or at 45° for 3 h (4%) [133];

  in refluxing ethylene dichloride (84°) for 1.5 h (37%) [134];
- with an aluminium chloride and sodium chloride mixture, at 140–200° (50%) [26];
- with aluminium iodide in refluxing acetonitrile (82°) for 10 h (46%) [33];

- with aluminium bromide in chlorobenzene at 110° for 4 h (17%) [32];
- with ferric chloride in a boiling water bath for 6 h (28%) [35];
- with titanium tetrachloride at 60° for 18 h (83%) [27] or in refluxing nitromethane for 30 min (36%) [36];
- with stannic chloride at 120° for 18 h (7%) [14];
- with hydrofluoric acid at 55° for 4 h (70%) [135];
- with polyphosphoric acid at 80° for 2.5 h (25%) [136], at 100° (6%) [40] or in a boiling water bath for 30 min (6%) [137];
- with Nafion-H, a polymeric perfluorinated resin sulfonic acid, in refluxing nitrobenzene for 12 h (48%) [38];
- with Nafion-XR, a H^+-form ion exchange resin, at 150° for 4 h under nitrogen (33%) [39];
- with ion-exchanged stratified clay catalyst, in the presence of phenol, at 180° for 4 h (45%) [138];
- with K 10 Montmorillonite as catalyst for 5 h at 140° (80%) [42].

– Preparation by dealkylation,

- of p-methoxybenzophenone,
  with aluminium bromide in refluxing benzene for 4 h (95%) [18];
  with aluminium chloride at 200–210° for 1.5 h [139];
  with 48% hydrobromic acid in refluxing acetic acid (good yield) [19], for 12 h [140];
  with hydrochloric acid by heating between 145° and 150° during 3–4 h [122]. There is elimination of methyl chloride;
- of p-ethoxybenzophenone,
  with aluminium bromide in refluxing benzene for 4 h [18];
  with aluminium chloride [141]. The starting keto ether [141] was obtained by Friedel–Crafts acylation of phenetole with benzoyl chloride in the presence of aluminium chloride according to [139];
  with hydrobromic acid in boiling acetic acid for 2 days [142];
- of 3-tert-butyl-4-hydroxybenzophenone with aluminium chloride in refluxing benzene [143].

– Preparation by reaction of benzoyl chloride,

- with phenol,
  in the presence of aluminium chloride at 100–130° (77%) [26] or at 75° (19%) [144];
  in the presence of aluminium chloride in nitrobenzene at 60° (92%) [26], at 60° for 18 h (88%) [54] or at 45° for 3 h (22%) [133];
  in the presence of aluminium chloride in ethylene dichloride at 85° (5%) [26];
  in the presence of aluminium chloride in boiling carbon disulfide [145], (10%) [122];
  in the presence of titanium tetrachloride in nitrobenzene at 60° (76%) [26] or at 60° for 18 h (87%) [54];
  in the presence of zinc chloride (10%) [122] according to [146];

  - with phenyl borate in the presence of aluminium chloride in refluxing carbon disulfide for 2 h (45%) [55];
  - with p-nonylphenol in the presence of aluminium chloride at 160° for 30 min (57%) [147].

- Also obtained by reaction of benzoic acid with phenol,
  in the presence of polyphosphoric acid at 75° (91%) [26], at 75° for 3 h (51%) [148] or at 75° for 20 h (46%) [148]; in a boiling water bath for 10 min (<4%) [137]; at 100° (9%) [40] or at 100° for 20 min (16%) [149]; at 160° for 30 min (30%) [148];
  in the presence of boron trifluoride at 100° for 1 h (52%) [150];
  in the presence of stannic chloride at 120° for 18 h (20%) [14];
  in the presence of trifluoromethanesulfonic acid, in a tube stoppered, at r.t. for 1 h [151];
  in the presence of sulfophenyl group-containing polysiloxanes at 180° for 20 h (52%) [152];
  in the presence of Amberlyst-15 in refluxing chlorobenzene for 48 h (9%) [53].
- Also obtained by reaction of p-(benzoyloxy)benzophenone with phenol in the presence of aluminium chloride in refluxing ethylene dichloride for 4 h (91%) [134].
- Preparation by diazotization of p-aminobenzophenone, followed by hydrolysis of the diazonium salt obtained [6,61,122,142], (54%) [15].
- Also obtained by oxidation of p-hydroxydiphenylcarbinol with DDQ (2,3-dichloro-5, 6-dicyano-benzoquinone) in dioxane at r.t. for 15 min (82%) [153].
- Also obtained by reduction of p-hydroxybenzophenone 2,4-dinitrophenylhydrazone with stannous chloride dihydrate in the presence of concentrated hydrochloric acid in boiling acetic acid for 1 h (93%) [52].
- Also obtained during the particular Fries rearrangements of some aryl esters during which there is usually an elimination of alkyl group,

  - Also obtained by Fries rearrangement of p-nonylphenyl benzoate with aluminium chloride at 130–135° for 4 h (51%) [147];
  - Also obtained by Fries rearrangement of p-tert-butylphenyl benzoate with hydrofluoric acid at 55° for 1 h (26%) and at 25° for 2 h (15%) [135] or with aluminium chloride at 140° (major product) [154];
  - Also obtained (by-product) by Fries rearrangement of phenyl o-benzoyloxybenzoate with aluminium chloride at 180° for 3 h (26%) [155].

- Preparation by heating at 120–130° during 12 h a mixture of phenol, benzanilide imidochloride and aluminium chloride. The keto anil obtained was hydrolyzed with ice-cold hydrochloric acid (21%) [156]. The same reaction using anisole gave a 43% yield [156].
- Preparation by saponification,

  - of p-(benzoyloxy)benzophenone,
    with 10% sodium hydroxide at reflux for 3 h (good yield) [134];

with potassium hydroxide in ethanol [146,157,158], (51%) [128,159]. The starting keto ester was obtained by reaction of benzoyl chloride with phenyl benzoate in the presence of zinc chloride at 175–180° [128,157,159] or by reaction of benzotrichloride with the same ester in the presence of zinc oxide [157]. This same keto ester (m.p. 112°5) can be also prepared by heating a benzoyl chloride/phenol mixture in the presence of zinc powder [158];

- of p-(carbomethoxyoxy)benzophenone with N sodium hydroxide solution at reflux for 15 min [160].

– Also obtained by hydrolysis of,

- p-(chloroacetoxy)benzophenone with fuming nitric acid at r.t. for several days (27%) [161];
- p-benzoylphenyl 5-benzoylsalicylate [155].

– Also obtained by cleavage of benzaurine on heating its dilute aqueous sodium hydroxide solution (1%) in a water bath with bubbling air (44%) [162].

– Also obtained by photo-Fries rearrangement of phenyl benzoate,

- in isopropanol (41–42%) [64,66];
- in methanol (40%) [66], irradiation for 1 h (37–43%) [71] or for 12–24 h (30%) [65];
- in butanol (36%) [66];
- in ethanol, at 30° for 3 days (28%) [67] or at r.t. for 70 h (16%) [68];
- in water for 24 h (16%) [69]; adding soluble starch, amylose or α or β-cyclodextrin does not improve the yield (18–20%) [69]; in water, in the presence of SDS (sodium dodecyl sulfate), a micelle, one obtains a 23% yield [70];
- in benzene for 12–24 h (30%) [65], 1 h (37–43%) [71] or at 52° for 8 h (20%) [72];
- in hexane for 1 h (37–43%) [71];
- in pentane (13%) [64]; adding silica gel does not improve the yield (10%) [64];
- in cyclohexane at 55° for 24 h (29%) [72];
- in dioxane at 61° for 24 h (23%) [72];
- in ethyl ether (22%) [66].

– Also refer to: [79,84,163–168].

N.B.: K salt [169,170].

Isolation from natural sources

– From leaves of *Talauma mexicana* (Magnoliceae) [171];

– From leaves of *Yoloxochitl* (Talauma mexicana) [172].

m.p. 135–136° [14,130,133], 135° [19,27,52,93,140,160],
134–135° [148,156,162],
134° [3,24,26,35,113,126,139,146,150,157,172],
133°8–133°9 [159], 133°7 [142], 133°5–134°5 [125],

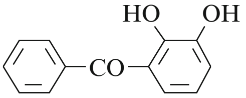
133–134° [70,155], 133–133°5 [15],
133° [6,18,137,145,170], 132°5 [173], 132–135° [174,175],
132–134° [136], 132–133°5 [134,135], 132–132°5 [91],
132° [72,149], 130–132° [67,161], 115–120° [131];
b.p.$_{24}$ 261° [142], b.p.$_{0.45}$ 168–170° [170];
^{1}H NMR [38,147,151], ^{13}C NMR [38],
IR [91,104,124,147,174,175], UV [91,93,110,113,176], MS [114,177];
pK_a [93,104,115]; TLC [116]; cryoscopic study [141].

1.2 Dihydroxybenzophenones

1.2.1 Hydroxy Groups Located on One Ring

(2,3-Dihydroxyphenyl)phenylmethanone

[52870-68-5] $C_{13}H_{10}O_3$ mol.wt. 214.22



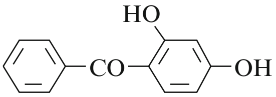
Syntheses

- Preparation by hydrolytic rearrangement of 2-benzoylacetyl-2,5-dimethoxytetrahydrofuran with refluxing 0.1 N hydrochloric acid in aqueous dioxane (95%) [178].
- Preparation by reaction of benzoic acid with pyrocatechol in the presence of Amberlyst-15 in refluxing chlorobenzene for 9 h (68%) [53].
- Preparation by refluxing 2,3-dimethoxybenzophenone with hydrobromic acid (d = 1.5) and acetic acid for 8 h [179].
- Also refer to: [180–182].

m.p. 65° [179], 64–65° [178]; Spectra (NA).

(2,4-Dihydroxyphenyl)phenylmethanone
(Resbenzophenone, Benzoresorcinol, Uvinul 400)

[131-56-6] $C_{13}H_{10}O_3$ mol.wt. 214.22



Syntheses

- Obtained by condensation of benzanilide with resorcinol in the presence of zinc chloride and phosphorous oxychloride at 130–140° for 1 h (25%) [183].
- Also obtained by condensation of benzamide with resorcinol in the presence of zinc chloride and phosphorous oxychloride at 100–140° for 1 h (22%) [183].
- The condensation of benzanilide imidochloride with resorcinol in the presence of aluminium chloride in ethyl ether gave a keto anil (92%). Which, by hydrolysis

with concentrated hydrochloric acid in refluxing ethanol for 3 h yielded the expected ketone (89%) [156].

- Also obtained by condensation of benzoic acid with resorcinol,
  - in the presence of hydrofluoric acid at 75° for 4 h in an autoclave (99%) [184];
  - in the presence of boron trifluoride at 160° for 2 h in a sealed tube (69%) [185] or in nitrobenzene at 80° for 30 min (96%) [186];
  - in the presence of stannic chloride at 140–150° for 30 min (30%) [183];
  - in the presence of zinc chloride (Nencki reaction), between 150° and 170° (for usually 15–30 min) [187–191], (20%) [192], (5%) [183];
  - in the presence of zinc chloride and phosphorous oxychloride at 65° for 3 h [193];
  - in the presence of zinc chloride and a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 40°. Then, during 1.5 h, phosphorous trichloride was added and the mixture heated at 60° for 16 h (91%) [194];
  - in the presence of phosphorous pentoxide at 120–130° for 15 min (poor yield) [183];
  - in the presence of Amberlite IR-120 at 160° for 2–3 h (25%) [195];
    Always by condensation of benzoic acid with resorcinol, but with stirring and azeotropic removal of water, in refluxing chlorobenzene (130°);
  - in the presence of ion exchange resins,
    - Dowex-50W-X-8 for 53 h (69%) [53];
    - Amberlyst-15 for 6 h (67%) [53], for 24 h (62%) [196]; the same reaction carried out in refluxing p-chlorotoluene (162°) for 2 h gave 59% yield [53];
    - Nafion-117 for 6 h (46%) [53] or for 24 h (45%) [196];
  - in the presence of Zeolite-H-beta for 72 h (42%) [53] or for 24 h (20%) [196]; the same reaction carried out in refluxing p-chlorotoluene (162°) for 16–18 h gave 70% yield [53] or in refluxing n-butylbenzene for 3 h under nitrogen gave 88% yield [53] and 40% yield [196];
  - in the presence of polyphosphoric acid or silicotungstic acid for 23 h (19%) [53];
  - in the presence of methanesulfonic acid for 23 h (17%) [53,196];
  - in the presence of phosphoric acid for 24 h (9%) [53,196].
- Also obtained by reaction of benzoyl chloride with resorcinol [197],
  - in the presence of zinc chloride and hydrochloric acid gas in chlorobenzene at 120° for 7 h (86%) [198] or without hydrochloric acid (72%) [198];
  - in the presence of aluminium chloride in ethylene dichloride at 65° for 5 h (75%) [199];
  - in the presence of aluminium chloride in nitrobenzene at r.t. for 48 h (70%) [200] or first at r.t., then at 70° for 5 h (83%) [201,202];
  - in the presence of aluminium chloride in refluxing carbon disulfide [145], for 4 h [203];

  - in the presence of Nafion-XR, a H+-form ion exchange resin, for 4 h under nitrogen at 160° [204] or at 150° (70%) [39].

- Preparation by demethylation of 2,4-dimethoxybenzophenone with aluminium bromide in refluxing benzene for 4 h (98%) [18]. Also obtained by using aluminium chloride in refluxing carbon disulfide for 30 min (3%) [205].
- Preparation by Fries rearrangement of,
  - resorcinol monobenzoate,
    with zinc chloride in the presence of hydrochloric acid gas in chlorobenzene at 120° for 7 h (88%) [198];
    with aluminium chloride at 180° for 2 h (68%) [155];
    with Nafion-XR, a H+-form ion exchange resin, at 150–160° for 4 h under nitrogen (72%) [39,204];
  - resorcinol dibenzoate with aluminium chloride between 100° and 150° [145].
- Preparation by condensation of benzoic anhydride with resorcinol,
  - in the presence of a few drops of concentrated sulfuric acid at 130° for some min (60%) [206];
  - in the presence of Amberlite IR-120, a cation exchange resin (sulfonic acid type), at 160° for 2–3 h (30%) [195];
  - in the presence of Zeokarb 225 at 160° for 2–3 h (30%) [195].
- Also obtained by decarboxylation,
- of 5-benzoyl-2,4-dihydroxybenzoic acid with dilute hydrochloric acid at 160–170° in a sealed tube [207] or by heating in a sealed tube [208];
- of 3-benzoyl-2,6-dihydroxybenzoic acid with a few drops of concentrated hydrochloric acid in boiling dilute acetic acid for 15–18 h [209].
- Also obtained by saponification of 2,4-di(benzoyloxy)benzophenone with 10% sodium hydroxide in refluxing ethanol for 2 h [203] or with potassium hydroxide in ethanol [210]. The starting diester was prepared by Fries rearrangement of resorcinol dibenzoate with zinc chloride at 100–120° [210].
- Also obtained by condensation of benzotrichloride with resorcinol,
  - in hot water [188] or in water at 65° for 4 h (42%) [202];
  - in 50% aqueous methanol at 70–80° (90%) [211] or in dilute methanol at 50° for 3 h (87%) [201,202];
  - in 50% aqueous isopropanol at 70–80° (90%) [211] or in dilute isopropanol between 30° and 80° for 2–4 h (80–84%) [202];
  - in 50% aqueous acetic acid solution (89%) [211] or in dilute acetic acid between 30° and 80° for 2–4 h (80–84%) [202];
  - in 50% aqueous dioxane solution (82%) [211] or in dilute dioxane between 30° and 80° for 2–4 h (80–84%) [202];
  - in ethanol [212] or in dilute ethanol between 30° and 80° for 2–4 h (80–84%) [202];
  - in dilute ethylcellosolve between 30° and 80° for 2–4 h (80–84%) [202];

- in hydrofluoric acid in the presence of water at −10° for 4 h, then at r.t. overnight (96%) [213]. The same reaction carried out in the presence of methanol or octanol at −10°, then at r.t. for 4 h yielded 96% and 93%, respectively [213].

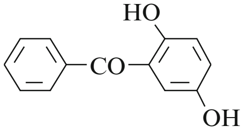
– Preparation by reaction of benzonitrile with resorcinol (Hoesch reaction) in the presence of zinc chloride and hydrochloric gas at 50° for 8 h (93%) [214] or during 12 days (89%) [215–217], or in ethyl ether at <5° for 3 h, then at 5° for 20 h (35%) [201,202].
– Also obtained by treatment of resorcinol with phenyl benzoate in the presence of boron trifluoride-etherate in refluxing benzene for 3 h (40%) [34].
– Also obtained from β-(4-benzoyl-3-hydroxyphenoxy)propionic acid by boiling for 5 min with 10% aqueous sodium hydroxide [218].
– Also obtained by photo-Fries rearrangement of resorcinol monobenzoate [219].
– Preparation by reaction of di(carbomethoxy)-β-resorcylic acid chloride with benzene in the presence of aluminium chloride at 60–70° for 1 h, then at 80° for 1 h (70%) [220].
– Also refer to: [78,84,146,168,221–233].

m.p. 148° [155], 146° [203], 145° [190,200,234–237],
144–146° [195,238], 144–145° [53,183,192,196,209],
144° [96,205,206,210,213,215], 143°5–144° [91],
143–144° [156,185,188,198,207,211,214], 143° [18,145,218],
142°6–144°2 [194], 142°5–143° [239], 142–144° [212], 141–143° [201,202];

^{1}H NMR [98], IR [91,201,202], UV [91,109–111,113,201,202,215,219,224,233, 235,236,240–243];
TLC [116,244]; HPLC [186,245]; pK_a [96,115];
polarographic study [117]; gel permeation chromatography [246,247];
vapour pressure [236,248].

(2,5-Dihydroxyphenyl)phenylmethanone ***(Quinbenzophenone)***

[2050-37-5] $C_{13}H_{10}O_3$ mol.wt. 214.22



Syntheses

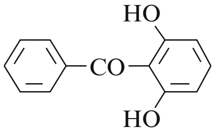
– Preparation by Fries rearrangement of hydroquinone dibenzoate with aluminium chloride [249,250], (good yield) [251], at 140° for 1 h (33%) [252], at 190–200° for 1 h 30 min (50%) [253] or at 200° for 20 min [20].
– Preparation by oxidation of 5-(benzoyloxy)-2,3-diphenyl-benzofuran with chromium trioxide in boiling acetic acid for 2 h, followed by saponification of the keto ester formed with boiling 8% sodium hydroxide in ethanol [254].
– Also obtained by hydrolysis of quinbenzophenone monobenzoate—5-(benzoyloxy)-2-hydroxy-benzophenone—(easily obtained by Fries rearrangement of hydroquinone mono or dibenzoate), with concentrated sulfuric acid at r.t. for overnight [252] or with 85% sulfuric acid [253].
– Also obtained by saponification of 2,5-di(benzoyloxy)benzophenone with alcoholic potassium hydroxide [255].

- Also obtained by reaction of benzoic acid with hydroquinone in the presence of boron trifluoride at 160° for 1 h in a sealed tube (61%) [185].
- Preparation by demethylation,
  - of 2-hydroxy-5-methoxybenzophenone with hydriodic acid in refluxing acetic acid and acetic anhydride mixture for 1.5 h (87%) [256];
  - of 2,5-dimethoxybenzophenone (SM),
    with boiling hydriodic acid [257];
    with an excess of hydrobromic acid [173];
    with boron tribromide in methylene chloride at 22° (78%) [258] or at r.t. for 12 h [20], according to [21]. SM was obtained by acylation of hydroquinone dimethyl ether with benzoyl chloride in methylene chloride in the presence of aluminium chloride at 0° (85%) [258].
- Preparation by reaction of phenyl benzoate with hydroquinone in the presence of boron trifluoride-etherate in refluxing benzene for 3 h (60%) [34].
- Also obtained by daylight irradiation of a 1,4-benzoquinone/benzaldehyde mixture [255] in benzene under nitrogen for 5 days (60%) [259].
- Also obtained by irradiation of α-hydroxybenzyl-1,4-benzoquinone in benzene for 4 days (65%) [260].
- Also refer to: [115,260–267].

m.p. 125–126°1 [256], 125° [249,250,253,255], 124–126° [257], 124–125° [252,254], 124° [185], 123–124° [260], 122° [173], 121–123° [259];
^{1}H NMR (Sadtler: standard n° 30288 M) [260],
IR (Sadtler: standard n° 57333) [260], UV [109–111];
TLC [116,259]; pK_a [115].

(2,6-Dihydroxyphenyl)phenylmethanone

[63411-81-4] $C_{13}H_{10}O_3$ mol.wt. 214.22



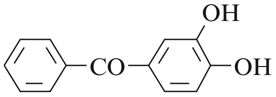
Synthesis

- Preparation by treatment of 8-benzoyl-7-hydroxy-4-methyl-coumarin (8-benzoyl-4-methylumbelliferone) [268,269] or of 8-benzoyl-7-hy droxy-4-phenylcoumarin (8-benzoyl-4-phenylumbelliferone) [192] with refluxing aqueous sodium hydroxide (64–78%) [268], (48%) [192].
- Also refer to: [270].

m.p. 135° [113,192,268]; ^{1}H NMR [98], UV [113].

(3,4-Dihydroxyphenyl)phenylmethanone

[10425-11-3] $C_{13}H_{10}O_3$ mol.wt. 214.22



Syntheses

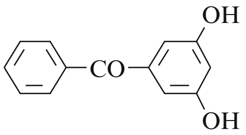
- Preparation by Fries rearrangement of pyrocatechol dibenzoate,

  - in the presence of aluminium chloride in nitrobenzene at 100° for 4 h (quantitative yield) [271] or heated on a steam bath for 6 h [272];
  - in the presence of Nafion-XR, a H^+-form ion exchange resin, at 175° for 4 h under nitrogen (38%) [39].

- Preparation in two steps by condensation of pyrocatechol with benzanilide imidochloride after acidic hydrolysis of the intermediate keto anil (30%) [156].
- Also obtained by demethylation,
  - of 3-hydroxy-4-methoxybenzophenone with boiling hydriodic acid (90%) [273];
  - of 3,4-dimethoxybenzophenone with boiling hydriodic acid [273] or with aluminium chloride in refluxing benzene for 6 h (64%) [274].
- Also obtained by reaction of benzoyl chloride with pyrocatechol dibenzoate in the presence of zinc chloride, followed by saponification of the 3,4-di(benzoyloxy) benzophenone formed with a boiling ethanolic sodium hydroxide solution [146].
- Also obtained by reaction of benzoyl chloride with pyrocatechol in the presence of phosphorous trichloride [275].
- Preparation by condensation of benzoic anhydride with pyrocatechol in the presence of zinc chloride at 180° for 5–6 h [276].
- Preparation by adding 30% hydrogen peroxide solution to the solution of 3-formyl-4-hydroxy-benzophenone in 10% sodium hydroxide and stirring the mixture at r.t. for 2 h (Dakin oxidation) (42%) [277].
- Also obtained by hydrolysis of 3-(benzoyloxy)-4-hydroxybenzophenone by heating gently with 50% sulfuric acid for 2 h (74%) [278].
- Also refer to: [232,279–283].

m.p. 148–149° [274], 147–148° [272], 145° [146],
134° [156,271,273,276,278], 133–134° [277];
hemihydrate [146], monohydrate [273,278];
Spectra (NA); pK_a [272]; molecular orbital studies [284].

(3,5-Dihydroxyphenyl)phenylmethanone



$C_{13}H_{10}O_3$ mol.wt. 214.22

Syntheses

- Preparation by reaction of diphenylcadmium with 3,5-di-acetoxybenzoyl chloride in refluxing benzene for 1 h. The keto ester obtained (66%) by saponification with refluxing 5% sodium hydroxide for 4–5 h gave the expected ketone (53%) [285].
- Also obtained by heating a mixture of 3,5-dicarbomethoxybenzoyl chloride and benzene in the presence of aluminium chloride, first at 70–75° for 1 h and at 75–80° for 45 min (37%). -Refer to: Chem. Abstr., **7**, 2563^2 (1913)^T.

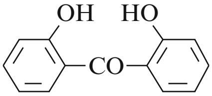
m.p. 160–162°^{T}, 148° (anhydrous) [285], 84° (monohydrate) [285];
Spectra (NA).

1.2.2 Hydroxy Groups Located on Both Rings

Symmetrical ketones

Bis(2-hydroxyphenyl)methanone

[835-11-0] $C_{13}H_{10}O_3$ mol.wt. 214.22



Syntheses

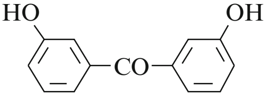
- Preparation by diazotization of 4,4′-diamino-2,2′-di-hydroxybenzophenone in diluted hydrochloric acid, followed by treatment with 50% phosphorous acid at 0° for 1 h, then at r.t. for 24 h (34%) [286].
- Also obtained by Fries rearrangement of phenyl o-hydroxybenzoate with aluminium chloride at 200° for 20 min [20].
- Also obtained from xanthone,
  - by heating with potassium hydroxide in ethanol at 180° for 4 h in a sealed tube [287,288];
  - by fusion with anhydrous potassium hydroxide [173,289], at 205–210° for 20 min [74], (64%) [290] or by heating at 200° (45%) [291].
- Also obtained by photo-Fries rearrangement of phenyl salicylate (salol) [219] or of phenyl carbonate in ethanol for 150 h (4%) [68].
- Also obtained by demethylation of 2,2′-dimethoxybenzophenone,
  - with boron trichloride in methylene chloride first at −70°, then at r.t. for 8 h (97%) [292];
  - with boron tribromide in methylene chloride at r.t. for 12 h [20], according to [21].
- Also obtained by hydrolysis of 2,2′-dihydroxybenzophenone ditosylate by treatment with 0.5 N sodium hydroxide in dilute methanol at r.t. for 8 days (98%) [293].
- Also refer to: [84,93,294–302].

N.B.: Ba, NH_4 [289] and K salts [288,289].

m.p. 62–63° [290], 59°5 [173], 59–60° [287,289,300,301],
55–57° [291], 54–56° [286]; b.p.$_{0.15}$ 128–132° [291];
^{1}H NMR [98,303], EPR [98], IR [291], UV [219,291], MS [114];
pK_a [115]; polarographic analysis [304]; HPLC [245].

Bis(3-hydroxyphenyl)methanone

[611-80-3] $C_{13}H_{10}O_3$ mol.wt. 214.22



Synthesis

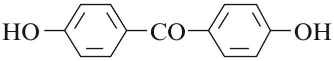
- Preparation from 3,3′-dinitrobenzophenone by reduction, diazotization of 3,3′-diaminobenzophenone obtained (m.p. 173–174°) [305], followed by hydrolysis of the diazonium salt obtained [288,301,305,306,307].

- Also refer to: [308,309].

  m.p. 163–164° [305], 162–163° [300,301]; IR [124].

Bis(4-hydroxyphenyl)methanone

[611-99-4] $C_{13}H_{10}O_3$ mol.wt. 214.22



Syntheses

- Preparation by demethylation of 4,4′-dimethoxy-benzophenone with aluminium bromide in refluxing benzene for 4 h (88%) [18].
- Also obtained by complete dealkylation of 4,4′-diethoxybenzophenone with hydrobromic acid in boiling acetic acid for 2 days [142].
- Preparation by hydrolysis of 4,4′-dichlorobenzophenone with aqueous sodium hydroxide in the presence of cuprous chloride or cupric chloride in an autoclave at 230–240° for 2 h (95–98%). The same reaction using cuprous oxide at 200° for 2 h gave a 82% yield [310,311]. Other methods using copper compounds for hydrolysis of the 4,4′-dichlorobenzophenone in the presence of sodium hydroxide [312,313], for 1 h at 270° in a steel autoclave (98–99%) [314].
- Preparation by Fries rearrangement of phenyl p-anisate (phenyl p-methoxybenzoate) with aluminium chloride at 140° (80%) [132] or at 160° [315].
- Also obtained by reaction of p-hydroxybenzoic acid with phenol,
  - in the presence of zinc chloride and a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 40°. Then, during 1.5 h, phosphorous trichloride was added and the mixture heated at 60° for 16 h (90%) [194];
  - in the presence of hydrofluoric acid at 75° in an autoclave (88%) [184];
  - in the presence of boron trifluoride in hydrofluoric acid (84%) [316] or in nitrobenzene at 80° for 30 min (67%) [186];
  - in the presence of polyphosphoric acid in a boiling water bath for 30 min (20%) [137] or at 100° for 20 min (47%) [149];
  - in the presence of trifluoromethanesulfonic acid at r.t. for overnight (93%) [151];
  - by heating in the presence of stannic chloride for 10 h [317].
- Also obtained by treatment of 4,4′-diphenoxybenzophenone with 16.7% sodium hydroxide at 300° for 30 min (57%) [318].
- Also obtained (poor yield) by Fries rearrangement of oxalic acid diphenyl ester with hydrofluoric acid in carbon tetrachloride at 80° for 4 h in an autoclave (<7%) [319].
- Also obtained by reaction of salicylic acid with phenol in the presence of stannic chloride (by-product) [317,320].

N.B.: According to Michael [317], the formation of this ketone was due to the presence of a small amount of p-hydroxybenzoic in the commercial salicylic acid used [321]. In contrast, Baeyer [320] claimed that this reaction proceeds via a transposition.

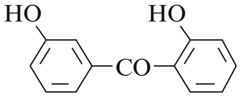
- Also obtained by oxidation of 4,4′-di-(benzoyloxy)diphenylmethane with chromium trioxide in refluxing acetic acid for 8 h, followed by saponification of the 4,4′-di-(benzoyloxy)benzophenone formed with potassium hydroxide in refluxing ethanol [301,322–324].
- Preparation by acylation of phenol with p-(trichloromethyl)phenyl p-(trichloromethyl)benzoate in methylene chloride in the presence of aluminium chloride at 0–5° over 1 h, then at r.t. for 1 h (yield 83%), followed by alkaline hydrolysis of the resulting keto ester (93–95%) [325].
- Also obtained by melting phenolphthalein,
    - with potassium hydroxide (75%) [326], according to [327];
    - with sodium hydroxide [190,328], (quantitative yield) [329].
- Also obtained by reduction of 4-hydroxy-4′-nitrobenzophenone with stannous chloride in the presence of hydrochloric acid, followed by diazotization of the 4-amino-4′-hydroxybenzophenone formed and hydrolysis of the diazonium salt obtained [322].
- Also obtained on treatment of aurin (p-rosolic acid) with water at high temperature, between 220° and 250° [163] or at >270° [330].
- Also obtained by treatment of rosaniline with water at high temperature (>270°) [330,331].
- Also obtained by reaction of carbon tetrachloride with phenol in the presence of zinc chloride at 120° (36%) [332].
- Also obtained by hydrogenation of 4,4′-dihydroxy-3,3′,5,5′-tetraiodobenzophenone in ethanol in the presence of 10% Pd/C and sodium acetate (66%) [333].
- Also refer to: [84,334–336].

N.B.: Na [88,314] and K salts [337].

m.p. 218° [151], 213°5 [142], 213–214° [18,333], 210°4–212° [194],
210–213° [316], 210–212° [338],
210° [163,190,237,300,301,311,323,324], 209–214° [184],
208–210° [322], 207° [137,149], 206° [113,315,319,328,329],
205°5–206°5 [317], 205–207° [330], 200° [331];
^{1}H NMR [151,316], IR [124,151,316,339], UV [113,333];
GLC [316]; HPLC [186,245]; polarographic study [304];
gel permeation chromatography [247].

Asymmetric ketones

(2-Hydroxyphenyl)(3-hydroxyphenyl)methanone



$C_{13}H_{10}O_3$ mol.wt. 214.22

Syntheses

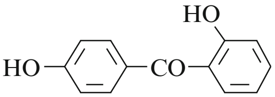
- Preparation by diazotization of 2,3′-diaminobenzophenone followed by hydrolysis of the obtained diazonium salt [301].

- Also obtained by Fries rearrangement of phenyl m-methoxy-benzoate (phenyl m-anisate) with aluminium chloride at 120° or 160° for 2 h [340].
- Also obtained (poor yield) by reaction of m-methoxybenzoyl chloride with phenol in the presence of aluminium chloride in nitrobenzene at 160° for 45 min (4%) [341].
- Preparation by demethylation of 2,3′-dimethoxybenzophenone in the presence of aluminium chloride in refluxing chlorobenzene for 1 h (86%) [341].
- Also refer to: [342,343].

m.p. 127° [340], 126° [301], 124–127° [341], 121–122° [300];
^{1}H NMR [341], IR [341], UV [341].

(2-Hydroxyphenyl)(4-hydroxyphenyl)methanone

[606-12-2] $C_{13}H_{10}O_3$ mol.wt. 214.22



Syntheses

- Preparation by diazotization of 2,4′-diamino-2′,4-di-hydroxybenzophenone, followed by decomposition of the diazonium salt obtained in the presence of 50% hypo-phosphorous acid (57%) [286].
- Preparation from 2-hydroxy-4′-nitrobenzophenone by reduction with stannous chloride and hydrochloric acid, followed by diazotization of the 4-amino-2′-hydroxybenzophenone formed and hydrolysis of the diazonium salt obtained [322].
- Preparation by hydrolysis of 2,4′-dichlorobenzophenone with sodium hydroxide for 1 h at 270° in a steel autoclave (98–99%) [314].
- Preparation by diazotization of 2,4′-diaminobenzophenone, followed by hydrolysis of the diazonium salt obtained [301].
- Also obtained by condensation of salicylic acid with phenol,
  - in the presence of stannic chloride at 115–125° (major product) [9,190,212,317,320,322,336,344];
  - in the presence of zinc chloride (poor yield) [317];
  - in the presence of hydrofluoric acid at 75° in an autoclave [184];
  - in the presence of boron trifluoride in nitrobenzene at 80° for 30 min (83%) [186];
  - in the presence of polyphosphoric acid at 100° for 20 min (2%) [149].
- Also obtained by Fries rearrangement,
  - of phenyl o-methoxybenzoate with aluminium chloride at 120–160° for 2 h [340], with aluminium chloride in the presence of phenol at 180° for 3 h (44%) [155];
  - of phenyl salicylate (salol),
    with aluminium chloride at 120–160° for 2 h [340], at 140° for 3 h (75%) [345] or at 180–182° for 3 h (53%) [155];
    with stannic chloride at 115–120° for 18 h (54%) [14];

  - of phenyl o-(benzoyloxy)benzoate with aluminium chloride in the presence of phenol at 180° for 3 h (36%) [155];
  - of o-(nicotinyloxy)phenyl benzoate with aluminium chloride at 140–145° for 2 h (by-product) (9%) [346].

– Preparation by dealkylation of 5-tert-butyl-2,4′-dimethoxybenzophenone with aluminium chloride in benzene at 65–70° for 45 h (75%) [9].
– Preparation by reaction of hydrobromic acid (d = 1.49) with 2,4′-di(4-methoxybenzoyloxy)-benzophenone in refluxing acetic acid for 2 h (86%) [19].
– Also obtained by hydrolysis of p-(salicyloyl)phenyl salicylate [346].
– Also obtained by treatment of 4-acetoxy-2′-methoxybenzophenone with aluminium chloride at 153–155° for 2 h (19%) [155]. There is simultaneously demethylation and hydrolysis of the acetoxy group.
– Also obtained by decarboxylation of 2-hydroxy-5-(salicyloyl)benzophenone by melting with potassium hydroxide (63%) [14].
– Also obtained by photo-Fries rearrangement,

  - of phenyl salicylate (salol) [219], in ethanol for 76 h (22%) [68];
  - of phenyl carbonate in ethanol for 150 h (11%) [68].

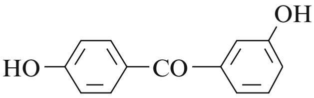
– Also refer to: [84,347–349].

N.B.: Na salt [88,314].

m.p. 150–151° [320], 150° [340], 148° [155,344,346], 147–149° [14],
147–148° [212], 146–147° [9,184], 145° [345], 144° [322],
143–145° [286], 143–144° [317,336],
142° [190,237,300,301], 141° [149], 138–141° [19];
^{1}H NMR (Sadtler: standard n° 38497 M),
IR (Sadtler: standard n° 65535 K), UV [219], MS [114];
gel permeation chromatography [302]; HPLC [186].

(3-Hydroxyphenyl)(4-hydroxyphenyl)methanone

[611-81-4] $C_{13}H_{10}O_3$ mol.wt. 214.22



Syntheses

– Obtained by Fries rearrangement of phenyl m-anisate without solvent in the presence of aluminium chloride for 2 h between 120° and 160° [340].
– Also obtained by reaction of m-hydroxybenzoic acid with phenol,

  - in the presence of polyphosphoric acid for 20 min at 100° (7%) [149];
  - in hydrofluoric acid at 30° under a pressure of 30 psi boron trifluoride in an autoclave for 4 h [350], (89%) [351].

– Also obtained by reaction of m-methoxybenzoyl chloride with phenol in the presence of aluminium chloride in nitrobenzene at 160° for 45 min (12%) [341].

- Preparation by diazotization of 3,4′-diaminobenzophenone (m.p. 121–122°) [305], followed by hydrolysis of the diazonium salt obtained [301,305].
- Preparation by demethylation of 3,4′-dimethoxybenzophenone with 48% hydrobromic acid in a acetic anhydride/acetic acid mixture (1:1) for 15 h at reflux (quantitative yield) [352].
- Also refer to: [353–355].

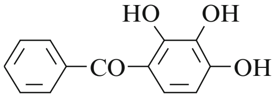
m.p. 203° [351], 200° [305], 198° [300], 197° [301], 196–197° [340],
195–200° [341], 195° [149], 193–194° [352];
Spectra (NA).

1.3 Trihydroxybenzophenones

1.3.1 Hydroxy Groups Located on One Ring

Phenyl(2,3,4-trihydroxyphenyl)methanone ***(Alizarine Yellow A)***

[1143-72-2] $C_{13}H_{10}O_4$ mol.wt. 230.22



Syntheses

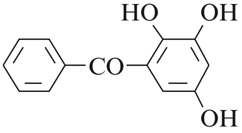
- Preparation by reaction of benzoic acid with pyrogallol,
  - in the presence of zinc chloride during 3 h at 145° [190,344,356];
  - in the presence of Amberlyst-15 (a strongly acid ion exchanger) in chlorobenzene during 10 h at 131–132° (60%) [357];
  - in the presence of Amberlite IR-120 or Zeokarb 225 (cation exchange resins, sulfonic acid type) during 3 h at 160° (14%) [195];
  - in the presence of boron trifluoride in ethyl ether at 0° (44%) [358,359].
- Preparation by reaction of benzoic anhydride with pyrogallol [195,344,356],
  - in the presence of concentrated sulfuric acid or polyphosphoric acid during 15 min at reflux (14%) [195];
  - in the presence of zinc chloride at 145° [344,356];
  - in the presence of Amberlite IR 120 or Zeokarb 225 during 3 h at 160° (16%) [195].
- Preparation by reaction of benzoyl chloride with pyrogallol [39,344,356],
  - in the presence of Nafion-XR, a H^+-form ion exchange resin, during 4 h at 150° under nitrogen (78%) [39];
  - in the presence of zinc chloride at 145° [344,356].
- Preparation by reaction of benzotrichloride with pyrogallol [360], in the presence of zinc chloride at 145° [344,361].
- Preparation by reaction of 2,3,4-triacetoxybenzoyl chloride with benzene in the presence of aluminium chloride at 40° for 4 h (55%) [362].

- Preparation in two steps by condensation of pyrogallol with benzanilide imidochloride after acidic hydrolysis of the intermediate keto anil (31%) [156].
- Also obtained by hydrolysis of gallacetophenone monobenzoate in the presence of concentrated sulfuric acid during 4 h into a cooling system (10%) [363].
- Preparation by Fries rearrangement of,
  - 2,3-dihydroxyphenyl benzoate with Nafion-XR during 4 h at 150° under nitrogen (81%) [39];
  - pyrogallol tribenzoate with aluminium chloride during 2 h at 160–170° (15%) [363].
- Also refer to: [267,280,281,364–368].

N.B.: K, Pb and Na salts are obtained according to [344].

m.p. 146° [358,359], 141–143° [195], 140–141° [156,344], 140° [363], 139–140° [190,237], 138–139° [362];
UV [190,359,369]; pK_a [369].

Phenyl(2,3,5-trihydroxyphenyl)methanone



$C_{13}H_{10}O_4$ mol.wt. 230.22

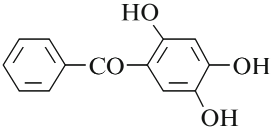
Synthesis

- Refer to: [220].

m.p. and Spectra (NA).

Phenyl(2,4,5-trihydroxyphenyl)methanone

[14894-91-8]



$C_{13}H_{10}O_4$ mol.wt. 230.22

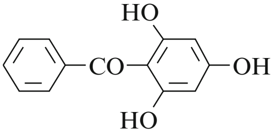
Synthesis

- Preparation by hydrolysis of 2,4,5-triacetoxybenzophenone with hydrochloric acid in refluxing ethanol for 2 h (77%) [256].

m.p. 129°1 [256]; Spectra (NA).

Phenyl(2,4,6-trihydroxyphenyl)methanone *(Phlorbenzophenone)*

[3555-86-0]



$C_{13}H_{10}O_4$ mol.wt. 230.22

Syntheses

- Preparation by Fries rearrangement of phloroglucinol tribenzoate with aluminium chloride at 160–170° for 2 h (95%) [363].
- Preparation by reaction of benzonitrile with phloroglucinol in the presence of zinc chloride and hydrochloric acid in ethyl ether at r.t. for overnight, followed by hydrolysis of the ketimine salt formed with dilute sulfuric acid [370], (65%) [371] (Hoesch reaction).

- Preparation in two steps by condensation of phloroglucinol with benzanilide imidochloride after acidic hydrolysis of the intermediate keto anil (39%) [156].
- Preparation by reaction of benzoyl chloride with phloroglucinol in the presence of aluminium chloride in a ethyl ether/nitrobenzene mixture [372].
- Preparation by condensation of benzoyl chloride with 1,3,5-trimethoxybenzene, followed by demethylation [373].
- Also obtained by reaction of phenyl benzoate with phloroglucinol in the presence of boron trifluoride-etherate in refluxing benzene for 3 h (25%) [34].
- Preparation by demethylation of 2,4,6-trimethoxybenzophenone with hydriodic acid (d = 1.7) in acetic anhydride at 115–120° for 4 h (83%) [374].
- Also refer to: [166,167,375,376].

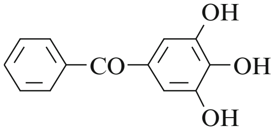
Isolation from natural sources

- From *Helichrysum triplinerve* (Asteraceae) [377];
- From *genus Leontonyx* [378].

m.p. 170–171° [374], 165° [34,156,363,371], 164–165° [372], 164° [370]; ^{1}H NMR [374], UV [370].

Phenyl(3,4,5-trihydroxyphenyl)methanone

[60487-86-7] $C_{13}H_{10}O_4$ mol.wt. 230.22



Synthesis

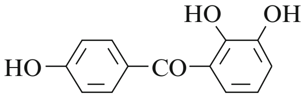
- Preparation by reaction of tri(carbomethoxy) galloyl chloride with benzene in the presence of aluminium chloride at 70–80° for 1.5 h, followed by saponification of the keto ester formed with N sodium hydroxide in a water bath for 15 min (65%) [160].
- Also refer to: [220,379].

m.p. 177–178° [160]; Spectra (NA).

1.3.2 Hydroxy Groups Located on Both Rings

(2,3-Dihydroxyphenyl)(4-hydroxyphenyl)methanone

[129726-78-9] $C_{13}H_{10}O_4$ mol.wt. 230.22



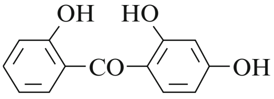
Synthesis

- Preparation by total demethylation of 2,3,4′-trimethoxy-benzophenone with hydrobromic acid (d = 1.5) in refluxing acetic acid [179].

m.p. 169° [179]; Spectra (NA).

(2,4-Dihydroxyphenyl)(2-hydroxyphenyl)methanone

[13087-18-8] $C_{13}H_{10}O_4$ mol.wt. 230.22



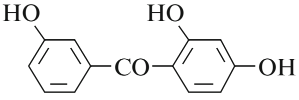
Syntheses

- Preparation by diazotization of 4,4′-diamino-2,2′-di-hydroxybenzophenone, followed by treatment of the diazonium salt formed with 50% hypophosphorous acid (26%) [286].
- Also obtained (poor yield) by action of salicylic acid on resorcinol at 195–200° for 15 h, without a dehydrating agent [190,317,336,344] or in the presence of zinc chloride and phosphorous oxychloride (40%) [380].
- Also obtained (poor yield) by condensation of o-acetoxybenzonitrile with resorcinol in the presence of zinc chloride and hydrochloric acid in ethyl ether at 0° (Hoesch reaction), followed by hydrolysis of the intermediate compound obtained with boiling 0.5 N sodium hydroxide (<5%) [381]. In this reaction, the major compound was 3-hydroxyxanthone (18%).
- Also refer to: [84,226] and [382] (Japanese patent).

m.p. 134–135° [380], 133–134° [317,336,381], 132–133° [286], 130–132° [190,237];
UV [190,303,380], MS [114]; TLC [116];
paper chromatography [383].

(2,4-Dihydroxyphenyl)(3-hydroxyphenyl)methanone

[837-60-5] $C_{13}H_{10}O_4$ mol.wt. 230.22



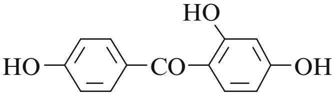
Synthesis

- Preparation by total demethylation of 2,3′,4-trimethoxy-benzophenone,
  - with 60% hydrobromic acid in refluxing acetic acid for 6 h [341,343], (87%) [291];
  - with aluminium chloride in boiling chloroform for 2.5 h [291].
- Also refer to: [293,384,385].

m.p. 178–182° [291], 178–180° [385];
^{1}H NMR [341], IR [291,385], UV [291,385], MS [385].

(2,4-Dihydroxyphenyl)(4-hydroxyphenyl)methanone

[1470-79-7] $C_{13}H_{10}O_4$ mol.wt. 230.22



Syntheses

- Preparation by reaction of 4-hydroxybenzoic acid with resorcinol,
  - in the presence of zinc chloride at 160° (Nencki reaction) [188,190];
  - in the presence of zinc chloride and phosphorous oxychloride for 4 days at r.t. (78%) [191];

- in the presence of zinc chloride and a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 27°. Then, during 2 h, phosphorous trichloride was added between 27° and 37° and the mixture heated at 60° for 16 h (quantitative yield) [194];
- in the presence of hydrofluoric acid at 75° in an autoclave [184].

– Preparation by reaction of β-resorcylic acid with phenol in the presence of zinc chloride and a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 27° [194], (40%) [380]. Then, during 2 h, phosphorous trichloride was added between 27° and 37° and the mixture heated at 60° for 16 h [194].

– Also refer to: [298,386–393].

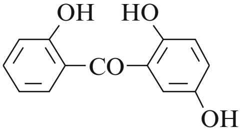
m.p. 203°4–204° [194], 200–201° [188,191], 200° [190,237], 199–200° [380];

dihydrate [188]; IR [394], UV [190,380,394], MS [114];

TLC [116], paper chromatography [383].

(2,5-Dihydroxyphenyl)(2-hydroxyphenyl)methanone

[183106-13-0] $C_{13}H_{10}O_4$ mol.wt. 230.22



Syntheses

– Preparation by total demethylation of 2,2′,5-trimethoxy-benzophenone with boron tribromide in methylene chloride at 0° under nitrogen atmosphere for 3 h (85%) [395].

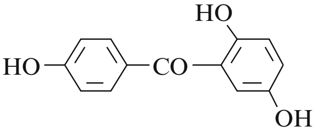
– Preparation by condensation of salicylic acid with hydroquinone in the presence of zinc chloride for 45 min at 125–140° (Nencki reaction). – Refer to: Chem. Abstr., **44**, 1271f (1950)^{T}.

m.p. 149–150° [395], 98°^{T}. One of the reported melting points is obviously wrong.

^{1}H NMR [395], IR [395], UV [395], MS [395]; TLC [395].

(2,5-Dihydroxyphenyl)(4-hydroxyphenyl)methanone

[120506-56-1] $C_{13}H_{10}O_4$ mol.wt. 230.22



Syntheses

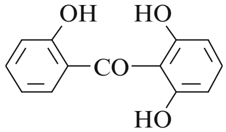
– Preparation by heating a mixture of 4-hydroxybenzoic acid and hydroquinone in nitrobenzene in the presence of boron trifluoride at 80° for 30 min (64%) [186]. **N.B.**: In the patent, this compound was erroneously called 3′,4,5′-tri-hydroxybenzophenone (assay 5, table page 3) [186].

– Preparation by Friedel–Crafts acylation of hydroquinone dimethyl ether with p-anisoyl chloride ($AlCl_3/CS_2$/ 3 h at r.t.), followed by demethylation ($AlCl_3$/ Toluene/ 1 h at 120°) (65%). -Refer to: Chem. Abstr., **44**, 1271f (1950)^{T}.

m.p. 162°^{T}; Spectra (NA).

(2,6-Dihydroxyphenyl)(2-hydroxyphenyl)methanone

[82-69-9] $C_{13}H_{10}O_4$ mol.wt. 230.22



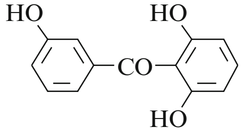
Synthesis

- Preparation by hydrolysis of 7-hydroxy-8-(o-hydroxybenzoyl)-4-methylcoumarin with sodium hydroxide in dilute ethanol (52%).
- Refer to: Chem. Abstr., **114**, 42490n (1991)^T.

m.p. 155°^{T}; IR^T.

(2,6-Dihydroxyphenyl)(3-hydroxyphenyl)methanone

[21554-76-7] $C_{13}H_{10}O_4$ mol.wt. 230.22



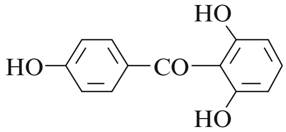
Synthesis

- Preparation by demethylation of 2,3′,6-trimethoxybenzophenone in the presence of aluminium chloride in refluxing chlorobenzene for 1 h (47%) [341].
- Also refer to: [342,343].

m.p. 124–125° [341]; IR [341], UV [341].

(2,6-Dihydroxyphenyl)(4-hydroxyphenyl)methanone

[131425-90-6] $C_{13}H_{10}O_4$ mol.wt. 230.22



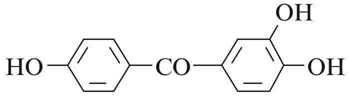
Synthesis

- Preparation by hydrolysis of 7-hydroxy-8-(p-hydroxy-benzoyl)-4-methylcoumarin (SM) with sodium hydroxide in refluxing dilute ethanol (57%). SM was obtained by Fries rearrangement of 7-(p-methoxybenzoyloxy)-4-methylcoumarin with aluminium chloride, first at 190°, then at 200° for 1.5 h (52%, m.p. 181°).
- Refer to: Chem. Abstr., **114**, 42490n (11991)^T.

m.p. 170°^{T}; IR^T.

(3,4-Dihydroxyphenyl)(4-hydroxyphenyl)methanone

$C_{13}H_{10}O_4$ mol.wt. 230.22



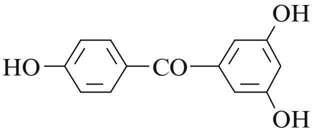
Synthesis

- Preparation by Friedel–Crafts acylation of pyrocatechol dimethyl ether with p-anisoyl-chloride ($AlCl_3$/CS_2/ 3 h at r.t.), followed by demethylation ($AlCl_3$/toluene/ 1 h at 120°) (65%).
- Refer to: Chem. Abstr., **44**, 1271f (1950)^T.

m.p. 205°^{T}; Spectra (NA).

(3,5-Dihydroxyphenyl)(4-hydroxyphenyl)methanone

[129020-58-2] $C_{13}H_{10}O_4$ mol.wt. 230.22



Synthesis

- Refer to: [396].

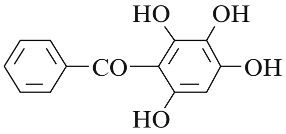
m.p. and Spectra (NA).

1.4 Tetrahydroxybenzophenones

1.4.1 Hydroxy Groups Located on One Ring

Phenyl(2,3,4,6-tetrahydroxyphenyl)methanone

[198879-06-0] $C_{13}H_{10}O_5$ mol.wt. 246.22



Synthesis

- Preparation by Friedel–Crafts acylation of 1,2,3,5-tetra-hydroxybenzene with benzoyl chloride (46%).
- Refer to: Chem. Abstr., **128**, 3540y (1998)^{T}.

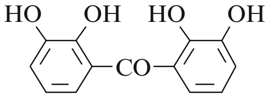
m.p. (NA); ^{1}H NMR^{T}, ^{13}C NMR^{T}, MS^{T}.

1.4.2 Hydroxy Groups Located on Both Rings

Symmetrical ketones

Bis(2,3-dihydroxyphenyl)methanone

[35042-50-3] $C_{13}H_{10}O_5$ mol.wt. 246.22



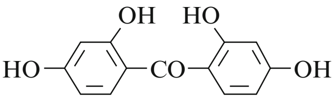
Synthesis

- Preparation by demethylation of 2′-hydroxy-2,3,3′-tri-methoxybenzophenone with 48% hydrobromic acid in refluxing acetic acid for 5 h (78%) [397].

m.p. 121–122° [397,398]; IR [397], UV [397].

Bis(2,4-dihydroxyphenyl)methanone *(Uvinul-D-50)*

[131-55-5] $C_{13}H_{10}O_5$ mol.wt. 246.22



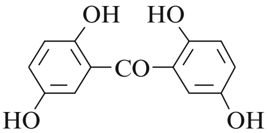
Syntheses

- Obtained (small amount) by melting a fluorescin chloride/sodium hydroxide mixture in the presence of a very few water at 230–240° for 2–3 h [399,400].
- Preparation by reaction of phosgene with resorcinol in the
- Presence of zirconium chloride in nitrobenzene at 70° (74%) [401].
- Preparation by Fries rearrangement of 3-hydroxyphenyl 2,4-dihydroxybenzoate with zirconium chloride in nitrobenzene at 70° (94%) [401].
- Preparation by reaction of β-resorcylic acid with resorcinol,
  - in the presence of zinc chloride and phosphorous oxychloride at 80–90° for 45 min, or at r.t. for 48 h (75%) [402], in sulfolane for 2 h at 50° (92%) [403];
  - in the presence of zinc chloride and a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 60° for 1 h. Then, during 1.5 h, phosphorous trichloride was added and the mixture heated at 60° for 8.5 h (85%) [194];
  - in the presence of boron trifluoride in nitrobenzene at 80° for 30 min (59%) [186].
- Preparation by condensation of 2,4-diacetoxybenzonitrile with resorcinol in the presence of zinc chloride and hydrochloric acid in ethyl ether (Hoesch reaction). The 2,4-diacetoxy-2′,4′-di-hydroxybenzophenone imine hydrochloride thus formed (m.p. 195° (d)) was hydrolyzed with boiling 25% aqueous sulfuric acid for 15 min [381], (33%) [404].
- Also refer to: [78,79,84,225,227,228,235,387,405–408].

m.p. 201° [235], 200–201° [194], 198° [236], 196–198° [402,404], 193–195° [113,399];
UV [113,235,236,240–242];
TLC [116]; HPLC [186]; vapour pressure [236];
gel permeation chromatography [247].

Bis(2,5-dihydroxyphenyl)methanone

$C_{13}H_{10}O_5$ mol.wt. 246.22



Syntheses

- Obtained by gently heating b-isoeuxanthone (2,7-di-hydroxyxanthone) with anhydrous potassium hydroxide [287].

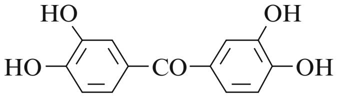
- This benzophenone can be obtained by total dealkylation of its tetramethyl ether by the usual methods. This one (m.p. 109°) was prepared by reaction of dimethylgentisic acid chloride (2,5-dimethoxybenzoyl chloride) with hydroquinone dimethyl ether (2,5-dimethoxybenzene) in the presence of aluminium chloride in carbon disulfide [409].

  N.B.: Pb salt [287].

  m.p. and Spectra (NA).

Bis(3,4-dihydroxyphenyl)methanone

[61445-49-6] $C_{13}H_{10}O_5$ mol.wt. 246.22



Synthesis

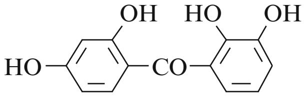
- Refer to: [280,281,309,410].

m.p. and Spectra (NA).

Asymmetric ketones

(2,3-Dihydroxyphenyl)(2,4-dihydroxyphenyl)methanone

[37728-10-2] $C_{13}H_{10}O_5$ mol.wt. 246.22



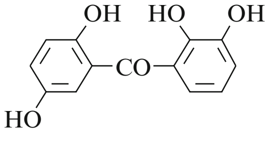
Synthesis

- Preparation by reaction of 2,3-dihydroxybenzoic acid with resorcinol in the presence of freshly fused zinc chloride and phosphorous oxychloride at 65–70° for 3 h (31%) [398].
- Also refer to: [79,280,411].

m.p. 160–161° [398]; IR [398], UV [398].

(2,3-Dihydroxyphenyl)(2,5-dihydroxyphenyl)methanone

[35040-37-0] $C_{13}H_{10}O_5$ mol.wt. 246.22



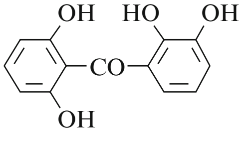
Synthesis

- Preparation by demethylation of 2-hydroxy-2′,3′,5-tri-methoxybenzophenone with 48% hydrobromic acid in refluxing acetic acid for 4 h (64%) [397].

m.p. 190–191° [397]; ^{1}H NMR [397], IR [397].

(2,3-Dihydroxyphenyl)(2,6-dihydroxyphenyl)methanone

[25577-01-9] $C_{13}H_{10}O_5$ mol.wt. 246.22



Syntheses

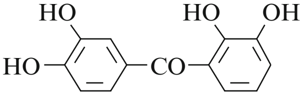
- Preparation by reaction of boron tribromide with 2,2′,3′-tris[(ethoxycarbonyl)oxy]-6-methoxybenzophenone (SM) in methylene chloride at r.t. for 5 h under nitrogen (61%) [412]. SM was obtained by reaction of ethyl chloroformate with 2,2′,3′-trihydroxy-6-methoxybenzophenone in the presence of potassium carbonate in refluxing acetone for 5–6 h (91%).

– Also obtained (by-product) by demethylation of 2,2′,3′,6-tetramethoxybenzophenone with boron tribromide in benzene at r.t. for 5 h (16%) [412].

m.p. 173–174° [412]; ^{1}H NMR [412], IR [412], UV [412], MS [412].

(2,3-Dihydroxyphenyl)(3,4-dihydroxyphenyl)methanone

[37728-15-7] $C_{13}H_{10}O_5$ mol.wt. 246.22



Syntheses

– Preparation by reaction of 2,3-dihydroxybenzoic acid with pyrocatechol in the presence of freshly fused zinc chloride and phosphorous oxychloride at 65–70° for 3 h (32%) [398].

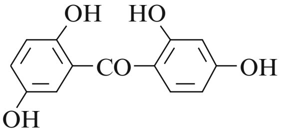
– Preparation by Friedel–Crafts acylation of veratrole with 2,3-dimethoxybenzoyl chloride in the presence of aluminium chloride at 30-40° for 16 h. The 2,3,3′,4′-tetramethoxybenzophenone formed was demethylated by heating with pyridinium chloride [281].

– Also refer to: [79,280,411].

m.p. 200° [281], 141–142° [398]. One of the reported melting points is obviously wrong. UV [398].

(2,4-Dihydroxyphenyl)(2,5-dihydroxyphenyl)methanone

[61234-44-4] $C_{13}H_{10}O_5$ mol.wt. 246.22



Syntheses

– Preparation by demethylation of 2,4,5′-trihydroxy-2′-methoxybenzophenone with hydrobromic acid in boiling acetic acid for 2 h (79%) [413].

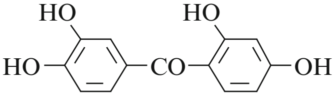
– Preparation [414] according to [415].

– Also refer to: [343].

m.p. 237° [413]; Spectra (NA).

(2,4-Dihydroxyphenyl)(3,4-dihydroxyphenyl)methanone

[61445-50-9] $C_{13}H_{10}O_5$ mol.wt. 246.22



Synthesis

– Preparation by reaction of protocatechuic acid with resorcinol,

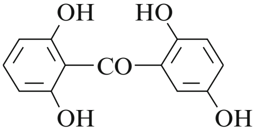
  - in the presence of zinc chloride at 160° [188,190] (Nencki reaction);
  - in the presence of zinc chloride and phosphorous oxychloride for 4 days at r.t. (69%) [191].

– Also refer to: [280,281].

m.p. 201–202° [188,191], 199–200° [190,237]; dihydrate [188]; UV [190].

(2,5-Dihydroxyphenyl)(2,6-dihydroxyphenyl)methanone

[88331-62-8] $C_{13}H_{10}O_5$ mol.wt. 246.22



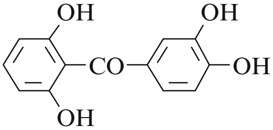
Synthesis

- Refer to: [267,368].

m.p. and Spectra (NA).

(2,6-Dihydroxyphenyl)(3,4-dihydroxyphenyl)methanone

[25576-99-2] $C_{13}H_{10}O_5$ mol.wt. 246.22



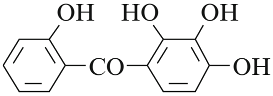
Synthesis

- Preparation by demethylation of 2,3′,4′,6-tetramethoxy-benzophenone with boron tribromide in methylene chloride at r.t. for 24 h (51%) [412].
- Also refer to: [416].

m.p. 113–119° (d) [412];
^{1}H NMR [412], IR [412], UV [412], MS [412].

(2-Hydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[42204-63-7] $C_{13}H_{10}O_5$ mol.wt. 246.22

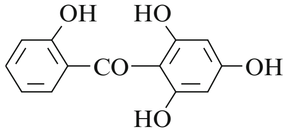


Syntheses

- Preparation by condensation of salicylic acid with pyrogallol,
  - in the presence of zinc chloride at 145° for 3 h (Nencki reaction) [190,344];
  - in the presence of zinc chloride and phosphorous oxychloride at 60–70° for 2 h (50%) [402].
- Also obtained by condensation of o-acetoxybenzonitrile with pyrogallol in the presence of zinc chloride and hydrochloric acid in ethyl ether at 0° (Hoesch reaction). The intermediate compound obtained was hydrolyzed in boiling water for 1 h (19%) [381].
- Also refer to: [79,411].

N.B.: Na salt [344].
m.p. (sesquihydrate) 103–104° [402], 102° [344], 100° [381];
(anhydrous) 149° [344,381,402], 145–147° [190];
UV [190].

(2-Hydroxyphenyl)(2,4,6-trihydroxyphenyl)methanone



$C_{13}H_{10}O_5$ mol.wt. 246.22

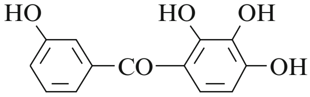
Synthesis

- Obtained by reaction of salicylonitrile with phloroglucinol (Hoesch reaction) Karrer [417]. **N.B.**: Nevertheless, Nishikawa and Robinson [418] were unable to confirm Karrer′s view of the structure of the isolated product.
- Also refer to: [87].

m.p. and Spectra (NA).

(3-Hydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[105443-53-6]



$C_{13}H_{10}O_5$ mol.wt. 246.22

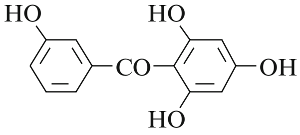
Synthesis

- Preparation by benzoylation of pyrogallol with m-hydroxy-benzoic acid,
  - in the presence of Amberlyst-15 in refluxing toluene under azeotropical removal of water [419];
  - in the presence of boron trifluoride or its complexes [420].
- Also refer to: [419–421] (Japanese patents).

m.p. and Spectra (NA).

(3-Hydroxyphenyl)(2,4,6-trihydroxyphenyl)methanone

[26271-33-0]



$C_{13}H_{10}O_5$ mol.wt. 246.22

Syntheses

- Preparation by demethylation of 2,4,6-trihydroxy-3′-methoxybenzophenone with aluminium chloride in refluxing chlorobenzene for 1 h (95%) [341,343,422].
- Preparation from phloroglucinol [414] according to [415].
- Also obtained (small quantities) by degradation of Gentisein in the presence of sodium hydroxide and potassium hydroxide (1 g each) at 300–310° under nitrogen for 3 h [296].
- Preparation by reaction of m-hydroxybenzonitrile with phloroglucinol (38%) (Hoesch reaction) [418].
- Also refer to: [342,413].

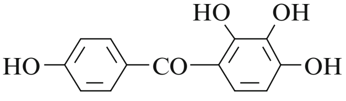
Isolation from natural source

- From fresh *Gentiana lutea* rhizome (Gentianaceae) [296,422,423].

m.p. 246° (d) [418], 235–238° [296,341,422];
^{1}H NMR [341,422], IR [341,422], UV [341,422];
TLC [422]; GC [422].

(4-Hydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[31127-54-5] $C_{13}H_{10}O_5$ mol.wt. 246.22



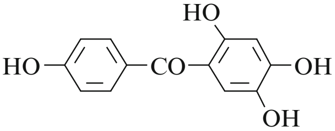
Syntheses

- Preparation by benzoylation of pyrogallol with p-hydroxybenzoic acid in the presence of Amberlyst-15 in refluxing toluene for 21 h under azeotropical removal of water (86%) [419].
- Preparation by Friedel–Crafts acylation of pyrogallol trimethyl ether with p-anisoyl chloride ($AlCl_3/CS_2$/ 3 h at r.t.), followed by demethylation ($AlCl_3$/toluene/ 1 h at 120°) (65%).
- Refer to: Chem. Abstr., **44**, 1271f (1950)^T.
- Also refer to: [424–427] and Chem. Abstr., **129**, 88025c, 129007u, 142608k, 237674t, 237675u, 237676v (1998).

m.p. 219°^{T}; Spectra (NA).

(4-Hydroxyphenyl)(2,4,5-trihydroxyphenyl)methanone

[58115-12-1] $C_{13}H_{10}O_5$ mol.wt. 246.22



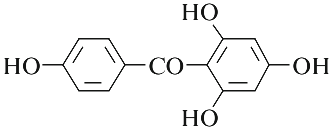
Synthesis

- Obtained by total demethylation of 2-hydroxy-4,4′,5-tri-methoxybenzophenone with hydriodic acid in acetic anhydride [428].

unstable compound [428]; m.p. and Spectra (NA).
m.p. (of tetraacetate) 132–134° [428].

(4-Hydroxyphenyl)(2,4,6-trihydroxyphenyl)methanone *(Iriflophenone)*

[52591-10-3] $C_{13}H_{10}O_5$ mol.wt. 246.22



Synthesis

- Preparation by reaction of p-hydroxybenzonitrile with phloroglucinol [429], (15%) [418] (Hoesch reaction).

Isolation from natural sources

- From branchwood of *Morus alba* (Moraceae) [171,430,431];
- From rhizomes of *Iris Germanica Linn* (white flowered variety) [432];
- From rhizoma of *Iris florentina* (Iridaceae) [433].

N.B.: Dihydrate [418,431].
m.p. 210° [418,431], 208–210° [432], 207–208° [433];
^{1}H NMR [433], IR [433], UV [433], MS [432,433].

1.5 Pentahydroxybenzophenones

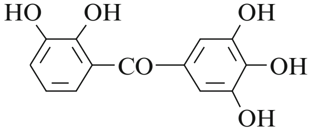
1.5.1 Hydroxy Groups Located on One Ring

Only one ketone possible, not yet described.

1.5.2 Hydroxy Groups Located on Both Rings

(2,3-Dihydroxyphenyl)(3,4,5-trihydroxyphenyl)methanone

[114415-01-9] $C_{13}H_{10}O_6$ mol.wt. 262.22



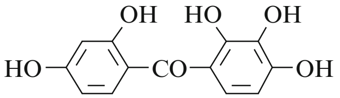
Synthesis

- Preparation by reaction of gallic acid with pyrocatechol in chlorobenzene in the presence of methanesulfonic acid for 6 h between 65° and 75° (90%) or by using boron trifluoride-etherate instead of methanesulfonic acid as catalyst (10%).
- Refer to: Chem. Abstr., **108**, 204327v (1988).

  m.p. and Spectra (NA).

(2,4-Dihydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[92379-42-5] $C_{13}H_{10}O_6$ mol.wt. 262.22



Syntheses

- Preparation by reaction of 2,3,4-trihydroxybenzoic acid with resorcinol,
  - in the presence of zinc chloride and phosphorous oxychloride at 70–80° for 1.5 h (65%) [402];
  - in the presence of zinc chloride and a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 27°. Then, during 2 h, phosphorous trichloride was added between 27° and 37° and the mixture heated at 60° for 16 h [194].
- Preparation by reaction of β-resorcylic acid with pyrogallol,
  - in the presence of zinc chloride and phosphorous oxychloride at 70–80° for 1 h [402], in sulfolane at 50° for 2 h [403];
  - in the presence of zinc chloride and a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 27°. Then, during 2 h, phosphorous trichloride was added between 27° and 37° and the mixture heated at 60° for 16 h [194];
  - in the presence of boron trifluoride in tetrachloroethane at 110° for 1 h [420].
- Also obtained by condensation of 2,4-diacetoxybenzonitrile with pyrogallol in the presence of zinc chloride and hydrochloric acid in ethyl ether (Hoesch reaction).

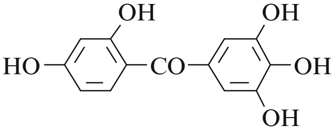
The intermediate compound obtained was hydrolyzed in boiling water for 2 h (22%) [381].

- Also refer to: [298,391,425,426,434–437].

m.p. 187–188° [402], 187° [381]; dihydrate [381];
Spectra (NA); TLC [116].

(2,4-Dihydroxyphenyl)(3,4,5-trihydroxyphenyl)methanone

[10425-09-9] $C_{13}H_{10}O_6$ mol.wt. 262.22



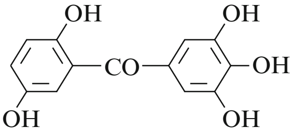
Syntheses

- Preparation by reaction of gallic acid with resorcinol in the presence of zinc chloride at 145° for 3 h [190] (Nencki reaction).
- Preparation by condensation of 2,4-dihydroxybenzoic acid with pyrogallol in the presence of zinc chloride for 45 min at 125–140° (Nencki reaction).
- Refer to: Chem. Abstr., **44**, 1271f (1950)^T.
- Also refer to: [280,281].

m.p. 253°^{T}, 243–245° [190,237]; UV [190]; TLC [116].

(2,5-Dihydroxyphenyl)(3,4,5-trihydroxyphenyl)methanone

$C_{13}H_{10}O_6$ mol.wt. 262.22



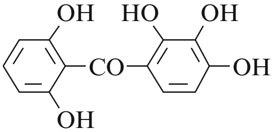
Synthesis

- Preparation by the Friedel–Crafts reaction.
- Refer to: Chem. Abstr., **65**, 18519h (1966)^T.

m.p. (NA); UV^T.

(2,6-Dihydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[112232-16-3] $C_{13}H_{10}O_6$ mol.wt. 262.22



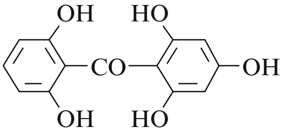
Synthesis

- Refer to: [438] (Japanese patent).

m.p. and Spectra (NA).

(2,6-Dihydroxyphenyl)(2,4,6-trihydroxyphenyl)methanone

$C_{13}H_{10}O_6$ mol.wt. 262.22



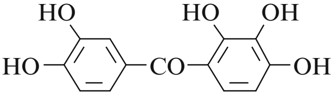
Synthesis

- Obtained (poor yield) by treatment of 1,3,8-trihydroxy-xanthone (m.p. 265°) with aqueous potassium hydroxide for 30 min at 240–250° (6%).
- Refer to: Chem. Abstr., **50**, 15523^b (1956)^T.

m.p. 162°^{T}; Spectra (NA).

(3,4-Dihydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[61445-51-0] $C_{13}H_{10}O_6$ mol.wt. 262.22



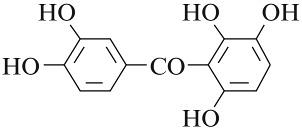
Synthesis

– Refer to: [280,281].

m.p. and Spectra (NA).

(3,4-Dihydroxyphenyl)(2,3,6-trihydroxyphenyl)methanone

[25577-03-1] $C_{13}H_{10}O_6$ mol.wt. 262.22



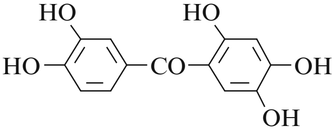
Synthesis

– Preparation by demethylation of 2,3,3′, 4′,6-pentamethoxy-benzophenone with boron tribromide in methylene chloride at r.t. for 8 h (70%) [412].

m.p. 131–135° [412];
^{1}H NMR [412], IR [412], UV [412], MS [412].

(3,4-Dihydroxyphenyl)(2,4,5-trihydroxyphenyl)methanone

[61445-52-1] $C_{13}H_{10}O_6$ mol.wt. 262.22



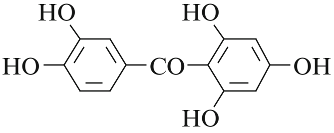
Synthesis

– Preparation by reaction of 3,4-(diacetoxy) benzonitrile with hydroxyhydroquinone in the presence of zinc chloride and hydrochloric acid in a chloroform and ethyl ether mixture, treatment with 10% sulfuric acid, followed by hydrolysis of the ketimine sulfate formed with boiling water for 1 h (21%) [439] and Chem. Abstr., **22**, 4519^7 (1928).
– Also refer to: [280,281].

m.p. 242° [439] and Chem. Abstr., **22**, 4519^7 (1928);
Spectra (NA).

(3,4-Dihydroxyphenyl)(2,4,6-trihydroxyphenyl)methanone
(Maclurin, Morin)

[519-34-6] $C_{13}H_{10}O_6$ mol.wt. 262.22



Syntheses

– Preparation by reaction of 3,4-dihydroxy-benzonitrile with phloroglucinol in the presence of zinc chloride and hydrochloric acid in ethyl ether (Hoesch reaction), first at r.t. for 1 h, then at 50–60° for 4 h (37%) [440].
– Also obtained by condensation of protocatechuic acid with phloroglucinol [441].
– Also refer to: [166,167,341–343,423,442–444].

Isolation from natural sources

- From heartwood of *Symphonia globulifera* L. (Guttiferae) [445];
- From yellow wood of *Maclura tinctoria* L. D. Don (Moraceae) [171,446], so called *Morus tinctoria* L. *(Maclura aurantiaca Nutt.)* [447,448] or *Chlorophora tinctoria* L. Gaud [430], (major product) [431,447];
- From bark of *Laguncularia racemosa* Garten (Moraceae) [171];
- From yellow sapwood of *Acacia catechu* and *Acacia catechuoides* (Moraceae) [171,449];
- From yellow sapwood of *Acacia sundra* (Moraceae) [171,449];
- From branches [171] or sawdust (small amounts) [431] of *Morus Alba* Linn. (Moraceae);
- From bark of *Coto* (Lauraceae) (main component) [450].

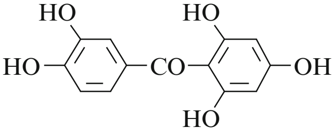
N.B.: Ba [446,451], Ca [446] and Pb salts [444,446].
monohydrate [440,442], (Wagner) [446]; sesquihydrate (Delffs) [446];

m.p. 226–230° [341], 222–224° [430], 220–222° [449], 220–221° [431], 220° [440]; ^{1}H NMR [341], IR [341], UV [341]; TLC [341].

(3,4-Dihydroxyphenyl)(2,4,6-trihydroxyphenyl-*1,3,5*- $^{14}C_3$)methanone

(Maclurin-1,3,5- $^{14}C_3$*)*

[75629-21-9] $C_{13}H_{10}O_6$ mol.wt. 268.22



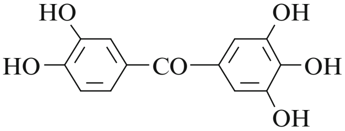
Synthesis

- Preparation by reaction of protocatechuonitrile (3,4-di-hydroxybenzonitrile) with phloroglucinol-2,4,6-^{14}C (Hoesch reaction).
- Refer to: Chem. Abstr., **93**, 217975b (1980)^T.

m.p. 199–200°^{T}; Sp. act. 3.05 x 10^7 dpm/mM^T.

(3,4-Dihydroxyphenyl)(3,4,5-trihydroxyphenyl)methanone

[56609-45-1] $C_{13}H_{10}O_6$ mol.wt. 262.22



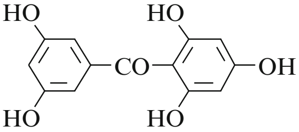
Synthesis

- Preparation by reaction of gallic acid with pyrocatechol in the presence of zinc chloride for 1 h at 140–145° [452] (Nencki reaction).
- Also refer to: [280,281,453,454].

m.p. 266° [452]; UV [452].

(3,5-Dihydroxyphenyl)(2,4,6-trihydroxyphenyl)methanone

[53250-52-5] $C_{13}H_{10}O_6$ mol.wt. 262.22



Synthesis

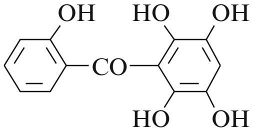
- Not yet described.

Isolation from natural source

- From the heartwood of *Garcinia pedunculata* (Guttiferae) [167,443].

  m.p. 258–260° [167]; MS [167].

(2-Hydroxyphenyl)(2,3,5,6-tetrahydroxyphenyl)methanone



$C_{13}H_{10}O_6$ mol.wt. 262.22

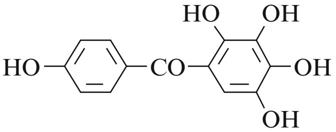
Synthesis

- Refer to: [87].

m.p. and Spectra (NA).

(4-Hydroxyphenyl)(2,3,4,5-tetrahydroxyphenyl)methanone

[112232-17-4]



$C_{13}H_{10}O_6$ mol.wt. 262.22

Synthesis

- Refer to: [438] (Japanese patent).

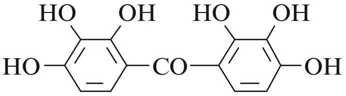
m.p. and Spectra (NA).

1.6 Hexahydroxybenzophenones

Symmetrical ketones

Bis(2,3,4-trihydroxyphenyl)methanone

[75440-84-5]



$C_{13}H_{10}O_7$ mol.wt. 278.22

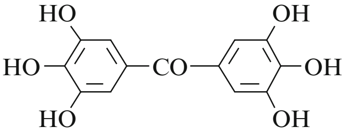
Syntheses

- Preparation by reaction of 2,3,4-trihydroxybenzoic acid with pyrogallol in the presence of zinc chloride for 45 min at 125–140° (Nencki reaction).
- Refer to: Chem. Abstr., **44**, 1271f (1950)^{T}.
- Preparation by heating a mixture of 2,3,4-trihydroxybenzoic acid, pyrogallol and excess of phosphorous oxychloride with zinc chloride for 2 h at 70–80° (73%). -Refer to: Chem. Abstr., **50**, 1787g (1956); **51**, 8736a (1957)^{TT}.
- Also refer to: Chem. Abstr., **102**, 73168v (1985) and **118**, 90911e (1993).

m.p. 244–245°^{TT}, 240°^{T}; Spectra (NA).

Bis(3,4,5-trihydroxyphenyl)methanone

[111621-53-5] $C_{13}H_{10}O_7$ mol.wt. 278.22



Synthesis

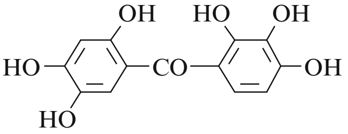
- One of metabolites of tannic acid formed on in vitro incubation with rat liver microsomes.
- Refer to: Chem. Abstr., **108**, 1953y (1988)^T.

m.p. (NA); ^{1}H NMR^T, IR^T, UV^T, MS^T.

Asymmetric ketones

(2,3,4-Trihydroxyphenyl)(2,4,5-trihydroxyphenyl)methanone

[153812-71-6] $C_{13}H_{10}O_7$ mol.wt. 278.22



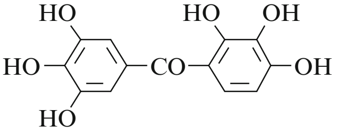
Synthesis

- Refer to: Chem. Abstr., **120**, 204680c (1994).

m.p. and Spectra (NA).

(2,3,4-Trihydroxyphenyl)(3,4,5-trihydroxyphenyl)methanone *(Exifone, Adlone)*

[52479-85-3] $C_{13}H_{10}O_7$ mol.wt. 278.22



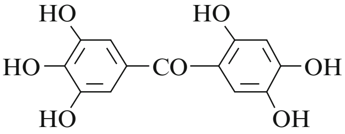
Synthesis

- Preparation by condensation of gallic acid with pyrogallol,
  - in the presence of zinc chloride at 120° (good yield) [455] or at 145° for 3 h [190] (Nencki reaction);
  - in the presence of zinc chloride and phosphorous oxychloride at 80° for 2 h, via Fries rearrangement [281].
- Also refer to: [280,365,454,456] and Chem. Abstr., **80**, 103486u (1974); **89**, 186079y (1978); **106**, 207622g (1987); **107**, 109260p (1987); **109**, 86223d (1988); **112**, 104863f (1990); **125**, 5333v (1996); **129**, 88025c (1998).

m.p. 275–280° [190,237], 272–273° [455], 270° [281];
^{1}H NMR -Refer to: Chem. Abstr., **115**, 196535p (1991), UV [190,237];
pK_a [369]; HPLC -Refer to: Chem. Abstr., **112**, 48178x (1990).

(2,4,5-Trihydroxyphenyl)(3,4,5-trihydroxyphenyl)methanone

[119427-61-1] $C_{13}H_{10}O_7$ mol.wt. 278.22



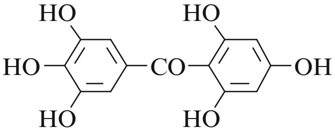
Synthesis

– Refer to: [281,298,435].

m.p. and Spectra (NA).

(2,4,6-Trihydroxyphenyl)(3,4,5-trihydroxyphenyl)methanone

[112005-19-3] $C_{13}H_{10}O_7$ mol.wt. 278.22



Synthesis

– Refer to: [438,457] (Japanese patent).

m.p. and Spectra (NA).

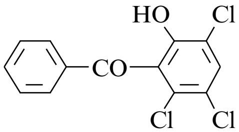
Chapter 2
Substituted Hydroxybenzophenones
(Class of METHANONES)

2.1 Monohydroxybenzophenones

2.1.1 Substituents Located on the Hydroxylated Ring

Phenyl(2,3,5-trichloro-6-hydroxyphenyl)methanone

[7396-96-5] $C_{13}H_7Cl_3O_2$ mol.wt. 301.56



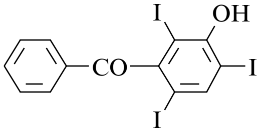
Synthesis

– Preparation by Fries rearrangement of 2,4,5-trichlorophenyl benzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 143–144° [458]; Spectra (NA).

(3-Hydroxy-2,4,6-triiodophenyl)phenylmethanone

[91692-34-1] $C_{13}H_7I_3O_2$ mol.wt. 575.91



Syntheses

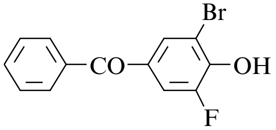
– Preparation by treating a solution of 3-hydroxybenzo-phenone in methanolic sodium hydroxide with iodine mono-chloride in aqueous sodium chloride (75%) [120,459].

– Also obtained by iodination of 3-hydroxybenzophenone with iodine monochloride [460].

m.p. 190° [120,459]; Spectra (NA).

(3-Bromo-5-fluoro-4-hydroxyphenyl)phenylmethanone

[579-15-7] $C_{13}H_8BrFO_2$ mol.wt. 295.11



Synthesis

– Preparation by reaction of bromine with 3-fluoro-4-hydroxybenzophenone in acetic acid [461].

m.p. 187° [461]; Spectra (NA).

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
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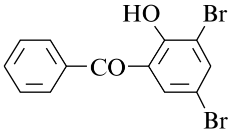
(3,5-Dibromo-2-hydroxyphenyl)phenylmethanone

[111277-24-8] $C_{13}H_8Br_2O_2$ mol.wt. 356.01



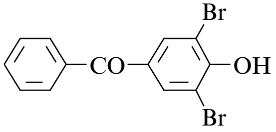
Syntheses

- Preparation by adding a solution of bromine in chloroform to a solution of 2-hydroxybenzophenone in ethanol [35,61,127,462].
- Preparation by Friedel–Crafts acylation of benzene with 3,5-dibromo-2-hydroxybenzoyl chloride in the presence of aluminium chloride in a steam bath for 1 h (70%) [462] or at 80° (>80%) [463].
- Also obtained (poor yield) by alkaline condensation of benzotrichloride with 2,4-dibromophenol in 30% aqueous sodium hydroxide solution at 80–85° (8%) [127].
- Also refer to: [73,464,465].

m.p. 129–130° [463], 128–129° [127], 126° [35,61], 121–122° [462];
Spectra (NA).

(3,5-Dibromo-4-hydroxyphenyl)phenylmethanone

[26733-16-4] $C_{13}H_8Br_2O_2$ mol.wt. 356.01



Syntheses

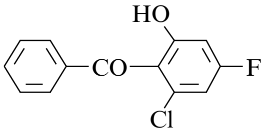
- Preparation by reaction of bromine with 4-hydroxybenzo-phenone in acetic acid [140,169,466], in chloroform [127] or in water [140].
- Preparation by adding a 5% solution of bromine in potassium bromide to a solution of 4-hydroxybenzophenone in dilute aqueous potassium hydroxide at 5° [140].
- Also obtained by treatment of a mixture of sodium acetate and 4-methoxybenzophenone with bromine in a sealed tube at 140° [140].
- Preparation by Fries rearrangement of 2,6-dibromophenyl benzoate with aluminium chloride without solvent at 120° for 3 h (40%) [467].
- Also obtained (poor yield) by alkaline condensation of benzotrichloride with 2,4,6-tribromophenol in 30% aqueous sodium hydroxide solution at 90–95° (2%) [127].
- Also refer to: [468].

N.B.: K salt [169].

m.p. 178° [467], 155° [140], 152–153° [466], 151–152° [169], 150–152° [127];
MS [177]; voltammetric studies [469].

(2-Chloro-4-fluoro-6-hydroxyphenyl)phenylmethanone

[169781-84-4] $C_{13}H_8ClFO_2$ mol.wt. 250.66



Syntheses

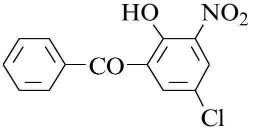
- Obtained by Fries rearrangement of 3-chloro-5-fluorophenyl benzoate with aluminium chloride at 200° for 20 min [20].

– Also obtained by demethylation of 2-chloro-4-fluoro-6-methoxybenzophenone with boron tribromide in methylene chloride at r.t. for 12 h [20], according to [21].

m.p. and Spectra (NA).

(5-Chloro-2-hydroxy-3-nitrophenyl)phenylmethanone

[85052-26-2] $C_{13}H_8ClNO_4$ mol.wt. 277.66



Synthesis

– Preparation by nitration of 5-chloro-2-hydroxy-benzo-phenone,

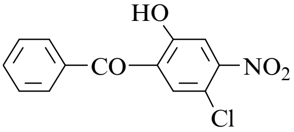
- with 26% nitric acid in acetic acid at r.t. for 12 h (50%) [470];
- with nitric acid (d = 1.38) in acetic acid and methylene chloride at r.t. for overnight [471];
- with 60% nitric acid in the presence of one drop of concentrated sulfuric acid at r.t. for 25 min (89%) [472].

– Also refer to: [473].

m.p. 70°5–71° [470], 68–70° [471], 56–58° [472];
^{1}H NMR [472], IR [470,472], MS [472].

(5-Chloro-2-hydroxy-4-nitrophenyl)phenylmethanone

[66306-91-0] $C_{13}H_8ClNO_4$ mol.wt. 277.66



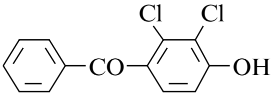
Synthesis

– Refer to: [474].

m.p. and Spectra (NA).

(2,3-Dichloro-4-hydroxyphenyl)phenylmethanone

[62967-12-8] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



Synthesis

– Preparation by demethylation of 2,3-dichloro-4-methoxy-benzophenone (SM),

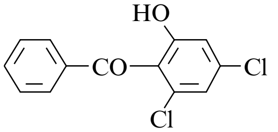
- with aluminium chloride, in refluxing methylene chloride overnight [475] or in refluxing benzene for 5 h [476];
- with pyridinium chloride at 180° for 2 h (99%) [477].

SM was obtained by Friedel–Crafts acylation of 2,3-dichloroanisole with benzoyl chloride in the presence of aluminium chloride in methylene chloride [475], first at 0° for 30 min, then at r.t. for 6 h (42%) [477] or in ethylene dichloride for 2 h [476].

m.p. 123–126° [475]; Spectra (NA).

(2,4-Dichloro-6-hydroxyphenyl)phenylmethanone

[34199-75-2] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



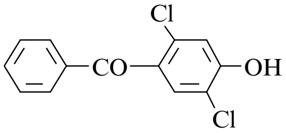
Synthesis

- Preparation by Fries rearrangement of 3,5-dichlorophenyl benzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 156–157° [458]; Spectra (NA).

(2,5-Dichloro-4-hydroxyphenyl)phenylmethanone

[123574-94-7] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



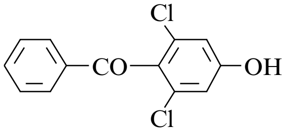
Synthesis

- Preparation by Friedel–Crafts acylation of 2,5-dichloro-phenol with benzoyl chloride in the presence of aluminium chloride in refluxing ethylene dichloride for 33 h (61%) [478].
- Also refer to: [479].

m.p. 161–163° [478]; ^{1}H NMR [478], IR [478], MS [478].

(2,6-Dichloro-4-hydroxyphenyl)phenylmethanone

[34183-13-6] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



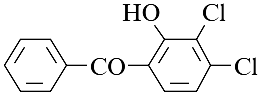
Synthesis

- Obtained by Fries rearrangement of 3,5-dichlorophenyl benzoate with aluminium chloride in chlorobenzene at 140–150° for 20 min or in nitrobenzene at 75° for 24 h [480].

m.p. 135–136° [480]; Spectra (NA).

(3,4-Dichloro-2-hydroxyphenyl)phenylmethanone

[54923-64-7] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



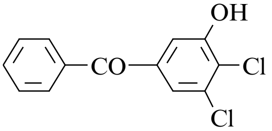
Synthesis

- Obtained by chromic oxidation (CrO_3) of 6,7-dichloro-3-phenylbenzofuran, followed by saponification of obtained keto ester [481].

m.p. 141°5 [481]; IR [481].

(3,4-Dichloro-5-hydroxyphenyl)phenylmethanone

[113730-38-4] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



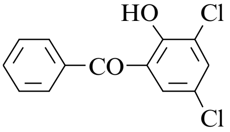
Synthesis

- Preparation by demethylation of 3,4-dichloro-5-methoxy-benzophenone with boron tribromide in methylene chloride at 5° for 1 h, then at r.t. for 20 min (98%) [482].

m.p. 177° [482]; hemihydrate [482]; ^{1}H NMR [482].

(3,5-Dichloro-2-hydroxyphenyl)phenylmethanone

[7396-92-1] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



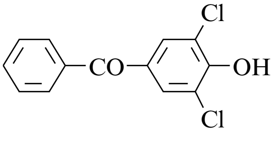
Syntheses

- Preparation by adding aluminium chloride into a solution of 3,5-dichloro-2-hydroxybenzoyl chloride in benzene and heating at 60° for 3–4 h (60–70%) [483].
- Also obtained by chromic oxidation (CrO_3) of 5,7-dichloro-3-phenylbenzofuran, followed by saponification of the obtained keto ester [481].
- Preparation by reaction of benzoyl chloride with 2,4-dichlorophenol in the presence of aluminium chloride at 180° for 23 min (46%) [484].
- Preparation by Fries rearrangement of 2,4-dichlorophenyl benzoate (SM) with aluminium chloride at 140° for 30 min (quantitative yield). SM was obtained by heating benzoyl chloride with aluminium tris(2,4-dichlorophenoxide) in a water bath for 30 min [485].
- Also refer to: [464,486–488].

m.p. 116° [483], 115° [481], 114–115° [485], 113–114° [484]; IR [481], UV [241].

(3,5-Dichloro-4-hydroxyphenyl)phenylmethanone

[34183-06-7] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



Syntheses

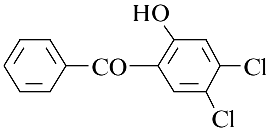
- Preparation by Fries rearrangement of 2,6-dichlorophenyl benzoate with aluminium chloride,
  - without solvent at 154° for 2.5 h (71%) [489];
  - in chlorobenzene at 140–150° for 20 min [480];
  - in nitrobenzene at 75° for 24 h [480].
- Preparation by demethylation of 3,5-dichloro-4-methoxybenzophenone with 48% hydrobromic acid in refluxing acetic acid [140].
- Preparation by passing chlorine into a solution of p-hydroxybenzophenone and sodium acetate in acetic acid during some hours [140].

- Also obtained by Fries rearrangement of 4-tert-butyl-2,6-dichlorophenyl benzoate with aluminium chloride at 140° (sole isolated reaction product). There is a total dealkylation [154].
- Also refer to: [298].

m.p. 149–150° [480], 148° [140], 145–146° [489]; Spectra (NA).

(4,5-Dichloro-2-hydroxyphenyl)phenylmethanone

[58430-25-4] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



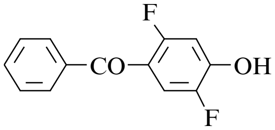
Synthesis

- Preparation by Friedel–Crafts acylation of 3,4-dichloro-phenol with benzoyl chloride in the presence of aluminium chloride at 180° for 35 min [75].

m.p. 115–116° [75]; Spectra (NA).

(2,5-Difluoro-4-hydroxyphenyl)phenylmethanone

[179018-49-6] $C_{13}H_8F_2O_2$ mol.wt. 234.20



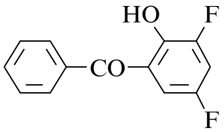
Synthesis

- Preparation by Fries rearrangement of 2,5-difluorophenyl benzoate with aluminium chloride at 160° for 2 h [490,491].

m.p. (NA); MS [490,491].

(3,5-Difluoro-2-hydroxyphenyl)phenylmethanone

[183280-20-8] $C_{13}H_8F_2O_2$ mol.wt. 234.20



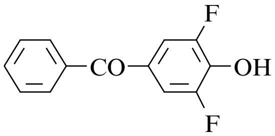
Synthesis

- Preparation by Fries rearrangement of 2,4-difluorophenyl benzoate with aluminium chloride at 150–180° for 20 min (35%) [492].

m.p. 83°5 [492]; ^{1}H NMR [492], UV [492], MS [492].

(3,5-Difluoro-4-hydroxyphenyl)phenylmethanone

[179018-48-5] $C_{13}H_8F_2O_2$ mol.wt. 234.20

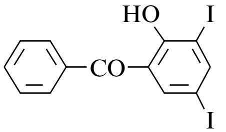


Synthesis

- Preparation by Fries rearrangement of 2,6-difluorophenyl benzoate with aluminium chloride at 160° for 2 h [490,491].

m.p. (NA); MS [490,491].

(2-Hydroxy-3,5-diiodophenyl)phenylmethanone



$C_{13}H_8I_2O_2$ mol.wt. 450.01

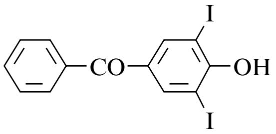
Synthesis

- Preparation by reaction of 3,5-diiodosalicylic acid chloride with benzene in the presence of aluminium chloride in carbon disulfide at 50° [493].
- Also refer to: [464,494].

m.p. 116° [493]; Spectra (NA).

(4-Hydroxy-3,5-diiodophenyl)phenylmethanone

[70036-74-7] $C_{13}H_8I_2O_2$ mol.wt. 450.01

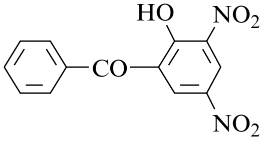


Synthesis

- Preparation by treatment of 4-hydroxybenzophenone with iodine monochloride,
  - in the presence of sodium acetate in acetic acid [140];
  - at steam bath temperature [460,495].

m.p. 145° [140]; Spectra (NA).

(2-Hydroxy-3,5-dinitrophenyl)phenylmethanone



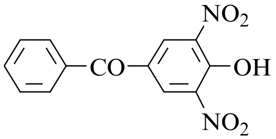
$C_{13}H_8N_2O_6$ mol.wt. 288.22

Synthesis

- Obtained by action of potassium hydroxide with 2-bromo-3,5-dinitrobenzophenone (SM) in ethanol. SM was prepared by Friedel–Crafts acylation of benzene with 2-bromo-3,5-dinitrobenzoyl chloride in the presence of aluminium chloride (97%, m.p. 153–154°). Refer to: Chem. Abstr., **20**, 1229^8 (1926)^T.
- **N.B.**: K salt, m.p. 180–200° (d)^T.

m.p. and Spectra (NA).

(4-Hydroxy-3,5-dinitrophenyl)phenylmethanone



$C_{13}H_8N_2O_6$ mol.wt. 288.22

Syntheses

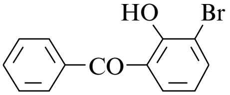
- Preparation by heating a solution of 4-hydroxybenzo-phenone in nitric acid (d = 1.4) at 40° for 30 min [140].
- Preparation by reaction of 10% aqueous sodium hydroxide with 4-chloro-3,5-dinitrobenzophenone

(SM) in refluxingethanol for 20 min (75%). SM was prepared from p-chlorobenzoic acid by a three-step synthesis: at first, nitration of this acid with nitric acid/concentrated sulfuric acid at 140° for 1.5 h. The 4-chloro-3,5-dinitrobenzoic acid formed (95%, m.p. 159°), on treatment with phosphorous pentachloride in refluxing benzene gave the 4-chloro-3,5-dinitrobenzoyl chloride. Then, by adding aluminium chloride to the reaction mixture and heating this one in a water bath for 30 min, SM was obtained (90%, m.p. 118°) [496].

m.p. 138° [140], 136° [496]; Spectra (NA).

(3-Bromo-2-hydroxyphenyl)phenylmethanone

[147321-82-2] $C_{13}H_9BrO_2$ mol.wt. 277.11



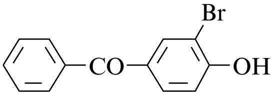
Synthesis

- Refer to: [497].

m.p. and Spectra (NA); TLC [497]; HPLC [497].

(3-Bromo-4-hydroxyphenyl)phenylmethanone

[89899-44-5] $C_{13}H_9BrO_2$ mol.wt. 277.11



Syntheses

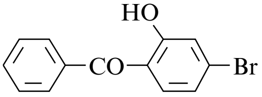
- Preparation by bromination of p-hydroxybenzophenone with bromine [466], in acetic acid [140,169] or in chloroform [127].
- Preparation by demethylation of 3-bromo-4-methoxy-benzophenone (SM) with 48% hydrobromic acid in refluxing acetic acid. SM was prepared by reaction of bromine with 4-methoxybenzophenone in the presence of sodium acetate in acetic acid at 100° for 6 h [140].
- Also obtained (poor yield) by aqueous alkaline condensation of benzotrichloride with 4-bromo-phenol or 2,4-dibromophenol in 30% sodium hydroxide solution at 80–85° (<3%) [127].
- Also obtained (poor yield) by reaction of benzoyl chloride with o-bromophenol in the presence of ferric chloride [466].
- Also refer to: [429,498].

N.B.: K salt [169].

m.p. 183° [140], 182–183° [127], 180–181° [169,466]; Spectra (NA); polarographic study [499]; voltammetric study [500]; TLC [497]; HPLC [497].

(4-Bromo-2-hydroxyphenyl)phenylmethanone

[6723-04-2] $C_{13}H_9BrO_2$ mol.wt. 277.11



Syntheses

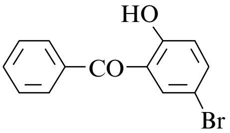
- Preparation by oxidation of 6-bromo-2,3-diphenylbenzo-furan with chromium trioxide in boiling acetic acid, followed by saponification of the obtained keto ester—(2-benzoyloxy)-4-bromobenzophenone—with sodium hydroxide in refluxing ethanol [44,501], (70%) [502].

- Also obtained by Fries rearrangement of m-bromophenyl benzoate with aluminium chloride [23], at 200° for 20 min [20].
- Also obtained by demethylation of 4-bromo-2-methoxybenzophenone with boron tribromide in methylene chloride at r.t. for 12 h [20], according to [21].

m.p. 83° [501,502], 79° [44]; IR [44,103,501], UV [503].

(5-Bromo-2-hydroxyphenyl)phenylmethanone

[55082-33-2] $C_{13}H_9BrO_2$ mol.wt. 277.11



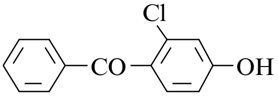
Syntheses

- Preparation by Fries rearrangement of p-bromophenyl benzoate with aluminium chloride without solvent at 150° for 15 min (42%) [462].
- Preparation by oxidation of 5-bromo-2,3-diphenylbenzo-furan with chromium trioxide in boiling acetic acid, followed by saponification of the obtained keto ester—2-(benzoyloxy)-5-bromobenzophenone—with sodium hydroxide [44] with potassium hydroxide in refluxing ethanol (65%) [504].
- Also obtained (poor yield) by alkaline condensation of benzotrichloride with p-bromophenol or 2,4-dibromophenol in 30% aqueous sodium hydroxide solution at 80–85° (2%) [127].
- Also refer to: [505].

m.p. 111–112° [462], 110° [504], 109° [44]; IR [44].

(2-Chloro-4-hydroxyphenyl)phenylmethanone

[81375-00-0] $C_{13}H_9ClO_2$ mol.wt. 232.67



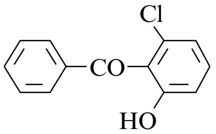
Syntheses

- Preparation by Fries rearrangement of 3-chlorophenyl benzoate without solvent,
  - with aluminium chloride for 2 h at 150° (25%) [506] or at 160° [490,491] or for 15 min at 175° (8%) [36];
  - with titanium tetrachloride for 15 min at 175° (17%) [36].
- Preparation by diazotization of 4-amino-2-chlorobenzophenone, followed by hydrolysis of the diazonium salt so obtained (66%) [6].

m.p. 118–119° [506], 115° [6]; MS [490,491].

(2-Chloro-6-hydroxyphenyl)phenylmethanone

[81374-99-4] $C_{13}H_9ClO_2$ mol.wt. 232.67



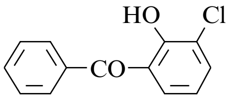
Synthesis

– Obtained by reaction of sodium iodide with 2-chloro-6-methoxybenzophenone (SM) in the presence of trimethyl-silyl chloride in acetonitrile into an autoclave at 130° for 24 h (36%). SM was prepared from 2-amino-6-chlorotoluene by a five-step synthesis [6].

m.p. 105° [6]; ^{13}C NMR [6].

(3-Chloro-2-hydroxyphenyl)phenylmethanone

[35582-86-6] $C_{13}H_9ClO_2$ mol.wt. 232.67



Syntheses

– Preparation by decarboxylation of 2-(3-chloro-2-hydroxy-benzoyl)benzoic acid in quinoline at 250° during 15 min in the presence of silver carbonate (80%) [507,508].

– Also obtained from o-chlorophenol,

  - by reaction with benzoyl chloride in the presence of aluminium chloride in tetrachloroethane for 3 h at 120–130° (17%) [508];
  - by reaction with benzoic acid in the presence of Amberlyst-15 in refluxing chlorobenzene during 70 h (18%) [53].

– Also obtained by chromic oxidation (CrO_3) of 7-chloro-3-phenylbenzofuran, followed by saponification of the obtained keto ester [481].

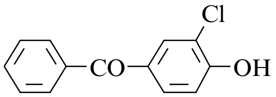
– Also obtained (by-product) by Fries rearrangement of 2-chlorophenyl benzoate with aluminium chloride without solvent at 152–155° for 2 h (6%) [506].

– Also refer to: [82,83,509,510].

m.p. 95° [506], 93° [481], 92°5–93° [508], 91°7–92°5 [507]; IR [481,507], UV [507].

(3-Chloro-4-hydroxyphenyl)phenylmethanone

[55191-20-3] $C_{13}H_9ClO_2$ mol.wt. 232.67



Syntheses

– Preparation by Fries rearrangement of o-chlorophenyl benzoate with aluminium chloride without solvent for 2 h at 152–155° (88%) [506] or at 160° [490,491].

– Also obtained by Fries rearrangement of 4-tert-butyl-2-chlorophenyl benzoate with aluminium chloride without solvent for 10 min at 140° (28%) [154].

– Preparation by condensation of benzoyl chloride,

  - with 2-chlorophenol in the presence of aluminium chloride in tetrachloroethane for 3 h at 120–130° [507,508], (46%) [508] or by heating in the presence of ferric chloride without solvent [511];

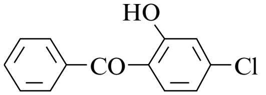
- with 2-chloroanisole in the presence of aluminium chloride in tetrachloroethane for 3 h at 120–130° (48%) [508].

– Preparation by heating 3-chloro-4-methoxybenzophenone in the presence of aluminium chloride in tetrachloroethane for 1 h at 100–110° (98%) [508].
– Also obtained by reaction of benzotrichloride (1 mol) with o-chlorophenol (1 mol) in the presence of water (1 mol) in solution of hydrofluoric acid (85%) [213].
– Also refer to: [512,513].

m.p. 182° [506], 181° [213], 180–181° [508], 179°6–181°3 [507], 179–180° [154], 176° [511]; IR [507], MS [490,491,514].

(4-Chloro-2-hydroxyphenyl)phenylmethanone

[2985-80-0] $C_{13}H_9ClO_2$ mol.wt. 232.67



Syntheses

– Preparation by Fries rearrangement of m-chlorophenyl benzoate,

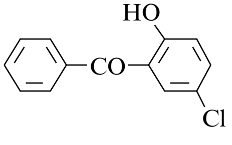
- with aluminium chloride [23,117], without solvent, at 140° for 1 h (54%) [96], at 150° for 2 h (58%) [506], at 175° for 15 min (80%) [36] or at 200° for 20 min [20];
- with titanium tetrachloride for 15 min at 175° (67%) [36].

– Also obtained by degradation of 6-chloro-2,3-diphenylbenzofuran with chromium trioxide in boiling acetic acid during 40 min or 2 h, followed by saponification of the 2-benzoyloxy-4-chloro-benzophenone so formed with 2 N or 4 N sodium hydroxide in refluxing aqueous ethanol for 1 h or 15 min [44,515].
– Preparation by reaction of 4-chloro-2-methoxybenzoyl chloride (SM) with benzene in the presence of aluminium chloride at r.t. [6] according to [14]. SM was obtained from 2-amino-4-chlorotoluene by a four-step synthesis.
– Also obtained by demethylation of 4-chloro-2-methoxybenzophenone with boron tribromide in methylene chloride at r.t. for 12 h [20], according to [21].
– Also refer to: [84,481,516].

m.p. 77° [506], 76–77° [515], 74°5 [44], 74° [6,96];
^{1}H NMR [99], IR [44,106,107,515], UV [99,109–111];
pK_a [96,115]; polarographic study [117]; TLC [116].

(5-Chloro-2-hydroxyphenyl)phenylmethanone *(Light Absorber HCB)*

[85-19-8] $C_{13}H_9ClO_2$ mol.wt. 232.67



Syntheses

– Preparation by Fries rearrangement of p-chlorophenyl benzoate with aluminium chloride [23],

- without solvent [517], at 180° for 10 min (85%) [518], at 155–157° for 2 h (90%) [506], at 130–140° for 1 h [8], (72%) [519] or at 100–150° for 0.5–3 h (68%) [29];

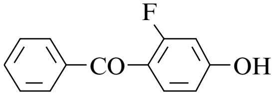
  - in refluxing chlorobenzene for 4 h (14%) [520].

- Preparation by photo-Fries rearrangement of p-chlorophenyl benzoate in benzene, in cyclohexane or in isopropanol at 53–54° for 19 h (51%, 37% and 29% yields, respectively) [72].
- Preparation from p-chlorophenol,
  - by reaction with benzoyl chloride in the presence of aluminium chloride without solvent at 180–195° for 35 min (87%) [484], in boiling carbon disulfide [145] or in tetrachloroethane at 120–130° for 3 h (10%) [508];
  - by reaction with benzoic acid in the presence of boron trifluoride at 180° for 4 h, in a sealed tube (70%) [521].
- Preparation by reaction of benzoyl chloride with p-chloroanisole in the presence of aluminium chloride in tetrachloroethane at 120–130° for 3 h (60%) [508].
- Preparation by reaction of chromium trioxide with 5-chloro-2,3-diphenylbenzofuran in refluxing acetic acid, followed by alkaline hydrolysis of the keto ester so obtained [44], (quantitative yield) [45,522].
- Preparation from 2-chlorothioxanthen-9-one 10,10-dioxide (SM) by a three-step synthesis: SM by refluxing in a solution of 2% sodium hydroxide in dioxane/water mixture (65:35) for 4 h gave the 2-(2-hydroxy-5-chlorobenzoyl) phenylsulfinic acid (37%). The former, by reaction with mercuric chloride in refluxing aqueous acetic acid for 4 h, led to the 2-chloromercuri-2′-hydroxy-5′-chloro-benzophenone (74%). Removal of the chloromercury group was achieved with concentrated hydrochloric acid in refluxing ethanol for 2 h (84%) [62].
- Also obtained by reaction of 5-chloro-2-hydroxybenzaldehyde with iodobenzene by using a catalyst system of palladium chloride/lithium chloride in the presence of sodium carbonate in N,N-dimethylformamide at 100° for 6 h (51%) [51].
- Also refer to: [8,73,75,77,82,83,89,225,472,523–530].

m.p. 97° [506], 96–97° [519], 96° [518], 95°5–96° [96], 95–95°5 [508],
95° [44], 94–95° [45,520,522], 94° [145,236,521],
93°7–95° [72], 93–94° [62,517], 93° [89], 82–85° [29];
b.p.$_{0.3}$ 147–149° [521];
^{1}H NMR [51,99], EPR [98], IR [44,62,72,89,106,107],
UV [99,109–111,236,240,241], MS [51]; pK_a [96,115,531]; TLC [116]; polarographic study [117]; vapour pressure [236].

(2-Fluoro-4-hydroxyphenyl)phenylmethanone

[179018-47-4] $C_{13}H_9FO_2$ mol.wt. 216.21



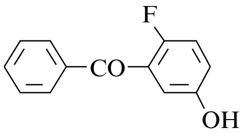
Synthesis

- Preparation by Fries rearrangement of m-fluorophenyl benzoate with aluminium chloride at 160° for 2 h [490,491].

m.p. (NA); MS [490,491].

(2-Fluoro-5-hydroxyphenyl)phenylmethanone

[145300-05-6] $C_{13}H_9FO_2$ mol.wt. 216.21



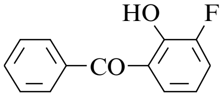
Synthesis

- Preparation by aromatization of 5-benzoyl-4-fluoro-3-cyclo-hexenone in the presence of cupric bromide and lithium bromide in refluxing acetonitrile during 1 h (70%) [118].

m.p. 102° [118]; ^{1}H NMR [118], IR [118].

(3-Fluoro-2-hydroxyphenyl)phenylmethanone

[183280-19-5] $C_{13}H_9FO_2$ mol.wt. 216.21



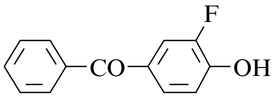
Synthesis

- Preparation by Fries rearrangement of o-fluorophenyl benzoate with aluminium chloride at 150–180° for 20 min (25%) [492].

m.p. 56° [492]; ^{1}H NMR [492], UV [492], MS [492].

(3-Fluoro-4-hydroxyphenyl)phenylmethanone

[365-14-0] $C_{13}H_9FO_2$ mol.wt. 216.21



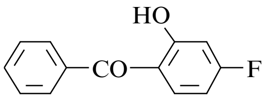
Syntheses

- Preparation by demethylation of 3-fluoro-4-methoxy-benzophenone with refluxing pyridinium chloride for 30 min [461].
- Preparation by Fries rearrangement of o-fluorophenyl benzoate with aluminium chloride at 160° for 2 h [490,491].

m.p. 123° [461]; MS [490,491].

(4-Fluoro-2-hydroxyphenyl)phenylmethanone

[169781-83-3] $C_{13}H_9FO_2$ mol.wt. 216.21



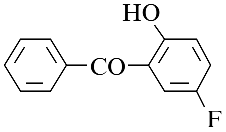
Syntheses

- Obtained by Fries rearrangement of m-fluorophenyl benzoate with aluminium chloride at 200° for 20 min [20].
- Also obtained by demethylation of 4-fluoro-2-methoxy-benzophenone with boron tribromide in methylene chloride at r.t. for 12 h [20], according to [21].

m.p. and Spectra (NA).

(5-Fluoro-2-hydroxyphenyl)phenylmethanone

[362-47-0] $C_{13}H_9FO_2$ mol.wt. 216.21



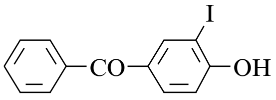
Syntheses

- Preparation by demethylation of 5-fluoro-2-methoxybenzophenone with refluxing pyridinium chloride [532].
- Preparation by Fries rearrangement of p-fluorophenyl benzoate with aluminium chloride [23].
- Also refer to: [525].

m.p. 77° [532]; Spectra (NA).

(4-Hydroxy-3-iodophenyl)phenylmethanone

[170744-87-3] $C_{13}H_9IO_2$ mol.wt. 324.12



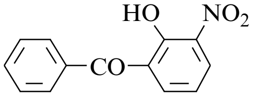
Syntheses

- Preparation by demethylation of 3-iodo-4-methoxybenzo-phenone with 48% hydrobromic acid in refluxing acetic acid [140].
- Preparation by treating an alkaline solution of 4-hydroxy-benzophenone with 5% iodine in aqueous solution of potassium bromide [140].
- Preparation by reaction of iodine monochloride with 4-hydroxybenzophenone in acetic acid solution at r.t. [495].
- Also refer to: [533].

m.p. 184° [140]; Spectra (NA).

(2-Hydroxy-3-nitrophenyl)phenylmethanone

[182499-95-2] $C_{13}H_9NO_4$ mol.wt. 243.22



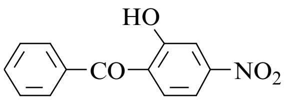
Synthesis

- Obtained by reaction of a 68% nitric acid/96% sulfuric acid mixture with o-hydroxybenzophenone first at <10°, then at r.t. for 2 h (32%) [492].
- Also refer to: [534].

m.p. 88°5 [492]; ^{1}H NMR [492], UV [492], MS [492].

(2-Hydroxy-4-nitrophenyl)phenylmethanone

[1834-88-4] $C_{13}H_9NO_4$ mol.wt. 243.22



Syntheses

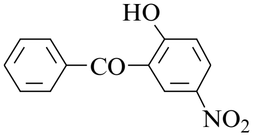
- Preparation by oxidation of 6-nitro-2,3-diphenylbenzofuran with chromium trioxide in acetic acid, followed by saponification of the keto ester so obtained (2-benzoyloxy-4-nitrobenzophenone) in the presence of 2 N sodium hydroxide in refluxing ethanol for 15 min (60%) [535].

- Preparation by reaction of 2-hydroxy-4-nitrobenzoyl chloride with benzene in the presence of aluminium chloride in nitrobenzene at 100–110° for 2.5 h (60%) [536].
- Also obtained (poor yield) by Fries rearrangement of m-nitrophenyl benzoate with aluminium chloride at 170° for 2 h (11%) [536].
- Also obtained (poor yield) by reaction of benzoyl chloride with m-nitrophenol in the presence of aluminium chloride at 170° for 2 h (10%) [537].

m.p. 115° [89], 114–115° [535], 112–113° [96,536], 108° [537];
^{1}H NMR [99], IR [103,104], UV [99,109–111];
pK_a [96,104,115,538]; TLC [116].

(2-Hydroxy-5-nitrophenyl)phenylmethanone

[18803-19-5] $C_{13}H_9NO_4$ mol.wt. 243.22



Syntheses

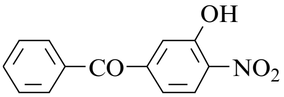
- Preparation by nitration of o-hydroxybenzophenone,
  - with 65% nitric acid in acetic acid at r.t. for 24 h (32%) [96];
  - with a 68% nitric acid/96% sulfuric acid mixture first at <10°, then at r.t. for 2 h (32%) [492].
- Preparation by reaction of potassium hydroxide with 2-chloro-5-nitrobenzophenone,
  - in the presence of a few water at 150–160° for 5–6 h (quantitative yield) [539], (56%) [8];
  - in refluxing aqueous ethylene glycol (135°) for 6 h (17%) (by-product) [523].
- Preparation by oxidation of 5-nitro-2,3-diphenylbenzofuran with chromium trioxide in acetic acid, followed by saponification of the keto ester obtained—2-(benzoyloxy)-5-nitrobenzophenone—with 2 N sodium hydroxide in refluxing ethanol [535].
- Also obtained (by-product) by diazotization of 2-amino-5-nitrobenzophenone (4%). The 2-nitrofluorenone was the major compound obtained (75%) [539].
- Also obtained (poor yield) by Fries rearrangement of p-nitrophenyl benzoate with aluminium chloride in nitrobenzene at 170° for 1 h (4%) [540].
- Preparation from 2-nitrothioxanthen-9-one 10,10-dioxide (SM) by a three-step synthesis: SM by refluxing in a solution of 2% sodium hydroxide in dioxane/water mixture (65:35) for 2 h, gave the 2-(2-hydroxy-5-nitrobenzoyl)phenylsulfinic acid (89%). The former by reaction with mercuric chloride in refluxing aqueous acetic acid for 4 h led to the 2-chloromercuri-2′-hydroxy-5′-nitrobenzophenone (82%). Removal of the chloromercury group was achieved with concentrated hydrochloric acid in refluxing ethanol for 2 h (84%) [62].

- Also obtained by reaction of 2-hydroxy-5-nitrobenzaldehyde with iodobenzene by using a catalyst system of palladium chloride/lithium chloride in the presence of sodium carbonate in N,N-di-methylformamide at 120° for 22 h (58%) [51].
- Preparation by dealkylation of 2-(2-hydroxyethoxy)-5-nitrobenzophenone (SM) in methylene chloride with boron tribromide at r.t. for 18 h (94%) [523]. SM was obtained by reaction of potassium hydroxide with 2-chloro-5-nitrobenzophenone in refluxing aqueous ethylene glycol (135°) for 6 h (65%).
- Also refer to: [429,541–547].

m.p. 128–129° [523], 126°5 [492], 124–124°5 [539], 124° [96,535], 123–124° [62], 122–124° [540], 122–123° [8];
^{1}H NMR [51,98,99], UV [99,109–111], MS [51];
pK_a [96,115,538]; TLC [116].

(3-Hydroxy-4-nitrophenyl)phenylmethanone

[182499-94-1] $C_{13}H_9NO_4$ mol.wt. 243.22



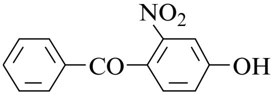
Synthesis

- Refer to: [534].

m.p. and Spectra (NA).

(4-Hydroxy-2-nitrophenyl)phenylmethanone

$C_{13}H_9NO_4$ mol.wt. 243.22



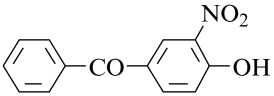
Synthesis

- Obtained (poor yield) by Fries rearrangement of m-nitro-phenyl benzoate with titanium tetrachloride in benzene or in nitromethane at 40° for 25 h (3%) [36].

m.p. and Spectra (NA).

(4-Hydroxy-3-nitrophenyl)phenylmethanone

[5464-98-2] $C_{13}H_9NO_4$ mol.wt. 243.22



Syntheses

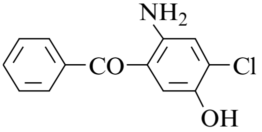
- Preparation by treatment of 4-methylamino-3-nitrobenzo-phenone with boiling 10% aqueous sodium hydroxide for 48 h (52%) [548].
- Preparation by demethylation of 4-methoxy-3-nitrobenzo-phenone with 48% hydrobromic acid in refluxing acetic acid [140].
- Preparation by nitration of 4-hydroxybenzophenone with nitric acid (d = 1.5) in an acetic acid and anhydride acetic mixture at 50–60° [140] or with nitric acid (d = 1.4) at 0° for 2 h [140].
- Also obtained (poor yield) by Fries rearrangement of o-nitrophenyl benzoate with aluminium chloride in nitrobenzene at 165–175° for 1 h (<4%) [540].

- Also obtained from 4-bromo-3-nitrobenzophenone (m.p. 124°) by heating with a mixture of sodium acetate and acetamide at 175–200° for 2 h (good yield) [549].
- Also refer to: [36,550,551].

m.p. 120–121° [549], 94° [140], 91–92° [540], 90° [548]. One of the reported melting points is obviously wrong. Spectra (NA).

(2-Amino-4-chloro-5-hydroxyphenyl)phenylmethanone

[62492-59-5] $C_{13}H_{10}ClNO_2$ mol.wt. 247.68



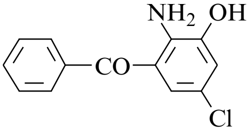
Synthesis

- Preparation by demethylation of 2-amino-4-chloro-5-methoxybenzophenone with boron tribromide in methylene chloride at r.t. for 4 h (86%) [552].

m.p. 165–167° [552]; ^{1}H NMR [552].

(2-Amino-5-chloro-3-hydroxyphenyl)phenylmethanone

[28363-58-8] $C_{13}H_{10}ClNO_2$ mol.wt. 247.68



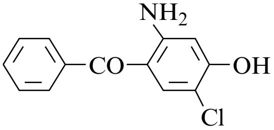
Synthesis

- Obtained by hydrolysis of 2-benzoyl-4-chloro-6-hydroxy-benzanilide with refluxing 2 N aqueous sodium hydroxide for 5 h (80–82%) [552,553].
- Also refer to: [554].

m.p. 166–168° [553], 166–167° [552]; ^{1}H NMR [553].

(2-Amino-5-chloro-4-hydroxyphenyl)phenylmethanone

[62492-60-8] $C_{13}H_{10}ClNO_2$ mol.wt. 247.68



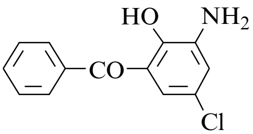
Synthesis

- Preparation by adding acetic anhydride to an ice cooled solution of 2-amino-5-chloro-4-methoxy-benzophenone in 57% aqueous hydriodic acid and heating the resulting mixture at reflux for 12 h (75%) [552].

m.p. 151–153° [552]; ^{1}H NMR [552]; TLC [552].

(3-Amino-5-chloro-2-hydroxyphenyl)phenylmethanone

[85052-43-3] $C_{13}H_{10}ClNO_2$ mol.wt. 247.68



Synthesis

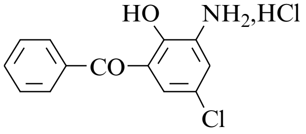
- Obtained from 5-chloro-2-hydroxy-3-nitrobenzophenone,
  - by reduction with stannous chloride and hydrochloric acid in refluxing methanol for 6 h [471];

- by hydrogenation in the presence of 10% Pd/C in a chloroform/ethanol mixture for 2 h [472].

m.p. 94–95° [471], 93–95° [472]; ^{1}H NMR [472], IR [472], MS [472]; TLC [472].

(3-Amino-5-chloro-2-hydroxyphenyl)phenylmethanone *(Hydrochloride)*

[85052-44-4] $C_{13}H_{10}ClNO_2$,HCl mol.wt. 284.15



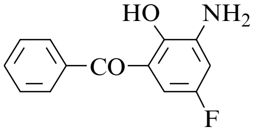
Synthesis

– Obtained by treatment of 3-amino-5-chloro-2-hydroxy-benzophenone with 2 N hydrochloric acid (27%) [472].

m.p. and Spectra (NA).

(3-Amino-5-fluoro-2-hydroxyphenyl)phenylmethanone

$C_{13}H_{10}FNO_2$ mol.wt. 231.23



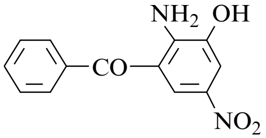
Synthesis

– Refer to: [472].

m.p. and Spectra (NA).

(2-Amino-3-hydroxy-5-nitrophenyl)phenylmethanone

[60302-91-2] $C_{13}H_{10}N_2O_4$ mol.wt. 258.23



Synthesis

– Not yet described.

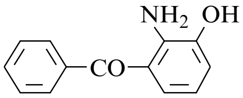
Isolation from natural source

– Major metabolite appearing mainly as conjugated compound, in the urine of rabbits fed nitrazepam. It was isolated after enzymatic hydrolysis with β-glucuro-nidase. -Refer to: Chem. Abstr., **85**, 116484v (1976)^T; **90**, 197343b (1979)^T.

m.p. (NA); ^{1}H NMR^T, IR^T, UV^T, MS^T.

(2-Amino-3-hydroxyphenyl)phenylmethanone

$C_{13}H_{11}NO_2$ mol.wt. 213.24

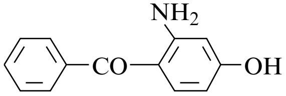


Synthesis

– Preparation by treatment of 4-benzoyl-2,3-dihydro-benzoxazol-2-one (SM) with refluxing 20% hydrochloric acid for 10 h (68%). SM was obtained by UV light irradiation of N-benzoyl-2,3-dihydrobenzoxazol-2-one in acetonitrile for 48 h (21%) [555].

m.p. 129°5–130°5 [555]; Spectra (NA).

(2-Amino-4-hydroxyphenyl)phenylmethanone



$C_{13}H_{11}NO_2$ mol.wt. 213.24

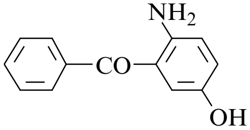
Synthesis

- Refer to: [396].

m.p. and Spectra (NA).

(2-Amino-5-hydroxyphenyl)phenylmethanone

[17562-32-2]



$C_{13}H_{11}NO_2$ mol.wt. 213.24

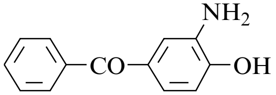
Syntheses

- Preparation by demethylation of 2-amino-5-methoxy-benzophenone with boiling 48% hydrobromic acid [556], for 8 h (85%) [557].
- Also obtained by UV light irradiation of 3-phenyl-2,1-benz-isoxazole in 66% sulfuric acid at 80–90° (88–95%) [121].
- Also obtained by heating N-benzoyl-p-methoxyaniline with bismuth chloride (5 mol excess) at 200–230° for 3 min (72%) [558].

m.p. 127–128° [556,557], 127° [121]; UV [558], MS [558]; TLC [558].

(3-Amino-4-hydroxyphenyl)phenylmethanone

[42404-41-1]



$C_{13}H_{11}NO_2$ mol.wt. 213.24

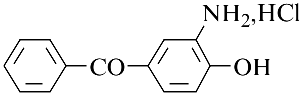
Syntheses

- Preparation by hydrolysis of 3-(acetylamino)-4-hydroxy-benzophenone with refluxing 10 N hydrochloric acid for 30 min (80%) [559].
- Preparation by hydrolysis of 3-benzamido-4-hydroxy-benzophenone (SM) with concentrated hydrochloric acid in refluxing acetic acid for 16 h (50%). SM was obtained by Fries rearrangement of 2-benzamidophenyl benzoate with aluminium chloride for 3 h at 160° under nitrogen (20%, m.p. 210°) [560].

m.p. 164–165° [559], 154° [560]; ^{1}H NMR [559], IR [559].

(3-Amino-4-hydroxyphenyl)phenylmethanone *(Hydrochloride)*

[87855-75-2]



$C_{13}H_{11}NO_2$,HCl mol.wt. 249.70

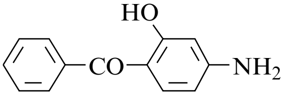
Synthesis

- Refer to: [555,561] (Japanese patents).

m.p. and Spectra (NA).

(4-Amino-2-hydroxyphenyl)phenylmethanone

[3333-96-8] $C_{13}H_{11}NO_2$ mol.wt. 213.24



Syntheses

– Obtained by hydrolysis of 4-acetamido-2-hydroxybenzo-phenone,

- with boiling 5 N hydrochloric acid [562];
- with refluxing 48% hydrobromic acid [557].

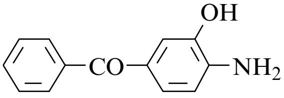
– Preparation by hydrolysis of 4-benzamido-2-hydroxybenzophenone (SM) with concentrated hydrochloric acid in refluxing acetic acid for 16 h (61%). SM was obtained by Fries rearrangement of 3-benzamidophenyl benzoate with aluminium chloride for 3 h at 160° (27%, m.p. 155°) [560].

– Also refer to: [563].

m.p. 127–128° [557], 125° [560,562]; Spectra (NA).

(4-Amino-3-hydroxyphenyl)phenylmethanone

[31684-63-6] $C_{13}H_{11}NO_2$ mol.wt. 213.24



Synthesis

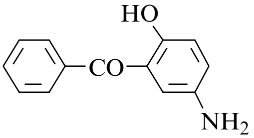
– Preparation from 6-benzoyl-2(3*H*)-benzoxazolinone,

- by alkaline hydrolysis with boiling 10% aqueous sodium hydroxide solution for 4 h (90–100%) [559,564,565], (80%) [566];
- by treatment with refluxing 20% hydrochloric acid during 40 h (88%) [555].

m.p. 164° [559,565,566], 134–135° [555]. One of the reported melting points is obviously wrong. ^{1}H NMR [559], IR [559], MS [564].

(5-Amino-2-hydroxyphenyl)phenylmethanone

[119798-76-4] $C_{13}H_{11}NO_2$ mol.wt. 213.24



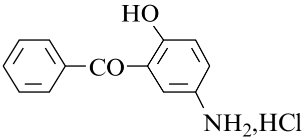
Syntheses

– Preparation by hydrolysis of 5-benzamido-2-hydroxy-benzophenone (SM) with concentrated hydrochloric acid in refluxing acetic acid for 16 h (71%). SM was obtained by Fries rearrangement of 4-benzamidophenyl benzoate with aluminium chloride for 3 h at 160° under nitrogen (40%, m.p. 168°) [560].

– Also obtained from the corresponding hydrochloride by treatment with sodium carbonate in aqueous solution [567].

m.p. 107° [560,567]; Spectra (NA).

(5-Amino-2-hydroxyphenyl)phenylmethanone *(Hydrochloride)*



$C_{13}H_{11}NO_2$,HCl mol.wt. 249.70

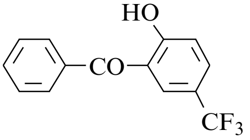
Synthesis

- Obtained by electrolytic reduction of m-nitrobenzophenone in concentrated sulfuric acid for 30 h, followed by action of hydrochloric acid gas in ethyl ether on the amino ketone so obtained [567].

m.p. and Spectra (NA).

[2-Hydroxy-5-(trifluoromethyl)phenyl]phenylmethanone

[72083-16-0] $C_{14}H_9F_3O_2$ mol.wt. 266.22



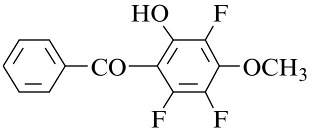
Syntheses

- Preparation by adding phenylmagnesium bromide to 2-methoxy-5-(trifluoromethyl)benzonitrile, followed by hydrolysis of the intermediate imino compound formed, then demethylation of 2-methoxy-5-(trifluoromethyl)-benzophenone so obtained [568].
- Preparation by demethylation of 2-methoxy-5-(trifluoromethyl)benzophenone with boron trichloride in methylene chloride at –60° for 1 h, then at r.t. [569].

m.p. 84–85° [569]; Spectra (NA).

Phenyl(3,5,6-trifluoro-2-hydroxy-4-methoxyphenyl)methanone

[32541-21-2] $C_{14}H_9F_3O_3$ mol.wt. 282.22



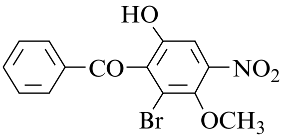
Synthesis

- Preparation by partial demethylation of 2,4-dimethoxy-3,5,6-trifluorobenzophenone in methylene chloride in the presence of aluminium chloride at 20° for 3–6 h (94%) [570].

m.p. 56–58° [570]; ^{1}H NMR [570], IR [570], UV [570].

(2-Bromo-6-hydroxy-3-methoxy-4-nitrophenyl)phenylmethanone

[40990-74-7] $C_{14}H_{10}BrNO_5$ mol.wt. 352.14



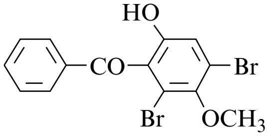
Syntheses

- Preparation by saponification of 2-(benzoyloxy)-6-bromo-5-methoxy-4-nitrobenzophenone (SM) with potassium hydroxide in ethanol (71%). SM was obtained by oxidation of 4-bromo-5-methoxy-6-nitro-2,3-diphenylbenzofuran with chromium trioxide in boiling acetic acid for 30 min [571].
- Also obtained by hydrolysis of 6-bromo-5-methoxy-4-nitro-2-(4-nitrobenzoyloxy)benzophenone [571].

m.p. 126° [571]; Spectra (NA).

(2,4-Dibromo-6-hydroxy-3-methoxyphenyl)phenylmethanone

[40990-66-7] $C_{14}H_{10}Br_2O_3$ mol.wt. 386.04



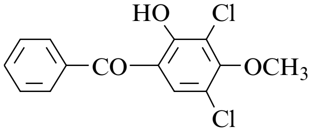
Synthesis

- Preparation by saponification of 2-(benzoyloxy)-4,6-di-bromo-5-methoxybenzophenone (SM) with potassium hydroxide in ethanol (65%). SM was obtained by oxidation of 4,6-dibromo-5-methoxy-2,3-diphenylbenzofuran with chromium trioxide in refluxing acetic acid for 30 min [571].

m.p. 138° [571]; Spectra (NA).

(3,5-Dichloro-2-hydroxy-4-methoxyphenyl)phenylmethanone

[158547-83-2] $C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14



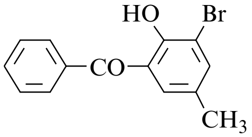
Synthesis

- Formation (trace) by 30 min UV light irradiation of oxybenzone in chlorinated water containing 5 ppm Cl [572].

m.p. (NA); MS [572]; GC [572].

(3-Bromo-2-hydroxy-5-methylphenyl)phenylmethanone

[6723-09-7] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



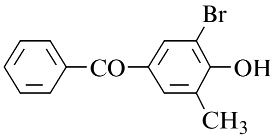
Synthesis

- Preparation by bromination of 2-hydroxy-5-methylbenzophenone [502], in aqueous acetic acid (86%) [573].
- Also refer to: [574–577].

m.p. 82° [501], 80–82° [502], 77–78° [573]; IR [103].

(3-Bromo-4-hydroxy-5-methylphenyl)phenylmethanone

$C_{14}H_{11}BrO_2$ mol.wt. 291.14



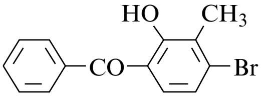
Synthesis

- Preparation by action of bromine on 4-hydroxy-3-methyl-benzophenone in acetic acid [578].

m.p. 130–131° [578]; Spectra (NA).

(4-Bromo-2-hydroxy-3-methylphenyl)phenylmethanone

[6758-89-0] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



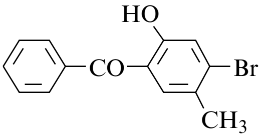
Synthesis

– Obtained by chromic oxidation (CrO_3) of 6-bromo-7-methyl-2,3-diphenylbenzofuran, followed by alkaline hydrolysis of keto ester so obtained [501,502].

m.p. 96° [501,502]; IR [103,501], UV [503].

(4-Bromo-2-hydroxy-5-methylphenyl)phenylmethanone

[6723-07-5] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



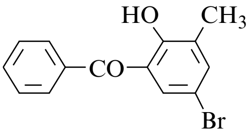
Synthesis

– Preparation by reaction of chromium trioxide with 6-bromo-5-methyl-2,3-diphenylbenzofuran in acetic acid, followed by saponification of the keto ester formed (2-benzoyloxy-4-bromo-5-methylbenzophenone) with sodium hydroxide in ethanol [501], (73%) [502].

m.p. 122° [501,502]; ^{1}H NMR [100], IR [100], UV [503].

(5-Bromo-2-hydroxy-3-methylphenyl)phenylmethanone

[6723-13-3] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



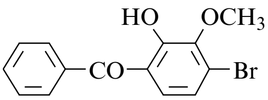
Synthesis

– Preparation by bromination of 2-hydroxy-3-methylbenzophenone [501], in chloroform [502].

m.p. 83° [501,502]; IR [103,106,107,501].

(4-Bromo-2-hydroxy-3-methoxyphenyl)phenylmethanone

[65202-49-5] $C_{14}H_{11}BrO_3$ mol.wt. 307.14



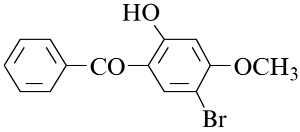
Synthesis

– Preparation by saponification of 2-(benzoyloxy)-4-bromo-3-methoxybenzophenone (SM) with potassium hydroxide in boiling ethanol (70%). SM was obtained by oxidation of 6-bromo-7-methoxy-2,3-diphenylbenzofuran with chromium trioxide in boiling acetic acid (65%) [579].

m.p. 240–243° [579]; Spectra (NA).

(5-Bromo-2-hydroxy-4-methoxyphenyl)phenylmethanone

[3286-93-9] $C_{14}H_{11}BrO_3$ mol.wt. 307.14



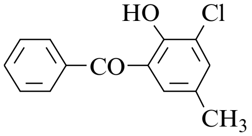
Synthesis

– Preparation from 2-(benzoyloxy)-5-bromo-4-methoxy-benzophenone (SM) by treatment with potassium hydroxide in refluxing ethanol for 1 h (68%). SM was obtained by oxidation of 5-bromo-6-methoxy-2,3-di-phenylbenzofuran with chromium trioxide in refluxing acetic acid for 30 min [571].

m.p. 125° [571]; Spectra (NA).

(3-Chloro-2-hydroxy-5-methylphenyl)phenylmethanone

$C_{14}H_{11}ClO_2$ mol.wt. 246.69



Syntheses

– Preparation by Fries rearrangement of 2-chloro-4-methyl-phenyl benzoate with aluminium chloride without solvent,

- at 140° for 10 min (92%) [132];
- at 120–130° for 1 h (67%) [519];
- at 160° for 15 min (14%) [580] (here, by using an old aluminium chloride).

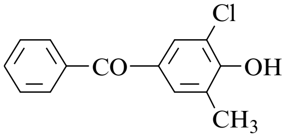
– Also obtained by action of aluminium chloride with esters mixtures^T without solvent at 150° for 15 min [580],

^{T}2-chloro-4-methylphenyl benzoate and p-tolyl acetate (50% yield);
^{T}p-tolyl benzoate and 2-chloro-4-methylphenyl acetate (40% yield);
^{T}2-chloro-4-methylphenyl benzoate and mesityl acetate (14% yield).

m.p. 81° [519], 71° [132,580]; Spectra (NA).

(3-Chloro-4-hydroxy-5-methylphenyl)phenylmethanone

[34183-08-9] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



Synthesis

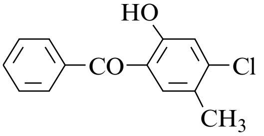
– Preparation by Fries rearrangement of 2-chloro-6-methyl-phenyl benzoate with aluminium chloride,

- in chlorobenzene at 140–150° for 20 min or in nitrobenzene at 75° for 24 h [480];
- without solvent at 160° for 45 min (45%) [581].

m.p. 126–127° [480]; Spectra (NA).

(4-Chloro-2-hydroxy-5-methylphenyl)phenylmethanone

[33561-92-1] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



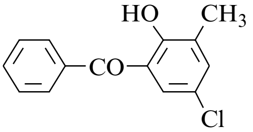
Syntheses

- Obtained by Fries rearrangement of 3-chloro-4-methyl-phenyl benzoate with aluminium chloride at 200° for 20 min [20].
- Also obtained by demethylation of 4-chloro-2-methoxy-5-methylbenzophenone with borontribromide in methylene chloride at r.t. for 12 h [20], according to [21].
- Also obtained by oxidation of 6-chloro-5-methyl-2,3-diphenylbenzofuran with chromium trioxide in boiling acetic acid for 2 h, followed by saponification of the 2-(benzoyloxy)-4-chloro-5-methyl-benzophenone formed with 2 N sodium hydroxide in boiling ethanol for 15 min [515].
- Also refer to: [107,582].

m.p. 108–109° [515]; IR [107,515,582].

(5-Chloro-2-hydroxy-3-methylphenyl)phenylmethanone

[53347-30-1] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



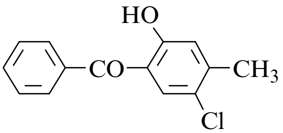
Synthesis

- Refer to: [83] (compound **7e**) and [82,583].

m.p. and Spectra (NA).

(5-Chloro-2-hydroxy-4-methylphenyl)phenylmethanone

[68751-90-6] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



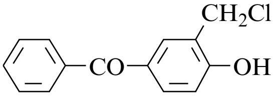
Syntheses

- Preparation by Fries rearrangement of 4-chloro-3-methyl-phenyl benzoate in the presence of,
  - aluminium chloride [23] without solvent at 140° for 10 min (quantitative yield) [132];
  - Nafion-XR, a H^+-form ion exchange resin, at 175° for 4 h (37%) [39,204].
- Preparation by oxidation of 5-chloro-6-methyl-2,3-diphenylbenzofuran with chromium trioxide in boiling acetic acid for 40 min, followed by saponification of the keto ester so obtained with 4 N sodium hydroxide in refluxing ethanol for 1 h [44].
- Also obtained by reaction of benzoyl chloride with 4-chloro-3-methylphenol in the presence of Nafion-XR at 175° for 4 h (28%) [39].
- Also refer to: [77,584,585].

m.p. 142° [132], 140° [44]; IR [44].

[3-(Chloromethyl)-4-hydroxyphenyl]phenylmethanone

[14898-76-1] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



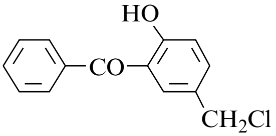
Syntheses

- Preparation by passing hydrogen chloride through a mixture of 40% formaldehyde solution, concentrated hydrochloric acid and p-hydroxybenzophenone in acetic acid at r.t. for 2 h (64%) [277].
- Other chloromethylation process [586], (73%) [164].
- Also refer to: [587].

m.p. 156° (d) [277]; Spectra (NA).

[5-(Chloromethyl)-2-hydroxyphenyl]phenylmethanone

[120973-82-2] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



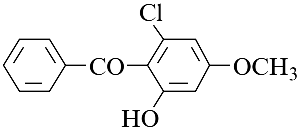
Synthesis

- Refer to: [588].

m.p. and Spectra (NA).

(2-Chloro-6-hydroxy-4-methoxyphenyl)phenylmethanone

[136741-50-9] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



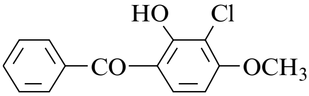
Synthesis

- Preparation by reaction of benzoyl chloride with 5-chlororesorcinol dimethyl ether in the presence of aluminium chloride in ethylene dichloride (72%) [589].

m.p. 96–98° [589]; Spectra (NA).

(3-Chloro-2-hydroxy-4-methoxyphenyl)phenylmethanone

[158547-82-1] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



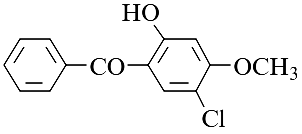
Synthesis

- Formation (trace) by 30 min UV light irradiation of oxybenzone in chlorinated water containing 5 ppm Cl [572].

m.p. (NA); MS [572]; GC [572].

(5-Chloro-2-hydroxy-4-methoxyphenyl)phenylmethanone

[3286-91-7] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



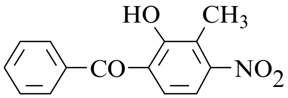
Syntheses

- Preparation by reaction of benzoyl chloride with 4-chlororesorcinol dimethyl ether in the presence of aluminium chloride in ethylene dichloride (66%) [589].
- Formation (trace) by 30 min UV light irradiation of oxybenzone in chlorinated water containing 5 ppm Cl [572].

m.p. 118–119° [589]; ^{1}H NMR [589], IR [589], MS [572,589]; GC [572].

(2-Hydroxy-3-methyl-4-nitrophenyl)phenylmethanone

[4072-22-4] $C_{14}H_{11}NO_4$ mol.wt. 257.25



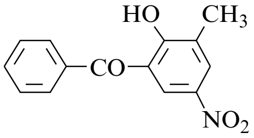
Synthesis

- Preparation by oxidation of 7-methyl-6-nitro-2,3-diphenyl-benzofuran with chromium trioxidein acetic acid, followed by saponification of the keto ester so formed—the 2-(benzoyloxy)-3-methyl-4-nitrobenzophenone—with sodium hydroxide in boiling dilute ethanol (70%) [590].

m.p. 98° [590], 97° [89]; ^{1}H NMR [100], IR [100,103,107,582], UV [503].

(2-Hydroxy-3-methyl-5-nitrophenyl)phenylmethanone

[18619-93-7] $C_{14}H_{11}NO_4$ mol.wt. 257.25



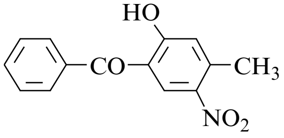
Synthesis

- Preparation by reaction of nitric acid (d = 1.42) with 2-hydroxy-3-methylbenzophenone in acetic acid under stirring overnight at r.t. (70%) [590].

m.p. 122° [590]; IR [103].

(2-Hydroxy-4-methyl-5-nitrophenyl)phenylmethanone

[68430-99-9] $C_{14}H_{11}NO_4$ mol.wt. 257.25



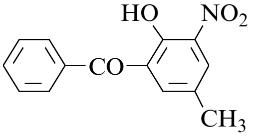
Synthesis

- Refer to: [591].

m.p. and Spectra (NA).

(2-Hydroxy-5-methyl-3-nitrophenyl)phenylmethanone

[4072-26-8] $C_{14}H_{11}NO_4$ mol.wt. 257.25



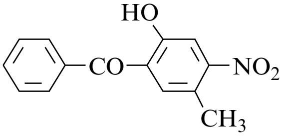
Synthesis

- Preparation by nitration of 2-hydroxy-5-methyl-benzo-phenone with nitric acid (d = 1.42) in acetic acid overnight at r.t. (82%) [590] or at 32° [592].
- Also refer to: [574,593].

m.p. 68° [89], 67–68° [590]; IR [103].

(2-Hydroxy-5-methyl-4-nitrophenyl)phenylmethanone

[4072-24-6] $C_{14}H_{11}NO_4$ mol.wt. 257.25



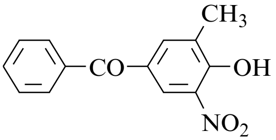
Synthesis

- Preparation by oxidation of 5-methyl-6-nitro-2,3-diphenyl-benzofuran with chromium trioxide in acetic acid, followed by saponification of the keto ester so formed—the 2-(benzoyloxy)-5-methyl-4-nitrobenzophenone—with sodium hydroxide in boiling dilute ethanol (86%) [590].

m.p. 92° [89,590]; IR [103,107,582].

(4-Hydroxy-3-methyl-5-nitrophenyl)phenylmethanone

[103555-87-9] $C_{14}H_{11}NO_4$ mol.wt. 257.25



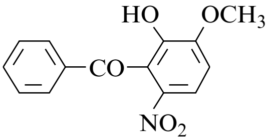
Synthesis

- Preparation from o-methylanisole in three steps: at first, acylation of o-methylanisole with benzoyl chloride in the presence of ferric chloride during 3 h at 20°. Then, demethylation of the 4-methoxy-3-methylbenzophenone so formed by treatment with refluxing 55% hydriodic acid for 10 h and subsequent nitration of the 4-hydroxy-3-methylbenzophenone so obtained with a concentrated nitric acid/concentrated sulfuric acid mixture with ice cooling (57%) [594].

m.p. and Spectra (NA).

(2-Hydroxy-3-methoxy-6-nitrophenyl)phenylmethanone

[65202-46-2] $C_{14}H_{11}NO_5$ mol.wt. 273.25



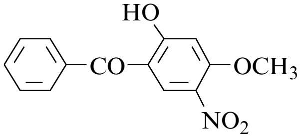
Synthesis

- Preparation by saponification of 2-(benzoyloxy)-3-methoxy-6-nitrobenzophenone (SM) with potassium hydroxide in boiling ethanol (70%). SM was obtained by oxidation of 7-methoxy-4-nitro-2,3-diphenylbenzofuran with chromium trioxide in boiling acetic acid (80%) [579].

m.p. 72–73° [579]; Spectra (NA).

(2-Hydroxy-4-methoxy-5-nitrophenyl)phenylmethanone

[41123-21-1] $C_{14}H_{11}NO_5$ mol.wt. 273.25



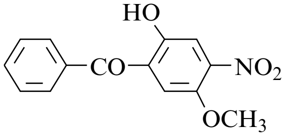
Synthesis

- Preparation by saponification of 2-(benzoyloxy)-4-methoxy-5-nitrobenzophenone (SM) with potassium hydroxide in ethanol (67%). SM was obtained by oxidation of 6-methoxy-5-nitro-2,3-diphenylbenzofuran with chromium trioxide in boiling acetic acid for 30 min [571].

m.p. 132° [571]; ^{1}H NMR [98].

(2-Hydroxy-5-methoxy-4-nitrophenyl)phenylmethanone

[40990-72-5] $C_{14}H_{11}NO_5$ mol.wt. 273.25



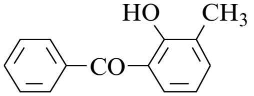
Synthesis

- Preparation by saponification of 2-(benzoyloxy)-5-methoxy-4-nitrobenzophenone (SM) with potassium hydroxide in ethanol (78%). SM was obtained by oxidation of 5-methoxy-6-nitro-2,3-diphenylbenzofuran with chromium trioxide in boiling acetic acid for 30 min [571].

m.p. 102° [571]; Spectra (NA).

(2-Hydroxy-3-methylphenyl)phenylmethanone

[4072-08-6] $C_{14}H_{12}O_2$ mol.wt. 212.25



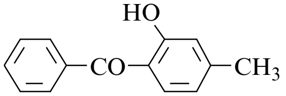
Syntheses

- Preparation by reaction of 2-hydroxy-3-methylbenzoyl chloride with benzene in the presence of aluminium chloride at 40–50° for 4 h (72%) [595], (23%) [92].
- Preparation by oxidation of 7-methyl-2,3-diphenylbenzofuran with chromium trioxide in refluxing acetic acid for 40 min, followed by hydrolysis of the keto ester so obtained (2-benzoyloxy-3-methylbenzophenone) with 4 N sodium hydroxide in refluxing ethanol for 1 h [44], (50%) [590].
- Preparation by reaction of benzoic acid with o-cresol in the presence of Amberlyst-15 in refluxing chlorobenzene for 47 h (50%) [53].
- Also obtained (by-product) by Fries rearrangement of o-tolyl benzoate in the presence of aluminium chloride in refluxing chlorobenzene for 4 h (<6%) [596].
- Also obtained (poor yield) by reaction of benzoyl chloride with o-tolyl borate in the presence of aluminium chloride in refluxing carbon disulfide for 2 h (2%) [55].
- Preparation by reaction between (o-tolyloxy)magnesium bromide complexed with HMPT and benzaldehyde in refluxing benzene for 48 h (54%) [50].

– Also refer to: [597].
yellow oil [50];
m.p. 48° [89], 47–48° [590], 47° [44];
b.p.$_2$ 152–155° [595], b.p.$_{0.1}$ 114–117° [92];
^{1}H NMR (Sadtler: standard n° 57894 M) [50,100],
IR (Sadtler: standard n° 84942 K) [50,89,100,103], UV [597], MS [50].

(2-Hydroxy-4-methylphenyl)phenylmethanone

[3098-18-8] $C_{14}H_{12}O_2$ mol.wt. 212.25



Syntheses

– Preparation by Friedel–Crafts acylation of m-cresol,

- with benzoic acid in the presence of boron trifluoride at 160° for 2 h (94%) [150] or in the presence of Amberlyst-15 in refluxing chlorobenzene for 65 h (37%) [53];
- with benzoyl chloride in the presence of aluminium chloride at 75° [144], in nitrobenzene at 60° for 18 h (8%) [54] or with titanium tetrachloride in the same conditions (17%) [54].

– Preparation by Fries rearrangement of m-tolyl benzoate,

- in the presence of aluminium chloride,

  without solvent
  at 200° for 5 min (81%) [598] or 20 min [20], at 175° for 15 min (95%) [132], (82%) [36], (31%) [599], at 140–150° [600], at 140° for 15 min (89%) [27], at 130–134° for 24 h (18%) [4] at 90° for a short time (50%) [601], at 60–70° for 1 h (40%) [602],
  with solvent
  in nitrobenzene, at 60–63° for 13–18 h (15%) [27,31,603];

- in the presence of titanium tetrachloride,

  without solvent
  at 175° for 15 min (75%) [36], at 140° for 15 min (82%) [27],
  with solvent
  in nitrobenzene at 60° for 18 h (27%) [27].

– Preparation from 2-methoxy-4-methylbenzophenone by demethylation,

- with refluxing pyridinium chloride for 1 h (74%) [604];
- with boron tribromide in methylene chloride at r.t. for 12 h [20], according to [21].

– Preparation by dehydrogenation of 6-benzoyl-3-methyl-2-cyclohexen-1-one by heating at reflux for 30 min in the presence of 5% Pd/BaSO$_4$ catalyst [605].

– Preparation by oxidation of various substituted benzofurans^T with chromium trioxide in boiling acetic acid, followed by saponification of the keto ester so formed (good yields), ^{T}6-methyl-2,3-diphenylbenzofuran [44,606], 6-methyl-

3-phenyl-2-(4-methoxyphenyl)-benzofuran or 6-methyl-3-phenyl-2-(4-methylphenyl) benzofuran [46].

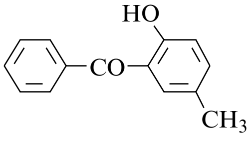
- Also obtained by isomerization of 4-hydroxy-2-methylbenzophenone with aluminium chloride at 180–190° for 20 min (92%) [132].
- Also obtained by condensation of m-cresol with benzotrichloride in the presence of aqueous sodium hydroxide in a water bath for 4 h (7%) [48].
- Also obtained (poor yield) by diazotization of 2-amino-4-methylbenzophenone, followed by hydrolysis of the diazonium salt so obtained (18%) [607].
- Also obtained by reaction of benzoyl chloride with m-tolyl borate in the presence of aluminium chloride in refluxing carbon disulfide for 2 h (19%) [55].
- Also obtained by photo-Fries rearrangement of m-tolyl benzoate,
  - in water in the presence of sodium dodecyl sulfate (a micelle) for 7 h under nitrogen (73%) [70];
  - in ethanol or in the presence of β-cyclodextrin solid for 12–24 h (29% and 96% yields, respectively) [65].
- Preparation by reaction of 2-methoxy-4-methylbenzoyl chloride with benzene in the presence of aluminium chloride in tetrachloroethane [6], according to [14].
- Also refer to: [76,226,608].

yellow oil [598];

m.p. 65° [89,609], 64° [600], 63° [27,144,599,601,603,606], 62°5 [150],
61–63° [70], 61–62° [604], 61° [6,602], 60–61° [96],
60° [46,48,607], 59–60° [605], 59° [44], 58° [4];
b.p.$_{15}$ 233–234° [602], b.p.$_{17}$ 230–240° [604], b.p.$_{14}$ 195–215° [599,601];
^{1}H NMR (Sadtler: standard n° 20031 M) [98,99,598],
IR (Sadtler: standard n° 47040) [44,70,103,609], UV [31,99,109–111,609];
TLC [116]; pK_a [96,115]; polarographic study [117].

(2-Hydroxy-5-methylphenyl)phenylmethanone

[1470-57-1] $C_{14}H_{12}O_2$ mol.wt. 212.25



Syntheses

- Preparation by Fries rearrangement of p-tolyl benzoate,
  - with aluminium chloride [23],

without solvent

at 180° for 10 min (80%) [518], at <150° for 8 min (94%) [132], at 140° (71%) [97], according to [462], at 140° for 10–15 min (quantitative yield) [132], (88%) [27], (80%) [599], at 140° for 20 min, then for a short time at 200° (94%) [601,610], at 140° for 1 h [96], at 130–140° for 1 h (88%) [519] or at 130° for 30 min (55%) [611],

with solvent
in refluxing o-dichlorobenzene for 3 h or in refluxing p-chlorotoluene for 1 h (89%) [520];
in refluxing 1,2,4-trichlorobenzene for 1 h (85%) [520];
in refluxing chlorobenzene for 1 h (63%) [520] or for 4 h (82%) [520], (37%) [596];
in refluxing nitromethane for 30 min (12%) [36];

- with titanium tetrachloride,

  without solvent
  at 140° for 15 min (78%) [27],
  with solvent
  in refluxing nitromethane for 10 min (41%) [36];

- with beryllium chloride at 145–150° for 30 min, then at 160° for a short time (69%) [612,613];
- with Nafion-XR, a H^+-form ion exchange resin, at 150° for 4 h under nitrogen (52%) [39];
- with Nafion-H, a polymeric perfluorinated resin sulfonic acid in refluxing nitrobenzene for 12 h (70%) [38];
- with hydrofluoric acid at 55° for 4 h or 6 h (52% and 76% yields, respectively) [135].

– Preparation by photo-Fries rearrangement of p-tolyl benzoate,

- in water in the presence of sodium dodecyl sulfate (a micelle) for 7 h under nitrogen (95%) [70];
- in benzene at 55° for 19 h (53%) [72];
- in pentane (37%) or in the presence of silica gel (42%) [64];
- in isopropanol at 57° for 20 h (34%) [72];
- in cyclohexane at 52° for 23 h (25%) [72];
- in dioxane at 63° for 24 h (20%) [72];
- in ethanol in the presence of sulfuric acid for 70 h [614].

– Preparation by condensation of benzotrichloride with p-cresol,

- in the presence of aluminium chloride in carbon disulfide for 2 h (75%) [125]. The 6,12-diphenyl-2,8-dimethyl-6,12-epoxy-*6H,12H*-dibenzo[b,f][1,5] dioxocin, an intermediate compound, was also formed under these conditions (29%). This "dioxocin", by hydrolysis with concentrated sulfuric acid at r.t., gave the expected ketone (91%) [125];
- in the presence of aqueous sodium hydroxide in a water bath for 4 h (33%) [48] or in 32–40% aqueous sodium hydroxide at 70–80° (67%) [615].

– Preparation by Friedel–Crafts acylation of p-cresol with benzoyl chloride,

- in the presence of aluminium chloride,
  without solvent
  at 180° for 30 min (64–72%) [616],

with solvent

in tetrachloroethane at 100–110° for 4 h (80%) [15,617];

in nitrobenzene at 60° for 18 h (33%) [54];

in carbon disulfide, in addition with ethyl iodide, by heating in a water bath [190]. An intermediate compound develops at the start of reaction (4-methylphenetole);

- in the presence of titanium tetrachloride, in nitrobenzene at 60° for 18 h (51%) [54].

- Preparation by reaction of chromium trioxide with 2,3-diphenyl-5-methylbenzofuran in acetic acid at 60° for 2.5 h [43], at 80° for 3 h [606] or at reflux for 40 min [44], followed by saponification of the keto ester formed with sodium hydroxide [43,44], (74%) [606].

  The same ketone was also obtained by chromic oxidation of 5-methyl-2-(4-methylphenyl)-3-phenylbenzofuran or of 5-methyl-2-(4-methoxyphenyl)-3-phenylbenzofuran, followed by saponification of the keto ester so formed with potassium hydroxide [46].
- Preparation by diazotization of 2-amino-5-methylbenzophenone, followed by hydrolysis of the diazonium salt so obtained (25%) [607].
- Also obtained by reaction of benzoyl chloride,
  - with p-methylanisole in the presence of aluminium chloride in carbon disulfide [618];
  - with p-methylphenetole in the presence of aluminium chloride, followed by treatment of the 2-ethoxy-5-methylbenzophenone so formed with aluminium chloride in carbon disulfide at 60–70° for 8 h [141,619], according to [139];
  - with p-tolyl borate in the presence of aluminium chloride in refluxing carbon disulfide for 2 h (15%) [55].
- Also obtained (poor yield) by photo-Fries rearrangement of 2-methoxy-4-methylphenyl benzoate (creosol benzoate) in benzene or ethanol for 4 h (6–7%) [620].
- As an historical curiosity, this compound has also been obtained by Fries rearrangement of p-tolyl benzoate with aluminium chloride in the presence of another aromatic compounds, at 150° for 15 min [580]: mesitol (83%); 2,6-dimethylphenyl acetate (53%); 2-chloro-4-methylphenyl acetate (20–34%); mesityl acetate (18%).
- Also refer to: [36,38,73,77,78,82,83,530,574,592,615,621–628].

m.p. 87° [519,601], 85° [27,169], 84°5 [617], 84–85° [70], 84° [15,48,89,96,132,580,599,607,609,613,619,629], 83°6–84° [125], 83°5–85° [97,135], 83°5–84° [520,611], 83–83°5 [618], 83° [518], 82–83° [43], 82–82°5 [615], 82° [190,237,590,606], 81–84° [92], 81° [44,46], 79–82° [72], 70–72° [610];

b.p.$_{1.5}$ 153–156° [97], b.p.$_{1-2}$ 119–122° [125];

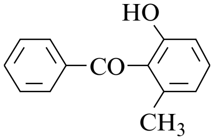
^{1}H NMR [97,99,100,610,615,629], ^{13}C NMR [97],

IR [44,97,100,103,104,106,107,610,615,629],

UV [99,100,109–111,615,629,630], MS [615];
pK_a [96,104,115,531,629];
TLC [116,278]; gas chromatography study [631];
polarographic study [117]; cryoscopic study [141].

(2-Hydroxy-6-methylphenyl)phenylmethanone

[50597-28-9] $C_{14}H_{12}O_2$ mol.wt. 212.25



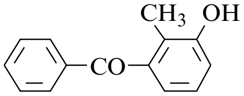
Syntheses

- Preparation from 4-methyl-2,3-diphenylbenzofuran by oxidation with chromium trioxide in acetic acid at 60° for 3 h, followed by saponification of the keto ester so formed with 10% sodium hydroxide in boiling ethanol [632,633].
- Preparation by Fries rearrangement of m-cresyl benzoate with trifluoromethanesulfonic acid in a sealed tube at 170° for 24 h (49%) [37].
- Also obtained from 2-methyl-6-nitroaniline by a eight-step synthesis. The final step consists in making to react sodium iodide and trimethylsilyl chloride with 2-methoxy-6-methylbenzophenone in acetonitrile at 130° for 24 h into an autoclave (15%) [6].
- Also obtained by photo-Fries rearrangement of m-cresyl benzoate in ethanol for 24 h (25%) and in the presence of β-cyclodextrin (34%) [65].

m.p. 73° [6], 57–63° [632]; ^{13}C NMR [6], MS [6].

(3-Hydroxy-2-methylphenyl)phenylmethanone

[74167-87-6] $C_{14}H_{12}O_2$ mol.wt. 212.25



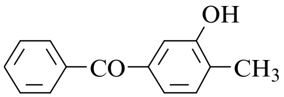
Synthesis

- Preparation by diazotization of 3-amino-2-methylbenzophenone, followed by hydrolysis of the diazonium salt obtained (56%) [119], according to [634].

m.p. 123° [119]; Spectra (NA).

(3-Hydroxy-4-methylphenyl)phenylmethanone

$C_{14}H_{12}O_2$ mol.wt. 212.25



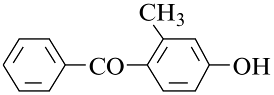
Synthesis

- Preparation by diazotization of 3-amino-4-methylbenzo-phenone, followed by hydrolysis of the diazonium salt obtained (76%) [635].

m.p. 132–133° [635]; Spectra (NA).

(4-Hydroxy-2-methylphenyl)phenylmethanone

[10425-07-7] $C_{14}H_{12}O_2$ mol.wt. 212.25



Syntheses

– Preparation by Fries rearrangement of m-tolyl benzoate,

- in the presence of aluminium chloride,

  without solvent

  at 60–70° for 1 h, then at r.t. for 24 h (62%) [602]; at 90° for a short time (32%) [601]; at 120° for 80 min (40%) [132]; at 130–134° for 24 h (21%) [4] or at 160° for 2 h [490,491],

  with solvent

  in nitrobenzene at 25–30° for 90 h (45%) [31]; at 60° for 5 h (60%) [132], (47%) [603], for 13 h (51%) [603] or for 18 h (67%) [27]; or at 62–63° for 18 h (42%) [31];
  in chlorobenzene at reflux for 4 h (46%) [596];
  in nitromethane at reflux for 30 min (33%) [36];

- in the presence of titanium tetrachloride, in refluxing nitromethane for 30 min (44%) [36];
- in the presence of Nafion-XR, a H^+-form ion exchange resin, at 150° for 4 h (67%) [39].

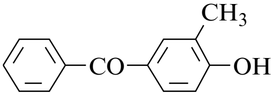
– Also obtained from p-benzoylthymol by elimination of isopropyl group with aluminium chloride in chlorobenzene at r.t. for 20 h, then at 50° for 4 h (80%) [636].
– Preparation by demethylation of 4-methoxy-2-methylbenzophenone with refluxing pyridinium chloride for 1 h (74%) [604].
– Also obtained by condensation of benzotrichloride with m-cresol in the presence of aqueous sodium hydroxide in a water bath for 4 h (6%) [48].
– Preparation by reaction of benzoyl chloride with m-cresol in the presence of aluminium chloride or titanium tetrachloride in nitrobenzene at 60° for 18 h (67% and 61% yields, respectively) [54]. The same reaction with aluminium chloride without solvent at 75° gave a 10% yield [144].
– Also obtained (poor yield) by reaction of benzoyl chloride with m-tolyl borate in the presence of aluminium chloride in refluxing carbon disulfide for 2 h (5%) [55].
– Preparation by acylation of m-tolyl benzoate with benzoyl chloride in the presence of zinc chloride at 130° for 1 h, followed by saponification of the p-keto ester so formed with sodium hydroxide in boiling ethanol [637].
– Also obtained by photo-Fries rearrangement of m-tolyl benzoate in ethanol during 24 h (30%) [65].

m.p. 135–136° [4], 130° [6], 129° [27,144,601–604],
128° [48,636,637]; b.p.$_{15}$ 235–240° [602], b.p.$_{13}$ 220–240° [601], b.p.$_{17}$ 230–240° [604];

^{1}H NMR (Sadtler: standard n° 20030 M), ^{13}C NMR [6],
IR (Sadtler: standard n° 47039), UV [31,602], MS [490,491].

(4-Hydroxy-3-methylphenyl)phenylmethanone

[5326-42-1] $C_{14}H_{12}O_2$ mol.wt. 212.25



Syntheses

- Preparation by Fries rearrangement of o-tolyl benzoate,
  - with aluminium chloride,

    without solvent

    at 160° for 1 h [164], (90–91%) [601,638] or 2 h [490,491]; at 140° for 15 min (quantitative yield) [132], (86%) [27], (70%) [639], (10%) [599] and for 4 h [573],

    with solvent

    in nitrobenzene at 60° for 18 h (91%) [27], in phenyl ether at 170° for 30 min (68%) [640], in refluxing nitromethane for 30 min (60%) [36] or in refluxing chlorobenzene for 4 h (45%) [596];
  - with titanium tetrachloride, in nitrobenzene at 60° for 18 h (89%) [27] or in refluxing nitromethane for 30 min (86%) [36];
  - with Nafion-XR, a H^+-form ion exchange resin, at 175° for 4 h under nitrogen (45%) [39].
- Also obtained by photo-Fries rearrangement of o-tolyl benzoate in ethanol between 60 and 75 h (20%) [641].
- Preparation by Friedel–Crafts acylation of o-cresol with benzoyl chloride,
  - in the presence of aluminium chloride in nitrobenzene at 60° for 18 h (90%) [54] or without solvent [642], at 75° (31%) [144];
  - in the presence of titanium tetrachloride in nitrobenzene at 60° for 18 h (87%) [54].
- Also obtained by reaction of benzoyl chloride,
  - with o-bromotoluene in the presence of aluminium chloride at 75° for 5 h (38%) [144];
  - with o-tolyl borate in the presence of aluminium chloride in refluxing carbon disulfide for 2 h (51%) [55];
  - with o-tolyl benzoate in the presence of zinc chloride at 130° for 1 h, followed by saponification of the p-keto ester formed with sodium hydroxide in boiling ethanol [643].
- Also obtained by treatment of p-benzoylcarvacrol with aluminium chloride in chlorobenzene, first at r.t. for 20 h, then at 50° for 4 h (68%) [636].
- Also obtained by reaction of benzotrichloride with o-cresol in the presence of aqueous sodium hydroxide at 80° several hours [644] or in a water bath for 4 h (22%) [48].
- Also obtained by heating a mixture of benzoic acid and o-cresol in the presence of Tonsil [645].

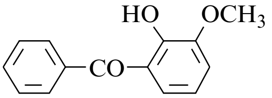
- Preparation by diazotization of 4-amino-3-methylbenzophenone, followed by hydrolysis of the resulting diazonium salt (76%) [635].
- Also refer to: [646,647].

m.p. 174–175° [635], 173–175° [490,491], 173–174° [599,601,641], 173° [27,144,636,640], 172–173° [48], 172–172°5 [643], 172° [132,573,645], 170–171° [644], 169° [639], 163° [642];

b.p.$_{12-15}$ 240–260° [599,601,638]; Spectra (NA).

(2-Hydroxy-3-methoxyphenyl)phenylmethanone

[65202-31-5] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

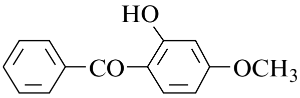
- Preparation by saponification of 2-benzoyloxy-3-methoxy-benzophenone (SM) with potassium hydroxide in refluxing ethanol for 1 h (73%) [579]. SM was obtained by oxidation of 7-methoxy-2,3-diphenylbenzofuran with chromium trioxide in refluxing acetic acid for 30 min (78%).
- Preparation by reaction of phenylmagnesium bromide with 2-hydroxy-3-methoxybenzonitrile in ethyl ether in a water bath for 2.5 h (88%) [648].
- Also obtained by reaction of 2-hydroxy-3-methoxybenzaldehyde with iodobenzene by using a catalyst system of palladium chloride/lithium chloride in the presence of sodium carbonate in

N,N-dimethylformamide at 100° for 8 h (70%) [51].

b.p.$_3$ 220–225° [648]; m.p. 59° [648]; ^{1}H NMR [51], MS [51].

(2-Hydroxy-4-methoxyphenyl)phenylmethanone *(Cyasorb UV-9, Oxybenzone, Sumisorb 110)*

[131-57-7] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

- Preparation by partial methylation of 2,4-dihydroxy-benzophenone [649,650],
  - with methyl iodide in the presence of sodium hydroxide [189];
  - with a methyl halide [651];
  - with dimethyl sulfate in the presence of sodium hydroxide [189] or alkaline solution [212,652].
- Also obtained by partial demethylation of 2,4-dimethoxybenzophenone (SM),
  - with aluminium chloride in refluxing carbon disulfide for 30 min [85,146,188,653], (42%) [205]. SM was prepared by reaction of benzoyl chloride with resorcinol dimethyl ether in the presence of aluminium chloride (good yield) [205];
  - with aluminium chloride in nitrobenzene [85,654];
  - with aluminium chloride or aluminium bromide in chlorobenzene at 90–95° (good yield) [655];

  - with excess beryllium chloride in refluxing toluene for 3 h (90%) [395];
  - in refluxing 40% hydrobromic acid for 5 h [85].
- Preparation by Friedel–Crafts acylation of resorcinol dimethyl ether with benzoyl chloride,
  - in the presence of aluminium chloride in ethylene dichloride (85%) [589] or in ethyl ether [656];
  - in the presence of aluminium chloride in a chlorobenzene/ N,N-dimethylformamide mixture (22:1) at 115° [235,657], (74%) [215];
  - in the presence of a zinc chloride/aluminium chloride mixture in ethylene dichloride, first between 0° and 5°, then at 10° for 1 h and at 65° for 6 h (71%) [658].
- Preparation by saponification of 2-(benzoyloxy)-4-methoxybenzophenone (SM) with sodium hydroxide [44] or potassium hydroxide [238] in refluxing ethanol for 1 h. SM was obtained by oxidation of 6 methoxy-2,3-diphenylbenzofuran with chromium trioxide in boiling acetic acid for 30–40 min [44].
- Preparation by Fries rearrangement of m-methoxyphenyl benzoate,
  - in the presence of aluminium chloride [23];
  - in the presence of hydrochloric acid and a small amount of ferric chloride (60%) [659].
- Preparation by reaction of benzotrichloride with resorcinol monomethyl ether in hydrofluoric acid in the presence of water at 0°, then at r.t. for 7 h (55%) [213].
- Preparation by Friedel–Crafts acylation of resorcinol monomethyl ether with benzoyl chloride,
  - in the presence of boron trichloride in benzene, first at −10°, then at reflux for 10 h (85%) [660];
  - in the presence of titanium tetrachloride in benzene, first at −10°, then at reflux for 14 h (77%) [660];
  - in the presence of iron powder at 240–260° for 2 h (50%) [661];
  - in the presence of ferric chloride-nitromethane complex at 185–195° for 20 min (51%) [661].
- Preparation by reaction of benzoyl chloride with resorcinol dimethyl ether,
  - in chlorobenzene in the presence of titanium tetrachloride for 1 h at 120° (78%) [662];
  - without solvent or in o-dichlorobenzene, in the presence of ferric chloride, at 160–200° for 7–11 h (by-product) [663].
- Also obtained from β-(2-benzoyl-5-methoxyphenoxy)propionic acid by heating on a steam bath with a 10% aqueous sodium hydroxide solution for some minutes [218].
- Preparation from 2-iodo-5-methoxyphenyl benzoate by rearrangement on treatment with n-butyl-lithium in a mixture of ethyl ether, hexane and tetrahydrofuran at −70° for 2 h, then treatment with saturated aqueous ammonium chloride solution (<16%) [58].
- Also obtained by reaction of benzoic acid with resorcinol monomethyl ether,

- in the presence of polyphosphoric acid on a water bath for 20 min (27%) [626];
- in the presence of boron trifluoride at 80° for 30 min (59%) [186].

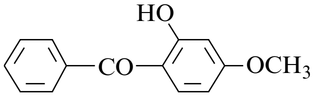
– Also refer to: [73,75,77,84,87,194,221,222,225,226,228,231,233,530,572,598, 625,664–673].

N.B.: Na [589,674], Sn salts [212].

oil [626];
m.p. 69° [662], 66° [113,652], 65° [215,660,661], 64–66° [589], 64° [212,236,650], 63–65° [395], 63–64° [238,658], 63° [218], 62–63° [239], 62° [96,205], 61° [44,93], 55° [213];
b.p. 359–361° [239]; ^{1}H NMR [58,98,99,303,395,675], IR [44,85,394,395,650], UV [93,99,109–111,113,233,235,236,240–243,394,395,650]; GC [631]; HPLC [245]; TLC [116,244,395]; polarographic study [117]; pK_a [93,96,115,531]; vapour pressure [236,248]; gel permeation chromatography [247].

(2-Hydroxy-4-methoxyphenyl)phenylmethanone-^{14}C

[17655-53-7] $C_{14}H_{12}O_3$ mol.wt. 230.25



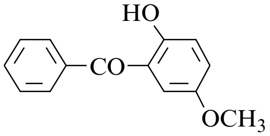
Synthesis

– Refer to: [676].

m.p. and Spectra (NA).

(2-Hydroxy-5-methoxyphenyl)phenylmethanone *(UV 9)*

[14770-96-8] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

– Preparation by Friedel–Crafts acylation of hydroquinone dimethyl ether with benzoyl chloride in the presence of aluminium chloride [173], in carbon disulfide first at 25° for 48 h, then at 50° for 30 min (55%) [256] or at r.t. for 48 h (by-product) [409], (10%) [677].

– Also obtained by selective demethylation of 2,5-dimethoxybenzophenone (SM),

- with hydriodic acid [257,409];
- with excess beryllium chloride in refluxing toluene for 3 h (90%) [395];
- with aluminium chloride [678], in nitromethane at 20° for 24 h (65%) [679] or in benzene under nitrogen at 80° for 12 h (90%) [258,680].

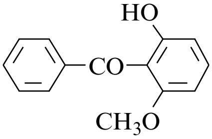
N.B.: SM was prepared by reaction of benzoyl chloride with hydroquinone dimethyl ether, either in the presence of stannic chloride in nitromethane at 20° for 1 h (78–94%) [679] or in the presence of aluminium chloride in carbon disulfide at r.t. for 48 h (78%) [677], (74–82%) [409].

- Preparation by partial methylation of 2,5-dihydroxybenzophenone with dimethyl sulfate in the presence of sodium hydroxide in dilute ethanol at 80–90° for 30 min (33%) [681].
- Also obtained by Fries rearrangement of p-methoxyphenyl benzoate with titanium tetrachloride without solvent at 120° for 1 h (20–35%) [679].
- Preparation by oxidation of 5-methoxy-2,3-diphenylbenzofuran with chromium trioxide in boiling acetic acid for 30–40 min, followed by saponification of the keto ester formed, the 2-(benzoyloxy)-5-methoxybenzophenone with sodium hydroxide in refluxing ethanol for 1 h [44,682]. The same result was obtained by using 5-methoxy-2-(4-methoxyphenyl)-3-phenylbenzofuran or 5-methoxy-2-(4-methylphenyl)-3-phenylbenzofuran [46].
- Also obtained (poor yield) by UV light irradiation of 2,5-dimethoxybenzophenone in carbon tetrachloride for 400 h (3%) [678].
- Also refer to: [20,530,671,683,684].

m.p. 84–85°5 [256], 84° [173,679], 83°5 [680], 83–84° [205], 82–85° [257], 82–84° [395], 82–83° [96], 81°5–82° [681], 81–82° [678], 81° [44,46,682], 78° [409,677];
^{1}H NMR (Sadtler: standard n° 30289 M) [98,99,395,678],
EPR [98], IR (Sadtler: standard n° 57334) [44,395,678,679],
UV [99,109–111,395,679];
pK_a [96,115]; TLC [116,395,678]; polarographic study [117].

(2-Hydroxy-6-methoxyphenyl)phenylmethanone

[20034-63-3] $C_{14}H_{12}O_3$ mol.wt. 228.25



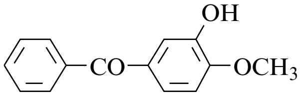
Syntheses

- Preparation from 2-iodo-3-methoxyphenyl benzoate by rearrangement on treatment with n-butyllithium in a mixture of ethyl ether, hexane and tetrahydrofuran at −70° for 2 h, followed by treatment with saturated aqueous ammonium chloride (51%) [58].
- Also obtained by Fries rearrangement of m-methoxyphenyl benzoate in the presence of trifluoro-methanesulfonic acid in tetrachloroethane at 170° for 24 h in a sealed tube (78%) [37].
- Preparation by partial methylation of 2,6-dihydroxybenzophenone with dimethyl sulfate,
  - in the presence of potassium carbonate in refluxing acetone for 7 h [270];
  - in the presence of potassium hydroxide in benzene in a water bath for 1 h (61%) [269].
- Preparation by partial demethylation of 2,6-dimethoxybenzophenone with aluminium chloride in benzene at 0° for 2 h (43%) [269].
- Also refer to: [171,685].

m.p. 140–141° [269], 140° [270], 106–107° [58]. There is discrepancy between the two melting points.
^{1}H NMR [58], IR [58], MS [58]; TLC [269].

(3-Hydroxy-4-methoxyphenyl)phenylmethanone

[66476-03-7] $C_{14}H_{12}O_3$ mol.wt. 228.25



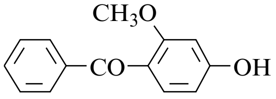
Syntheses

- Preparation from 3-(benzoyloxy)-4-methoxybenzophenone (SM) (m.p. 95°5–96°5) [686] by saponification [162] with sodium hydroxide in refluxing ethanol (good yield) [273,686] or with potassium hydroxide in refluxing methanol (98%) [687]. SM was obtained by Friedel–Crafts acylation of guaiacol benzoate with benzoyl chloride in the presence of zinc chloride [162,273,686] or in the presence of stannic chloride in nitromethane for 1 h at 20° (82%) [687].
- Preparation by partial methylation of 3,4-dihydroxybenzophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 90 min (70%) [274].
- Also refer to: [688,689].

m.p. 132°5–133° [274], 131–132° [162,273,686], 117° [687];
^{1}H NMR (Sadtler: standard n° 28215 M) [274],
IR (Sadtler: standard n° 55287) [687], UV [274,687].

(4-Hydroxy-2-methoxyphenyl)phenylmethanone

[21112-64-1] $C_{14}H_{12}O_3$ mol.wt. 228.25



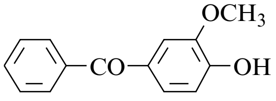
Syntheses

- Preparation by saponification of 4-(benzoyloxy)-2-methoxy-benzophenone (SM) with potassium hydroxide in refluxing aqueous ethanol for 1 h (95%) [238]. SM was obtained from resbenzophenone by a two-step synthesis (4-O-benzoylation and 2-O-methylation).
- Also obtained from β-(4-benzoyl-3-methoxyphenoxy)propionic acid by heating with 10% aqueous sodium hydroxide [189,218].

m.p. 124° [189,218], 123°5–124°5 [238]; ^{1}H NMR (Sadtler: standard n° 28221 M), IR (Sadtler: standard n° 55293) [238], UV [238].

(4-Hydroxy-3-methoxyphenyl)phenylmethanone

[51439-89-5] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

- Preparation by decarboxylation of 2-(4-hydroxy-3-methoxy-benzoyl)benzoic acid in the presence of cupric acetate mono-hydrate in quinoline at 250–254° for 35 min (72%) [690].
- Also obtained by Friedel–Crafts acylation,
  - of veratrole with benzoyl chloride in the presence of aluminium chloride in carbon disulfide overnight at r.t. [691];
  - of guaiacol with benzoic acid in the presence of polyphosphoric acid at 120° for 1 h [490,491].

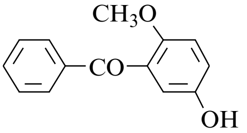
- Also obtained by cleavage of 3,3′-dimethoxybenzaurine[T] on heating its dilute aqueous sodium hydroxide solution (1%) in a water bath with air bubbling [162].

  N.B.: [T]Synonyms: 4-hydroxy-3,3′-dimethoxyfuchsone and 4-[(4-hydroxy-3-methoxyphenyl)-phenylmethylene]-3-methoxy-2,5-cyclohexadien-1-one.
- Also refer to: [688,689].

m.p. 100–101°5 [690], 97–98° [162,691]; IR [690,692], UV [690], MS [490,491].

(5-Hydroxy-2-methoxyphenyl)phenylmethanone

[80427-34-5] $C_{14}H_{12}O_3$ mol.wt. 228.25



Synthesis

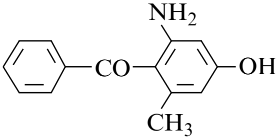
- Preparation by acylation of p-methoxyphenyl benzoate with benzoyl chloride in the presence of stannic chloride in nitromethane at 20° for 2 days, followed by saponification of the resulting keto ester—5-(benzoyloxy)-2-methoxy-benzophenone—with sodium hydroxide in refluxing methanol for 1 h (90%) [679].
- Also claimed to be obtained by partial demethylation of 2,5-dimethoxybenzophenone with hydriodic acid [409]. Nevertheless, the structure of this compound was erroneous. In that case, it probably was its isomer the 2-hydroxy-5-methoxybenzophenone [257].

m.p. 111° [679], 78° [409]. One of the reported melting points is obviously wrong.

^{1}H NMR (Sadtler: standard n° 30290 M), IR (Sadtler: standard n° 57335) [679], UV [679].

(2-Amino-4-hydroxy-6-methylphenyl)phenylmethanone

[54439-89-3] $C_{14}H_{13}NO_2$ mol.wt. 227.26



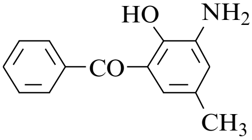
Synthesis

- Preparation from 3′-methyl-5-phenyl-3,5′-diisoxazolyl-methane by performing hydrogenolysis and subsequent hydrolysis with hydrochloric acid (86%) [693].

m.p. 162° [693]; ^{1}H NMR [693], MS [693].

(3-Amino-2-hydroxy-5-methylphenyl)phenylmethanone

$C_{14}H_{13}NO_2$ mol.wt. 227.26



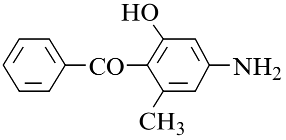
Synthesis

- Refer to: [472].

m.p. and Spectra (NA).

(4-Amino-2-hydroxy-6-methylphenyl)phenylmethanone

[54439-92-8] $C_{14}H_{13}NO_2$ mol.wt. 227.26



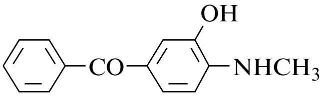
Synthesis

– Obtained (trace) from 5-methyl-3′-phenyl-3,5′-diisoxazolyl-methane by performing hydrogenolysis and subsequent hydrolysis with hydrochloric acid [693].

m.p. 125° [693]; ^{1}H NMR [693], MS [693].

[3-Hydroxy-4-(methylamino)phenyl]phenylmethanone

[54903-59-2] $C_{14}H_{13}NO_2$ mol.wt. 227.26



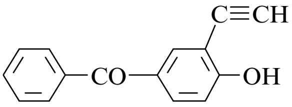
Synthesis

– Preparation from 6-benzoyl-3-methylbenzoxazolinone by alkaline hydrolysis with boiling 10% aqueous sodium hydroxide solution [564], (90–100%) [565], 85% [566].

m.p. 165° [565,566]; Spectra (NA).

(3-Ethynyl-4-hydroxyphenyl)phenylmethanone

[183589-15-3] $C_{15}H_{10}O_2$ mol.wt. 222.24



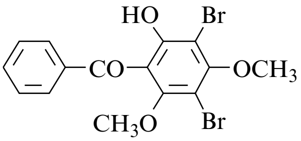
Synthesis

– Obtained by treatment of p-benzoyl-o-[(trimethylsilyl)-ethynyl]phenol in methanol in the presence of potassium fluoride at 25° for 1 h (75%) [533].

m.p. 145–146° [533]; ^{1}H NMR [533], ^{13}C NMR [533], IR [533], MS [533].

(3,5-Dibromo-2-hydroxy-4,6-dimethoxyphenyl)phenylmethanone

$C_{15}H_{12}Br_2O_4$ mol.wt. 416.07



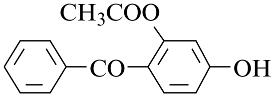
Synthesis

– Preparation by reaction of bromine with monobromo-hydrocotoin in chloroform [694].

m.p. 95° [694]; Spectra (NA).

[2-(Acetyloxy)-4-hydroxyphenyl]phenylmethanone

[145747-24-6] $C_{15}H_{12}O_4$ mol.wt. 256.26



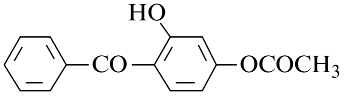
Synthesis

- Obtained by enzymatic deacetylation of 2,4-diacetoxy-benzophenone in the presence of porcine pancreas lipase in a tetrahydrofuran/n-butanol mixture at 40–45° (70%) [695].

m.p. and Spectra (NA).

[4-(Acetyloxy)-2-hydroxyphenyl]phenylmethanone

[18803-24-2] $C_{15}H_{12}O_4$ mol.wt. 256.26



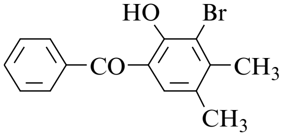
Syntheses

- Preparation by partial hydrolysis of 2,4-diacetoxy-benzophenone in the presence of trifluoroacetic acid containing 5% of water at 65° for 5 min (98%) [696].
- Preparation by reaction of acetyl chloride (1 equiv) with resbenzophenone [697].
- Also refer to: [96,99,109,116].

m.p. 93°6–94° [697], 92–93° [96]; ^{1}H NMR [99], UV [99,109]; TLC [116]; pK_a [96].

(3-Bromo-2-hydroxy-4,5-dimethylphenyl)phenylmethanone

[143815-12-7] $C_{15}H_{13}BrO_2$ mol.wt. 305.17



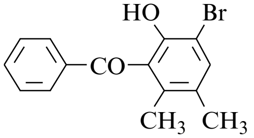
Syntheses

- Preparation by Fries rearrangement of 2-bromo-4,5-di-methylphenyl benzoate with aluminium chloride without solvent at 140° for 2 h (24%) [698].
- Preparation by reaction of bromine with 2-hydroxy-4,5-di-methylbenzophenone in boiling acetic acid [578].

m.p. 134–135° [578,698]; ^{1}H NMR (Sadtler: standard n° 59390 M) [698], IR (Sadtler: standard n° 86546 K) [698], UV [698], MS [698].

(3-Bromo-2-hydroxy-5,6-dimethylphenyl)phenylmethanone

[143815-11-6] $C_{15}H_{13}BrO_2$ mol.wt. 305.17



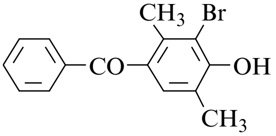
Syntheses

- Preparation by Fries rearrangement of 2-bromo-4,5-di-methylphenyl benzoate with titanium tetrachloride without solvent at 140° for 2 h (60%) [698].

– Also obtained (by-product) by Fries rearrangement of 2-bromo-4,5-dimethylphenyl benzoate with aluminium chloride without solvent at 140° for 2 h (10%) [698].

m.p. 157–158° [698]; ^{1}H NMR (Sadtler: standard n° 59392 M) [698], IR (Sadtler: standard n° 86548 K) [698], UV [698], MS [698].

(3-Bromo-4-hydroxy-2,5-dimethylphenyl)phenylmethanone



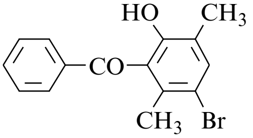
$C_{15}H_{13}BrO_2$ mol.wt. 305.17

Synthesis

– Preparation by reaction of bromine with 4-hydroxy-2,5-di-methylbenzophenone in acetic acid [578].

m.p. 115–116° [578]; Spectra (NA).

(3-Bromo-6-hydroxy-2,5-dimethylphenyl)phenylmethanone



$C_{15}H_{13}BrO_2$ mol.wt. 305.17

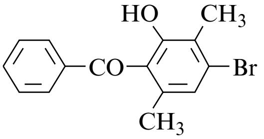
Synthesis

– Preparation by bromination of 2-hydroxy-3,6-dimethyl-benzophenone in chloroform [501,502].

m.p. 133° [501,502]; IR [103,501].

(4-Bromo-2-hydroxy-3,6-dimethylphenyl)phenylmethanone

[6721-06-8]



$C_{15}H_{13}BrO_2$ mol.wt. 305.17

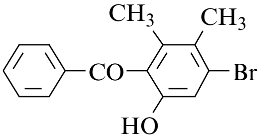
Synthesis

– Obtained by chromic oxidation (CrO_3) of 6-bromo-4,7-di-methyl-2,3-diphenylbenzofuran, followed by saponification of the keto ester so obtained [501,502].

m.p. 117° [501,502]; IR [103,501], UV [503].

(4-Bromo-6-hydroxy-2,3-dimethylphenyl)phenylmethanone

[143815-13-8]



$C_{15}H_{13}BrO_2$ mol.wt. 305.17

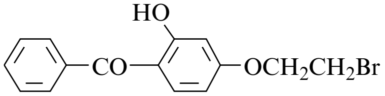
Synthesis

– Obtained (by-product) by Fries rearrangement of 2-bromo-4,5-dimethylphenyl benzoate with aluminium chloride without solvent at 140° for 2 h (22%) [698].

m.p. 194–195° [698]; ^{1}H NMR (Sadtler: standard n° 59394 M) [698], IR (Sadtler: standard n° 86550 K) [698], UV [698], MS [698].

[4-(2-Bromoethoxy)-2-hydroxyphenyl]phenylmethanone

[18902-63-1] $C_{15}H_{13}BrO_3$ mol.wt. 321.17



Synthesis

– Preparation by reaction of ethylene dibromide with resbenzophenone,

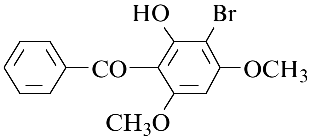
- in the presence of sodium hydroxide in refluxing dilute ethanol for 15 h (47%) [238,699];
- in the presence of sodium methoxide in diisobutylketone between 130° and 140° (78%) [700].

– Also refer to: [701–703].

m.p. 90–95° [700], 87–88° and 97–98° [238,699]; double melting point (two allotropic forms); Spectra (NA).

(3-Bromo-2-hydroxy-4,6-dimethoxyphenyl)phenylmethanone

$C_{15}H_{13}BrO_4$ mol.wt. 337.17



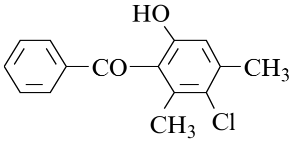
Synthesis

– Preparation by reaction of bromine with hydrocotoin in chloroform [694].

m.p. 147° [694]; Spectra (NA).

(3-Chloro-6-hydroxy-2,4-dimethylphenyl)phenylmethanone

[34174-02-2] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



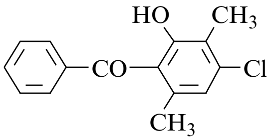
Synthesis

– Preparation by Fries rearrangement of 4-chloro-3,5-di-methylphenyl benzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 203–204° [458]; Spectra (NA).

(4-Chloro-2-hydroxy-3,6-dimethylphenyl)phenylmethanone

[33561-94-3] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



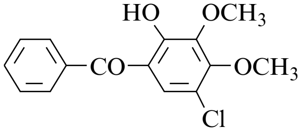
Synthesis

– Obtained by oxidation of 6-chloro-4,7-dimethyl-2,3-di-phenylbenzofuran with chromium trioxide in boiling acetic acid for 2 h, followed by saponification of the resulting 2-(benzoyloxy)-4-chloro-3,6-dimethylbenzophenone with 2 N sodium hydroxide in boiling ethanol for 15 min [515].

m.p. 102° [515]; IR [515].

(5-Chloro-2-hydroxy-3,4-dimethoxyphenyl)phenylmethanone

[140665-35-6] $C_{15}H_{13}ClO_4$ mol.wt. 292.72



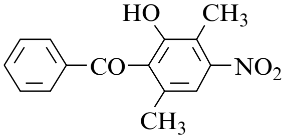
Synthesis

- Preparation by reaction of sulfuryl chloride with 2-hydroxy-3,4-dimethoxybenzophenone in methylene chloride overnight at r.t. (70%) [704].

m.p. 84–85° [704]; ^{1}H NMR [704], IR [704], MS [704].

(2-Hydroxy-3,6-dimethyl-4-nitrophenyl)phenylmethanone

[18619-94-8] $C_{15}H_{13}NO_4$ mol.wt. 271.27



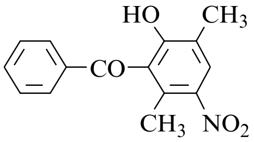
Synthesis

- Refer to: [103,107,108,582].

m.p. (NA); IR [103,107,108,582].

(2-Hydroxy-3,6-dimethyl-5-nitrophenyl)phenylmethanone

[18619-95-9] $C_{15}H_{13}NO_4$ mol.wt. 271.27



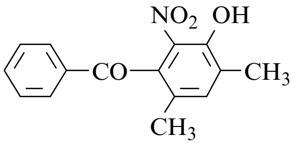
Synthesis

- Refer to: [103].

m.p. (NA); IR [103].

(3-Hydroxy-4,6-dimethyl-2-nitrophenyl)phenylmethanone

[20010-69-9] $C_{15}H_{13}NO_4$ mol.wt. 271.27



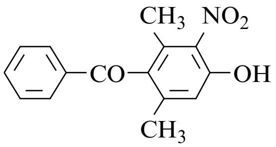
Synthesis

- Obtained by treatment of 5-amino-2,4-dimethylbenzo-phenone (SM) in nitric acid (d = 1.34) with an aqueous sodium nitrite solution between 0° and 10°, then at r.t. for 2 h and at 50° for 3 h (43%). SM was prepared by acylation of 2,4-dimethylacetamide with benzoyl chloride in carbon disulfide in the presence of aluminium chloride (62%, m.p. 101°) [705].

m.p. 119°5–120° [705]; Spectra (NA).

(4-Hydroxy-2,6-dimethyl-3-nitrophenyl)phenylmethanone

[100923-75-9] $C_{15}H_{13}NO_4$ mol.wt. 271.27



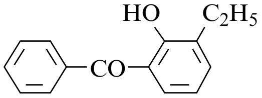
Synthesis

- Refer to: [706].

m.p. (NA); ^{1}H NMR [706,707], ^{13}C NMR [708], IR [706].

(3-Ethyl-2-hydroxyphenyl)phenylmethanone

[56394-91-3] $C_{15}H_{14}O_2$ mol.wt. 226.27



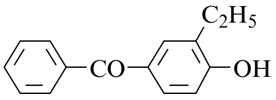
Synthesis

- Preparation by reaction of 3-ethylsalicylic acid chloride (3-ethyl-2-hydroxybenzoyl chloride) with benzene in the presence of aluminium chloride overnight at 100° (58%) [92,709–711].
- Preparation by Fries rearrangement of o-ethylphenyl benzoate (SM) with aluminium chloride at 160–170° for 30 min (61%). SM was obtained by reaction of benzoyl chloride with aluminium tris(o-ethylphenoxide) [485].

b.p.$_{0.14}$ 123–126° [709,710], b.p.$_{0.4}$ 123–126° [92,711],
b.p.$_3$ 165–167 [485]; n_D^{22} = 1.6081 [709,710]; IR [709,710].

(3-Ethyl-4-hydroxyphenyl)phenylmethanone

[67217-94-1] $C_{15}H_{14}O_2$ mol.wt. 226.27



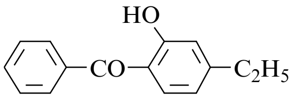
Syntheses

- Preparation by decarboxylation of 2-(3′-ethyl-4′-hydroxy-benzoyl)benzoic acid in the presence of cupric acetate in refluxing quinoline (254°) for 40 min (98%) [712].
- Also obtained (by-product) by Fries rearrangement of o-ethylphenyl benzoate with aluminium chloride at 160–170° for 30 min (12%) [485].

m.p. 138–140°2 [712], 136° [485]; Spectra (NA).

(4-Ethyl-2-hydroxyphenyl)phenylmethanone

[78473-50-4] $C_{15}H_{14}O_2$ mol.wt. 226.27



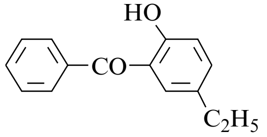
Synthesis

- Preparation by Fries rearrangement of m-ethylphenyl benzoate in the presence of aluminium chloride without solvent at 140° for 4 h [713].
- Also refer to: [714].

oil [713]; b.p. and Spectra (NA).

(5-Ethyl-2-hydroxyphenyl)phenylmethanone

[3132-42-1] $C_{15}H_{14}O_2$ mol.wt. 226.27



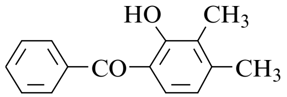
Syntheses

- Preparation by Fries rearrangement of p-ethylphenyl benzoate with aluminium chloride at 140° for 4 h [713].
- Preparation by reaction of benzoyl chloride with p-ethyl-phenol in the presence of aluminium chloride in tetra-chloroethane at 105° for 22 h (60%) [92].
- Preparation by condensation of benzotrichloride with p-ethylphenol in the presence of 32–40% aqueous sodium hydroxide at 70–80° (70%) [615].
- Also refer to: [715,716].

m.p. 74° [713], 69°5–70° [615], 69–72° [92];
^{1}H NMR [615], IR [615], UV [615], MS [615];
gas chromatography study [631].

(2-Hydroxy-3,4-dimethylphenyl)phenylmethanone

[14770-98-0] $C_{15}H_{14}O_2$ mol.wt. 226.27



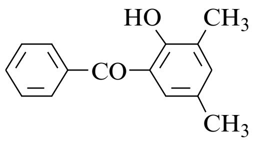
Syntheses

- Preparation by Fries rearrangement of 2,3-dimethylphenyl benzoate in the presence of aluminium chloride at 180° for 10 min (60%) [518].
- Preparation by oxidation of 6,7-dimethyl-2,3-diphenylbenzofuran with chromium trioxide in boiling acetic acid, followed by saponification of the keto ester so obtained (2-benzoyloxy-3,4-di-methylbenzophenone) [44,682,717].
- Also refer to: [76].

m.p. 45° [717], 42° [44,518,682,718]; IR [44,717].

(2-Hydroxy-3,5-dimethylphenyl)phenylmethanone

[16762-34-8] $C_{15}H_{14}O_2$ mol.wt. 226.27



Syntheses

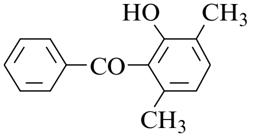
- Preparation by Fries rearrangement of 2,4-dimethylphenyl benzoate in the presence of aluminium chloride (major product) [719], at 130–140° for 4 h (50%) [720], at 140–160° for 20 min (83%) [721] or at 180° for 10 min (75%) [518].
- Preparation by chromic oxidation of various substituted 5,7-dimethylbenzofurans (5,7-dimethyl-2,3-diphenylbenzofuran [44]; 5,7-dimethyl-2-(4-methylphenyl)-3-phenylbenzofuran [46] and 2-(4-methoxyphenyl)-5,7-dimethyl-3-phenylbenzofuran [46]), followed by saponification of the resulting keto esters.

- Preparation by Friedel–Crafts acylation of 2,4-dimethylanisole or of 2,4-dimethylphenetole with benzoyl chloride in the presence of aluminium chloride in refluxing carbon disulfide (10% and 30% yields, respectively) [719].
- Preparation by reaction of benzoyl chloride with 2,4-dimethylphenol in the presence of aluminium chloride in nitrobenzene at 50–60° for 18 h (27%) [721].
- Also refer to: [4].

oil [44,46]; m.p. 40–41° [720,722];
b.p.$_{20}$ 202° [719], b.p.$_{12}$ 198–200° [518], b.p.$_{17}$ 192–194° [720]; IR [44].

(2-Hydroxy-3,6-dimethylphenyl)phenylmethanone

[4072-17-7] $C_{15}H_{14}O_2$ mol.wt. 226.27



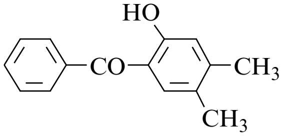
Synthesis

- Preparation by oxidation of 4,7-dimethyl-2,3-diphenyl-benzofuran with chromium trioxide in acetic acid at 60°, followed by hydrolysis of the keto ester so obtained (2-benzoyloxy-3,6-dimethylbenzophenone) with 10% sodium hydroxide [632,723].
- Also refer to: [76,633].

m.p. 104° [89,609,723], 103–104° [632];
IR [89,103,723], UV [609].

(2-Hydroxy-4,5-dimethylphenyl)phenylmethanone

[4072-14-4] $C_{15}H_{14}O_2$ mol.wt. 226.27



Syntheses

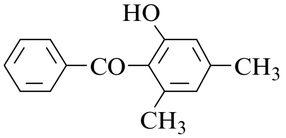
- Preparation by reaction of chromium trioxide with 5,6-dimethyl-2,3-diphenylbenzofuran [44,606] or 5,6-di-methyl-2-(4-methylphenyl)-3-phenylbenzofuran [46] in acetic acid, followed by saponification of the keto ester so formed (2-benzoyloxy-4,5-dimethylbenzophenone).
- Preparation by Fries rearrangement of 3,4-dimethylphenyl benzoate without solvent, in the presence of,
  - aluminium chloride between 120° and 150° (quantitative yield) [724] or at 140° for 20 min (66%) [462];
  - titanium tetrachloride at 140° for 2 h (75%) [698].
- Also obtained by isomerization of 2-hydroxy-4,6-dimethylbenzophenone in the presence of aluminium chloride between 140° and 180° for several hours (quantitative yield) [724]. There is a methyl group migration.
- Preparation by reaction of benzoyl chloride with 3,4-dimethylphenyl benzoate in the presence of zinc chloride at 110°, followed by saponification of the keto ester formed (2-benzoyloxy-4,5-di-methylbenzophenone) with sodium hydroxide in boiling ethanol [725].

- Preparation by reaction of pyridinium chloride with 2-methoxy-4,5-dimethylbenzophenone by boiling for 50 min [726].
- Also obtained by reaction of benzoyl chloride with 4-methylthymol methyl ether (4,5-dimethyl-2-isopropylanisole) in the presence of aluminium chloride in carbon disulfide at r.t. for 23 h [726].
- Preparation by saponification of 2-benzoyloxy-4,5-dimethylbenzophenone with potassium hydroxide in refluxing ethanol for 1 h (64%) [504].
- Also obtained from 3,4-dimethylbenzophenone by nitration with nitric acid in acetic anhydride at 70°, then at 5° overnight. The diene obtained (2-benzoyl-4,5-dimethyl-4-nitro-1,4-dihydrophenyl acetate as cis and trans mixture) was added to a concentrated solution of sodium methoxide in methanol [727].
- Also obtained (by-product) by reaction of aluminium chloride with 2-bromo-4,5-dimethylphenyl benzoate without solvent at 140° for 2 h (7%) [698].
- Also obtained (by-product) by treatment of 2-bromo-4,5-dimethylbenzophenone with 25% aqueous ammonium hydroxide in ethanol into an autoclave at 180–190° for 5 h under 40 atmospheres (15%) [728].
- Preparation by reaction of 3,4-xylenol with benzoic acid in the presence of boron trifluoride-ethyl ether complex, at 128–129° for 7 min, followed by treatment of the difluoroboroxy chelate formed with boiling aqueous ethanol for 15 min (63%) [729].
- Also refer to: [504,730,731].

m.p. 112–113°5 [727], 111° [89,724], 110–111° [462,606,698,725,728,729], 110° [726], 108° [44,46];
^{1}H NMR (Sadtler: standard n° 59389 M) [698,727,729],
IR (Sadtler: standard n° 86545 K) [44,46,89,103,698,726,727,729],
UV [698,729], MS [698,727].

(2-Hydroxy-4,6-dimethylphenyl)phenylmethanone

[2929-45-5] $C_{15}H_{14}O_2$ mol.wt. 226.27



Syntheses

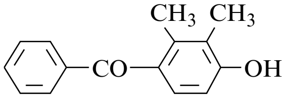
- Preparation by reaction of benzoyl chloride with 3,5-di-methylphenol in the presence of aluminium chloride in nitromethane at 25° for 3 h, then at reflux for 1 h [102], (45%) [732].
- Preparation by Fries rearrangement of 3,5-dimethylphenyl benzoate with aluminium chloride [6,632,733], without solvent between 120° and 150° (quantitative yield) [724], (55%) [733] or in nitrobenzene at 62–63° for 18 h (80%) [31].
- Preparation by oxidation of 4,6-dimethyl-2,3-diphenylbenzofuran with chromium trioxide in acetic acid at 60°, followed by saponification of the keto ester so obtained (2-(benzoyloxy)-4,6-di-methylbenzophenone) with 10% sodium hydroxide in boiling ethanol [632].
- Also obtained by reaction of benzoic acid with 3,5-dimethylphenol in the presence of boron trifluoride in a sealed tube at 160° for 2 h (58%) [150].

- Also obtained (by-product) by treatment of 2-bromo-4,5-dimethylphenyl benzoate with aluminium chloride at 140° for 2 h (8%) [698].
- Also refer to: [4,37,76,633,734].

m.p. 143–143°5 [31], 143° [724], 142–143° [732], 142° [150,632], 141–142° [698],
140° [6,89,609,735], 139–140° [733], 134° [94,95];
^{1}H NMR [94,95,101,102,698,707,735],
^{13}C NMR [101], IR [94,95,103,698,735,736],
UV [698,735,737], MS [698];
pK_a [735]; polarographic study [117]; thermal behaviour [94,95].

(4-Hydroxy-2,3-dimethylphenyl)phenylmethanone

[107931-09-9] $C_{15}H_{14}O_2$ mol.wt. 226.27



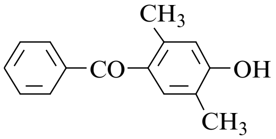
Synthesis

- Preparation by demethylation of 2,3-dimethyl-4-methoxy-benzophenone (SM) with pyridinium chloride at 180° for 2 h. SM was prepared by reaction of phenylmagnesium bromide with 2,3-dimethyl-4-methoxybenzaldehyde in ethyl ether at 0° for 30 min [477].

m.p. and Spectra (NA).

(4-Hydroxy-2,5-dimethylphenyl)phenylmethanone

[62262-03-7] $C_{15}H_{14}O_2$ mol.wt. 226.27



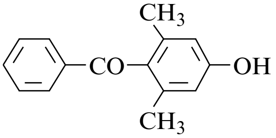
Syntheses

- Preparation by Friedel–Crafts acylation of 2,5-dimethyl-phenyl benzoate with benzoyl chloride in the presence of zinc chloride, followed by saponification of the p-keto ester so obtained with sodium hydroxide in boiling ethanol (quantitative yield) [725].
- Preparation by reaction of hydriodic acid with 4-methoxy-2,5-dimethylbenzophenone in boiling acetic acid (56%) [719].
- Also obtained (by-product) by Fries rearrangement of 2,5-dimethylphenyl benzoate with aluminium chloride at 130–140° for 4 h [719].
- Also refer to: [734].

m.p. 166–167° [719,738]; Spectra (NA).

(4-Hydroxy-2,6-dimethylphenyl)phenylmethanone

[81375-01-1] $C_{15}H_{14}O_2$ mol.wt. 226.27



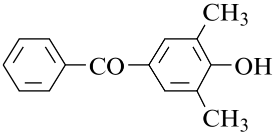
Syntheses

- Preparation by reaction of sodium iodide with 4-benzyloxy-2,6-dimethylbenzophenone (SM) in the presence of trimethylsilyl chloride in acetonitrile into an autoclave at 130° for 24 h (60%). SM was prepared from 4-bromo-3,5-di-methylphenol by a four-step synthesis [6].
- Preparation by diazotization of 4-amino-2,6-dimethylbenzophenone, followed by hydrolysis of the diazonium salt obtained (34%) [732].
- Also refer to [706,739].

m.p. 115° [6,732]; b.p.$_{0.01}$ 153–155° [6];
^{1}H NMR [706,707,732], ^{13}C NMR [6,708], IR [706,732].

(4-Hydroxy-3,5-dimethylphenyl)phenylmethanone

[5336-56-1] $C_{15}H_{14}O_2$ mol.wt. 226.27

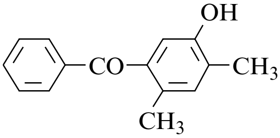


Syntheses

- Preparation by decarboxylation of 2-(4-hydroxy-3,5-di-methylbenzoyl)benzoic acid in the presence of cupric acetate monohydrate in refluxing quinoline for 30 min (98%) [740].
- Preparation by demethylation of 4-methoxy-3,5-dimethyl-benzophenone (SM) with aluminium chloride without solvent at 100–110° for 3 h [741]. SM was obtained by Friedel–Crafts acylation of 2,6-dimethylanisole with benzoyl chloride according to usual method.
- Preparation by Fries rearrangement of 2,6-dimethylphenyl benzoate with aluminium chloride without solvent [742], at 130–140° (46%) [743].
- Also obtained by photo-Fries rearrangement of 2,6-dimethylphenyl benzoate in isopropanol at 26° for 24 h (33%) [64,72] or in pentane in the presence of silica gel (23%) [64].
- Also obtained (poor yield) by reaction of cumene hydroperoxide with 4-benzyl-2,6-dimethyl-phenol and air bubbling in the presence of cobalt phthalate in cumene at 80–100° for 100 h (6%) [744].
- Also refer to: [4,429,745–747].

m.p. 145° [742], 143°3–144°5 [740], 143° [744], 142–142°5 [743], 141–142° [741], 139–141°6 [72];
^{1}H NMR [742].

(5-Hydroxy-2,4-dimethylphenyl)phenylmethanone



$C_{15}H_{14}O_2$ mol.wt. 226.27

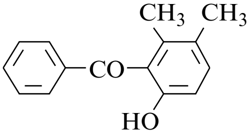
Syntheses

- Preparation by Friedel–Crafts acylation of 2,4-dimethyl-phenyl benzoate with benzoyl chloride in the presence of zinc chloride in boiling chloroform, followed by saponification of the 5-(benzoyloxy)-2,4-dimethyl-benzophenone so obtained with sodium hydroxide in refluxing ethanol (45%) [748].
- Also obtained (poor yield) by Fries rearrangement of 2,4-dimethylphenyl benzoate with aluminium chloride without solvent at 130–140° for 4 h (4%) [720].
- Obtained (by-product) by Friedel–Crafts acylation,
  - of 2,4-dimethylanisole with benzoyl chloride in the presence of aluminium chloride in refluxing carbon disulfide for 3–5 h (8%) [719];
  - of 2,4-dimethylphenetole with benzoyl chloride in the presence of aluminium chloride in boiling carbon disulfide [719].
- Preparation by demethylation of 2,4-dimethyl-5-methoxybenzophenone,
  - with hydriodic acid (d = 1.7) in boiling acetic acid at 130–140° for 2 h (70%) [719];
  - with aluminium chloride in boiling carbon disulfide for 8 h (32%) [719].

m.p. 145–146° [748], 140–141° [719,720]; Spectra (NA).

(6-Hydroxy-2,3-dimethylphenyl)phenylmethanone

[108478-10-0] $C_{15}H_{14}O_2$ mol.wt. 226.27



Syntheses

- Preparation by oxidation of 4,5-dimethyl-2,3-diphenyl-benzofuran with chromium trioxide in acetic acid, followed by alkaline hydrolysis of the resulting keto ester with refluxing 10% sodium hydroxide in ethanol for 2 h [632].
- Also obtained by saponification of 2,3-dimethyl-6-benzoyl-oxybenzophenone with refluxing 10% sodium hydroxide for 30 min [632].
- Preparation by debromination of 3-bromo-2-hydroxy-5,6-dimethylbenzophenone in the presence of copper powder in caproic acid at 220° for 15 min (82%) [698].
- Also obtained (poor yield) by Fries rearrangement of 3,4-dimethylphenyl benzoate with titanium tetrachloride without solvent at 140° for 2 h (5%) [698].
- Also refer to: [633].

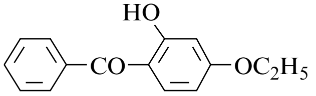
m.p. 124–125° [698], 114–115° [632];

^{1}H NMR (Sadtler: standard n° 59393 M) [698],

IR (Sadtler: standard n° 86549 K) [698], UV [698], MS [698].

(4-Ethoxy-2-hydroxyphenyl)phenylmethanone

[15889-70-0] $C_{15}H_{14}O_3$ mol.wt. 242.27



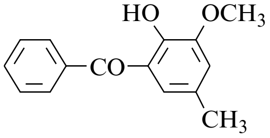
Syntheses

- Preparation by reaction of ethyl bromide with resbenzo-phenone, in the presence of sodium hydroxide in dilute ethanol, first at 30–40° for 1 h, then at 65–75° for 16 h (35%) [189].
- Preparation by reaction of ethyl p-toluenesulfonate with resbenzophenone in the presence of potassium carbonate in boiling water for 8 h (75%) [201,202].
- Preparation by Friedel–Crafts acylation of 1,3-diethoxybenzene with benzoyl chloride in the presence of a zinc chloride/aluminium chloride mixture, first between 0° and 5°, then at 10° for 1 h and finally at 65° for 6 h [658].
- Also refer to: [222,655,667].

m.p. 54°5 [189], 50–52° [201,202]; Spectra (NA).

(2-Hydroxy-3-methoxy-5-methylphenyl)phenylmethanone

[17603-92-8] $C_{15}H_{14}O_3$ mol.wt. 242.27



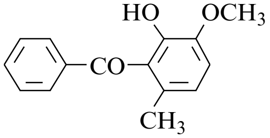
Synthesis

- Preparation by photo-Fries rearrangement of 2-methoxy-4-methylphenyl benzoate in benzene or in ethanol during 4 h (40% and 63% yields, respectively) [620].

m.p. (NA); ^{1}H NMR [620], IR [620].

(2-Hydroxy-3-methoxy-6-methylphenyl)phenylmethanone

[129103-91-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



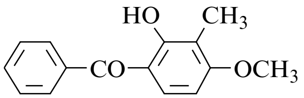
Synthesis

- Preparation from 2-iodo-6-methoxy-3-methylphenyl benzoate by rearrangement on treatment with n-butyllithium in a mixture of ethyl ether, hexane and tetrahydrofuran at −70° for 2 h, then treatment with saturated aqueous ammonium chloride (55%) [58].

m.p. 128–129°5 [58]; ^{1}H NMR [58], IR [58].

(2-Hydroxy-4-methoxy-3-methylphenyl)phenylmethanone

[83803-88-7] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

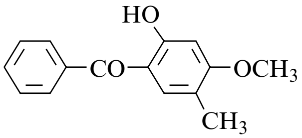
- Preparation by methylation of 2,4-dihydroxy-3-methyl-benzophenone with methyl iodide in the presence of potassium carbonate in refluxing acetone for 2 h [749].

- Also obtained by methylation of resbenzophenone with methyl iodide in the presence of potassium hydroxide in methanol at 0° (26%) [750]. There is an introduction of one methyl group on the nucleus.
- Also obtained by methylation of resbenzophenone [649] according to the method [751].
- Also refer to: [221].

m.p. 125° [649,749,750], 124–125° [113];
UV [113].

(2-Hydroxy-4-methoxy-5-methylphenyl)phenylmethanone

[59954-97-1] $C_{15}H_{14}O_3$ mol.wt. 242.27



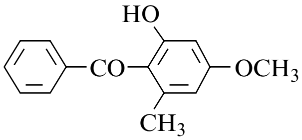
Syntheses

- Preparation by reaction of benzoyl chloride with 4-methyl-resorcinol dimethyl ether in the presence of aluminium chloride in ethylene dichloride (97%) [589].
- Preparation by oxidation of 6-methoxy-5-methyl-2,3-di-phenylbenzofuran with chromium trioxide in refluxing acetic acid, followed by alkaline hydrolysis of the keto ester so obtained (2-benzoyloxy-4-methoxy-5-methylbenzophenone) with potassium hydroxide in refluxing ethanol (73%) [752].

m.p. 188° [752], 88–89° [589]. One of the reported melting points is obviously wrong. Spectra (NA).

(2-Hydroxy-4-methoxy-6-methylphenyl)phenylmethanone

[23573-43-5] $C_{15}H_{14}O_3$ mol.wt. 242.27



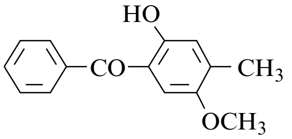
Syntheses

- Preparation by partial methylation of 2,4-dihydroxy-6-methylbenzophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 4 h (74%) [753].
- Also obtained by UV light irradiation of 3-methoxy-5-methyl-phenyl benzoate in ethanol at 20° for 70 h (21%) [754].

m.p. 93–94° [753], 87–88° [754]; ^{1}H NMR [643,754], IR [754], UV [754].

(2-Hydroxy-5-methoxy-4-methylphenyl)phenylmethanone

[59954-92-6] $C_{15}H_{14}O_3$ mol.wt. 242.27



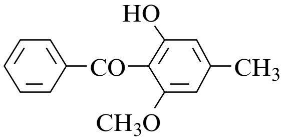
Synthesis

- Preparation by oxidation of 5-methoxy-6-methyl-2,3-di-phenylbenzofuran with chromium trioxide in refluxing acetic acid for 30 min, followed by alkaline hydrolysis of the resulting keto ester (2-benzoyloxy-5-methoxy-4-methyl-benzophenone) with potassium hydroxide in refluxing ethanol (75%) [752].

m.p. 114° [752]; Spectra (NA).

(2-Hydroxy-6-methoxy-4-methylphenyl)phenylmethanone

[23565-66-4] $C_{15}H_{14}O_3$ mol.wt. 242.27



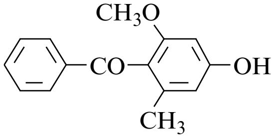
Synthesis

– Obtained by UV light irradiation of 3-methoxy-5-methyl-phenyl benzoate in ethanol at 20° for 70 h (13%) [754].

m.p. 70–71° [754]; ^{1}H NMR [754], IR [754], UV [754].

(4-Hydroxy-2-methoxy-6-methylphenyl)phenylmethanone

[23565-67-5] $C_{15}H_{14}O_3$ mol.wt. 242.27



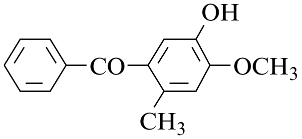
Synthesis

– Obtained by UV light irradiation of 3-methoxy-5-methyl-phenyl benzoate in ethanol at 20° for 70 h (8%) [754].

m.p. 123–124° [754]; ^{1}H NMR [754], IR [754], UV [754].

(5-Hydroxy-4-methoxy-2-methylphenyl)phenylmethanone

$C_{15}H_{14}O_3$ mol.wt. 242.27



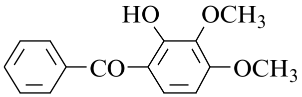
Synthesis

– Preparation by saponification of 5-(benzoyloxy)-4-methoxy-2-methylbenzophenone (SM) (m.p. 95–96°) with sodium hydroxide in boiling dilute ethanol (quantitative yield). SM was obtained by condensation of benzoyl chloride with creosol benzoate in the presence of zinc chloride [755].

m.p. 150° [755]; Spectra (NA).

(2-Hydroxy-3,4-dimethoxyphenyl)phenylmethanone

[7508-32-9] $C_{15}H_{14}O_4$ mol.wt. 258.27



Syntheses

– Preparation by reaction of benzoyl chloride with pyrogallol trimethyl ether,

- in the presence of aluminium chloride in ethylene dichloride between 0° and 5°, then at r.t. for 3 h and finally at reflux for 1 h (96%) [704] or for 2 h (47%) [598] and also in refluxing methylene chloride for 2 h (22%) [756].

N.B.: This benzophenone is, by mistake, named, 2-hydroxy-3,4-dimethoxybenzaldehyde (synthetic example 20, compound **a**, page 39), in the patent PCT Int. Appl. WO 96 05,188 of 22 February 1996 [598].

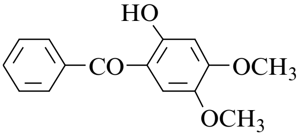
- in the presence of zinc chloride (good yield) [686], at 100° for 1 h [757];
- in the presence of mercuric chloride instead of aluminium chloride without solvent at 115° (reflux) for 1–3 h (40%) [758,759].

- Preparation by reaction of methyl iodide with 2,3,4-trihydroxybenzophenone monosodium salt in the presence of sodium carbonate at 160° for 6 h [344]. By using lead salt instead of sodium salt in the same conditions, the yield was decreased.
- Also obtained by reaction of methyl iodide with 2,3,4-trihydroxybenzophenone in the presence of lithium carbonate in N,N-dimethylformamide at 30° for 15 h under nitrogen (17%) [369].
- Also obtained by reaction of dimethyl sulfate with 2,3-dihydroxy-4-methoxybenzophenone or with 2,4-dihydroxy-3-methoxybenzophenone in the presence of alkali [379].

m.p. 185–187° [369]. This melting point is obviously wrong. 132–134° [704], 131° [344,756], 130–131° [686,757], 127–128° [598], 120–121° [379]; ^{1}H NMR [369,598,704], ^{13}C NMR [369], IR [704], UV [369], MS [369,704]; pK_a [369].

(2-Hydroxy-4,5-dimethoxyphenyl)phenylmethanone

[36896-99-8] $C_{15}H_{14}O_4$ mol.wt. 258.27



Syntheses

- Preparation by partial methylation of 2,5-dihydroxy-4-methoxybenzophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone [656,760].
- Preparation by partial demethylation of 2,4,5-trimethoxy-benzophenone with aluminium chloride in boiling carbon disulfide for 1 h [761,762].
- Also obtained by Friedel–Crafts acylation of 1,2,4-trimethoxybenzene with benzoyl chloride in the presence of aluminium chloride,
  - in carbon disulfide (by-product) [763], (15%) [761,762];
  - in ethylene dichloride between 0° and 5°, then at r.t. for 4 h and finally at reflux for 2 h (97%) [704].
- Preparation by reaction of benzoyl chloride with 3,4-dimethoxyphenol in the presence of boron trichloride in methylene chloride at r.t. for 2.5 h (51%) [598].

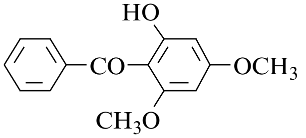
N.B.: This benzophenone is, by mistake named, 2-hydroxy-4,5-dimethoxybenzaldehyde (synthetic example 1, compound **h**, page 21), in the patent PCT Int. Appl. WO 96 05,188 of 22 February 1996 [598].

- Also obtained from O-methyldalbergin (6,7-dimethoxy-4-phenylcoumarin) (SM) by oxidation with neutral permanganate. SM was prepared by methylation of dalbergin (6-hydroxy-7-methoxy-4-phenylcoumarin), itself isolated from *Dalbergia sissoo* [764].
- Also refer to: [530,670].

m.p. 109–110° [656,704], 106–107° [760–762], 104–105° [598];
^{1}H NMR [598,704,760], IR [704], MS [704].

(2-Hydroxy-4,6-dimethoxyphenyl)phenylmethanone *(Hydrocotoin)*

[34425-64-4] $C_{15}H_{14}O_4$ mol.wt. 258.27



Syntheses

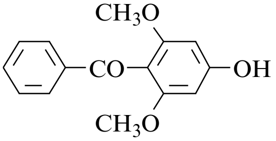
- Preparation from 2-bromo-3,5-dimethoxyphenyl benzoate on treatment with sec-butyllithium in a mixture of ethyl ether, hexane and tetrahydrofuran at −70° for 2 h, followed by treatment with saturated aqueous ammonium chloride (47%) [58].
- Preparation by Friedel–Crafts acylation of phloroglucinol trimethyl ether with benzoyl chloride in the presence of aluminium chloride [753], in ethyl ether for 8 h [416].
- Preparation by action of boron trichloride with 2,4,6-trimethoxybenzophenone in methylene chloride for 30 min at r.t. under nitrogen (92%) [171].
- Preparation by heating a mixture of benzoyl chloride and 3,5-dimethoxyphenyl benzoate in the presence of zinc chloride in benzene, followed by saponification of the keto ester so formed [220,765].
- Also obtained (poor yield) by reaction of benzonitrile with phloroglucinol dimethyl ether in the presence of zinc chloride and hydrochloric acid in ethyl ether (Hoesch reaction) [653], (3%) [766].
- Also obtained from 5,7-dimethoxy-4-phenylcoumarin,
  - by oxidation with potassium permanganate in acetone for 30 min (<5%) [767];
  - by ozonolysis in a mixture of carbon tetrachloride and chloroform (25%) [767].
- Also obtained (poor yield) by UV light irradiation of 2,4,6-trimethoxybenzophenone in carbon tetrachloride for 500 h (3%) [678].
- Also refer to: [379,686,768–770].

Isolation from natural sources

- From the heartwood of *Allanblackia fluoribunda* Oliver (Guttiferae; subfamily Clusioideae) [171];
- From the Paracotobark or *Aniba pseudocoto* Rusby (Kostermans) (Lauraceae) [694,771];
- From the *Coto* bark (Lauraceae) [772].

m.p. 99–100° [753], 98° [447,694,766,773,774], 97–98° [678], 96–98° [171], 96–97° [58], 95–96° [767], 93–95° [765]; ^{1}H NMR [58,171,416,678], IR [171], UV [171,767], MS [58,171]; GLC [678].

(4-Hydroxy-2,6-dimethoxyphenyl)phenylmethanone



$C_{15}H_{14}O_4$ mol.wt. 258.27

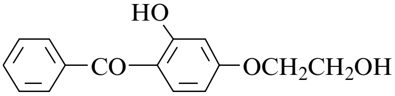
Syntheses

- Preparation by saponification of 4-(benzoyloxy)-2,6-di-methoxybenzophenone with 10% potassium hydroxide in methanol at r.t. for 2 h [775].
- Also obtained by reaction of benzonitrile with phloroglucinol dimethyl ether in the presence of zinc chloride and hydrochloric acid (14%) (Hoesch reaction) [766].
- Also refer to: [379].

m.p. 178–179° [775], 177° [766]; Spectra (NA).

[2-Hydroxy-4-(2-hydroxyethoxy)phenyl]phenylmethanone

[16909-78-7]



$C_{15}H_{14}O_4$ mol.wt. 258.27

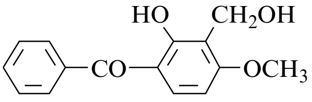
Syntheses

- Preparation by reaction of ethylene chlorohydrin (2-chloroethanol) with resbenzophenone,
  - in the presence of sodium hydroxide, in boiling water for 6 h (40%) [238] or in water at 70° for 18 h (91%) [776];
  - in the presence of sodium carbonate in refluxing dilute ethanol for 16 h (58%) [777].
- Preparation by reaction of ethylene carbonate with resbenzophenone,
  - in the presence of a quaternary ammonium salt (for example benzyltriethylammonium chloride) at 140–150° [778], for 11 h (93%) [779];
  - in the presence of an alkaline metal or alkaline earth metal salt as catalyst (sodium ethylenediamine tetraacetate (0.02 mol); calcium citrate monohydrate (0.02 mol); sodium nitriloacetate (0.02 mol); sodium oleate (0.05 mol); sodium stearate (0.05 mol)), for 7–8 h at 155° (92–93% yields) [780];
  - in the presence of sodium methoxide in diisobutylketone for 50 min at 140° [700].
- Preparation by treatment of resbenzophenone with ethylene oxide,
  - in methanol in the presence of sodium methoxide in autoclave at 150°, under 16.4 atmospheres, for 6 h (54%) [777];
  - in the presence of aqueous potassium hydroxide at 105–110° into an autoclave (<90 psig) under nitrogen (99%) [781].
- Also refer to: [782–788] and also [789–793] (Japanese patents).

m.p. 93°5–95°5 [794], 92–93° [777], 91°5–97°5, this gap of 6° C appears in the patent U.S. US 4,978,797 [781], 91–92° [238], 89–90° [93];
UV [93]; gel chromatography [247]; pK_a [93]; TLC [116].

[2-Hydroxy-3-(hydroxymethyl)-4-methoxyphenyl]phenylmethanone

[80604-76-8] $C_{15}H_{14}O_4$ mol.wt. 258.27



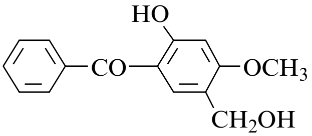
Synthesis

- Obtained by adding a 27% solution of formaldehyde to a solution of 2-hydroxy-4-methoxybenzophenone in aqueous sodium hydroxide and tetrahydrofuran then stirring the mixture at r.t. for 5.5 h (31%) [795,796].
- Also refer to: [665].

m.p. and Spectra (NA).

[2-Hydroxy-5-(hydroxymethyl)-4-methoxyphenyl]phenylmethanone

[80501-48-0] $C_{15}H_{14}O_4$ mol.wt. 258.27



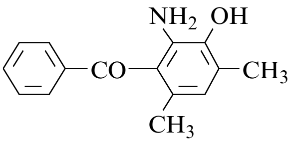
Synthesis

- Preparation by adding a 37% solution of formaldehyde to a solution of 2-hydroxy-4-methoxybenzophenone in aqueous sodium hydroxide and tetrahydrofuran then stirring the mixture at r.t. for 24 h (75%) [795]. In the same conditions, but using a 27% solution of formaldehyde and stirring the mixture at r.t. for 5.5 h, the keto alcohol was obtained in a yield of only 47% [795,796].
- Also refer to: [665,797].

m.p. and Spectra (NA).

(2-Amino-3-hydroxy-4,6-dimethylphenyl)phenylmethanone

$C_{15}H_{15}NO_2$ mol.wt. 241.29



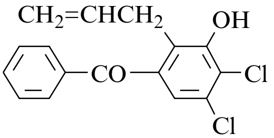
Synthesis

- Preparation by reduction of 2-nitro-3-hydroxy-4,6-dimethyl-benzophenone with sodium hydrosulfite in dilute ethanol (67%) [705].

m.p. 125° [705]; Spectra (NA).

[4,5-Dichloro-3-hydroxy-2-(2-propenyl)phenyl]phenylmethanone

[113730-42-0] $C_{16}H_{12}Cl_2O_2$ mol.wt. 307.18



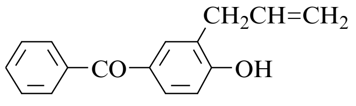
Synthesis

- Obtained by heating 5-(allyloxy)-3,4-dichlorobenzo-phenone at 235° for 8 min (67%) (Claisen rearrangement) [482].

m.p. 105° [482]; ^1H NMR [482].

[4-Hydroxy-3-(2-propenyl)phenyl]phenylmethanone

[73720-75-9]



$C_{16}H_{14}O_2$ mol.wt. 238.29

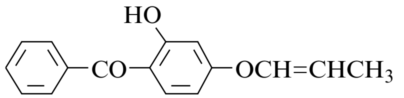
Synthesis

- Preparation by Claisen rearrangement of 4-(allyloxy)-benzophenone in refluxing phenyl ether for 1 h (73%) [798] or in diethylaniline at 207–218° for 3.5 h (47%) [799].

- Also refer to: [429].

m.p. 129–130°6 [798]; UV [798].

[2-Hydroxy-4-(1-propenyloxy)phenyl]phenylmethanone



$C_{16}H_{14}O_3$ mol.wt. 254.29

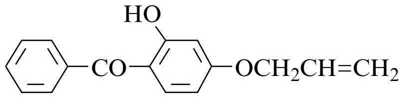
Synthesis

- Obtained (poor yield) by reaction of epichlorohydrin with resbenzophenone in the presence of aqueous potassium hydroxide at r.t. for 18 h (<6%) [800].

m.p. 97–98°5 [800]; Spectra (NA).

[2-Hydroxy-4-(2-propenyloxy)phenyl]phenylmethanone

[2549-87-3]



$C_{16}H_{14}O_3$ mol.wt. 254.29

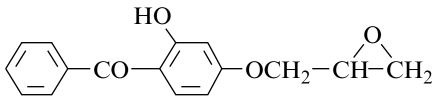
Synthesis

- Preparation by reaction of allyl chloride (3-chloro-propene) with resbenzophenone (90%), according to [801].

m.p. 65–68° [801]; Spectra (NA); TLC [116].

[2-Hydroxy-4-(oxiranylmethoxy)phenyl]phenylmethanone

[19389-82-3]



$C_{16}H_{14}O_4$ mol.wt. 270.28

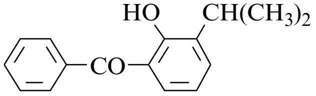
Synthesis

- Preparation by reaction of epichlorohydrin with 2,4-dihydroxybenzophenone in the presence of an aqueous potassium hydroxide solution at 88° for 2 h [802].

m.p. 99–100° [803], 98°5–99° [802]; IR [802], UV [802]; TLC [116].

[2-Hydroxy-3-(1-methylethyl)phenyl]phenylmethanone

[33621-54-4] $C_{16}H_{16}O_2$ mol.wt. 240.30



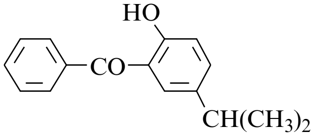
Synthesis

– Preparation by oxidation of 7-isopropyl-2,3-diphenyl-benzofuran with chromium trioxide in acetic acid at 70° for 2 h, followed by alkaline hydrolysis of the resulting keto ester (2-benzoyloxy-3-isopropylbenzophenone) in boiling dilute ethanol for 15 min [717].

Yellow liquid [717]; b.p.$_{3-5}$ 170–175° [717]; IR [717].

[2-Hydroxy-5-(1-methylethyl)phenyl]phenylmethanone

[20401-89-2] $C_{16}H_{16}O_2$ mol.wt. 240.30



Syntheses

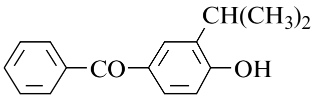
– Preparation by Fries rearrangement of p-isopropylphenyl benzoate with aluminium chloride [117].

– Also obtained by photo-Fries rearrangement of p-isopropyl-phenyl benzoate in pentane, with or without silica gel (32–35%) [64].

m.p. (NA); ^{1}H NMR [99], IR [99]; TLC [116]; polarographic study [117].

[4-Hydroxy-3-(1-methylethyl)phenyl]phenylmethanone

[83938-73-2] $C_{16}H_{16}O_2$ mol.wt. 240.30



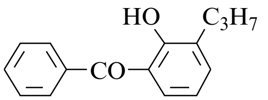
Synthesis

– Preparation by decarboxylation of 2-(4-hydroxy-3-iso-propylbenzoyl)benzoic acid in the presence of cupric acetate monohydrate in refluxing quinoline for 45 min (97%) [804].

m.p. 134°1–135°2 [804]; ^{1}H NMR [804], IR [804], UV [804].

(2-Hydroxy-3-propylphenyl)phenylmethanone

[108294-70-8] $C_{16}H_{16}O_2$ mol.wt. 240.30



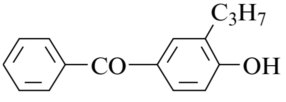
Synthesis

– Preparation by reaction of 2-hydroxy-3-propylbenzoyl chloride with benzene in the presence of aluminium chloride in refluxing carbon disulfide (42%) [92].

m.p. 34–35° [92]; Spectra (NA).

(4-Hydroxy-3-propylphenyl)phenylmethanone

[183013-50-5] $C_{16}H_{16}O_2$ mol.wt. 240.30



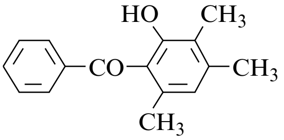
Synthesis

- Preparation by catalytic hydrogenation of 3-allyl-4-hydroxy-benzophenone (SM). SM was obtained by reaction of allyl bromide with 4-hydroxybenzophenone in the presence of potassium carbonate, followed by Claisen rearrangement of the 4-(allyloxy)benzophenone so formed [168].

m.p. and Spectra (NA).

(2-Hydroxy-3,4,6-trimethylphenyl)phenylmethanone

[33621-48-6] $C_{16}H_{16}O_2$ mol.wt. 240.30



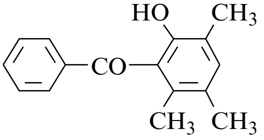
Synthesis

- Obtained by oxidation of 2,3-diphenyl-4,6,7-trimethyl-benzofuran with chromium trioxide in acetic acid at 65° for 1.5 h, then saponification of the resulting keto ester, the 2-(benzoyloxy)-3,4,6-trimethylbenzophenone, with 10% sodium hydroxide in boiling dilute ethanol for 30 min [723].
- Also refer to: [76].

m.p. 102° [723]; IR [723].

(2-Hydroxy-3,5,6-trimethylphenyl)phenylmethanone

[33634-16-1] $C_{16}H_{16}O_2$ mol.wt. 240.30



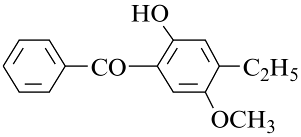
Synthesis

- Obtained by oxidation of 2,3-diphenyl-4,5,7-trimethyl-benzofuran with chromium trioxide in acetic acid at 65° for 1.5 h, then saponification of the resulting keto ester, the 2-(benzoyloxy)-3,5,6-trimethylbenzophenone, with 10% sodium hydroxide in boiling dilute ethanol for 30 min [723].

m.p. 127° [723]; IR [723].

(4-Ethyl-2-hydroxy-5-methoxyphenyl)phenylmethanone

[59623-15-3] $C_{16}H_{16}O_3$ mol.wt. 256.30



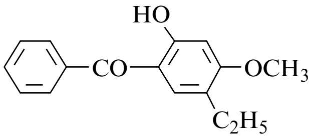
Synthesis

- Preparation by saponification of 2-(benzoyloxy)-4-ethyl-5-methoxybenzophenone (SM) with potassium hydroxide in refluxing ethanol for 1 h (73%). SM was obtained by oxidation of 6-ethyl-5-methoxy-2,3-diphenylbenzofuran with chromium trioxide in refluxing acetic acid for 30 min (80%, m.p. 140°) [370].

m.p. 124° [370]; Spectra (NA).

(5-Ethyl-2-hydroxy-4-methoxyphenyl)phenylmethanone

[59623-21-1] $C_{16}H_{16}O_3$ mol.wt. 256.30



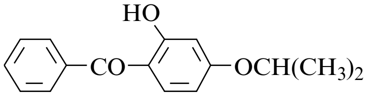
Synthesis

- Preparation by saponification of 2-(benzoyloxy)-5-ethyl-4-methoxybenzophenone (SM) with potassium hydroxide in refluxing ethanol for 1 h (67%). SM was obtained by oxidation of 5-ethyl-6-methoxy-2,3-diphenylbenzofuran with chromium trioxide in refluxing acetic acid for 30 min (64%, m.p. 181°) [370].

m.p. 170° [370]; Spectra (NA).

[2-Hydroxy-4-(1-methylethoxy)phenyl]phenylmethanone

$C_{16}H_{16}O_3$ mol.wt. 256.30



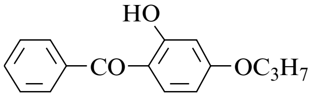
Syntheses

- Preparation by reaction of isopropyl bromide with resbenzophenone in the presence of potassium hydroxide in dilute ethanol at 75–80° for 15 h (38%) [800].
- Preparation by Friedel–Crafts acylation of 1,3-diisopropoxybenzene with benzoyl chloride in the presence of a zinc chloride/aluminium chloride mixture, first between 0° and 5°, then at 10° for 1 h and finally at 65° for 6 h [658].

m.p. 42–42°5 [800]; Spectra (NA).

(2-Hydroxy-4-propoxyphenyl)phenylmethanone

[3088-11-7] $C_{16}H_{16}O_3$ mol.wt. 256.30



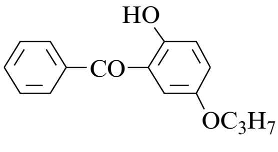
Synthesis

- Preparation by reaction of n-propyl bromide with resbenzophenone in the presence of sodium hydroxide in dilute ethanol at 70–80° for 13 h (30%) [189].
- Also refer to: [222].

m.p. 67° [189]; Spectra (NA).

(2-Hydroxy-5-propoxyphenyl)phenylmethanone

$C_{16}H_{16}O_3$ mol.wt. 256.30



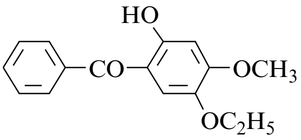
Synthesis

- Preparation by reaction of n-propyl bromide with 2,5-di-hydroxybenzophenone in the presence of sodium hydroxide in dilute ethanol at 70–80° for 28 h (25%) [681].

m.p. and Spectra (NA).

(5-Ethoxy-2-hydroxy-4-methoxyphenyl)phenylmethanone

[52811-38-8]



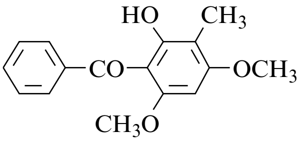
$C_{16}H_{16}O_4$ mol.wt. 272.30

Synthesis

- Preparation by partial ethylation of 2,5-dihydroxy-4-methoxybenzophenone with diethyl sulfate in the presence of potassium carbonate in refluxing acetone for 5 h (75%) [763].

m.p. 100–102° [763]; ^1H NMR [763], IR [763], UV [763].

(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)phenylmethanone



$C_{16}H_{16}O_4$ mol.wt. 272.30

Syntheses

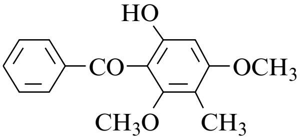
- Preparation by reaction of methyl iodide with 2,4,6-tri-hydroxy-3-methylbenzophenone or with 2,6-dihydroxy-4-methoxy-3-methylbenzophenone in the presence of potassium carbonate in boiling acetone for 8 h [805,806].
- Preparation by reaction of diazomethane with 2,4,6-trihydroxy-3-methylbenzophenone in ethyl ether (86%) [806].
- Also refer to: [807,808].

Isolation from natural source

- From the leaves of *Leptospermum luehmannii* (F. M. Bailey) (Myrtaceae) (minor product) [171,449,806,809].

m.p. 137° [806,809], 136–137° [805]; ^1H NMR [806], UV [806].

(2-Hydroxy-4,6-dimethoxy-5-methylphenyl)phenylmethanone



$C_{16}H_{16}O_4$ mol.wt. 272.30

Synthesis

- Not yet described.

Isolation from natural source

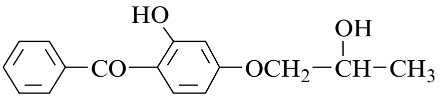
- From the leaves or rhizomes of *Agonis luehmannii* (now *Leptospermum luehmannii*) (F. M. Bailey) (Myrtaceae) (major product) [171,449,806,809].

m.p. 110° [806], 104° [809];

^1H NMR [806], IR [806], UV [806]; GC [806].

[2-Hydroxy-4-(2-hydroxypropoxy)phenyl]phenylmethanone

[22546-86-7]



$C_{16}H_{16}O_4$ mol.wt. 272.30

Synthesis

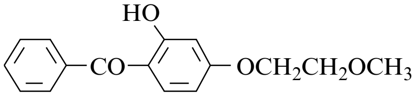
- Preparation by reaction of propylene oxide with resbenzophenone in the presence of aqueous potassium hydroxide into an autoclave (140°) under nitrogen (66%) [781].

m.p. 78–80° [781]; Spectra (NA).

[2-Hydroxy-4-(2-methoxyethoxy)phenyl]phenylmethanone

[27992-95-6] $C_{16}H_{16}O_4$ mol.wt. 272.30

Syntheses



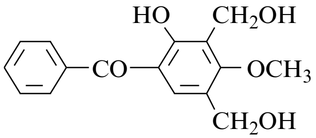
- Preparation by reaction of 2-methoxyethyl chloride (2-chloroethyl methyl ether) with resbenzophenone (91%), according to [801].
- Preparation by reaction of 2-methoxyethylene bromide with 2,4-dihydroxybenzophenone in the presence of sodium carbonate in acetone at 50–55° (74%) [810].

m.p. 40–41° [801]; Spectra (NA).

[2-Hydroxy-3,5-di(hydroxymethyl)-4-methoxyphenyl]phenylmethanone

[80501-47-9] $C_{16}H_{16}O_5$ mol.wt. 288.30

Synthesis



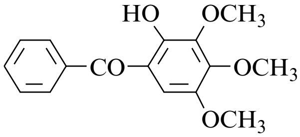
- Obtained (poor yield) by adding a 27% solution of formaldehyde to a solution of 2-hydroxy-4-methoxy-benzophenone in aqueous sodium hydroxide and tetrahydrofuran then stirring the mixture at r.t. for 5.5 h (3%) [795,796].

m.p. and Spectra (NA).

(2-Hydroxy-3,4,5-trimethoxyphenyl)phenylmethanone

[42833-88-5] $C_{16}H_{16}O_5$ mol.wt. 288.30

Synthesis



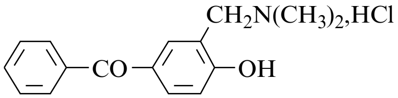
- Preparation by reaction of benzoyl chloride with 1,2,3,4-tetramethoxybenzene in the presence of aluminium chloride in ethyl ether for 22 h [416].

m.p. (NA); ^{1}H NMR [416].

[3-[(Dimethylamino)methyl]-4-hydroxyphenyl]phenylmethanone *(Hydrochloride)*

[82506-20-5] $C_{16}H_{17}NO_2$,HCl mol.wt. 291.78

Synthesis

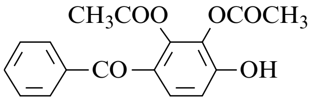


- Preparation by aminomethylation of p-hydroxy-benzophenone with dimethylamine and formaline in water at 35–40° for 4 h (52%) [811].

m.p. 77° [811]; ^{1}H NMR [811], IR [811].

[2,3-Bis(acetyloxy)-4-hydroxyphenyl]phenylmethanone

[177703-36-5] $C_{17}H_{14}O_6$ mol.wt. 314.29



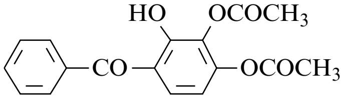
Synthesis

- Obtained by action of acetic anhydride with 2,3,4-tri-hydroxybenzophenone in the presence of lithium carbonate in N,N-dimethylformamide at r.t. for 15 h under nitrogen (12%) [369].

m.p. (NA); ^{1}H NMR [369].

[3,4-Bis(acetyloxy)-2-hydroxyphenyl]phenylmethanone

[177703-35-4] $C_{17}H_{14}O_6$ mol.wt. 314.29



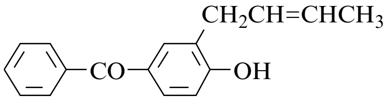
Synthesis

- Obtained by action of acetic anhydride with 2,3,4-tri-hydroxybenzophenone in the presence of lithium carbonate in N,N-dimethylformamide at r.t. for 15 h under nitrogen (28%) [369].

m.p. (NA); ^{1}H NMR [369].

[3-(2-Butenyl)-4-hydroxyphenyl]phenylmethanone

[96825-03-5] $C_{17}H_{16}O_2$ mol.wt. 252.31



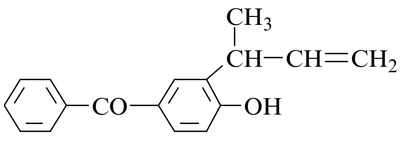
Synthesis

- Preparation by condensation of p-hydroxybenzo-phenone with 1,3-buta-diene in the presence of orthophosphoric acid in petroleum ether at 30–35° for 24 h (77%) [769].

m.p. 104–106° [769]; ^{1}H NMR [769].

[4-Hydroxy-3-(1-methyl-2-propenyl)phenyl]phenylmethanone

[73720-57-7] $C_{17}H_{16}O_2$ mol.wt. 252.31



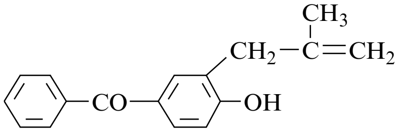
Synthesis

- Preparation by Claisen rearrangement of 4-(γ-methallyloxy)benzophenone [799].
- Also refer to: [457].

m.p. and Spectra (NA).

[4-Hydroxy-3-(2-methyl-2-propenyl)phenyl]phenylmethanone

[112005-09-1]



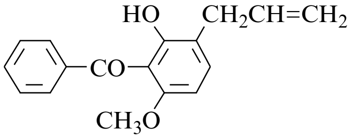
$C_{17}H_{16}O_2$ mol.wt. 252.31

Synthesis

- Preparation by heating 4-(β-methallyloxy)-benzophenone in diethylaniline for 3.5 h between 207° and 218° (73%) (Claisen rearrangement) [799].
- Also refer to: [457].

m.p. 131–133° [799]; ^{1}H NMR [799].

[2-Hydroxy-6-methoxy-3-(2-propenyl)phenyl]phenylmethanone



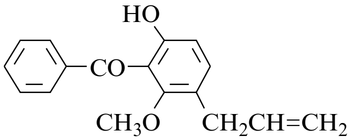
$C_{17}H_{16}O_3$ mol.wt. 268.31

Syntheses

- Preparation by Claisen rearrangement of 2-allyloxy-6-methoxybenzophenone in boiling diethylaniline for 40 min (40%) [269].
- Also obtained (by-product) by partial methylation of 3-allyl-2,6-dihydroxybenzophenone with dimethyl sulfate in boiling benzene in the presence of potassium hydroxide for 10 h (16%) [269].

b.p.$_3$ 192–194° [269]; $d_4^{20} = 0.888$ [269]; $n_D^{20} = 1.5960$ [269]; TLC [269]; Spectra (NA).

[6-Hydroxy-2-methoxy-3-(2-propenyl)phenyl]phenylmethanone



$C_{17}H_{16}O_3$ mol.wt. 268.31

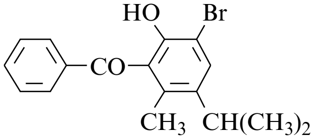
Syntheses

- Preparation by partial methylation of 3-allyl-2,6-di-hydroxybenzophenone with dimethyl sulfate in boiling benzene in the presence of potassium hydroxide for10 h (50%) [269].
- Obtained (by-product) by Claisen rearrangement of 2-allyloxy-6-methoxybenzophenone in boiling diethylaniline for 40 min (10%) [269].

yellow oil [269]; b.p. and Spectra (NA); TLC [269].

[3-Bromo-2-hydroxy-6-methyl-5-(1-methylethyl)phenyl]phenylmethanone

[143815-17-2]



$C_{17}H_{17}BrO_2$ mol.wt. 333.22

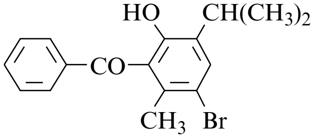
Synthesis

- Preparation by Fries rearrangement of 2-bromo-5-methyl-4-isopropylphenyl benzoate with titanium tetrachloride without solvent at 140° for 2 h (62%) [698].

m.p. 119–120° [698]; ^{1}H NMR (Sadtler: standard n° 59400 M) [698], IR (Sadtler: standard n° 86556 K) [698], UV [698], MS [698].

[3-Bromo-6-hydroxy-2-methyl-5-(1-methylethyl)phenyl]phenylmethanone

[16846-17-6]



$C_{17}H_{17}BrO_2$ mol.wt. 333.22

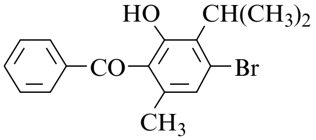
Synthesis

- Preparation by bromination of 2-hydroxy-6-methyl-3-iso-propylbenzophenone [501], with bromine in chloroform at r.t. for 6 h [812].

m.p. 129° [501], 128–129° [812], 74° [735]. One of the reported melting points is obviously wrong.
^{1}H NMR [735], IR [103,501,735], UV [735]; pK_a [735]; polarographic study [117].

[4-Bromo-2-hydroxy-6-methyl-3-(1-methylethyl)phenyl]phenylmethanone

[16846-13-2]



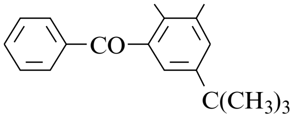
$C_{17}H_{17}BrO_2$ mol.wt. 333.22

Synthesis

- Preparation by reaction of chromium trioxide with 6-bromo-4-methyl-2,3-diphenyl-7-iso-propylbenzofuran in boiling acetic acid, followed by saponification of the keto ester so formed [501], by action of potassium hydroxide in ethanol in a water bath for 1 h [812].

m.p. 91° [501,812]; IR [103,813].

[3-Chloro-5-(1,1-dimethylethyl)-2-hydroxyphenyl]phenylmethanone



$C_{17}H_{17}ClO_2$ mol.wt. 288.77

Synthesis

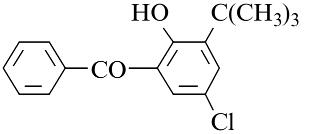
- Obtained (poor yields) by photo-Fries rearrangement of two substituted phenyl esters^{T} in benzene [154],^{T}4-tert-butyl-2-chlorophenyl benzoate (7%);

^{T}4-tert-butyl-2,6-dichlorophenyl benzoate (11%). In this case, one chlorine atom was eliminated.

m.p. 55–59° [154]; b.p.$_{0.2}$ 150° [154]; Spectra (NA).

[5-Chloro-3-(1,1-dimethylethyl)-2-hydroxyphenyl]phenylmethanone

[52196-47-1] $C_{17}H_{17}ClO_2$ mol.wt. 288.77



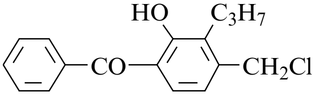
Synthesis

– Refer to: [531].

m.p. and Spectra (NA); pK_a [531].

[4-(Chloromethyl)-2-hydroxy-3-propylphenyl]phenylmethanone

[97582-40-6] $C_{17}H_{17}ClO_2$ mol.wt. 288.77



Synthesis

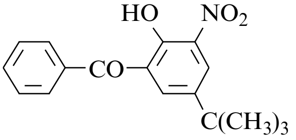
– Preparation by reaction of ethyl chloroformate with 4-(dimethylamino)methyl-2-hydroxy-3-propylbenzo-phenone in toluene cooled with an ice water bath for 2 h, then at r.t. for 16 h (55%) [814].

– Also refer to: [168,815].

m.p. 47–49° [814]; Spectra (NA); HPLC [814].

[5-(1,1-Dimethylethyl)-2-hydroxy-3-nitrophenyl]phenylmethanone

[85052-33-1] $C_{17}H_{17}NO_4$ mol.wt. 299.33



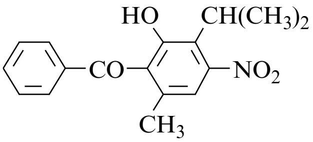
Synthesis

– Preparation by reaction of a concentrated nitric acid/concentrated sulfuric acid mixture with 5-tert-butyl-2-hydroxybenzophenone in methylene chloride at 5° for 30 min (42%) [472].

m.p. (NA); ^{1}H NMR [472], MS [472].

[2-Hydroxy-6-methyl-3-(1-methylethyl)-4-nitrophenyl]phenylmethanone

$C_{17}H_{17}NO_4$ mol.wt. 299.33



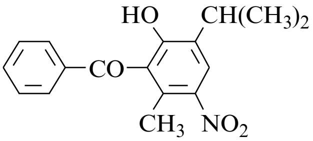
Synthesis

– Refer to: [103,503].

m.p. (NA); IR [103], UV [503].

[2-Hydroxy-6-methyl-3-(1-methylethyl)-5-nitrophenyl]phenylmethanone

$C_{17}H_{17}NO_4$ mol.wt. 299.33



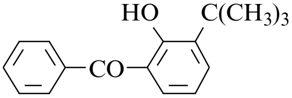
Synthesis

– Refer to: [103].

m.p. (NA); IR [103].

[3-(1,1-Dimethylethyl)-2-hydroxyphenyl]phenylmethanone

[24248-99-5] $C_{17}H_{18}O_2$ mol.wt. 254.33



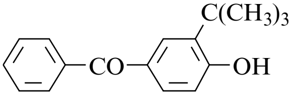
Syntheses

- Preparation by oxidation of 7-tert-butyl-2,3-diphenyl-benzofuran with chromium trioxide in acetic acid at 70° for 2 h, followed by alkaline hydrolysis of the keto ester obtained (2-benzoyloxy-3-tert-butylbenzophenone) in boiling diluted ethanol for 15 min [717].
- Also obtained by photo-Fries rearrangement of 2-tert-butylphenyl benzoate in benzene (35%) [143].
- Also obtained by reaction between (o-tert-butylphenoxy)magnesium bromide complexed with HMPT and benzaldehyde in refluxing benzene for 48 h (18%) [50].
- Also refer to: [597].

pale yellow viscous oil [143], m.p. 55° [717], 53° [50]; b.p.$_{0.01}$ 92° [143]; ^{1}H NMR [50,143], IR [50,143,717], MS [50].

[3-(1,1-Dimethylethyl)-4-hydroxyphenyl]phenylmethanone

[16928-03-3] $C_{17}H_{18}O_2$ mol.wt. 254.33



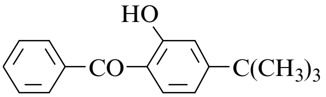
Syntheses

- Obtained by partial dealkylation of 3,5-di-tert-butyl-4-hydroxybenzophenone by UV light irradiation in cyclohexane (50%) [143].
- Also obtained by photo-Fries rearrangement of 2-tert-butylphenyl benzoate in benzene (20%) [143].
- Preparation by Friedel–Crafts acylation of o-tert-butylphenol with benzoyl chloride in ethylene dichloride in the presence of titanium tetrachloride, first with ice cooling, then for 5 h at r.t. (44%) [816].

m.p. 179–180° [143]; ^{1}H NMR [143], IR [143].

[4-(1,1-Dimethylethyl)-2-hydroxyphenyl]phenylmethanone

[39000-51-6] $C_{17}H_{18}O_2$ mol.wt. 254.33



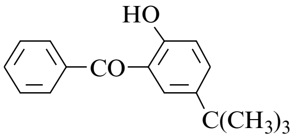
Syntheses

- Obtained by Fries rearrangement in hydrofluoric acid,
  - of m-tert-butylphenyl benzoate at 25° for 2 h (37%) [135];
  - of 3,5-di-tert-butylphenyl benzoate at 55° for 4 h (10%) [135].

m.p. 81–82° [135]; Spectra (NA).

[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]phenylmethanone

[10425-05-5] $C_{17}H_{18}O_2$ mol.wt. 254.33



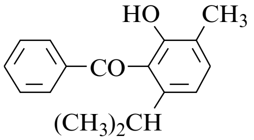
Syntheses

- Preparation by Fries rearrangement of p-tert-butylphenyl benzoate with,
  - titanium tetrachloride without solvent at 140° for 15 min (47%) [24] or in nitromethane at r.t. for 7 days (29%) [24];
  - aluminium chloride without solvent at 180° for 15 min (35%) [24].
- Preparation by demethylation of 5-tert-butyl-2-methoxybenzophenone in the presence of,
  - in a refluxing mixture of 47% hydrobromic acid (2 vol) and 57% hydriodic acid (1 vol) in acetic acid (10 vol) (89%) [817];
  - aluminium chloride in benzene at 50–60° for 5 h (63%) [9].
- Also obtained by UV light irradiation of two substituted phenyl esters in benzene: p-tert-butylphenyl benzoate (45%) [154], 2-chloro-4-tert-butylphenyl benzoate. In this last case, there is an elimination of chlorine atom (21%) [154].
- Also obtained by photo-Fries rearrangement of p-tert-butylphenyl benzoate in ethanol for 10 h or in ethyl ether [818].
- Preparation by reaction of benzotrichloride with p-tert-butylphenol in the presence of 30% aqueous sodium hydroxide at 75–80° for 30 min, and hydrolysis of the resulting ester as a side product by steam distillation (56–65%) [97,615].
- Preparation by treatment of o-hydroxybenzophenone at 120° with a mixture of isobutylene/nitrogen (1:1) or with tert-butyl chloride in the presence a macroreticular acid ion exchanger as catalyst (Wofatit OK 80) for 1 h (52%) [819].
- Also refer to: [79,87,472,597,820].

m.p. 67–68° [9], 67° [24,154], 63–65° [97], 52–53° [615];
b.p.$_{12}$ 195–197° [9], b.p.$_2$ 164–166° [97], b.p.$_{0.15}$ 125–130° [819];
^{1}H NMR [97,615], ^{13}C NMR [97], IR [24,97,154,615], UV [24,615], MS [615]; gas chromatography study [631]; TLC [116].

[2-Hydroxy-3-methyl-6-(1-methylethyl)phenyl]phenylmethanone

[33829-50-4] $C_{17}H_{18}O_2$ mol.wt. 254.33



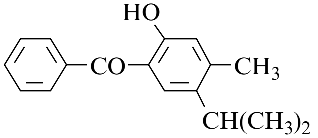
Synthesis

- Refer to: [100].

m.p. (NA); ^{1}H NMR [100], IR [100], UV [100].

[2-Hydroxy-4-methyl-5-(1-methylethyl)phenyl]phenylmethanone

[108974-20-5] $C_{17}H_{18}O_2$ mol.wt. 254.33



Syntheses

- Preparation by Fries rearrangement of p-thymyl benzoate with titanium tetrachloride at 140° for 2 h (82%) [698].
- Claimed to be prepared by two methods:
  - by reaction of 2-methoxy-4-methyl-5-isopropyl-benzoyl chloride (SM1) with benzene in the presence of aluminium chloride at 80° for 3 h, then at r.t. for 20 h (33%) [821];
  - from 2-methoxy-4-methyl-5-isopropylbenzophenone (SM2) by heating with pyridinium chloride for 1 h [821].

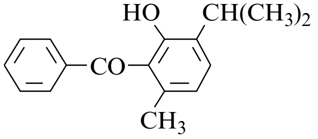
However, the structures of SM1 and SM2 were incorrect.

N.B.: All the results of reference [821] were erroneous. Only the first route was correct. The ^{1}H NMR spectra confirms the above structure [698].

m.p. 152°5 [821] (this melting point is incompatible with a non-vicinal ortho-acylphenol structure), 48–49° [698]; b.p.$_{20}$ 282–287° [821];
^{1}H NMR (Sadtler: standard n° 59398 M) [698], IR (Sadtler: standard n° 86554 K) [698], UV [698], MS [698].

[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]phenylmethanone

[4072-16-6] $C_{17}H_{18}O_2$ mol.wt. 254.33

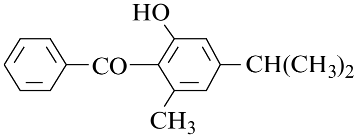


Syntheses

- Obtained by oxidation of 4-methyl-7-isopropyl-2,3-di-phenylbenzofuran with chromium trioxide in refluxing acetic acid (82%) [43,812], followed by saponification of the keto ester so formed [43,632,812].
- Obtained by refluxing mixture of benzoic acid/thymol/aluminium chloride for 12 h (76%) [822] (no reproductive reaction).
- Also refer to: [633].

m.p. 104° [735], 97° [89,501,609,632,812], 44° [822]. One of the reported melting points is obviously wrong.
^{1}H NMR [100,735], IR [100,103,107,108,582,735,813],
UV [100,735]; polarographic study [117]; pK_a [735].

[2-Hydroxy-6-methyl-4-(1-methylethyl)phenyl]phenylmethanone



$C_{17}H_{18}O_2$ mol.wt. 254.33

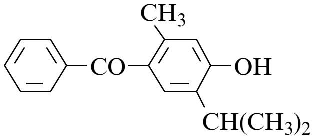
Synthesis

- Preparation (compound **II-f**) by saponification of 2-(benzoyloxy)-6-methyl-4-isopropylbenzophenone, itself obtained by chromic oxidation of 4-methyl-6-isopropyl-2,3-diphenylbenzofuran [633].

m.p. 80–83° [633]; IR [633].

[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]phenylmethanone

[28178-94-1]



$C_{17}H_{18}O_2$ mol.wt. 254.33

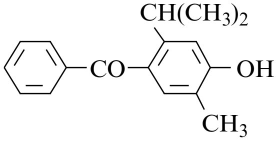
Syntheses

- Preparation by reaction of benzoyl chloride with thymol in the presence of aluminium chloride [812], (98%) [823], in nitrobenzene at r.t. overnight (good yield) [372].
- Preparation by demethylation of 4-methoxy-2-methyl-5-isopropylbenzophenone with pyridinium chloride at 205–215° for 1.5 h (95%) [824] or 15 min [825].
- Preparation by Fries rearrangement of thymyl benzoate with aluminium chloride in nitrobenzene at 60° for 5 h (70%) [132] or at 50° in the same conditions (21%) [131].
- Also obtained by reaction of benzotrichloride with thymol in the presence of stannic chloride at 60–65° for 15 h (20%) [823].
- Also obtained by saponification of 4-(benzoyloxy)-2-methyl-5-isopropylbenzophenone (SM) with sodium hydroxide in boiling ethanol [812]. SM was obtained by oxidation of 4-desylthymyl benzoate with chromium trioxide in boiling acetic acid.
- Also obtained (by-product) from p-thymyl benzoate by heating with aluminium chloride [826].
- Also refer to: [204,827] (Japanese patent).

m.p. 154° [824], 153° [132,372,812,825], 152°5 [826],
150–150°5 [823], 138–144° [131];
Spectra (NA).

[4-Hydroxy-5-methyl-2-(1-methylethyl)phenyl]phenylmethanone

[99821-75-7]



$C_{17}H_{18}O_2$ mol.wt. 254.33

Syntheses

- Preparation by Fries rearrangement of 2-methyl-5-isopropyl-phenyl benzoate,
  - in the presence of aluminium chloride in nitrobenzene at 60° for 5 h (60%) [132];
  - in the presence of Nafion-XR, a H^+-form ion exchange resin, at 150° for 4 h under nitrogen (31%) [39].

– Also obtained by reaction of benzoyl chloride with carvacrol,

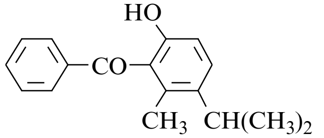
- in the presence of Nafion-XR at 150° for 4 h under nitrogen (20%) [39];
- in the presence of aluminium chloride in nitrobenzene at r.t. for 48 h (4%) [828].

– Also refer to: [204] (Japanese patent).

m.p. 172–173° [132], 126° [828]. One of the reported melting points is obviously wrong. Spectra (NA).

[6-Hydroxy-2-methyl-3-(1-methylethyl)phenyl]phenylmethanone

[108974-21-6] $C_{17}H_{18}O_2$ mol.wt. 254.33



Syntheses

– Preparation from 3-bromo-2-hydroxy-6-methyl-5-iso-propylbenzophenone (SM1) by reductive removal of bromine with copper powder in caproic acid at 220° for 15 min (83%) [698]. SM1 was obtained from p-thymol by a three-step synthesis (bromination, esterification and Fries rearrangement with titanium tetrachloride).

– Also obtained (by-product) by Fries rearrangement of p-thymyl benzoate with titanium tetrachloride at 140° for 2 h (10%) [698].

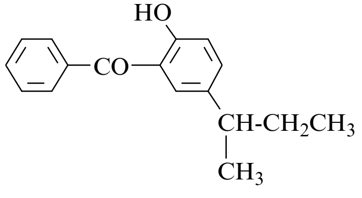
– Claimed to be prepared by heating 6-methoxy-2-methyl-3-isopropylbenzophenone (SM2) with pyridinium chloride at reflux for 1.5 h (85%) [821]. SM2 was obtained by Friedel–Crafts acylation of 3-methyl-4-isopropylanisole with benzoyl chloride in the presence of aluminium chloride in carbon disulfide at r.t. The structure of SM2 was erroneous. The true structure of SM2 must be the 2-methoxy-4-methyl-5-isopropylbenzophenone.

N.B.: All the results of reference [821] were erroneous. Only the first route was correct. The ^1H NMR spectra confirms the above structure [698].

m.p. 142–143° [698]; yellow viscous oil [821]; b.p.$_{14}$ 207° [821]; $n_D^{25.5}$ = 1.5950 [821]; ^1H NMR (Sadtler: standard n° 59401 M) [698], IR (Sadtler: standard n° 86557 K) [698], UV [698], MS [698].

[2-Hydroxy-5-(1-methylpropyl)phenyl]phenylmethanone

[59746-97-3] $C_{17}H_{18}O_2$ mol.wt. 254.33



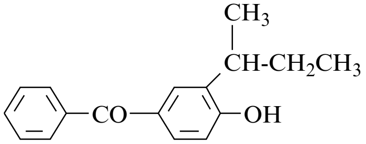
Synthesis

– Preparation by reaction of benzotrichloride with 4-sec-butylphenol in hydrofluoric acid in the presence of water at –10°, then between 0° and –10° for 2 h, at r.t. for 7 h and at 80° for 30 min into an autoclave (60%) [213].

yellow oil [213]; b.p.$_1$ 150–155° [213]; Spectra (NA).

[4-Hydroxy-3-(1-methylpropyl)phenyl]phenylmethanone

[124979-07-3] $C_{17}H_{18}O_2$ mol.wt. 254.33



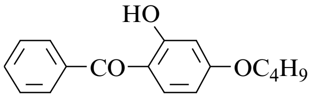
Synthesis

- Preparation by Friedel–Crafts acylation of 2-sec-butylphenol—2-(1-methylpropyl)phenol—with benzoyl chloride in ethylene dichloride in the presence of titanium tetrachloride, first at 0°, then at r.t. [816].

m.p. 95–96° [816]; Spectra (NA).

(4-Butoxy-2-hydroxyphenyl)phenylmethanone

[15131-43-8] $C_{17}H_{18}O_3$ mol.wt. 270.33



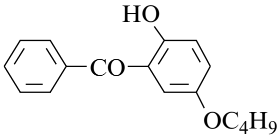
Syntheses

- Preparation by reaction of n-butyl bromide with resbenzophenone,
  - in the presence of sodium hydroxide in dilute ethanol in a water bath (40%) [189];
  - in the presence of potassium carbonate in cyclohexanone at 145° for 6 h (72%) [202], (48%) [201].
- Preparation by alkylation of resbenzophenone with a butyl halide [651].
- Preparation by Friedel–Crafts acylation of 1,3-di-n-butoxybenzene with benzoyl chloride,
  - in the presence of a zinc chloride/aluminium chloride mixture, first between 0° and 5°, then at 10° for 1 h and at 65° for 6 h [658];
  - in the presence of aluminium chloride in chlorobenzene at 90° [222].
- Preparation by partial dealkylation of 4-butoxy-2-methoxybenzophenone or 2,4-dibutoxy-benzophenone with aluminium chloride in chlorobenzene at 80–100° [655].
- Also refer to: [78,231,235,667,829].

m.p. 56–57° [189], 52–53° [201], 50–53° [202]; UV [235]; TLC [116]; pK_a [531].

(5-Butoxy-2-hydroxyphenyl)phenylmethanone

$C_{17}H_{18}O_3$ mol.wt. 270.33



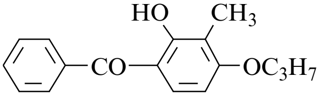
Synthesis

- Obtained by reaction of n-butyl bromide with 2,5-di-hydroxybenzophenone in the presence of sodium hydroxide in dilute ethanol at 80–85° for 35 h (21%) [681].

m.p. 42° [681]; Spectra (NA).

(2-Hydroxy-3-methyl-4-propoxyphenyl)phenylmethanone

[172479-20-8]



$C_{17}H_{18}O_3$ mol.wt. 270.33

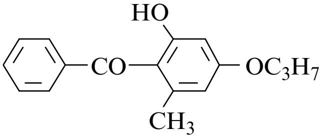
Synthesis

- Prepared by standard techniques [830].

m.p. (NA); UV [830].

(2-Hydroxy-6-methyl-4-propoxyphenyl)phenylmethanone

[172479-19-5]



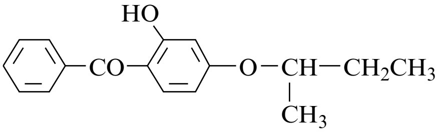
$C_{17}H_{18}O_3$ mol.wt. 270.33

Synthesis

- Prepared by standard techniques [830].

m.p. (NA); UV [830].

[2-Hydroxy-4-(1-methylpropoxy)phenyl]phenylmethanone



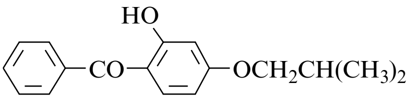
$C_{17}H_{18}O_3$ mol.wt. 270.33

Synthesis

- Preparation by reaction of sec-butyl bromide with resbenzophenone in the presence of potassium hydroxide in dilute ethanol at 80–90° for 20 h (29%) [800].

m.p. 41° [800]; Spectra (NA).

[2-Hydroxy-4-(2-methylpropoxy)phenyl]phenylmethanone



$C_{17}H_{18}O_3$ mol.wt. 270.33

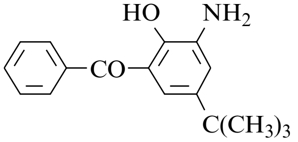
Synthesis

- Preparation by reaction of isobutyl bromide with resbenzophenone in the presence of potassium hydroxide in dilute ethanol at 80–92° for 18 h (23%) [800].

m.p. 100–100°5 [800]; Spectra (NA).

[3-Amino-5-(1,1-dimethylethyl)-2-hydroxyphenyl]phenylmethanone

[85052-51-3] $C_{17}H_{19}NO_2$ mol.wt. 269.34



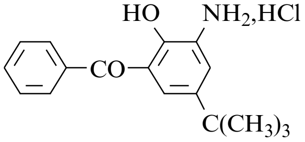
Synthesis

– Preparation by reduction of 5-tert-butyl-2-hydroxy-3-nitro-benzophenone with titanium trichloride in a benzene/tetrahydrofuran mixture for 2 h at r.t. (34%) [472].

m.p. 123–124° [472]; ^{1}H NMR [472], IR [472], MS [472]; TLC [472].

[3-Amino-5-(1,1-dimethylethyl)-2-hydroxyphenyl]phenylmethanone *(Hydrochloride)*

[85069-31-4] $C_{17}H_{19}NO_2,HCl$ mol.wt. 305.81



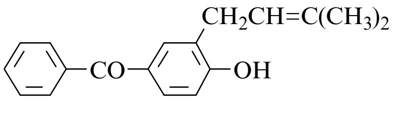
Synthesis

– Obtained by treatment of 3-amino-5-tert-butyl-2-hydroxy-benzophenone with 2 N hydrochloric acid [472].

m.p. and Spectra (NA).

[4-Hydroxy-3-(3-methyl-2-butenyl)phenyl]phenylmethanone

[63565-02-6] $C_{18}H_{18}O_2$ mol.wt. 266.34



Synthesis

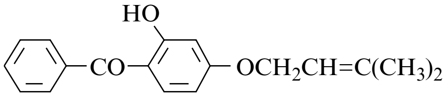
– Obtained (poor yield) by reaction of prenyl bromide with p-hydroxybenzophenone,

- in the presence of sodium methoxide in refluxing methanol for 4 h (8%) [831];
- in the presence of silver oxide in dioxane at r.t. for 2 h (13%) [832].

m.p. 77° [831], 76–77° [832]; ^{1}H NMR [831], IR [831], UV [831].

[2-Hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]phenylmethanone

[63564-99-8] $C_{18}H_{18}O_3$ mol.wt. 282.34



Synthesis

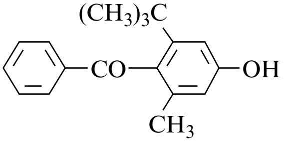
– Obtained by reaction of prenyl bromide with resbenzophenone,

- in the presence of potassium carbonate in refluxing acetone for 4 h (30%) [831];
- in the presence of sodium methoxide in refluxing methanol for 4 h [831].

m.p. 82° [831]; ^{1}H NMR [831], IR [831], UV [831].

[2-(1,1-Dimethylethyl)-4-hydroxy-6-methylphenyl]phenylmethanone

[133721-73-0] $C_{18}H_{20}O_2$ mol.wt. 268.36



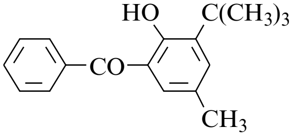
Synthesis

– Obtained by treating a solution of 2-(tert-butyl)-4-[[(tert-butyl)dimethylsilyl]oxy]-6-methylbenzophenone in ethanol with 2 N aqueous hydrochloric acid for 1 h at 75° in a sealed tube (94%) [833].

m.p. 150–152° [833]; ^{1}H NMR [833], IR [833], MS [833].

[3-(1,1-Dimethylethyl)-2-hydroxy-5-methylphenyl]phenylmethanone

[52196-46-0] $C_{18}H_{20}O_2$ mol.wt. 268.36



Synthesis

– The reaction of benzoyl chloride with a pentane solution of [AlMe(dbmp)$_2$] leads to acylation of one of the dbmp ligands and affords [AlMe(dbmp)(bhmbp)]. Hydrolysis of this complex with a saturated solution of ammonium chloride gave the attempted ketone (75%) [834].

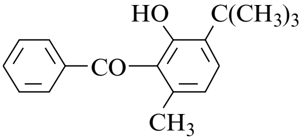
N.B.: Hdbmp = 2,6-di-tert-butyl-4-methylphenol;

Hbhmbp = 3-tert-butyl-2-hydroxy-5-methylbenzophenone.

m.p. >240° [834]; ^{1}H NMR [834], ^{13}C NMR [834]; IR [834], MS [834]; pK_a [531].

[3-(1,1-Dimethylethyl)-2-hydroxy-6-methylphenyl]phenylmethanone

[14963-84-9] $C_{18}H_{20}O_2$ mol.wt. 268.36



Synthesis

– Preparation by saponification of 2-(benzoyloxy)-3-tert-butyl-6-methylbenzophenone (SM) with sodium hydroxide in boiling ethanol for 2 h. SM was obtained by oxidation of 7-tert-butyl-4-methyl-2,3-diphenylbenzofuran with chromium trioxide in acetic acid at 60° (m.p. 127°) [633].

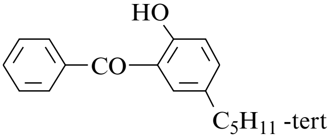
– Also refer to: [117,609].

m.p. 125–126° [735], 77° [633]. One of the reported melting points is obviously wrong.

^{1}H NMR [735], IR [735,813], UV [735]; pK_a [735]; polarographic study [117].

[5-(1,1-dimethylpropyl)-2-hydroxyphenyl]phenylmethanone

[110701-33-2] $C_{18}H_{20}O_2$ mol.wt. 268.36



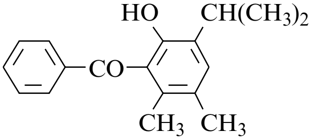
Synthesis

- Preparation by reaction of benzotrichloride with p-tert-pentylphenol in the presence of 30% aqueous sodium hydroxide at 75–80° for 30 min, and hydrolysis of the resulting ester as a side product (43%) [97].

m.p. 37–38°5 [97]; b.p.$_2$ 164–167° [97]; ^{1}H NMR [97], ^{13}C NMR [97], IR [97].

[2-Hydroxy-5,6-dimethyl-3-(1-methylethyl)phenyl]phenylmethanone

[109252-33-7] $C_{18}H_{20}O_2$ mol.wt. 268.36



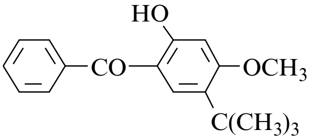
Synthesis

- Obtained by Friedel–Crafts acylation of 4-methylthymol (4,5-dimethyl-2-isopropylphenol) with benzoyl chloride in carbon disulfide in the presence of aluminium chloride at r.t. for 2 days (16%) [726].

b.p.$_{0.15}$ 148–149° [726]; m.p. 89–90° [735]; ^{1}H NMR [735], IR [735], UV [735]; polarographic study [117]; pK_a [735].

[5-(1,1-Dimethylethyl)-2-hydroxy-4-methoxyphenyl]phenylmethanone

$C_{18}H_{20}O_3$ mol.wt. 284.36



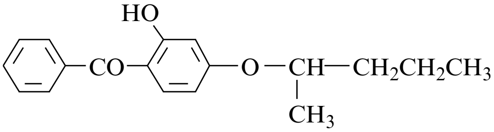
Synthesis

- Preparation by selective methylation of 5-tert-butyl-2,4-di-hydroxybenzophenone with dimethyl sulfate in refluxing methyl ethyl ketone for 7 h in the presence of potassium carbonate (81%) [835].

m.p. 93° [835,836]; Spectra (NA).

[2-Hydroxy-4-(1-methylbutoxy)phenyl]phenylmethanone

$C_{18}H_{20}O_3$ mol.wt. 284.36



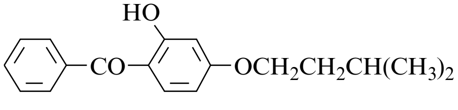
Synthesis

- Obtained by reaction of sec-amyl bromide with resbenzophenone in the presence of potassium hydroxide in dilute ethanol at 80–90° for 16 h (17%) [800].

m.p. 70–72° [800]; Spectra (NA).

[2-Hydroxy-4-(3-methylbutoxy)phenyl]phenylmethanone

[36130-62-8] $C_{18}H_{20}O_3$ mol.wt. 284.36



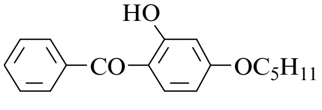
Syntheses

- Preparation by Friedel–Crafts acylation of 1,3-diisoamyloxybenzene with benzoyl chloride in the presence of a zinc chloride/aluminium chloride mixture, first between 0° and 5°, then at 10° for 1 h and at 65° for 6 h [658].
- Preparation by reaction of isoamyl chloride (1-chloro-3-methylbutane) with resbenzophenone (90%) [801].

m.p. 38–40° [801]; Spectra (NA).

[2-Hydroxy-4-(pentyloxy)phenyl]phenylmethanone

[83937-21-7] $C_{18}H_{20}O_3$ mol.wt. 284.36



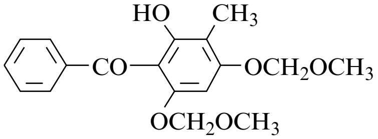
Synthesis

- Preparation by reaction of n-amyl bromide with resbenzophenone,
  - in the presence of potassium hydroxide in dilute ethanol at 80–90° for 18 h (25%) [800];
  - in the presence of potassium carbonate in refluxing acetone for 20 h [738].
- Also refer to: [837] (Japanese patent).

m.p. 49° [800], 45° [738]; Spectra (NA).

[2-Hydroxy-4,6-bis(methoxymethoxy)-3-methylphenyl]phenylmethanone

[74627-90-0] $C_{18}H_{20}O_6$ mol.wt. 332.35



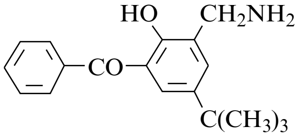
Synthesis

- Preparation by alkylation of 2,4,6-trihydroxy-3-methylbenzophenone with chloromethyl methyl ether in acetone in the presence of potassium carbonate for 1 h (55%) [838].

yellow oil [838]; b.p. (NA); ^{1}H NMR [838], MS [838]; TLC [838].

[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]phenylmethanone

[75060-99-0] $C_{18}H_{21}NO_2$ mol.wt. 283.37



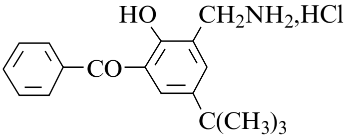
Synthesis

- Refer to: [817]; look at the hydrochloride below.

m.p. and Spectra (NA).

[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]phenylmethanone *(Hydrochloride)*

[75060-64-9] $C_{18}H_{21}NO_2$,HCl mol.wt. 319.83



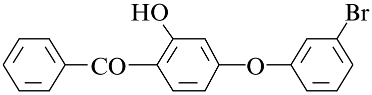
Synthesis

- Preparation by reaction of concentrated hydrochloric acid with 2-benzoyl-4-tert-butyl-6-(N-chloroacetyl-aminomethyl) phenol in refluxing ethanol for 20 h (92%) [817].

m.p. 227–228° [817]; Spectra (NA).

[4-(3-Bromophenoxy)-2-hydroxyphenyl]phenylmethanone

[35698-51-2] $C_{19}H_{13}BrO_3$ mol.wt. 369.21



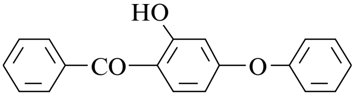
Synthesis

- Refer to: [839] (compound **9**).

m.p. (NA); UV [839].

(2-Hydroxy-4-phenoxyphenyl)phenylmethanone

[35698-39-6] $C_{19}H_{14}O_3$ mol.wt. 290.32



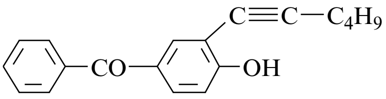
Synthesis

- Preparation by reaction of benzoyl chloride with 3-methoxydiphenyl ether in chlorobenzene in the presence of aluminium chloride, first at r.t., then at 90–95° for 4 h (75%) [839].

m.p. 64–65° [839]; UV [839].

[3-(1-Hexynyl)-4-hydroxyphenyl]phenylmethanone

[183589-20-0] $C_{19}H_{18}O_2$ mol.wt. 278.35



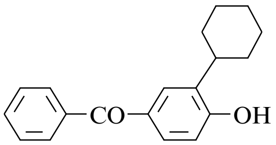
Synthesis

- Obtained by treatment of p-benzoyl-o-(hex-1-ynyl)phenyl acetate in acetone in the presence of 2 N hydrochloric acid at 60° for 20 h (30%) [533].

oil [533]; b.p. (NA); Spectra (NA).

(3-Cyclohexyl-4-hydroxyphenyl)phenylmethanone

[23299-02-7] $C_{19}H_{20}O_2$ mol.wt. 280.37



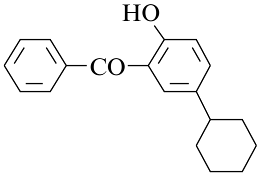
Synthesis

- Preparation by Friedel–Crafts acylation of 2-cyclohexyl-phenol with benzoyl chloride in ethylene dichloride in the presence of titanium tetrachloride, first at 0°, then at r.t. [816].

m.p. 199–200° [816]; Spectra (NA).

(5-Cyclohexyl-2-hydroxyphenyl)phenylmethanone

[3097-56-1] $C_{19}H_{20}O_2$ mol.wt. 280.37



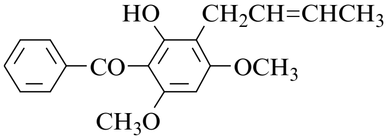
Synthesis

- Preparation by reaction of benzotrichloride with 4-cyclo-hexylphenol in hydrofluoric acid in the presence of water at −10°, then between 0° and −10° for 2 h, at r.t. for 7 h and at 80° for 30 min into an autoclave (50%) [213].

yellow oil [213]; b.p.$_1$ 175–180° [213]; Spectra (NA).

[3-(2-Butenyl)-2-hydroxy-4,6-dimethoxyphenyl]phenylmethanone

[96836-14-5] $C_{19}H_{20}O_4$ mol.wt. 312.37



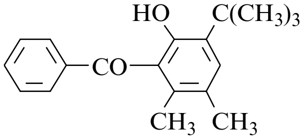
Synthesis

- Preparation by reaction of 2-hydroxy-4,6-di-methoxybenzophenone with 1,3-butadiene in the presence of orthophosphoric acid (70%) [769].

m.p. 93–95° [769]; ^{1}H NMR [769], IR [769].

[3-(1,1-Dimethylethyl)-2-hydroxy-5,6-dimethylphenyl]phenylmethanone

[14963-88-3] $C_{19}H_{22}O_2$ mol.wt. 282.38



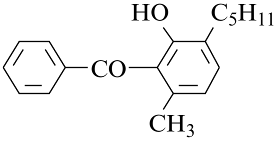
Synthesis

- Preparation by saponification of 2-(benzoyloxy)-3-tert-butyl-5,6-dimethylbenzophenone (SM) with sodium hydroxide in boiling ethanol for 2 h. SM was obtained by oxidation of 7-tert-butyl-4,5-dimethyl-2,3-diphenyl-benzofuran with chromium trioxide in acetic acid at 60° (m.p. 156–157°) [633].
- Also refer to: [813].

m.p. 91–92° [633], 89–90° [735]; ^{1}H NMR [735], IR [813], UV [735]; pK_a [735]; polarographic study [117].

(2-Hydroxy-6-methyl-3-pentylphenyl)phenylmethanone

[26940-71-6] $C_{19}H_{22}O_2$ mol.wt. 282.38



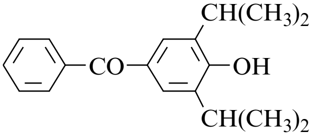
Synthesis

- Refer to: [117,609].

m.p. 74° [735]; ^{1}H NMR [735], IR [735], UV [735]; pK_a [735]; polarographic study [117].

[4-Hydroxy-3,5-bis(1-methylethyl)phenyl]phenylmethanone

[738-15-8] $C_{19}H_{22}O_2$ mol.wt. 282.38



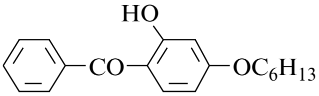
Synthesis

- Obtained by photo-Fries rearrangement of 2,6-di-isopropylphenyl benzoate (SM) in isopropyl alcohol (56%) [64,72], of SM absorbed on a silica gel-pentane (40%) or on dry silica gel (70%) [64].

m.p. 113°5–114° [72] (a phase change occurred at 98°5); Spectra (NA).

[4-(Hexyloxy)-2-hydroxyphenyl]phenylmethanone

[3293-97-8] $C_{19}H_{22}O_3$ mol.wt. 298.38



Synthesis

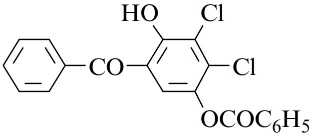
- Obtained by reaction of n-hexyl bromide with resbenzo-phenone,

- in the presence of sodium hydroxide in dilute ethanol in a water bath for 18–23 h (15%) [189];
- in the presence of potassium carbonate in refluxing acetone for 20 h [738].

m.p. 55°5 [189], 52° [738]; UV [243].

[5-(Benzoyloxy)-3,4-dichloro-2-hydroxyphenyl]phenylmethanone

$C_{20}H_{12}Cl_2O_4$ mol.wt. 387.22



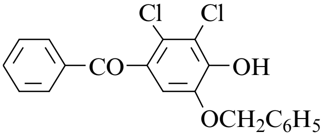
Synthesis

- Obtained by reaction of 2,3-dichloro-1,4-benzoquinone with benzaldehyde in the presence of benzoyl peroxide at 80° or in the absence of benzoyl peroxide at 155° [840].

m.p. and Spectra (NA).

[2,3-Dichloro-4-hydroxy-5-(phenylmethoxy)phenyl]phenylmethanone

[103843-60-3] $C_{20}H_{14}Cl_2O_3$ mol.wt. 373.23



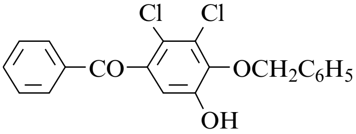
Synthesis

– Preparation by reaction of benzyl bromide with 2,3-di-chloro-4,5-dihydroxybenzophenone in the presence of sodium hydride in N,N-dimethylformamide at r.t. for 10–15 min (77%) [841,842].

m.p. 171–173° [841,842]; ^{1}H NMR [841,842], IR [841,842].

[2,3-Dichloro-5-hydroxy-4-(phenylmethoxy)phenyl]phenylmethanone

[103843-65-8] $C_{20}H_{14}Cl_2O_3$ mol.wt. 373.23



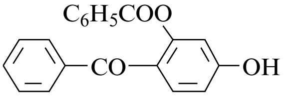
Synthesis

– Obtained by reaction of benzyl bromide with 2,3-di-chloro-4,5-dihydroxybenzophenone in the presence of sodium hydride in N,N-dimethylformamide at 100° for 2 h (34%) [841].
– Also refer to: [842].

m.p. 111–112° [841]; ^{1}H NMR [841], IR [841].

[2-(Benzoyloxy)-4-hydroxyphenyl]phenylmethanone

$C_{20}H_{14}O_4$ mol.wt. 318.33



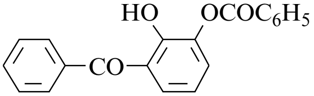
Synthesis

– Preparation from 2,4-dihydroxybenzophenone as starting material via the 2-hydroxy-4-methoxymethoxy-benzophenone and 2-benzoyloxy-4-methoxymethoxy-benzophenone [429].

m.p. and Spectra (NA).

[3-(Benzoyloxy)-2-hydroxyphenyl]phenylmethanone

[97971-72-7] $C_{20}H_{14}O_4$ mol.wt. 318.33



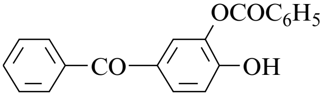
Synthesis

– Obtained by photo-Fries rearrangement of 1,2-di-(benzoyloxy)benzene in benzene for 8 h under nitrogen (15%) [843].

m.p. 74° [843]; ^{1}H NMR [843].

[3-(Benzoyloxy)-4-hydroxyphenyl]phenylmethanone

[76346-15-1] $C_{20}H_{14}O_4$ mol.wt. 318.33



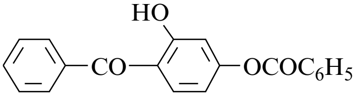
Synthesis

– Obtained (poor yield) by reaction of benzoyl peroxide with p-hydroxybenzophenone [144] in refluxing chloroform for 16 h (9%) [278].

m.p. 109–110° [278]; Spectra (NA); TLC [278].

[4-(Benzoyloxy)-2-hydroxyphenyl]phenylmethanone

[18803-25-3] $C_{20}H_{14}O_4$ mol.wt. 318.33



Syntheses

– Preparation by photo-Fries rearrangement of resorcinol dibenzoate in benzene for 8 h under nitrogen (35%) [843].

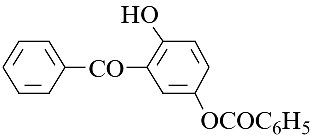
– Also obtained by reaction of benzoyl chloride,

- with resbenzophenone [697], in the presence of sodium hydroxide in refluxing ethanol/ethyl ether mixture for 6 h (22%) [238];
- with resorcinol [197].

m.p. 94°5–95°5 [844], 93°5–94°5 [238], 90° [697], 84° [96], 77–80° [843]; ^{1}H NMR [99], UV [99]; pK_a [96]; TLC [116].

[5-(Benzoyloxy)-2-hydroxyphenyl]phenylmethanone

[97971-74-9] $C_{20}H_{14}O_4$ mol.wt. 318.33



Syntheses

– Obtained by Fries rearrangement,

- of quinol monobenzoate in the presence of aluminium chloride (3.3 mol) at 140° for 1 h (14%) [252];
- of quinol dibenzoate in the presence of aluminium chloride (2 mol) at 190–200° for 1.5 h [253] or (3.3 mol or 5.5 mol of catalyst) at 140° for 1 h (33%) [252];
- of p-methoxyphenyl benzoate in the presence of aluminium chloride (3.3 mol) at 130–132° for 1 h or at 150–155° for 1 h (<9%) [845].

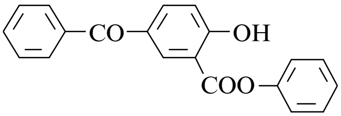
– Preparation by photo-Fries rearrangement of quinol dibenzoate in benzene for 8 h under nitrogen (30%) [843].

m.p. 213–214° [843], 96° [253], 94–95° [252]. There is a discrepancy between the different melting points indicated in literature. 94–96° are more likely, due to chelation.

^{1}H NMR [843].

Phenyl 5-benzoyl-2-hydroxybenzoate ***5-Benzoyl-2-hydroxybenzoic acid phenyl ester***

[124208-60-2] $C_{20}H_{14}O_4$ mol.wt. 318.33



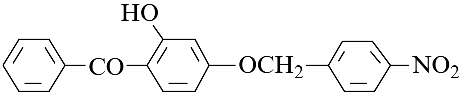
Synthesis

– Obtained by Fries rearrangement of phenyl 2-benzoyl-oxybenzoate with aluminium chloride without solvent at 142–145° for 2 h (31%) or in nitrobenzene as a solvent at r.t. for 2 days (17%) [155].

m.p. 82–84° [155]; Spectra (NA).

[2-Hydroxy-4-[(4-nitrophenyl)methoxy]phenyl]phenylmethanone

[36419-36-0] $C_{20}H_{15}NO_5$ mol.wt. 349.34



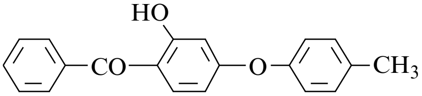
Synthesis

– Refer to: [235].

m.p. 172° [235]; UV [235].

[2-Hydroxy-4-(methylphenoxy)phenyl]phenylmethanone

[35698-46-5] $C_{20}H_{16}O_3$ mol.wt. 304.35



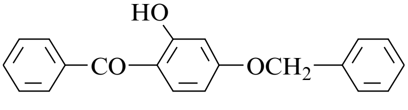
Synthesis

– Preparation by reaction of benzotrichloride with 3-hydroxy-4′-methyldiphenyl ether in solution of 2.5 N sodium hydroxide in the presence of potassium iodide at 80° [839].

m.p. 82–85° [839]; UV [839].

[2-Hydroxy-4-(phenylmethoxy)phenyl]phenylmethanone

[6079-76-1] $C_{20}H_{16}O_3$ mol.wt. 304.35



Synthesis

– Preparation by reaction of benzyl chloride with 2,4-dihydroxybenzophenone (resbenzophenone),

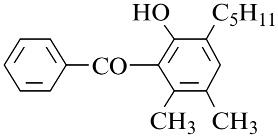
- in the presence of potassium carbonate in refluxing acetone [738], (34%) [846];
- in the presence of potassium carbonate and potassium iodide in N,N-dimethylformamide at 100° for 1.5 h (95%) [801].

– Also refer to: [228,847,848].

m.p. 121–122° [801], 120–121° [738,846,849]; Spectra (NA).

(2-Hydroxy-5,6-dimethyl-3-pentylphenyl)phenylmethanone

[26881-03-8] $C_{20}H_{24}O_2$ mol.wt. 296.41



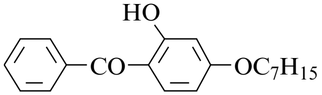
Synthesis

– Refer to: [117,609].

m.p. 85° [735]; ^{1}H NMR [735], IR [735], UV [735]; pK_a [735]; polarographic study [117].

[4-(Heptyloxy)-2-hydroxyphenyl]phenylmethanone *(Uvistat 247)*

[3550-43-4] $C_{20}H_{24}O_3$ mol.wt. 312.41



Synthesis

– Obtained by reaction of n-heptyl bromide with resbenzophenone,

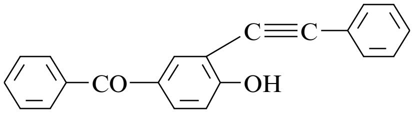
- in the presence of potassium hydroxide in dilute ethanol at 85–95° for 22 h (13%) [800];
- in the presence of potassium carbonate in refluxing acetone for 20 h [738].

– Also refer to: [850–853].

m.p. 40° [738,800]; Spectra (NA); gel permeation chromatography [247].

[4-Hydroxy-3-(phenylethynyl)phenyl]phenylmethanone

[183589-17-5] $C_{21}H_{14}O_2$ mol.wt. 298.34



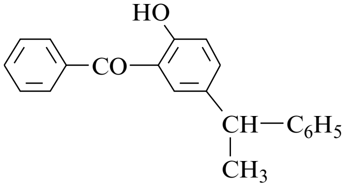
Synthesis

– Obtained by treatment of p-benzoyl-o-(phenyl-ethynyl)phenyl acetate in acetone in the presence of 2 N hydrochloric acid at 60° for 16 h (40%) [533].

m.p. 139–140° [533]; ^{1}H NMR [533], ^{13}C NMR [533], IR [533], MS [533].

[2-Hydroxy-5-(1-phenylethyl)phenyl]phenylmethanone

[125182-23-2] $C_{21}H_{18}O_2$ mol.wt. 302.37

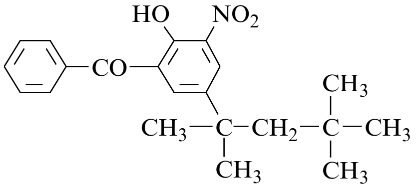


Synthesis

– Preparation by treatment of o-hydroxybenzophenone at 120° with styrene under nitrogen in the presence of a macroreticular acid ion exchanger as catalyst (Wofatit OK 80) for 1–2 h (90%) [819].

b.p.$_{0.15}$ 200–210° [819]; Spectra (NA).

[2-Hydroxy-3-nitro-5-(1,1,3,3-tetramethylbutyl)phenyl]phenylmethanone



$C_{21}H_{25}NO_4$ mol.wt. 355.44

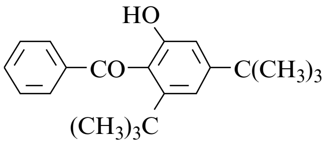
Synthesis

- Obtained by reaction of 65% nitric acid with 2-hydroxy-5-tert-octylbenzophenone in acetic acid at 20° [854].

oil [854]; b.p. and Spectra (NA).

[2,4-Bis(1,1-dimethylethyl)-6-hydroxyphenyl]phenylmethanone

[13113-73-0]



$C_{21}H_{26}O_2$ mol.wt. 310.44

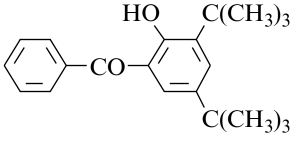
Synthesis

- Obtained by photo-Fries rearrangement of 3,5-di-tert-butylphenyl benzoate,
  - in benzene [105], (29%) [855]; after 27.5 h, it had decreased to 8% [855];
  - in ethanol for 3 h (48%) [818];
  - in isopropanol for 3 h (37%) and 52% after 6 h; after 17 h, it had decreased to 16% [818];
  - in a mixture of ethanol/ethyl ether (9:1) for 3 h (34%) [818];
  - in N,N-dimethylformamide for 3 h (9%); (16%) after 1 h [818];
  - in n-hexane and tetrahydrofuran for 3 h (8% and 12% yields, respectively) [818];
  - in dioxane and glyme (1,2-dimethoxyethane) for 3 h (2% and 5% yields, respectively) [818].
- Also refer to: [79].

m.p. 202°5 [855]; ^{1}H NMR [105], IR [105].

[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]phenylmethanone

[24242-58-8]



$C_{21}H_{26}O_2$ mol.wt. 310.44

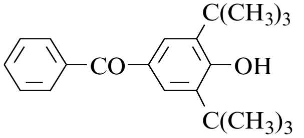
Syntheses

- Obtained from (3,5-di-tert-butyl-2-hydroxyphenyl)-phenylcarbinol by oxidation with DDQ (2,3-dichloro-5,6-dicyanobenzoquinone) in dioxane at r.t. for 16 h (71%) [153].
- Also obtained by photo-Fries rearrangement of 2,4-di-tert-butylphenyl benzoate in benzene under nitrogen (32%) [143].
- Preparation by treatment of o-hydroxybenzophenone at 120° with a mixture of isobutylene/nitrogen (1:1) or with tert-butyl chloride in the presence of a macroreticular acid ion exchanger as catalyst (Wofatit OK 80) for 10 h (80%) [819].

b.p.$_{0.15}$ 140–145° [819]; m.p. 61–62° [143], 60–62° [153];
^{1}H NMR [143], IR [143].

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]phenylmethanone

[7175-89-5] $C_{21}H_{26}O_2$ mol.wt. 310.44



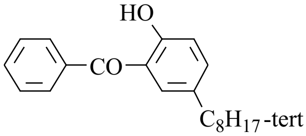
Syntheses

- Preparation by reaction of benzoyl chloride with 2,6-di-tert-butylphenol in the presence of aluminium chloride [856], (93%) [857], 70% [858], (50–55%) [859,860].
- Preparation by reaction of benzoic acid with 2,6-di-tert-butylphenol in the presence of trifluoroacetic anhydride at r.t. [861], for 3 h (65%) [862].
- Preparation by oxidation of (3,5-di-tert-butyl-4-hydroxyphenyl)phenylcarbinol with 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ) in dioxane at r.t. for 15 min (95%) [153].
- Preparation by reaction of benzoyl chloride with sodium 2,6-di-tert-butylphenoxide (SM) in dioxane between 60° and 80° (24%) [863]. SM was obtained by reaction of sodium with 2,6-di-tert-butylphenol in methanol, then solvent elimination at 80°.
- Also obtained by basic hydrolysis of α(3,5-di-tert-butyl-4-hydroxyphenyl)benzyl benzoate with potassium hydroxide in refluxing dilute ethanol for 30 min (90%) [863].
- Also refer to: [143].

m.p. 132–134° [861,862], 125°5–126°5 [858], 125–126° [863], 124–125° [859,860], 123–124° [857]; ^{1}H NMR [858,862], IR [858,860].

[2-Hydroxy-5-(1,1,3,3-tetramethylbutyl)phenyl]phenylmethanone

[4090-99-7] $C_{21}H_{26}O_2$ mol.wt. 310.44



Syntheses

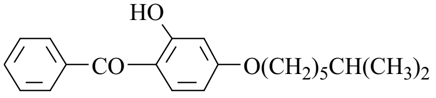
- Preparation by photo-Fries rearrangement of p-tert-octyl-phenyl benzoate (45%) [854] according to [154].
- Preparation by reaction of benzotrichloride with p-tert-octylphenol in the presence of 30% aqueous sodium hydroxide at 75–80° for 30 min, and hydrolysis of the resulting ester as a side product (61%) [615], (44%) [97]. The same reaction was carried out in the presence of sodium iodide, during 2.5 h at 80° (39%) [864].
- Preparation by reaction between (p-octylphenoxy)magnesium bromide complexed with HMPT and benzaldehyde in refluxing benzene for 48 h (36%) [50].

yellow oil [50]; m.p. 34–35° [615]; b.p.$_{2.5}$ 176–179° [97], b.p.$_1$ 140° [864]; ^{1}H NMR [50,97,615], ^{13}C NMR [97], IR [50,97,615], UV [615,864], MS [50,615]; gas chromatography study [631].

[2-Hydroxy-4-(isooctyloxy)phenyl]phenylmethanone

[33059-05-1]

$C_{21}H_{26}O_3$ mol.wt. 326.44



Synthesis

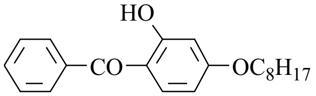
– Preparation by reaction of isooctyl chloride with resbenzophenone in the presence of sodium carbonate and sodium iodide in acetone for 4 h at 150° (97%) [865].

m.p. and Spectra (NA).

[2-Hydroxy-4-(octyloxy)phenyl]phenylmethanone *(Octabenzone, Cyasorb UV 531)*

[1843-05-6]

$C_{21}H_{26}O_3$ mol.wt. 326.44



Syntheses

– Preparation by reaction of n-octyl chloride with 2,4-di-hydroxybenzophenone,

  - in the presence of a mixture of sodium carbonate, triethylamine and potassium iodide in refluxing butanol for 15 h (90%) [866];
  - in the presence of potassium carbonate in cyclohexanone at 145° for 5 h (66%) [201,202];
  - in the presence of potassium hydroxide and antimony triiodide in diethylene glycol at 150° for 1 h (93%) [801];
  - in the presence of sodium bicarbonate and potassium iodide in 1-methylpyrrolidone for 2 h at 150° (96%) [867].

– Preparation by reaction of n-octyl bromide with 2,4-dihydroxybenzophenone,

  - in the presence of potassium carbonate,

    in water at 110–115° for 4 h (84%) [868];
    in cyclohexanone at 110° for 6 h (75%) [202];
    in methyl isobutyl ketone at 110° for 10 h (70%) [202];
    in methyl n-hexyl ketone at 161° for 4 h (71%) [202];
    in acetone at 58° for 20 h (54%) [202] or in refluxing acetone for 16 h [869].

  - in the presence of sodium hydroxide,

    in dilute ethanol in a water bath for 18–23 h (13–15%) [189];
    in cyclohexanone at 145° for 5 h (51%) [202].

– Preparation by alkylation of resbenzophenone with an octyl halide [651].

– Preparation by reaction of n-octyl p-toluenesulfonate with 2,4-dihydroxybenzophenone,

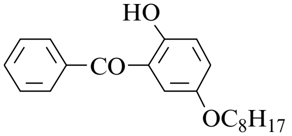
  - in the presence of potassium carbonate in boiling water for 5 h (74%) [201,202];
  - in the presence of sodium carbonate in boiling water for 8 h (63%) [202];
  - in the presence of potassium hydroxide in boiling water for 8 h (61%) [202].

- Preparation by reaction of n-octyl benzenesulfonate with 2,4-dihydroxybenzophenone in the presence of potassium carbonate in boiling water for 8 h (66%) [202].
- Preparation by reaction of benzoyl chloride with resorcinol dioctyl ether in chlorobenzene in the presence of titanium tetrachloride for 1 h at 120° (69%) [662].
- Also refer to: [77,224,226,228,231,235,667–669,802,837,852,870–880].

m.p. 50° [235], 48–50° [801], 48–49° [662], 47–48° [201,202], 45–46° [189,869,881]; ^{1}H NMR [99], IR [201,202], UV [99,201,202,235,243]; TLC [116,244]; gel permeation chromatography [247].

[2-Hydroxy-5-(octyloxy)phenyl]phenylmethanone

[4998-51-0] $C_{21}H_{26}O_3$ mol.wt. 326.44



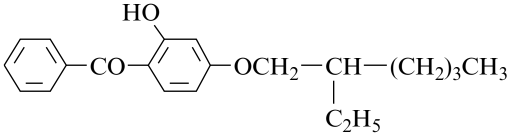
Synthesis

- Obtained (poor yield) by reaction of n-octyl bromide with 2,5-dihydroxybenzophenone in the presence of sodium hydroxide in dilute ethanol at 90–95° for 50 h (7%) [681].

m.p. 26–27° [681]; Spectra (NA).

[4-[(2-Ethylhexyl)oxy]-2-hydroxyphenyl]phenylmethanone *(Dastib 242)*

[2549-90-8] $C_{21}H_{26}O_3$ mol.wt. 326.44



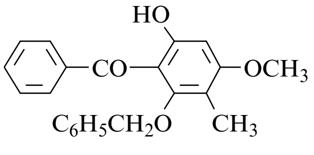
Synthesis

- Preparation by reaction of 1-chloro-2-ethylhexane with resbenzophenone in the presence of barium hydroxide and arsenic tribromide in dimethyl sulfoxide at 80° for 3 h (93%) [801].

b.p.$_1$ 213–216° [801]; ^{1}H NMR [99], UV [99]; gel permeation chromatography [246,247].

[6-Hydroxy-4-methoxy-3-methyl-2-(phenylmethoxy)phenyl]phenylmethanone

[74627-93-3] $C_{22}H_{20}O_4$ mol.wt. 348.40



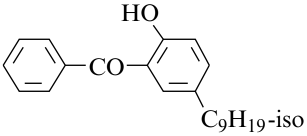
Synthesis

- Preparation by partial methylation of 2-benzyloxy-3-methyl-4,6-dihydroxybenzophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 10 min (96%) [838].

colourless oil [838]; b.p. (NA); ^{1}H NMR [838]; TLC [838].

(2-Hydroxy-5-isononylphenyl)phenylmethanone

[59802-03-8] $C_{22}H_{28}O_2$ mol.wt. 324.46



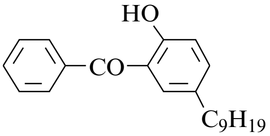
Synthesis

- Preparation by reaction of benzotrichloride with 4-iso-nonylphenol in hydrofluoric acid in the presence of water at −10°, then between −10° and 0° for 2 h, at r.t. for 7 h and at 80° for 30 min into an autoclave [213].

yellow oil [213]; b.p.$_1$ 190° [213]; Spectra (NA).

(2-Hydroxy-5-nonylphenyl)phenylmethanone

[58085-73-7] $C_{22}H_{28}O_2$ mol.wt. 324.46



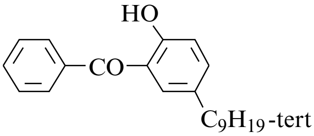
Syntheses

- Preparation by Fries rearrangement of p-nonylphenyl benzoate with aluminium chloride [882], without solvent at 140–145° for 30–50 min (52%) [147].
- Preparation by Friedel–Crafts acylation of p-nonylphenol with benzoyl chloride in the presence of aluminium chloride in ethylene dichloride at 160° for 30–60 min (37%) [147].
- Preparation by reaction of benzotrichloride with p-nonylphenol in the presence of 30% aqueous sodium hydroxide (60–70%) [883], at 75–80° for 30 min, and hydrolysis of the ester formed as a side product (42%) [97].
- Also refer to: [884,885].

m.p. 35–36° [97]; b.p.$_5$ 205–208° [97]; TLC [886], GC [886], HPLC [886].
^{1}H NMR [97,147], ^{13}C NMR [97], IR [97,147].

(2-Hydroxy-5-tert-nonylphenyl)phenylmethanone

[111547-84-3] $C_{22}H_{28}O_2$ mol.wt. 324.46



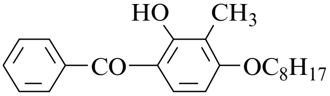
Synthesis

- Refer to: [631].

m.p. and Spectra (NA); gas chromatography study [631].

[2-Hydroxy-3-methyl-4-(octyloxy)phenyl]phenylmethanone

[52220-72-1]



$C_{22}H_{28}O_3$ mol.wt. 340.46

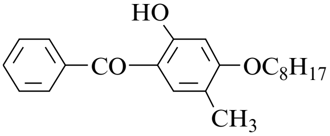
Synthesis

- Preparation by partial alkylation of 2,4-dihydroxy-3-methylbenzophenone with octyl chloride in the presence of sodium bicarbonate and potassium iodide in 1-methyl-pyrrolidone for 2 h at 150° (92%) [867].

m.p. and Spectra (NA).

[2-Hydroxy-5-methyl-4-(octyloxy)phenyl]phenylmethanone

[52220-73-2]



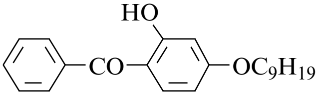
$C_{22}H_{28}O_3$ mol.wt. 340.46

Synthesis

- Preparation by partial alkylation of 2,4-dihydroxy-5-methylbenzophenone with octyl chloride in the presence of sodium bicarbonate and potassium iodide in 1-methyl-pyrrolidone for 2 h at 150° (91%) [867] or in refluxing methyl cellosolve [887].

m.p. and Spectra (NA).

[2-Hydroxy-4-(nonyloxy)phenyl]phenylmethanone



$C_{22}H_{28}O_3$ mol.wt. 340.46

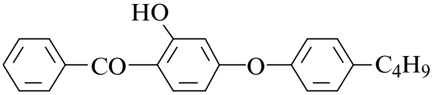
Synthesis

- Obtained by reaction of n-nonyl bromide with resbenzo-phenone in the presence of sodium hydroxide in dilute ethanol in a water bath for 18–23 h (28–30%) [189].
- Also refer to: [852].

m.p. 50°5 [189]; Spectra (NA).

[4-(4-Butylphenoxy)-2-hydroxyphenyl]phenylmethanone

[35698-49-8]



$C_{23}H_{22}O_3$ mol.wt. 346.94

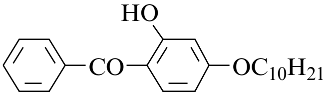
Synthesis

- Refer to: [839] (compound **7**).

m.p. (NA); UV [839].

[4-(Decyloxy)-2-hydroxyphenyl]phenylmethanone

[2162-63-2] $C_{23}H_{30}O_3$ mol.wt. 354.49



Synthesis

– Obtained by reaction of n-decyl bromide with resbenzo-phenone,

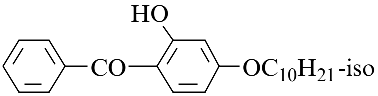
- in the presence of sodium hydroxide in dilute ethanol at 110–115° for 15 h (18%) [189];
- in the presence of potassium carbonate in refluxing acetone for 20 h [738].

– Also refer to: [226,228].

m.p. 50° [189], 47–50° [738]; IR [394], UV [394].

[2-Hydroxy-4-(isodecyloxy)phenyl]phenylmethanone

[55909-78-9] $C_{23}H_{30}O_3$ mol.wt. 354.49



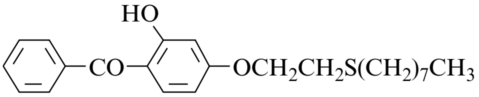
Synthesis

– Refer to: [247].

m.p. and Spectra (NA); gel permeation chromatography [247].

[2-Hydroxy-4-[2-(octylthio)ethoxy]phenyl]phenylmethanone

[36130-66-2] $C_{23}H_{30}O_3S$ mol.wt. 386.56



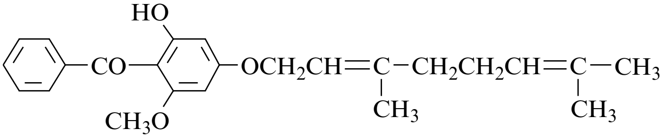
Synthesis

– Preparation by reaction of 2-chloroethyl octyl sulfide (1-(2-chloroethylsulfanyl)-octane) with resbenzophenone (82%), according to [801].

b.p.$_{0.2}$ 253–258° [801]; Spectra (NA).

[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2-hydroxy-6-methoxyphenyl] phenyl-methanone *(E)*

[140158-57-2] $C_{24}H_{28}O_4$ mol.wt. 380.48



Synthesis

– Not yet described.

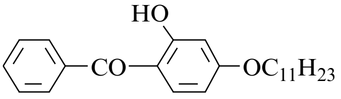
Isolation from natural sources

– From *Helichrysum triplinerve* (Asteraceae) [377];

– From *genus Leontonyx* [378].

m.p. (NA); ^{1}H NMR [377], MS [377].

[2-Hydroxy-4-(undecyloxy)phenyl]phenylmethanone



$\mathbf{C_{24}H_{32}O_3}$ mol.wt. 368.52

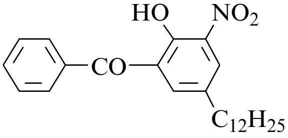
Synthesis

- Refer to: [888].

m.p. and Spectra (NA).

(5-Dodecyl-2-hydroxy-3-nitrophenyl)phenylmethanone

[35698-17-0]



$C_{25}H_{33}NO_4$ mol.wt. 411.54

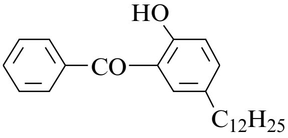
Synthesis

- Preparation by reaction of fuming nitric acid with 5-dodecyl-2-hydroxybenzophenone in acetic acid/acetic anhydride solution for 40 min at 15–22° (92%) [889–891].

b.p.$_{0.8}$ 210° [889,890,891]; Spectra (NA).

(5-Dodecyl-2-hydroxyphenyl)phenylmethanone

[35698-16-9]



$C_{25}H_{34}O_2$ mol.wt. 366.54

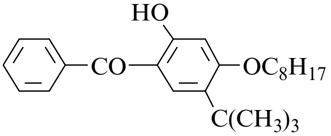
Synthesis

- Preparation by reaction of benzenyl trichloride (benzo-trichloride) with p-dodecylphenol in carbon disulfide inthe presence of aluminium chloride, first between −20° and −15°, then at 0–5° for 1 h (38%) [889–891].

m.p. and Spectra (NA).

[5-(1,1-Dimethylethyl)-2-hydroxy-4-(octyloxy)phenyl]phenylmethanone

[55913-02-5]



$C_{25}H_{34}O_3$ mol.wt. 382.54

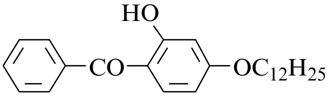
Synthesis

- Refer to: [247].

m.p. and Spectra (NA); gel permeation chromatography [247].

[4-(Dodecyloxy)-2-hydroxyphenyl]phenylmethanone

[2985-59-3]



$C_{25}H_{34}O_3$ mol.wt. 382.54

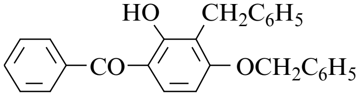
Syntheses

- Preparation by reaction of 1-bromododecane (dodecyl bromide or lauryl bromide) with resbenzophenone in the presence of potassium carbonate in refluxing acetone for 16 h [869] or for 20 h [738].

- Preparation by alkylation of resbenzophenone with a dodecyl halide [651].
- Also refer to: [78,228,667,829,892–894].

m.p. 49–50° [869] 48° [738]; EPR [98], IR [394], UV [394];
TLC [116]; gel permeation chromatography [247].

[2-Hydroxy-4-(phenylmethoxy)-3-(phenylmethyl)phenyl]phenylmethanone



$C_{27}H_{22}O_3$ mol.wt. 394.47

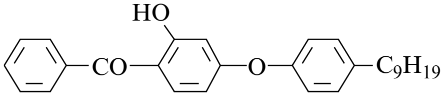
Synthesis

- Preparation by reaction of benzyl chloride with 3-benzyl-2,4-dihydroxybenzophenone in the presence of potassium carbonate in refluxing acetone for 8 h (31%) [846].

m.p. 92–93° [846]; Spectra (NA).

[2-Hydroxy-4-(4-nonylphenoxy)phenyl]phenylmethanone

[35698-50-1]



$C_{28}H_{32}O_3$ mol.wt. 416.56

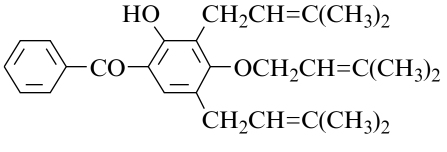
Synthesis

- Refer to: [839] (compound **8**).

m.p. (NA); UV [839].

[2-Hydroxy-3,5-bis(3-methyl-2-butenyl)-4-[(3-methyl-2-butenyl)oxy]phenyl]-phenylmethanone

[63565-03-7]



$C_{28}H_{34}O_3$ mol.wt. 418.58

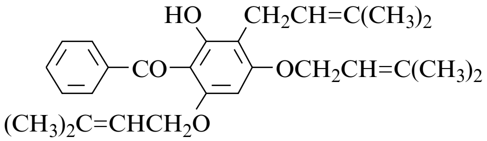
Synthesis

- Obtained (poor yield) by reaction of prenyl bromide with resbenzophenone in the presence of sodium methoxide in refluxing methanol for 4 h (<2%) [831].

m.p. 98° [831]; ^{1}H NMR [831], IR [831], UV [831].

[2-Hydroxy-3-(3-methyl-2-butenyl)-4,6-bis[(3-methyl-2-butenyl)oxy] phenyl]phenylmethanone

[63565-08-2]



$C_{28}H_{34}O_4$ mol.wt. 434.58

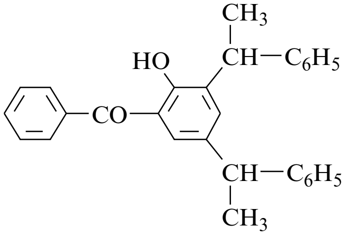
Synthesis

- Obtained by reaction of prenyl bromide with 2,4,6-trihydroxybenzophenone in the presence of potassium carbonate in refluxing acetone for 4 h (21%) [831].

m.p. 112° [831]; ^{1}H NMR [831], IR [831], UV [831].

[2-Hydroxy-3,5-bis(1-phenylethyl)phenyl]phenylmethanone

[125182-24-3]



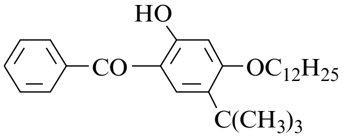
$C_{29}H_{26}O_2$ mol.wt. 406.52

Synthesis

- Preparation by treatment of o-hydroxybenzophenone at 120° with styrene under nitrogen in the presence of a macroreticular acid ion exchanger as catalyst(Wofatit OK 80) for 3–4 h (80%) [819].

b.p.$_{0.15}$ 220–225° [819]; Spectra (NA).

[5-(1,1-Dimethylethyl)-4-(dodecyloxy)-2-hydroxyphenyl]phenylmethanone



$C_{29}H_{42}O_3$ mol.wt. 438.65

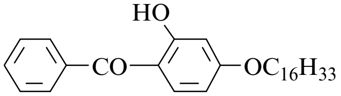
Synthesis

- Preparation by selective alkylation of 5-tert-butyl-2,4-dihydroxybenzophenone with dodecyl bromide in refluxing methyl ethyl ketone in the presence of potassium carbonate [835].

m.p. 74°5–75°5 [835,836]; Spectra (NA).

[4-(Hexadecyloxy)-2-hydroxyphenyl]phenylmethanone

[3457-17-8]



$C_{29}H_{42}O_3$ mol.wt. 438.65

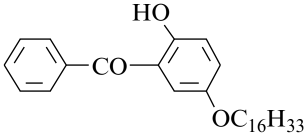
Synthesis

- Obtained by partial alkylation of resbenzophenone with hexadecyl bromide (cetyl bromide) in the presence of potassium carbonate in refluxing acetone for 20 h [738].
- Also refer to: [405] and [895] (Japanese patent).

m.p. 50° [738]; Spectra (NA).

[5-(Hexadecyloxy)-2-hydroxyphenyl]phenylmethanone

[131664-12-5]



$C_{29}H_{42}O_3$ mol.wt. 438.65

Synthesis

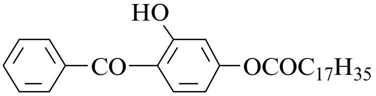
- Refer to: [896] (Japanese patent).

m.p. and Spectra (NA).

[2-Hydroxy-4-(octadecanoyloxy)phenyl]phenylmethanone

Octadecanoic acid, 4-benzoyl-3-hydroxyphenyl ester

[65953-50-6] $C_{31}H_{44}O_4$ mol.wt. 480.69



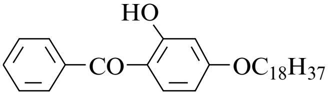
Synthesis

- Refer to: [243].

m.p. (NA); UV [243].

[2-Hydroxy-4-(octadecyloxy)phenyl]phenylmethanone

[3457-13-4] $C_{31}H_{46}O_3$ mol.wt. 466.70



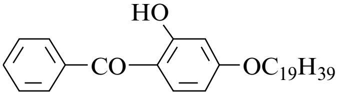
Syntheses

- Preparation by reaction of stearyl chloride (octadecyl chloride or 1-chlorooctadecane) with resbenzophenone in the presence of sodium hydroxide and phosphorous triiodide in aqueous diethylene glycol at 175° for 1.5 h (94%) [801].
- Preparation by alkylation of resbenzophenone with an octadecyl halide [651].
- Also refer to: [667].

m.p. 55–56° [801]; Spectra (NA); TLC [116].

[2-Hydroxy-4-(nonadecyloxy)phenyl]phenylmethanone

$C_{32}H_{48}O_3$ mol.wt. 480.73



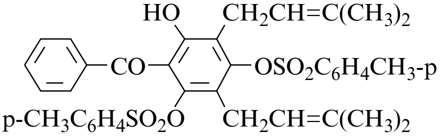
Synthesis

- Obtained by partial alkylation of resbenzophenone with nonadecyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [738].

m.p. 53° [738]; Spectra (NA).

[2-Hydroxy-3,5-bis(3-methyl-2-butenyl)-4,6-bis[[(4-methylphenyl)sulfonyl]oxy]phenyl]phenylmethanone

[83611-03-4] $C_{37}H_{38}O_8S_2$ mol.wt. 674.84



Synthesis

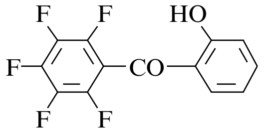
- Obtained by ditosylation of 2,4,6-trihydroxy-3,5-diprenylbenzophenone with p-toluenesulfonyl chloride in the presence of potassium carbonate in refluxing acetone for 8 h (39%) [373].

m.p. 57° [373]; ^{1}H NMR [373].

2.1.2 Substituents Located on the Other Ring

(2-Hydroxyphenyl)(2,3,4,5,6-pentafluorophenyl)methanone

[32541-24-5] $C_{13}H_5F_5O_2$ mol.wt. 288.17



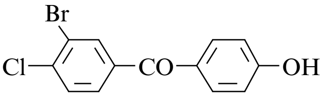
Synthesis

– Preparation by demethylation of 2,3,4,5,6-pentafluoro-2′-methoxybenzophenone (SM) in methylene chloride in the presence of aluminium chloride at 20° for 3–6 h (68%) [570]. SM was obtained in two steps: first, preparation of 2′-methoxy-2,3,4,5,6-pentafluorobenzhydrol by condensation of o-methoxybenzaldehyde with pentafluorophenylmagnesium bromide in boiling ethyl ether for 2 h (96%). Then, this “benzhydrol” was oxidized with chromium trioxide in acetic acid at 20° for 20 h (95%) [570].

m.p. 78–79° [570]; ^{1}H NMR [570], IR [570], UV [570].

(3-Bromo-4-chlorophenyl)(4-hydroxyphenyl)methanone

[78930-23-1] $C_{13}H_8BrClO_2$ mol.wt. 311.56



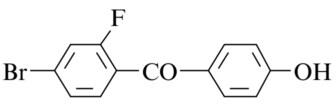
Synthesis

– Preparation by reaction of 3-bromo-4-chlorobenzoyl chloride with phenol in the presence of aluminium chloride [165].

m.p. 193–194° [165]; Spectra (NA).

(4-Bromo-2-fluorophenyl)(4-hydroxyphenyl)methanone

[192443-11-1] $C_{13}H_8BrFO_2$ mol.wt. 295.11



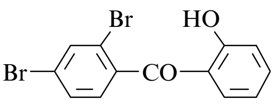
Synthesis

– Preparation by demethylation of 4-bromo-2-fluoro-4′-methoxybenzophenone (SM) with 62% aqueous hydrobromic acid in refluxing acetic acid (98%). SM was obtained by Friedel–Crafts acylation of anisole with 4-bromo-2-fluorobenzoyl chloride in nitrobenzene in the presence of aluminium chloride, first at temperature <6°, then at r.t. overnight (88%, m.p. 93–94°). -Refer to: Chem. Abstr., **127**, 108921f (1997)^T.

m.p. (NA); MS^T.

(2,4-Dibromophenyl)(2-hydroxyphenyl)methanone

$C_{13}H_8Br_2O_2$ mol.wt. 356.01



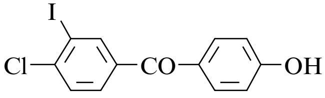
Synthesis

– Refer to: [87].

m.p. and Spectra (NA).

(4-Chloro-3-iodophenyl)(4-hydroxyphenyl)methanone

[83888-75-9] $C_{13}H_8ClIO_2$ mol.wt. 358.56



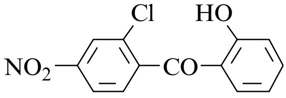
Synthesis

– Preparation by reaction of 4-chloro-3-iodobenzoyl chloride with phenol in the presence of aluminium chloride [165].

m.p. 191–192° [165]; Spectra (NA).

(2-Chloro-4-nitrophenyl)(2-hydroxyphenyl)methanone

[72090-64-3] $C_{13}H_8ClNO_4$ mol.wt. 277.66



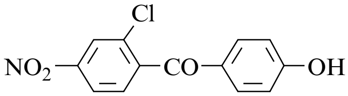
Synthesis

– Obtained by Fries rearrangement of phenyl 2-chloro-4-nitro-benzoate with aluminium chloride without solvent at 120° or at 160° [897].

m.p. and Spectra (NA).

(2-Chloro-4-nitrophenyl)(4-hydroxyphenyl)methanone

[72103-42-5] $C_{13}H_8ClNO_4$ mol.wt. 277.66



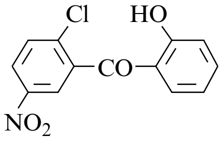
Synthesis

– Obtained by Fries rearrangement of phenyl 2-chloro-4-nitrobenzoate with aluminium chloride without solvent at 120° or at 160° [897].

m.p. and Spectra (NA).

(2-Chloro-5-nitrophenyl)(2-hydroxyphenyl)methanone

[72090-65-4] $C_{13}H_8ClNO_4$ mol.wt. 277.66



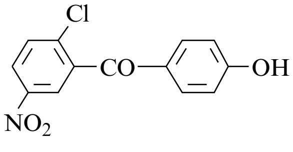
Synthesis

– Obtained by Fries rearrangement of phenyl 2-chloro-5-nitro-benzoate with aluminium chloride without solvent at 120° or at 160° [897].

m.p. and Spectra (NA).

(2-Chloro-5-nitrophenyl)(4-hydroxyphenyl)methanone

[72090-66-5] $C_{13}H_8ClNO_4$ mol.wt. 277.66



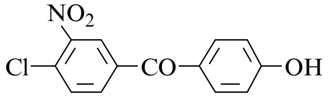
Synthesis

– Obtained by Fries rearrangement of phenyl 2-chloro-5-nitro-benzoate with aluminium chloride without solvent at 120° or at 160° [897].

m.p. and Spectra (NA).

(4-Chloro-3-nitrophenyl)(4-hydroxyphenyl)methanone

[93958-85-1] $C_{13}H_8ClNO_4$ mol.wt. 277.66



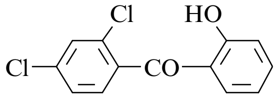
Synthesis

– Preparation by demethylation of 4-chloro-4′-methoxy-3-nitrobenzophenone with pyridinium chloride at 220–230° for 30 min (55%) [898].

m.p. 185° [898]; IR [898].

(2,4-Dichlorophenyl)(2-hydroxyphenyl)methanone

[46795-43-1] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



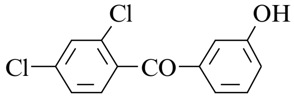
Synthesis

– Refer to: [87] (compound **VI-A**).

m.p. and Spectra (NA).

(2,4-Dichlorophenyl)(3-hydroxyphenyl)methanone

[62810-56-4] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



Syntheses

– Preparation by reaction of m-anisoyl chloride with m-di-chlorobenzene in the presence of excess aluminium chloride first at 20°, then at reflux for 2 h (71%) [119,899].

– Preparation by demethylation of 2,4-dichloro-3′-methoxybenzophenone with aluminium chloride in refluxing chlorobenzene [900].

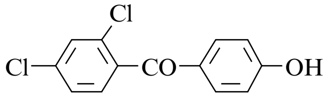
– Also obtained by diazotization of 3′-amino-2,4-dichlorobenzophenone followed by hydrolysis of the diazonium salt so obtained [900].

m.p. 135° [119,899], 70° [900]. One of the reported melting points is obviously wrong.

Spectra (NA).

(2,4-Dichlorophenyl)(4-hydroxyphenyl)methanone

[34183-01-2] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



Syntheses

– Preparation by reaction of 2,4-dichlorobenzoyl chloride with phenol in the presence of aluminium chloride [165].

– Preparation by reaction of 2,4-dichlorobenzotrichloride with phenol,

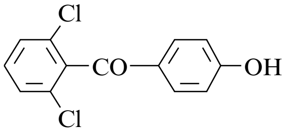
- in hydrofluoric acid in the presence of water at –10°, then at 15° overnight and at 80° for 30 min [213];
- in methylene chloride in the presence of aluminium chloride, first at 0–3° for 30 min, then at 20° for 3 h (52%) [901].

- Preparation by Fries rearrangement of phenyl 2,4-dichlorobenzoate with aluminium chloride in chlorobenzene at 140–150° for 20 min or in nitrobenzene at 75° for 24 h [480].

m.p. 140–141° [480], 134–135° [165], 132–135°5 [901]; Spectra (NA).

(2,6-Dichlorophenyl)(4-hydroxyphenyl)methanone

[61002-53-7] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



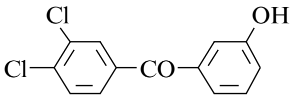
Synthesis

- Refer to: [902].

m.p. and Spectra (NA).

(3,4-Dichlorophenyl)(3-hydroxyphenyl)methanone

[62810-54-2] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



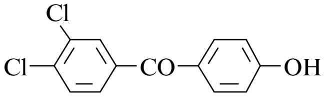
Syntheses

- Preparation by reaction of m-anisoyl chloride with o-di-chlorobenzene in the presence of excess aluminium chloride first at 20°, then at reflux for 2 h (41%) [119,899].
- Preparation by demethylation of 3,4-dichloro-3′-methoxybenzophenone with aluminium chloride in refluxing chlorobenzene [900].

m.p. 140° [900], 130° [119,899]; Spectra (NA).

(3,4-Dichlorophenyl)(4-hydroxyphenyl)methanone

[60013-02-7] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



Syntheses

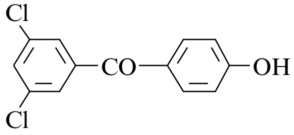
- Preparation by reaction of 3,4-dichlorobenzoyl chloride,
  - with phenol in the presence of aluminium chloride [165];
  - with phenetole in the presence of aluminium chloride in carbon disulfide at r.t. overnight (40%) [903];
  - with anisole in the presence of aluminium chloride at 70° for 4 h, followed by demethylation of the obtained ketone with 48% hydrobromic acid in refluxing acetic acid for 47 h [904].
- Preparation by dealkylation of 3,4-dichloro-4′-ethoxybenzophenone with aluminium chloride in refluxing carbon disulfide [903].

m.p. 172–174° [903], 158°5–170° [165]. A typing error probably occurred in the published data.

Spectra (NA).

(3,5-Dichlorophenyl)(4-hydroxyphenyl)methanone

[119427-60-0] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



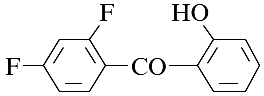
Synthesis

– Refer to: [298,513] and [435] (compound **155**).

m.p. and Spectra (NA).

(2,4-Difluorophenyl)(2-hydroxyphenyl)methanone

[46795-44-2] $C_{13}H_8F_2O_2$ mol.wt. 234.20



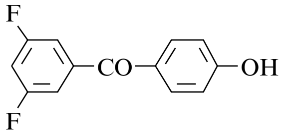
Synthesis

– Refer to: [87] (compound **V-A**).

m.p. and Spectra (NA).

(3,5-Difluorophenyl)(4-hydroxyphenyl)methanone

[148253-49-0] $C_{13}H_8F_2O_2$ mol.wt. 234.20



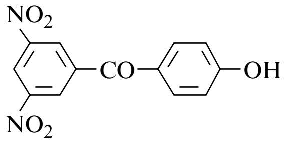
Synthesis

– Preparation by demethylation of 3,5-difluoro-4′-methoxy-benzophenone (SM) with 48% hydrobromic acid in refluxing acetic acid for 4 h (91%) [905,906]. SM was obtained by Friedel–Crafts acylation of anisole with 3,5-di-fluorobenzoyl chloride in ethylene dichloride in the presence of aluminium chloride at r.t. under nitrogen for 3 h (88%) [905].

m.p. 134–135° [905,906];
^{1}H NMR [905,906], ^{13}C NMR [905,906], IR [905,906], MS [905,906].

(3,5-Dinitrophenyl)(4-hydroxyphenyl)methanone

[51339-44-7] $C_{13}H_8N_2O_6$ mol.wt. 288.22



Syntheses

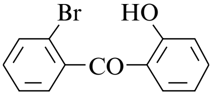
– Preparation by reaction of 3,5-dinitrobenzoyl chloride with phenetole in the presence of aluminium chloride in carbon disulfide, first between 0° and 5° for 8.5 h, then at r.t. overnight (60%) [903].

– Obtained (by-product) by reaction of 3,5-dinitrobenzoyl chloride with anisole in the presence of aluminium chloride [907].

m.p. 196–197° (d) [903], 176–177° [907]; Spectra (NA).

(2-Bromophenyl)(2-hydroxyphenyl)methanone

[99515-47-6] $C_{13}H_9BrO_2$ mol.wt. 277.11



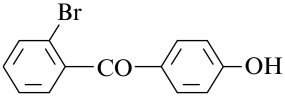
Synthesis

- Preparation from 2′-bromo-5-tert-butyl-2-methoxy-benzophenone by total dealkylation with aluminium chloride in benzene at 65–70° for 45 h (75%) [9], (60–80%) [22].
- Also refer to: [908].

m.p. 76–77° [9,22]; Spectra (NA).

(2-Bromophenyl)(4-hydroxyphenyl)methanone

$C_{13}H_9BrO_2$ mol.wt. 277.11



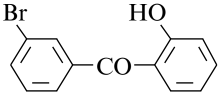
Synthesis

- Preparation by dealkylation of 2-bromo-4′-ethoxy-benzophenone with hydrobromic acid (d = 1.49) in boiling acetic acid for 2 days.
- Refer to: Chem. Abstr., **17**, 3497^5 (1923)^T.

b.p.$_{10}$ 260°^{T}; m.p. 114°^{T}; Spectra (NA).

(3-Bromophenyl)(2-hydroxyphenyl)methanone

$C_{13}H_9BrO_2$ mol.wt. 277.11



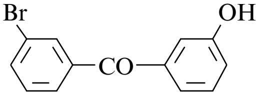
Synthesis

- Preparation by total dealkylation of 5-tert-butyl-3′-bromo-2-methoxybenzophenone with aluminium chloride in benzene at 65–70° for 45 h (60%) [9], 60–80% [22].

m.p. 77–78° [9,22]; Spectra (NA).

(3-Bromophenyl)(3-hydroxyphenyl)methanone

[62810-50-8] $C_{13}H_9BrO_2$ mol.wt. 277.11

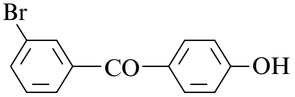


Synthesis

- Preparation by demethylation of 3-bromo-3′-methoxy-benzophenone with aluminium chloride in refluxing chlorobenzene [900].

m.p. 110° [900]; Spectra (NA).

(3-Bromophenyl)(4-hydroxyphenyl)methanone



$C_{13}H_9BrO_2$ mol.wt. 277.11

Synthesis

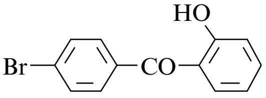
- Preparation by dealkylation of 3-bromo-4′-ethoxy-benzophenone (SM) with hydrobromic acid (d = 1.49) in boiling acetic acid for 2 days.

SM was obtained by action of m-bromobenzoyl chloride with phenetole in carbon disulfide in the presence of aluminium chloride at 55° (80%, m.p. 79°5). -Refer to: Chem. Abstr., **17**, 3497^{5} (1923)^{T}.

m.p. 171°^{T}; Spectra (NA).

(4-Bromophenyl)(2-hydroxyphenyl)methanone

[2038-92-8]



$C_{13}H_9BrO_2$ mol.wt. 277.11

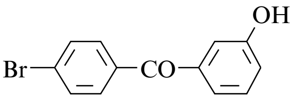
Syntheses

- Preparation by Fries rearrangement of phenyl p-bromo-benzoate [909], with aluminium chloride at 140° for 30 min (23%) [485].
- Preparation by reaction of o-methoxybenzoyl chloride with bromobenzene in the presence of aluminium chloride (Friedel–Crafts) [910].
- Preparation by diazotization of 2-amino-4′-bromobenzophenone, followed by hydrolysis of the diazonium salt so obtained (28%) [911]. The 3-bromofluorenone was the major compound obtained.

m.p. 98° [910,911], 90–92° [485]; ^{1}H NMR [910], IR [910].

(4-Bromophenyl)(3-hydroxyphenyl)methanone

[62810-46-2]



$C_{13}H_9BrO_2$ mol.wt. 277.11

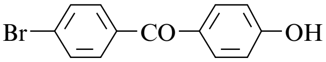
Syntheses

- Preparation by reaction of m-anisoyl chloride with bromo-benzene in the presence of an excess of aluminium chloride: first at 20°, then at reflux for 2 h (65%) [119,899].
- Preparation by Friedel–Crafts acylation of bromobenzene with m-nitrobenzoyl chloride, reduction of the obtained 4-bromo-3′-nitrobenzophenone and diazotization of the resulting 3-amino-4′-bromobenzophenone, followed by hydrolysis of the diazonium salt [900].
- Preparation by demethylation of 4-bromo-3′-methoxybenzophenone with aluminium chloride in refluxing chlorobenzene [900].
- Also refer to: [912].

m.p. 170° [900], 167° [119,899]; Spectra (NA).

(4-Bromophenyl)(4-hydroxyphenyl)methanone

[4369-50-0] $C_{13}H_9BrO_2$ mol.wt. 277.11



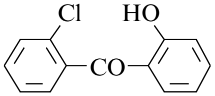
Syntheses

- Preparation by reaction of p-bromobenzoyl chloride with phenol in the presence of aluminium chloride [165].
- Preparation by isomerization of 2-hydroxy-4′-bromobenzophenone with trifluoromethanesulfonic acid at 110° for 3 h (42%) [909].
- Preparation by Fries rearrangement of phenyl p-bromobenzoate with aluminium chloride without solvent at 140° for 30 min (47%) [485] or in nitrobenzene at 60° for 40 h (67%) [913–915].
- Also obtained by reaction of hydrobromic acid with 4-bromo-4′-ethoxybenzophenone in refluxing acetic acid [142].
- Also obtained by diazotization of 4-amino-4′-bromobenzophenone, followed by hydrolysis of the obtained diazonium salt [916].
- Preparation by demethylation of 4-bromo-4′-methoxybenzophenone (SM) with aluminium chloride in refluxing benzene for 8 h. SM was obtained by Friedel–Crafts acylation of anisole with p-bromobenzoyl chloride [917].
- Also refer to: [512,513,902,918].

m.p. 192–193° [913–915], 191° [142,485,916], 187°5–191° [165]; IR [917]; TLC [917].

(2-Chlorophenyl)(2-hydroxyphenyl)methanone

[70288-96-9] $C_{13}H_9ClO_2$ mol.wt. 232.67



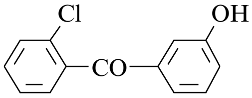
Syntheses

- Obtained by total dealkylation of 5-tert-butyl-2′-chloro-2-methoxybenzophenone with aluminium chloride in benzene at 65–70° for 45 h (60–80%) [22], (73%) [9].
- Also obtained by Fries rearrangement of phenyl o-chlorobenzoate (SM) with aluminium chloride [23,897], at 140° for 30 min (32%) [485]. SM was prepared by heating o-chlorobenzoyl chloride with aluminium tris(phenoxide) in a water bath for 30 min [485].
- Also obtained (poor yield) by reaction of o-chlorobenzoyl chloride with phenyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (3%) [55].
- Also refer to: [29,850].

m.p. 92° [485], 58–59° [9,22]. There is a discrepancy between the two melting points. Spectra (NA).

(2-Chlorophenyl)(3-hydroxyphenyl)methanone

[62810-53-1] $C_{13}H_9ClO_2$ mol.wt. 232.67



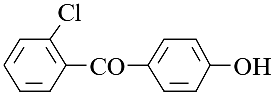
Synthesis

– Preparation by demethylation of 2-chloro-3′-methoxy-benzophenone with aluminium chloride in refluxing chlorobenzene [900].

m.p. 126° [900]; Spectra (NA).

(2-Chlorophenyl)(4-hydroxyphenyl)methanone

[55270-71-8] $C_{13}H_9ClO_2$ mol.wt. 232.67



Syntheses

– Preparation by Fries rearrangement of phenyl o-chloro-benzoate in the presence of aluminium chloride [897,919,920], without solvent for 2 h at 160° [920] or in nitrobenzene at 60° (31%) [919].

– Preparation by Fries rearrangement of phenyl o-chlorobenzoate (SM) with aluminium chloride at 140° for 30 min (56%). SM was obtained by heating o-chlorobenzoyl chloride with aluminium tris(phenoxide) in a water bath for 30 min [485].

– Preparation by reaction of o-chlorobenzoyl chloride,
  - with phenyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (57%) [55];
  - with phenol trimethylsilyl ether in the presence of aluminium chloride in refluxing methylene chloride for 2 h (54%) [921].

– Also obtained by reaction of o-chlorobenzoic acid with phenol in the presence of polyphosphoric acid for 20 min at 100° (13%) [149].

– Also obtained (trace) by reaction of EKONOL$^{(TM)}$, an aromatic polyester, behaves as a Friedel–Crafts acylating reagent, with chlorobenzene in triflic acid solution at 25° for 18 h (1%) [922].

– Also refer to: [542,902].

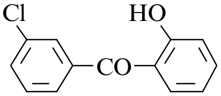
m.p. 165° [920], 128° [55], 119–121° [919], 118° [149], 112° [485], 102–104° [921];

There is a discrepancy between the various melting points.

Spectra (NA); HPLC [922].

(3-Chlorophenyl)(2-hydroxyphenyl)methanone

[72090-60-9] $C_{13}H_9ClO_2$ mol.wt. 232.67



Syntheses

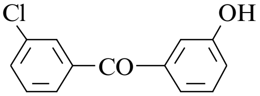
– Preparation by heating a mixture of 3-(3-chlorobenzoyl)-4-methoxybenzoic acid and pyridinium chloride at 200° for 20 h (65%) [923]. There are simultaneous demethylation and decarboxylation.

- Also obtained by Fries rearrangement of phenyl m-chlorobenzoate,
  - with aluminium chloride between 120° and 160° [924];
  - in the presence of Nafion-H, a polymeric perfluorinated resin sulfonic acid, in refluxing nitrobenzene for 12 h (21%) [38].

m.p. 89° [924], 87° [923]; ^{1}H NMR [38], ^{13}C NMR [38], IR [923].

(3-Chlorophenyl)(3-hydroxyphenyl)methanone

[62810-42-8] $C_{13}H_9ClO_2$ mol.wt. 232.67



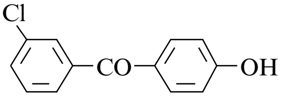
Synthesis

- Preparation by demethylation of 3-chloro-3′-methoxy-benzophenone with aluminium chloride in refluxing chlorobenzene (81%) [900].

m.p. 104° [900]; Spectra (NA).

(3-Chlorophenyl)(4-hydroxyphenyl)methanone

[61002-52-6] $C_{13}H_9ClO_2$ mol.wt. 232.67



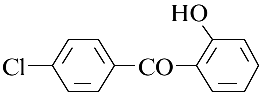
Syntheses

- Preparation by Fries rearrangement of phenyl m-chloro-benzoate,
  - in the presence of aluminium chloride without solvent at 120° or at 160° [924];
  - in the presence of Nafion-H, a polymeric perfluorinated resin sulfonic acid, in refluxing nitrobenzene for 12 h (54%) [38].
- Also obtained (poor yield) by reaction of m-chlorobenzoic acid with phenol in the presence of polyphosphoric acid at 100° for 20 min (5%) [149].
- Also obtained by reaction of ethyl nitrite with 2-amino-5-chloro-4′-hydroxybenzophenone in refluxing ethanol (elimination of amino group) [925].
- Also refer to: [542,897,902].

m.p. 172° [924], 169° [149], 161° [925]; ^{1}H NMR [38], ^{13}C NMR [38].

(4-Chlorophenyl)(2-hydroxyphenyl)methanone

[2985-79-7] $C_{13}H_9ClO_2$ mol.wt. 232.67



Syntheses

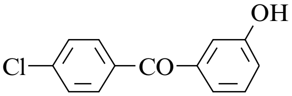
- Preparation by Fries rearrangement of phenyl p-chloro-benzoate,
  - with aluminium chloride without solvent between 120° and 160° [28,926], (29%) [28], at 200° for 20 min [20] or in refluxing chlorobenzene for 10 h (53%) [28];
  - with trifluoromethanesulfonic acid at 45–55° (6%) [927].
- Preparation by reaction of 2-hydroxybenzoyl chloride with chlorobenzene in the presence of aluminium chloride in refluxing carbon disulfide (72%) [92].

- Also obtained by photo-Fries rearrangement of phenyl p-chlorobenzoate in cyclohexane or in benzene between 46° and 52° (42–49%) [72].
- Also obtained by reaction of p-chlorobenzoyl chloride with phenyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (18%) [55].
- Preparation by treatment of 5-tert-butyl-4′-chloro-2-methoxybenzophenone with aluminium chloride in benzene at 65–70° during 45 h (60–80%) [9,927].
- Also obtained by reaction of salicylaldehyde with p-iodochlorobenzene by using a catalyst system of palladium chloride/lithium chloride in the presence of sodium carbonate in N,N-dimethyl-formamide at 100° for 2 h (57%) [51].
- Also obtained by demethylation of 4-chloro-2′-methoxybenzophenone with boron tribromide in methylene chloride at r.t. for 12 h [20], according to [21].
- Also refer to: [77].

m.p. 77–78° [926], 75–76° [72], 74–75° [9,22], 70–72° [92], 68–71°5 [28]; ^{1}H NMR [51], MS [51].

(4-Chlorophenyl)(3-hydroxyphenyl)methanone

[62810-39-3] $C_{13}H_9ClO_2$ mol.wt. 232.67



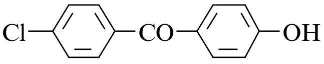
Synthesis

- Preparation by diazotization of 3-amino-4′-chlorobenzo-phenone (SM), followed by hydrolysis of the diazonium salt obtained (81%) [900], (84%) [634]. SM was obtained by Friedel–Crafts acylation of chlorobenzene with m-nitro-benzoyl chloride, followed by reduction of the resulting 4-chloro-3′-nitrobenzophenone [900].
- Also refer to: [928].

m.p. 154–155° [634], 154° [900]; Spectra (NA).

(4-Chlorophenyl)(4-hydroxyphenyl)methanone

[42019-78-3] $C_{13}H_9ClO_2$ mol.wt. 232.67



Syntheses

- Preparation by Fries rearrangement of phenyl p-chloro-benzoate,
  - in the presence of aluminium chloride, without solvent, at 160° for 5 min [490,491], between 120° and 160° (good yield) [926] or at 130° for 1 h, then at 160° for 1 h (10%) [28] or in refluxing chlorobenzene for 10 h (52%) [28];
  - in the presence of trifluoromethanesulfonic acid at 45–55° (94%) [927].
- Preparation by reaction of p-chlorobenzotrichloride with phenol in hydrofluoric acid in the presence of water at 0°, then at r.t. overnight (91%) [213].
- Also obtained by reaction of p-chlorobenzoyl chloride,
  - with phenol in the presence of aluminium chloride [165];
  - with anisole in the presence of aluminium chloride [929], at 70° for 4 h [904]. The 4′-chloro-4-methoxybenzophenone so formed [929], (67%) [904], gave

the expected ketone by demethylation with 48% hydrobromic acid in refluxing acetic acid for 47 h [904] or with aluminium chloride in refluxing chlorobenzene for 1.5 h [929];

- with phenyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (32%) [55].

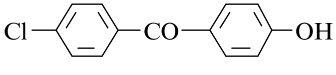
- Also obtained by reaction of p-chlorobenzoic acid with phenol,
- in the presence of hydrofluoric acid for 6 h at 75° in an autoclave under pressure (74%) [930];
  - in the presence of polyphosphoric acid at 100° for 20 min (2%) [149];
  - in the presence of a trifluoromethanesulfonic acid at r.t. for one day [151].
- Also obtained by reaction of p-hydroxybenzoic acid with chlorobenzene in the presence of trifluoromethanesulfonic acid at 100° for 5 days (50%) [151].
- Preparation by Friedel–Crafts acylation of chlorobenzene with p-anisoyl chloride in the presence of aluminium chloride at 120° for 3 h (50–70%) [931].
- Preparation by diazotization of 4-amino-4′-chlorobenzophenone [932], followed by hydrolysis of the diazonium salt so obtained [142].
- Also obtained by reaction of EKONOL$^{(TM)}$, an aromatic polyester as Friedel–Crafts reagent, with chlorobenzene in triflic acid solution at 25° for 18 h (28%) [922]. Similar results can be obtained using hydrofluoric acid/boron trifluoride or aluminium chloride in place of triflic acid [922].
- Also obtained (poor yield) by photo-Fries rearrangement of phenyl p-chlorobenzoate in benzene at 52° for 19 h (14%) or in cyclohexane at 46° for 19 h (6%) [72].
- Also obtained from 4-chloro-4′-fluorobenzophenone by reaction under nitrogen with potassium hydroxide in aqueous dimethyl sulfoxide at 60° for 18 h [170].
- Also refer to: [366,902,933–943].

N.B.: K salt [170].

m.p. 179–181° [165], 179°5 [170], 179°2 [142], 179° [149,213], 178°5–180°5 [930], 175–176° [926], 173–175° [931], 172°5–173°8 [72], 170–171° [28];
b.p.$_{13}$ 257° [142]; TLC [931]; HPLC [922];
^{1}H NMR [151,931], ^{13}C NMR [922], MS [922,931].

(4-Chlorophenyl)(4-hydroxyphenyl)methanone-^{14}C

[60044-21-5] $C_{13}H_9ClO_2$ mol.wt. 234.67

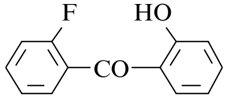


Synthesis

- Preparation by demethylation of ^{14}C-4′-chloro-4-methoxybenzophenone (SM) with 48% hydrobromic acid in refluxing acetic acid for 48 h (74%). SM was obtained by reaction of ^{14}C-p-chlorobenzoyl chloride with anisole in the presence of aluminium chloride at 50° for 3 h (82%) [944].

m.p. and Spectra (NA).

(2-Fluorophenyl)(2-hydroxyphenyl)methanone



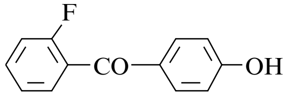
$C_{13}H_9FO_2$ mol.wt. 216.21

Synthesis

– Preparation by Fries rearrangement of phenyl o-fluoro-benzoate with aluminium chloride [23].

m.p. and Spectra (NA).

(2-Fluorophenyl)(4-hydroxyphenyl)methanone



$C_{13}H_9FO_2$ mol.wt. 216.21

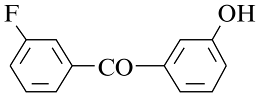
Synthesis

– Obtained (trace) by reaction of EKONOL$^{(TM)}$, an aromatic polyester as Friedel–Crafts reagent, with fluorobenzene in triflic acid solution at 75° for 2 h or at 25° for 18 h (1%) [922].

m.p. and Spectra (NA); HPLC [922].

(3-Fluorophenyl)(3-hydroxyphenyl)methanone

[62810-55-3]



$C_{13}H_9FO_2$ mol.wt. 216.21

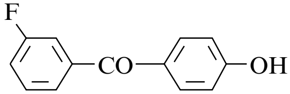
Synthesis

– Preparation by demethylation of 3-fluoro-3′-methoxy-benzophenone with aluminium chloride in refluxing chlorobenzene [900].

m.p. 64° [900]; Spectra (NA).

(3-Fluorophenyl)(4-hydroxyphenyl)methanone

[190728-34-8]



$C_{13}H_9FO_2$ mol.wt. 216.21

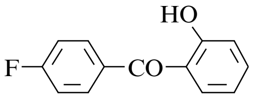
Synthesis

– Refer to: Chem. Abstr., **127**, 34137f (1997).

m.p. and Spectra (NA).

(4-Fluorophenyl)(2-hydroxyphenyl)methanone

[62666-37-9]



$C_{13}H_9FO_2$ mol.wt. 216.21

Syntheses

– Preparation by Fries rearrangement of phenyl p-fluoro-benzoate with aluminium chloride,

- without solvent at 200° for 20 min [20];
- in nitrobenzene at 140–145° for 3 h (35%) [909].

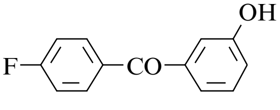
– Also obtained by demethylation of 4-fluoro-2′-methoxybenzophenone with boron tribromide in methylene chloride at r.t. for 12 h [20], according to [21].

- Also obtained (by-product) by reaction of p-fluorobenzoic acid with phenol in the presence of hydrofluoric acid for 6 h at 75° in an autoclave under pressure (5%) [930].
- Also refer to: [945].

m.p. and Spectra (NA).

(4-Fluorophenyl)(3-hydroxyphenyl)methanone

[62810-47-3] $C_{13}H_9FO_2$ mol.wt. 216.21



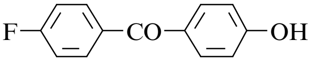
Syntheses

- Preparation by reaction of m-anisoyl chloride with fluoro-benzene in the presence of an excess of aluminium chloride: first at 20°, then at reflux for 2 h (43%) [119].
- Preparation by reaction of m-hydroxybenzoic acid with fluorobenzene in the presence of a hydrofluoric acid/boron trifluoride mixture at r.t. for 6 h into an autoclave under 30 psig of boron trifluoride (61%) [350].
- Preparation by diazotization of 3-amino-4′-fluorobenzophenone followed by hydrolysis of the obtained diazonium salt [900].
- Preparation by demethylation of 4-fluoro-3′-methoxybenzophenone with aluminium chloride in refluxing chlorobenzene [900].
- Also refer to: [899,946,947].

m.p. 105° [900,947], 103° [119,899], 102° [946], 99–99°5 [350];
^{1}H NMR [350], ^{13}C NMR [350], ^{19}F NMR [350].

(4-Fluorophenyl)(4-hydroxyphenyl)methanone

[25913-05-7] $C_{13}H_9FO_2$ mol.wt. 216.21



Syntheses

- Preparation by reaction of p-fluorobenzoyl chloride with phenol,
  - in the presence of aluminium chloride [165];
  - in the presence of boron trifluoride in hydrofluoric acid (83%) [316].
- Preparation by isomerization of 4-fluoro-2′-hydroxybenzophenone,
  - with trifluoromethanesulfonic acid at 120° for 5 h (75%) or with perfluoroethanesulfonic acid at 120° for 3 h (53%) [909];
  - by dissolution in toluene at 110° and the resultant solution cooled to 3° (97%) [945].
- Preparation by Fries rearrangement of phenyl p-fluorobenzoate,
  - with hydrofluoric acid between −10° and 0° (63%) [948];
  - with aluminium chloride without solvent at 160° for 5 min [490,491] or in nitrobenzene at 140–145° for 3 h (65%) [909].

- Preparation by reaction of p-fluorobenzoic acid with phenol,
  - in the presence of hydrofluoric acid for 6 h at 75° in an autoclave under pressure (90%) [930];
  - in the presence of boron trifluoride in hydrofluoric acid (64%) [316];
  - in the presence of trifluoromethanesulfonic acid overnight at r.t. (77%) [151].
- Also obtained by reaction of EKONOL$^{(TM)}$, an aromatic polyester, behaves as a Friedel–Crafts acylating reagent, with fluorobenzene in triflic acid solution at 75° for 2 h (74%) or at 25° for 18 h (67%) [922]. Similar results can be obtained using hydrofluoric acid/boron trifluoride or aluminium chloride in place of triflic acid [922].
- Preparation by reaction of 4,4′-difluorobenzophenone (1 mol) with potassium hydroxide (2 mol) in aqueous dimethyl sulfoxide at 60° for 18 h (81%) [170].
- Also refer to: [350,512,935,943,945,949–956].

N.B.: Na [949] and K salts [170,337].

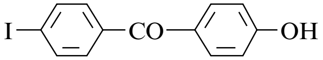
m.p. 170°6–172° [316], 169°5–171°5 [165], 169–171° [957], 168° [170], 166°5–168°5 [151];

^{1}H NMR [151,316], ^{13}C NMR [922], IR [151,316], MS [922,930];

GLC [316]; HPLC [922].

(4-Hydroxyphenyl)(4-iodophenyl)methanone

[113275-52-8] $C_{13}H_9IO_2$ mol.wt. 324.12



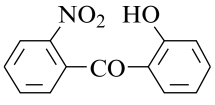
Synthesis

- Refer to: [512].

m.p. and Spectra (NA).

(2-Hydroxyphenyl)(2-nitrophenyl)methanone

[22293-32-9] $C_{13}H_9NO_4$ mol.wt. 243.22



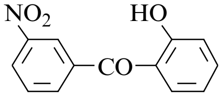
Synthesis

- Obtained by photo-Fries rearrangement of phenyl o-nitro-benzoate in ethanol during 60–75 h (23%) [641].
- Also refer to: [897].

m.p. 104° [641]; IR [641], UV [641].

(2-Hydroxyphenyl)(3-nitrophenyl)methanone

[36412-61-0] $C_{13}H_9NO_4$ mol.wt. 243.22



Syntheses

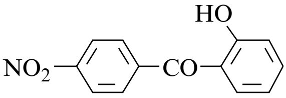
- Preparation by diazotization of 2-amino-3′-nitrobenzophenone followed by thermal decomposition of the 2-(3′-nitrobenzoyl)benzenediazonium fluoborate formed with 0.05 M sulfuric acid at 45° or 65° (67%) [17].

- Also obtained (poor yield) by Fries rearrangement of phenyl m-nitrobenzoate with aluminium chloride at 120° or 160° for 2 h (5%) [897,958].
- Also obtained by reaction of m-nitrobenzoyl chloride with anisole in the presence of aluminium chloride in refluxing carbon disulfide for 2 h (16%) [959]. In this reaction, 4-methoxy-3′-nitrobenzophenone was the major product.
- Also refer to: [960].

m.p. 101° [958], 96–97° [17], 93°5–94°5 [959]; IR [959], UV [959].

(2-Hydroxyphenyl)(4-nitrophenyl)methanone

[68223-20-1] $C_{13}H_9NO_4$ mol.wt. 243.22



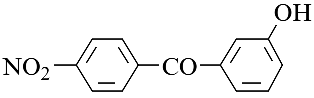
Syntheses

- Obtained by Fries rearrangement of phenyl p-nitrobenzoate with aluminium chloride [897],
  - without solvent at 160° (26%) [28], (15%) [961];
  - in refluxing chlorobenzene (8%) [28].
- Also obtained (by-product) by Fries rearrangement of phenyl p-nitrobenzoate (SM) with aluminium chloride at 140° for 30 min (16%). SM was obtained by heating p-nitrobenzoyl chloride with aluminium tris(phenoxide) in a water bath for 30 min [485].
- Also obtained by photo-Fries rearrangement of phenyl p-nitrobenzoate in ethanol during 60–75 h (19%) [641].
- Also obtained (by-product) by reaction of p-nitrobenzoyl chloride with phenetole in the presence of aluminium chloride in carbon disulfide [322,683,962].
- Also refer to: [963].

m.p. 114° [961], 112° [641], 111–113° [322], 111° [485], 108–110° [28];
IR [641], UV [641].

(3-Hydroxyphenyl)(4-nitrophenyl)methanone

[147029-77-4] $C_{13}H_9NO_4$ mol.wt. 243.22



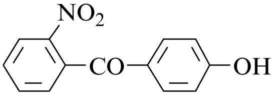
Synthesis

- Preparation by demethylation of 3-methoxy-4′-nitro-benzophenone with 62% hydrobromic acid in refluxing acetic acid for 4 h (70%) [964].

m.p. 117° [964]; Spectra (NA).

(4-Hydroxyphenyl)(2-nitrophenyl)methanone

[61101-88-0] $C_{13}H_9NO_4$ mol.wt. 243.22



Syntheses

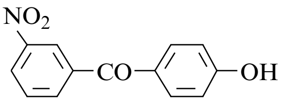
- Preparation by reaction of o-nitrobenzoyl chloride with (trimethylsilyl)phenol in the presence of stannic chloride in refluxing methylene chloride for 2 h (56%) [921].
- Also obtained (trace) by reaction of o-nitrobenzoic acid in the presence of polyphosphoric acid at 100° for 20 min (0.1%) [149].
- Also refer to: [897].

m.p. 165–167° [921], 122° [149]. There is a discrepancy between the two melting points.

Spectra (NA).

(4-Hydroxyphenyl)(3-nitrophenyl)methanone

[72090-63-2] $C_{13}H_9NO_4$ mol.wt. 243.22



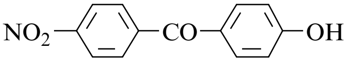
Syntheses

- Preparation by acylation of phenetole with m-nitrobenzoyl chloride in ethyl ether in the presence of aluminium chloride, then dealkylation of the 4-ethoxy-3′-nitrobenzophenone so formed with the same catalyst [141], in boiling carbon disulfide (60–70°) for 8 h [619] according to [139].
- Preparation by Fries rearrangement of phenyl m-nitrobenzoate with aluminium chloride [897] without solvent at 120° or at 160° for 2 h (32%) [958].
- Also obtained (poor yield) by reaction of m-nitrobenzoyl chloride with phenyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (9%) [55].
- Also obtained (trace) by reaction of m-nitrobenzoic acid with phenol in the presence of polyphosphoric acid at 100° for 20 min (1%) [149].
- Also refer to: [366,965,966].

m.p. 173° [619,958], 171° [149]; Spectra (NA); cryoscopic study [141].

(4-Hydroxyphenyl)(4-nitrophenyl)methanone

[18920-70-2] $C_{13}H_9NO_4$ mol.wt. 243.22



Syntheses

- Obtained by Fries rearrangement of phenyl p-nitro-benzoate with aluminium chloride [897],
  - without solvent at 140° for 30 min (52%) [485], at 120° for 2 h (21%) [961] or first at 130° for 1 h, then at 160° for 1 h (2%) [28];
  - in refluxing chlorobenzene for 5 h (24%) [28].

N.B.: Phenyl p-nitrobenzoate failed to undergo the Fries rearrangement in the presence of aluminium chloride [74].

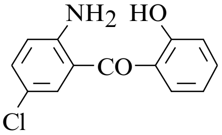
- Preparation by dealkylation,
  - of 4-methoxy-4′-nitrobenzophenone with aluminium chloride in boiling carbon disulfide or without solvent at 100–120° [322];
  - of 4-ethoxy-4′-nitrobenzophenone with aluminium chloride [141], in boiling carbon disulfide or without solvent at 100–120° [322].
- Preparation by reaction of p-nitrobenzoyl chloride with phenetole in the presence of aluminium chloride in ethyl ether [141] or in carbon disulfide. Simultaneous deethylation take place during the reaction [322].
- Also obtained (trace) by reaction of p-nitrobenzoic acid with phenol in the presence of polyphosphoric acid at 100° for 20 min [149].
- Also refer to: [366,902,967].

N.B.: Na salt [963].

m.p. 193–195° [28], 192° [961], 190–192° [322], 190° [149,485];
IR [124]; cryoscopic study [141].

(2-Amino-5-chlorophenyl)(2-hydroxyphenyl)methanone

[62492-57-3] $C_{13}H_{10}ClNO_2$ mol.wt. 247.68



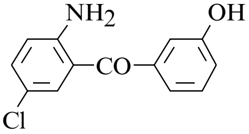
Synthesis

- Preparation by demethylation of 2-amino-5-chloro-2′-methoxybenzophenone with boron tribromide in methylene chloride at r.t. for 4 h (88%) [552].

m.p. 74–76° [552]; ^{1}H NMR [552].

(2-Amino-5-chlorophenyl)(3-hydroxyphenyl)methanone

[62492-58-4] $C_{13}H_{10}ClNO_2$ mol.wt. 247.68



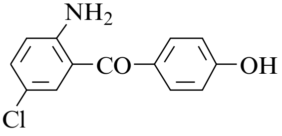
Synthesis

- Preparation by demethylation of 2-amino-5-chloro-3′-methoxybenzophenone with boron tribromide in methylene chloride at r.t. for 4 h (84%) [552].

m.p. 190–192° [552]; ^{1}H NMR [552].

(2-Amino-5-chlorophenyl)(4-hydroxyphenyl)methanone

[784-41-8] $C_{13}H_{10}ClNO_2$ mol.wt. 247.68



Syntheses

- Preparation by cleavage of 5-chloro-3-(p-hydroxyphenyl)-2,1-benzisoxazole (other name: 5-chloro-3-(p-hydroxy-phenyl)anthranil) (SM),
  - by heating at reflux with aluminium iodide for 50 min (87%) [968];

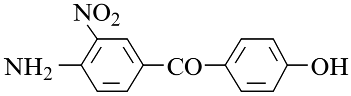
- by reaction with concentrated hydrochloric acid and an excess of tin in boiling ethanol or acetic acid. SM (m.p. 241°) was prepared by condensation of o-nitrobenzaldehyde with phenol in the presence of hydrogen chloride or phosphorous oxychloride in cold acetic acid [925];
- by hydrogenation in the presence of 10% Pd/C in ethyl acetate between 40° and 60° at a pressure of 3 atmospheres (quantitative yield) [969].

– Preparation by demethylation of 2-amino-5-chloro-4′-methoxybenzophenone with boron tribromide in methylene chloride at r.t. for 4 h (80%) [552].

m.p. 174° [925], 173–175° [969], 172–173° [968], 166–168° [552];
^{1}H NMR [552].

(4-Amino-3-nitrophenyl)(4-hydroxyphenyl)methanone

[60014-09-7] $C_{13}H_{10}N_2O_4$ mol.wt. 258.23



Synthesis

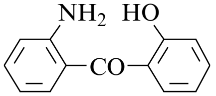
– Preparation by reaction of ammonia with 4-chloro-4′-hydroxy-3-nitrobenzophenone in dimethyl sulfoxide at 100° for 6 h (72%) [898].

– Also refer to: [970].

m.p. 218° [898]; IR [898], MS [898].

(2-Aminophenyl)(2-hydroxyphenyl)methanone

[13134-93-5] $C_{13}H_{11}NO_2$ mol.wt. 213.24



Syntheses

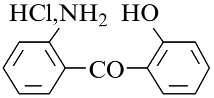
– Preparation by demethylation of 2-amino-2′-methoxybenzophenone (SM),

- with concentrated hydrobromic acid in refluxing acetic acid for 24 h (85%) [971];
- with aluminium chloride in refluxing benzene for 1 h (94%) [972]. SM was obtained according to [973].

– Also obtained by action of an excess ammonia on the 2,2′-dihydroxybenzophenone in ethanol [344].

m.p. 222° [344]; red oil [971], yellow [972];
^{1}H NMR [972], IR [972], MS [972]; TLC [972].

(2-Aminophenyl)(2-hydroxyphenyl)methanone *(Hydrochloride)*



$C_{13}H_{11}NO_2$,HCl mol.wt. 249.70

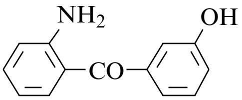
Synthesis

- Obtained from the corresponding amine [344].

m.p. 242° [344]; Spectra (NA).

(2-Aminophenyl)(3-hydroxyphenyl)methanone

[38824-12-3] $C_{13}H_{11}NO_2$ mol.wt. 213.24



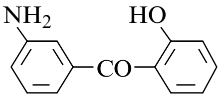
Synthesis

- Refer to: [974].

m.p. and Spectra (NA).

(3-Aminophenyl)(2-hydroxyphenyl)methanone

[35486-64-7] $C_{13}H_{11}NO_2$ mol.wt. 213.24



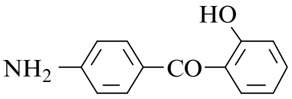
Synthesis

- Preparation by reduction of 2-hydroxy-3′-nitrobenzophenone with ammonium ferrous sulfate (86%) [959].
- Also refer to: [974,975].

m.p. 119–120° [959]; IR [959], UV [959].

(4-Aminophenyl)(2-hydroxyphenyl)methanone

[13134-94-6] $C_{13}H_{11}NO_2$ mol.wt. 213.24



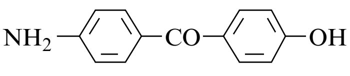
Synthesis

- Preparation by hydrogenation of 2-hydroxy-4′-nitro-benzophenone in the presence of Raney nickel in methanol (89%) [962].
- Also refer to: [563].

m.p. 138–139° [962]; Spectra (NA).

(4-Aminophenyl)(4-hydroxyphenyl)methanone

[14963-34-9] $C_{13}H_{11}NO_2$ mol.wt. 213.24



Synthesis

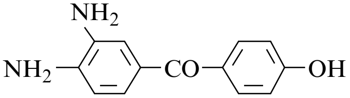
- Obtained by heating 4-hydroxy-4′-nitro-diphenylmethane (SM), sulfur and sodium hydroxide in 50% ethanol in a boiling water bath for 7 h (60%) [112]. In this reaction, oxidation of the methylene group to a carbonyl group occurred together with reduction of the nitro group. The oxidizing agent for the methylene group was the tetrasulfide [63]. SM was obtained by diazotization of 4-amino-4′-nitrodiphenylmethane (83%), according to [976].

- Also refer to: [176].

m.p. 184° [112]; UV [112,176].

(3,4-Diaminophenyl)(4-hydroxyphenyl)methanone

[93958-45-3] $C_{13}H_{12}N_2O_2$ mol.wt. 228.25



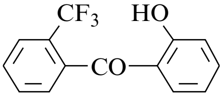
Synthesis

- Preparation by catalytic hydrogenation of 4-amino-4′-hydroxy-3-nitrobenzophenone in the presence of Raney nickel in ethanol in a Paar hydrogenator at 3 kg/cm^2 pressure for 4 h [898].

solid mass [898]; m.p. and Spectra (NA).

(2-Hydroxyphenyl)[2-(trifluoromethyl)phenyl]methanone

[205319-41-1] $C_{14}H_9F_3O_2$ mol.wt. 266.22



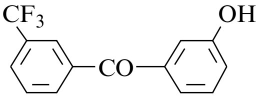
Synthesis

- Refer to: Chem. Abstr., **128**, 257232e (1998).

m.p. and Spectra (NA).

(3-Hydroxyphenyl)[3-(trifluoromethyl)phenyl]methanone

[62810-48-4] $C_{14}H_9F_3O_2$ mol.wt. 266.22



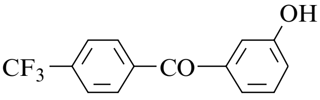
Synthesis

- Preparation by demethylation of 3-methoxy-3′-(trifluoro-methyl)benzophenone with refluxing pyridinium chloride or with 48% hydrobromic acid [900].

m.p. 78° [900]; Spectra (NA).

(3-Hydroxyphenyl)[4-(trifluoromethyl)phenyl]methanone

[21084-29-7] $C_{14}H_9F_3O_2$ mol.wt. 266.22



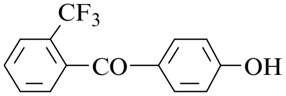
Syntheses

- Preparation by demethylation of 3-methoxy-4′-(trifluoro-methyl)benzophenone with refluxing pyridinium chloride for 90 min (72%) or with 48% hydrobromic acid [900].
- Preparation by dealkylation of 4-(trifluoromethyl)-3′-ethoxybenzophenone with pyridinium bromide at 210° for 0.5 h [977].

m.p. 130° [900], 127–128° [977]; Spectra (NA).

(4-Hydroxyphenyl)[2-(trifluoromethyl)phenyl]methanone

[190728-32-6] $C_{14}H_9F_3O_2$ mol.wt. 266.22



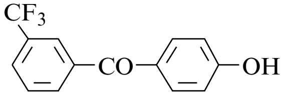
Synthesis

- Refer to: Chem. Abstr., **127**, 34137f (1997).

m.p. and Spectra (NA).

(4-Hydroxyphenyl)[3-(trifluoromethyl)phenyl]methanone

[732-55-8] $C_{14}H_9F_3O_2$ mol.wt. 266.22



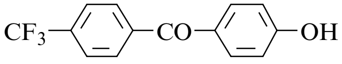
Syntheses

- Preparation by reaction of m-(trifluoromethyl) benzoyl fluoride with phenol in hydrofluoric acid at 100° for 6 h under 5 atmospheres (92%) [978].
- Preparation by reaction of m-(trifluoromethyl)benzoyl chloride with phenetole in the presence of aluminium chloride in carbon disulfide between 0° and 5°, then at r.t. for overnight (21%) [903].

m.p. 144–145° [903]; Spectra (NA).

(4-Hydroxyphenyl)[4-(trifluoromethyl)phenyl]methanone

[21084-27-5] $C_{14}H_9F_3O_2$ mol.wt. 266.22



Syntheses

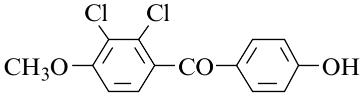
- Preparation by reaction of p-(trifluoromethyl)benzoyl chloride with phenol in the presence of aluminium chloride [165].
- Also obtained from 4-fluoro-4′-(trifluoromethyl)benzophenone by reaction under nitrogen with potassium hydroxide in aqueous dimethyl sulfoxide at 60° for 18 h [170].
- Preparation by dealkylation of 4-(trifluoromethyl)-4′-ethoxybenzophenone with pyridinium bromide at 210° for 0.5 h (77%) [977].
- Also refer to: [512,943].

N.B.: K salt [170].

m.p. 147° [170], 144–145° [977], 142–143° [165]; Spectra (NA).

(2,3-Dichloro-4-methoxyphenyl)(4-hydroxyphenyl)methanone

[92285-27-3] $C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14

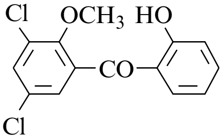


Synthesis

- Preparation by adding 2,3-dichloro-4-methoxy-4′-nitrobenzophenone in a mixture of acetaldoxime and sodium hydroxide in N,N-dimethylformamide cooled in an ice bath, and the mixture stirred overnight at r.t. (75%) [979].

m.p. 215–217° [979]; Spectra (NA).

(3,5-Dichloro-2-methoxyphenyl)(2-hydroxyphenyl)methanone



$C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14

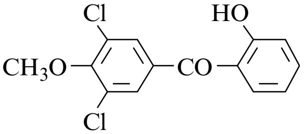
Synthesis

- Preparation from 2-iodophenyl 3,5-dichloro-2-methoxy-benzoate on treatment with n-butyllithium in a mixture of ethyl ether, hexane and tetrahydrofuran at −70° for 2 h, followed by treatment with saturated aqueous ammonium chloride (68%) [58].

pale yellow oil [58]; b.p. (NA); ^{1}H NMR [58], IR [58], MS [58].

(3,5-Dichloro-4-methoxyphenyl)(2-hydroxyphenyl)methanone

[129103-88-4] $C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14



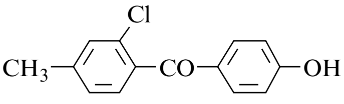
Synthesis

N.B.: This benzophenone was mentioned in the [Chem. Abstr., **113**, 131832x (1990)]. It has never been prepared by authors [58]. The paper actually concerns the 3,5-di-chloro-2′-hydroxy-2-methoxybenzophenone or(3,5-dichloro-2-methoxyphenyl)(2-hydroxyphenyl)-methanone. In entry 6 (table 1) of the paper [58], R_3 = H, but this information was not indicated in this one [980].

m.p. and Spectra (NA).

(2-Chloro-4-methylphenyl)(4-hydroxyphenyl)methanone

[98155-82-9] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



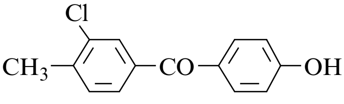
Synthesis

- Refer to: [901] (compound **33**).

m.p. and Spectra (NA).

(3-Chloro-4-methylphenyl)(4-hydroxyphenyl)methanone

[83885-15-8] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



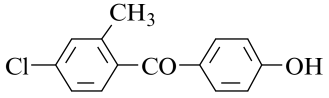
Synthesis

- Preparation by reaction of 3-chloro-4-methylbenzoyl chloride with phenol in the presence of aluminium chloride [165].

m.p. 153–154° [165]; Spectra (NA).

(4-Chloro-2-methylphenyl)(4-hydroxyphenyl)methanone

[98155-76-1] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



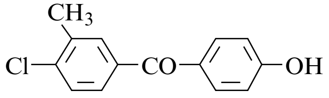
Synthesis

- Refer to: [901] (compound **9**).

m.p. and Spectra (NA).

(4-Chloro-3-methylphenyl)(4-hydroxyphenyl)methanone

[83885-20-5] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



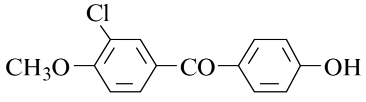
Synthesis

- Preparation by reaction of 4-chloro-3-methylbenzoyl chloride with phenol in the presence of aluminium chloride [165].

m.p. 154–156° [165]; Spectra (NA).

(3-Chloro-4-methoxyphenyl)(4-hydroxyphenyl)methanone

[83885-14-7] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



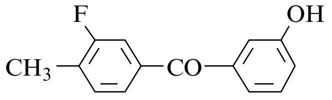
Synthesis

- Preparation by reaction of 3-chloro-4-methoxybenzoyl chloride with phenol in the presence of aluminium chloride [165].

m.p. 167–168° [165]; Spectra (NA).

(3-Fluoro-4-methylphenyl)(3-hydroxyphenyl)methanone

[62810-52-0] $C_{14}H_{11}FO_2$ mol.wt. 230.24



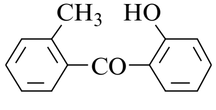
Synthesis

- Preparation by diazotization of 3′-amino-3-fluoro-4-methyl-benzophenone followed by hydrolysis of the diazonium salt obtained [900].

m.p. 120° [900]; Spectra (NA).

(2-Hydroxyphenyl)(2-methylphenyl)methanone

[51974-19-7] $C_{14}H_{12}O_2$ mol.wt. 212.25



Syntheses

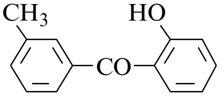
- Preparation by hydrogenation of 5-chloro-2-hydroxy-2′-methylbenzophenone in ethanolic solution in the presence of 10% Pd/C and potassium acetate at r.t. under atmosphere pressure (98%) [29].

- Also obtained by photo-Fries rearrangement of phenyl o-toluate in methanol or isopropanol (36–39%) and in benzene or ethyl ether (22%) [66].
- Also obtained (by-product) by diazotization of 2-amino-2′-methylbenzophenone with sodium nitrite in 5 N hydrochloric acid (3%). 1-methylfluorenone was the major product obtained in this reaction [981].

oil [29]; m.p. 65–67° [981]; ^{1}H NMR [29].

(2-Hydroxyphenyl)(3-methylphenyl)methanone

[33785-66-9] $C_{14}H_{12}O_2$ mol.wt. 212.25



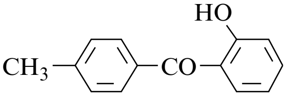
Syntheses

- Preparation from 2-methylthioxanthen-9-one 10,10-dioxide (SM) by a three-step synthesis: SM, by refluxing in 2% sodium hydroxide-65% dioxane-water solution for 18 h gave the 2-(2-hydroxybenzoyl)-4-methylphenylsulfinic acid (72%). The former, by reaction with mercuric chloride in refluxing acetic acid for 4 h led to the
- 2-chloromercuri-2′-hydroxy-5-methylbenzophenone (74%). Removal of the chloromercury group was achieved with concentrated hydrochloric acid in refluxing ethanol for 2 h (82%) [62].
- Also obtained by Fries rearrangement of phenyl m-toluate in the presence of aluminium chloride in refluxing carbon disulfide, then elimination of the solvent and heating at 150° for 3 h (20%) [62].
- Also obtained by reaction of m-toluoyl chloride with phenyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (13%) [55].

oil [62]; b.p.$_{0.9}$ 140–141° [62]; ^{1}H NMR [62], IR [62].

(2-Hydroxyphenyl)(4-methylphenyl)methanone

[19434-30-1] $C_{14}H_{12}O_2$ mol.wt. 212.25



Syntheses

- Preparation by reaction of pyridinium chloride on the 2-methoxy-4′-methylbenzophenone (SM) at reflux (33%). SM was obtained by reaction of p-tolunitrile on the o-methoxyphenylmagnesium bromide in ethyl ether (44%) [982].
- Preparation by Fries rearrangement of phenyl p-methylbenzoate,
  - with aluminium chloride in refluxing chlorobenzene for 10 h (40%) or without solvent, at 160° (22%) [28] or at 180° for 10 min (15%) [518];
  - with Nafion-H, a polymeric perfluorinated resin sulfonic acid, in refluxing nitrobenzene for 12 h (18%) [38].
- Also obtained by reaction of aluminium chloride with 5-tert-butyl-2-methoxy-4′-methyl-benzophenone in benzene at 65–70° for 45 h (60–80%) [9,22].
- Also obtained by reaction of 2-methoxybenzoyl chloride with toluene in the presence of aluminium chloride [5,9], (72%) [13], (47%) [17].

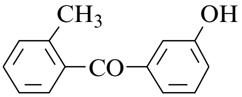
- Demethylation occurred during the Friedel–Crafts acylation, especially in the presence of ferric chloride at 130–140° [5].
- Also obtained by thermal decomposition of 2-(4′-methylbenzoyl)benzenediazonium fluoborate in 0.05 M sulfuric acid at 25° (36%) [17].
- Also refer to: [60].

m.p. 61°5 [13], 61–63° [982], 61–62° [17], 58–60° [28], 40° [518], 39–40° [9,22]. There is a discrepancy between the various melting points.

^{1}H NMR [38], ^{13}C NMR [38].

(3-Hydroxyphenyl)(2-methylphenyl)methanone

[147029-78-5] $C_{14}H_{12}O_2$ mol.wt. 212.25



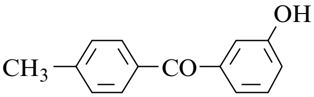
Synthesis

- Preparation by condensation of the Grignard reagent of anisole with o-toluoyl chloride, followed by demethylation of the resulting methyl ether (excellent yield) [964].

m.p. 112° [964]; Spectra (NA).

(3-Hydroxyphenyl)(4-methylphenyl)methanone

[62810-49-5] $C_{14}H_{12}O_2$ mol.wt. 212.25



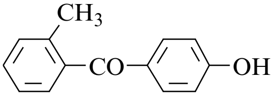
Syntheses

- Preparation by reaction of m-anisoyl chloride with toluene in the presence of an excess of aluminium chloride: first at 20°, then at reflux for 2 h (78%) [119,899].
- Preparation by diazotization of 3-amino-4′-methyl-benzophenone followed by hydrolysis of the diazonium salt obtained [900].
- Also obtained by demethylation of 3-methoxy-4′-methylbenzophenone with aluminium chloride in refluxing chlorobenzene [900].

m.p. 126° [119,899], 121° [900]; Spectra (NA).

(4-Hydroxyphenyl)(2-methylphenyl)methanone

[52981-01-8] $C_{14}H_{12}O_2$ mol.wt. 212.25



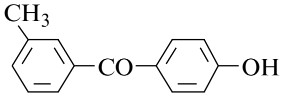
Syntheses

- Preparation by reaction of o-toluic acid with phenol in the presence of polyphosphoric acid at 100° for 20 min (47%) [149].
- Also obtained by photo-Fries rearrangement of phenyl o-toluate in methanol or isopropanol (21%) and in ethyl ether or benzene (6–8%) [66].

m.p. 96° [149]; Spectra (NA).

(4-Hydroxyphenyl)(3-methylphenyl)methanone

[71372-37-7] $C_{14}H_{12}O_2$ mol.wt. 212.25



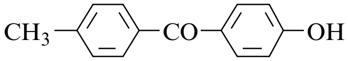
Syntheses

- Preparation by Fries rearrangement of phenyl m-toluate in the presence of aluminium chloride in refluxing carbon disulfide for 2 h, then elimination of the solvent and heating at 150° for 3 h (major product) [62].
- Preparation by reaction of m-toluoyl chloride with phenetole in the presence of aluminium chloride in carbon disulfide between 0° and 5° for 8 h, then at r.t. overnight (50%) [903].
- Also obtained by reaction of m-toluic acid with phenol in the presence of polyphosphoric acid at 100° for 20 min (19%) [149].
- Also obtained (poor yield) by reaction of m-toluoyl chloride with phenyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (5%) [55].

m.p. 166° [149], 165–166° [62], 163–164° [903]; Spectra (NA).

(4-Hydroxyphenyl)(4-methylphenyl)methanone

[134-92-9] $C_{14}H_{12}O_2$ mol.wt. 212.25



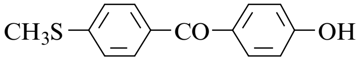
Syntheses

- Preparation by reaction of p-methylbenzoyl chloride with phenol in the presence of aluminium chloride [165].
- Preparation by reaction of p-toluic acid with phenol in the presence of polyphosphoric acid at 100° for 20 min (24%) [149].
- Preparation by Fries rearrangement of phenyl p-toluate,
  - with aluminium chloride without solvent at 130° for 1 h, then 160° for 1 h (20%) or in refluxing chlorobenzene for 7.5 h (57%) [28];
  - with Nafion-H, a polymeric perfluorinated resin sulfonic acid, in refluxing nitrobenzene for 12 h (45%) [38].
- Preparation by diazotization of 4-amino-4′-methylbenzophenone, followed by hydrolysis of the diazonium salt obtained (50%) [983].
- Also refer to: [984–988].

m.p. 171–173° [165], 170° [149], 166–167° [983], 161–162° [28]; ^{1}H NMR [38], ^{13}C NMR [38].

(4-Hydroxyphenyl)[4-(methylthio)phenyl]methanone

[83888-61-3] $C_{14}H_{12}O_2S$ mol.wt. 244.31



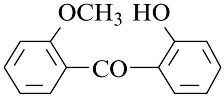
Synthesis

- Preparation by reaction of p-(methylthio)benzoyl chloride with phenol in the presence of aluminium chloride [165].

m.p. 133–134° [165]; Spectra (NA).

(2-Hydroxyphenyl)(2-methoxyphenyl)methanone

[21147-18-2] $C_{14}H_{12}O_3$ mol.wt. 228.25



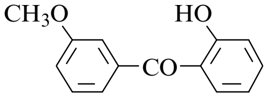
Syntheses

- Preparation from 2-iodophenyl 2-methoxybenzoate by treatment with n-butyllithium in a mixture of ethyl ether, hexane and tetrahydrofuran at –70° for 2 h, followed by treatment with saturated aqueous ammonium chloride (93%) [58].
- Preparation by hydrogenolysis of 2-(benzyloxy)-2′-methoxybenzophenone (SM) in ethyl acetate in the presence of 10% Pd/C at r.t. under atmospheric pressure (91%). SM was obtained by reaction of 2-(benzyloxy)benzaldehyde with 2-methoxyphenylmagnesium bromide in tetrahydrofuran [29].
- Also obtained by partial demethylation of 2,2′-dimethoxybenzophenone with boron trichloride in methylene chloride: first at –70°, then at r.t. for 30 min. From 2,2′-dimethoxybenzophenone, one methyl group is lost rapidly and a second somewhat more slowly [292].
- Also obtained (poor yield) by Fries rearrangement of phenyl o-anisate with aluminium chloride at 160° [897].
- Preparation by partial methylation of 2,2′-dihydroxybenzophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 5 h [989].
- Also refer to: [287].

oil [29,58]; m.p. 77° [989]; ^{1}H NMR [29,58], IR [58], MS [58,114].

(2-Hydroxyphenyl)(3-methoxyphenyl)methanone

[21554-73-4] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

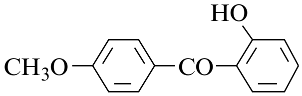
- Preparation from 2-methoxythioxanthen-9-one 10,10-dioxide (SM) by a three-step synthesis: SM by refluxing in 2% sodium hydroxide-65% dioxane-water solution for 18 h gave the 2-(2-hydroxybenzoyl)-4-methoxyphenylsulfinic acid (68%). The former by reaction with mercuric chloride in refluxing aqueous acetic acid for 4 h led to the 2-chloromercuri-2′-hydroxy-5-methoxybenzophenone (68%). Removal of the chloromercury group was achieved with concentrated hydrochloric acid in refluxing ethanol for 2 h (83%) [62].
- Also obtained by Fries rearrangement of phenyl m-anisate in the presence of aluminium chloride in refluxing carbon disulfide for 2 h, then elimination of the solvent and heating at 150° for 3 h (34%) [62].
- Also obtained by selective demethylation of 2,3′-dimethoxybenzophenone in the presence of boron trichloride in methylene chloride at r.t. for 30 min [341,343].
- Also obtained (poor yield) by reaction of 3-methoxybenzoyl chloride with anisole in the presence of aluminium chloride in refluxing carbon disulfide for 2 h [341].

- Also refer to: [897].

light yellow oil [62]; m.p. 40–42° [341]; b.p.$_{0.5}$ 148–149° [62]; ^{1}H NMR [62,341], IR [62,341], UV [341].

(2-Hydroxyphenyl)(4-methoxyphenyl)methanone

[18733-07-8] $C_{14}H_{12}O_3$ mol.wt. 228.25



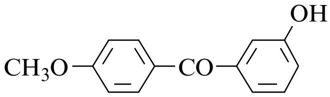
Syntheses

- Preparation from 2-iodophenyl p-anisate on treatment with n-butyllithium in a mixture of ethyl ether, hexane and tetrahydrofuran at −70° for 2 h, followed by treatment with saturated aqueous ammonium chloride (<18%) [58].
- Preparation by Fries rearrangement of phenyl p-anisate in the presence of aluminium chloride [897].
- Also obtained by reaction of salicylaldehyde with p-iodoanisole by using a catalyst system of palladium chloride/lithium chloride in the presence of sodium carbonate in N,N-dimethyl-formamide at 100° for 6 h (81%) [51].
- Preparation by partial demethylation of 2,4′-dimethoxybenzophenone with aluminium chloride in chlorobenzene at 80–100° [655].
- Also refer to: [626].

m.p. (NA); ^{1}H NMR [51], UV [235], MS [51].

(3-Hydroxyphenyl)(4-methoxyphenyl)methanone

[103203-53-8] $C_{14}H_{12}O_3$ mol.wt. 228.25



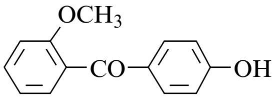
Syntheses

- Obtained by saponification of 3-acetoxy-4′-methoxy-benzophenone with 2 N sodium hydroxide in refluxing dilute ethanol for 30 min (98%). The starting material was obtained by Friedel–Crafts acylation of anisole with m-acetoxybenzoyl chloride in the presence of aluminium chloride in carbon disulfide at 5° for 20 h (15%) [990].
- Also obtained by selective demethylation of 3,4′-dimethoxybenzophenone in the presence of boron trichloride in methylene chloride at r.t. for 30 min [341].

m.p. 133° [990], 130–133° [341]; Spectra (NA).

(4-Hydroxyphenyl)(2-methoxyphenyl)methanone

[72090-61-0] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

- Preparation by reaction of o-methoxybenzoic acid with phenol in the presence of polyphosphoric acid at 100° for 20 min (61%) [149] or at 75–85° for 3 h (56%) [346].

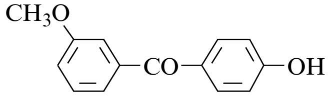
- Preparation by Fries rearrangement of phenyl o-methoxy-benzoate with polyphosphoric acid at 100° for 20 min (43%) [991].
- Also refer to: [155,897,992,993].

N.B.: Na salt [88].

m.p. 152–153° [991], 149° [149], 147–149° [346];
^{1}H NMR (Sadtler: standard n° 38490 M),
IR (Sadtler: standard n° 65528 K), UV [991].

(4-Hydroxyphenyl)(3-methoxyphenyl)methanone

[72090-62-1] $C_{14}H_{12}O_3$ mol.wt. 228.25



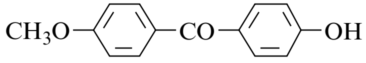
Syntheses

- Preparation by Fries rearrangement of phenyl m-anisate in the presence of aluminium chloride in refluxing carbon disulfide for 2 h, then elimination of the solvent and heating at 150° for 3 h (major product) [62].
- Preparation by reaction of m-methoxybenzoyl chloride with phenetole in the presence of aluminium chloride in carbon disulfide between 0° and 5°, then at r.t. overnight (51%) [903].
- Also obtained by reaction of m-methoxybenzoic acid with phenol in the presence of polyphosphoric acid at 100° for 20 min (15%) [149].
- Also refer to: [897].

m.p. 141–142° [903], 138° [149], 137–138° [62]; Spectra (NA).

(4-Hydroxyphenyl)(4-methoxyphenyl)methanone

[61002-54-8] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

- Preparation by reduction of 4-methoxy-4′-nitro-benzophenone with stannous chloride and hydrochloric acid, followed by diazotization of the resulting 4-amino-4′-methoxybenzophenone and hydrolysis of the diazonium salt [322].
- Preparation by Fries rearrangement of phenyl p-anisate,
  - with aluminium chloride,
    without solvent at 120° or at 160° [315];
    in nitromethane at 20° for 170 h (35%) [991];
    in nitrobenzene at 75° for 6 h (49%) [991] or at 80° for 1 h (21%) [994];
  - with titanium tetrachloride,
    without solvent at 95–100° for 30 min (11%) [991];
    in nitromethane at 20° for 170 h (76%) [991];
  - with stannic chloride in nitromethane at 20° for 170 h (14%) [991];
  - with polyphosphoric acid in a water bath for 30 min (43%) [137].

- Also obtained by reaction of p-anisic acid with phenol in the presence of polyphosphoric acid by heating in a water bath for 30 min (34%) [137] or at 100° for 20 min (75%) [149].
- Also obtained by reaction of p-hydroxybenzoic acid with anisole,
  - in the presence of polyphosphoric acid in a boiling water bath for 20 min (25%) [137];
  - in the presence of zinc chloride and a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 40°. Then, during 1.5 h, phosphorous trichloride was added and the mixture heated at 60° for 16 h (91%) [194];
  - in the presence of boron trifluoride in nitrobenzene at 80° for 30 min (74%) [186].
- Preparation by Friedel–Crafts acylation,
  - of phenol with p-methoxybenzoyl chloride in the presence of aluminium chloride [165];
  - of anisole with p-(acetoxy)benzoyl chloride in the presence of aluminium chloride in carbon disulfide at 15° for 1 h. The saponification of the 4-(acetoxy)-4′-methoxybenzophenone formed with sodium hydroxide in refluxing dilute ethanol for 30 min gave the expected ketone (95%) [990].
- Preparation by hydrolysis of 4-(4-anisoyloxy)-4′-methoxybenzophenone with concentrated sulfuric acid on standing for 10 min (79%) [137].
- Preparation from 4-fluoro-4′-methoxybenzophenone by reaction under nitrogen with potassium hydroxide in aqueous dimethyl sulfoxide at 60° for 18 h [170].
- Also refer to: [309,897,902,918,943,992,993,995–999].

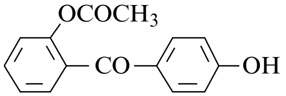
N.B.: Na [88] and K salts [170].

m.p. 155° [170], 154° [990], 153–154° [994], 151–152° [322], 151° [137,149,991], 150°6–151°8 [194], 150–151° [315], 114°5–145°5 [165]. A typing error probably occurred in the published data.

^{1}H NMR (Sadtler: standard n° 38498 M), IR (Sadtler: standard n° 65536 K), UV [991].

[2-(Acetyloxy)phenyl](4-hydroxyphenyl)methanone

[145723-29-1] $C_{15}H_{12}O_4$ mol.wt. 256.26



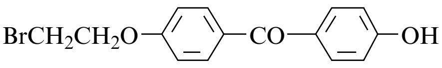
Synthesis

- Obtained by photooxygenation of 3-(4-hydroxyphenyl)-2-methylbenzofuran in methylene chloride at 5° (60%) [1000].

m.p. 125–126° [1000]; ^{1}H NMR [1000], ^{13}C NMR [1000], IR [1000], UV [1000].

[4-(2-Bromoethoxy)phenyl](4-hydroxyphenyl)methanone

[79578-62-4] $C_{15}H_{13}BrO_3$ mol.wt. 321.17



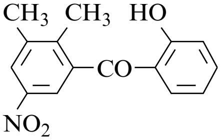
Synthesis

– Preparation by reaction of 4-hydroxybenzoic acid with β-bromophenetole [1001], in solution of a polyphosphoric acid/85% phosphoric acid/zinc chloride mixture. The solution was heated to 50–60°, phosphorous trichloride was added during 1 h and the mixture heated for 20 h at 70° (74%) [1002].

m.p. 139–142° [1002], 136–138° [1001]; ^{1}H NMR [1002], IR [1002], MS [1002].

(2,3-Dimethyl-5-nitrophenyl)(2-hydroxyphenyl)methanone

[110969-51-2] $C_{15}H_{13}NO_4$ mol.wt. 271.27



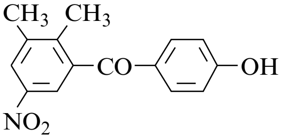
Synthesis

– Obtained by Fries rearrangement of phenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride without solvent at 160° for 2 h (17%) [1003].

m.p. 126° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2,3-Dimethyl-5-nitrophenyl)(4-hydroxyphenyl)methanone

[110969-52-3] $C_{15}H_{13}NO_4$ mol.wt. 271.27



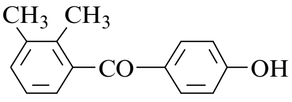
Synthesis

– Preparation by Fries rearrangement of phenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride without solvent at 160° for 2 h (55%) [1003].

m.p. 212° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2,3-Dimethylphenyl)(4-hydroxyphenyl)methanone

[134994-27-7] $C_{15}H_{14}O_2$ mol.wt. 226.27



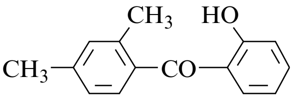
Synthesis

– Refer to: [513].

m.p. and Spectra (NA).

(2,4-Dimethylphenyl)(2-hydroxyphenyl)methanone

[143824-87-7] $C_{15}H_{14}O_2$ mol.wt. 226.27



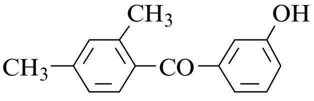
Synthesis

– Preparation by reaction of salicylic acid with m-xylene in hydrofluoric acid at 60° for 4 h in an autoclave (79%) [1004].

m.p. and Spectra (NA).

(2,4-Dimethylphenyl)(3-hydroxyphenyl)methanone

[74167-90-1] $C_{15}H_{14}O_2$ mol.wt. 226.27



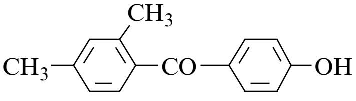
Syntheses

- Preparation by reaction of m-anisoyl chloride with m-xylene in the presence of excess aluminium chloride at first at 20°, then at reflux for 2 h (79%) [119,899].
- Preparation by Friedel–Crafts acylation of m-xylene with m-nitrobenzoyl chloride, reduction of the 2,4-dimethyl-3′-nitrobenzophenone so obtained and diazotization of the 3′-amino-2,4-dimethylbenzophenone formed, followed by hydrolysis of the diazonium salt [900].
- Also refer to: [912].

m.p. 116° [119,899]; Spectra (NA).

(2,4-Dimethylphenyl)(4-hydroxyphenyl)methanone

[116173-30-9] $C_{15}H_{14}O_2$ mol.wt. 226.27



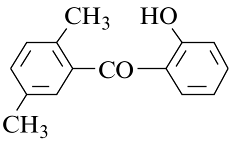
Synthesis

- Obtained by reaction of EKONOL(TM), an aromatic polyester as Friedel–Crafts reagent, with m-xylene in triflic acid solution at 25° for 18 h (90%) [922]. Similar results can be obtained using hydrofluoric acid/boron trifluoride or aluminium chloride in place of triflic acid [922].
- Also refer to: [997] (Japanese patent).

m.p. (NA); ^{13}C NMR [922], MS [922]; HPLC [922].

(2,5-Dimethylphenyl)(2-hydroxyphenyl)methanone

$C_{15}H_{14}O_2$ mol.wt. 226.27



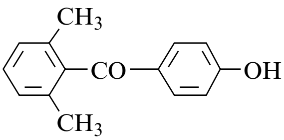
Synthesis

- Obtained by reaction of salicylic acid with p-xylene in hydrofluoric acid at 60° for 4 h in an autoclave (18%) [1004].

m.p. and Spectra (NA).

(2,6-Dimethylphenyl)(4-hydroxyphenyl)methanone

[61002-55-9] $C_{15}H_{14}O_2$ mol.wt. 226.27



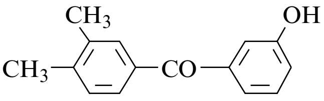
Synthesis

- Preparation by Friedel–Crafts acylation of anisole with 2,6-dimethylbenzoyl chloride in the presence of aluminium chloride [902].

m.p. 155° [902]; Spectra (NA).

(3,4-Dimethylphenyl)(3-hydroxyphenyl)methanone

[62810-57-5] $C_{15}H_{14}O_2$ mol.wt. 226.27



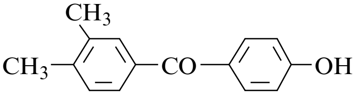
Synthesis

- Preparation by diazotization of 3′-amino-3,4-dimethyl-benzophenone followed by hydrolysis of the diazonium salt so obtained [900].

m.p. 116° [900]; Spectra (NA).

(3,4-Dimethylphenyl)(4-hydroxyphenyl)methanone

$C_{15}H_{14}O_2$ mol.wt. 226.27



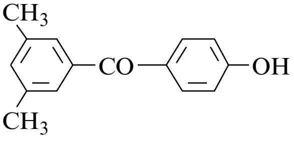
Syntheses

- Preparation by reaction of 3,4-dimethylbenzoyl chloride with phenol in the presence of aluminium chloride [165].
- Preparation by Friedel–Crafts acylation of o-xylene with p-anisoyl chloride in the presence of aluminium chloride in a boiling water bath for 1 h (major product, 40% yield) [1005].
- Preparation by demethylation of 3,4-dimethyl-4′-methoxybenzophenone with boiling pyridinium chloride (80%) [1005].

m.p. 131° [1005]; b.p.$_{17}$ 265–266° [1005]; Spectra (NA).

(3,5-Dimethylphenyl)(4-hydroxyphenyl)methanone

$C_{15}H_{14}O_2$ mol.wt. 226.27



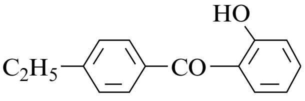
Synthesis

- Preparation by Friedel–Crafts acylation of anisole with 3,5-dimethylbenzoyl chloride, followed by demethylation of the resulting 4′-methoxy-3,5-dimethylbenzophenone with pyridinium bromide [429].

m.p. and Spectra (NA).

(4-Ethylphenyl)(2-hydroxyphenyl)methanone

[82520-51-2] $C_{15}H_{14}O_2$ mol.wt. 226.27



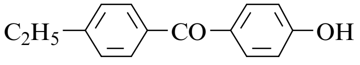
Synthesis

- Obtained (by-product) by action of o-anisoyl chloride with ethylbenzene [5],
  - in the presence of aluminium chloride between 25° and 60° for 2 h;
  - in the presence of ferric chloride at 130–140° for 5 h.

b.p.$_7$ 165–167° [5]; d_4^{20}= 1.1203 [5]; n_D^{20}= 1.6072 [5]; Spectra (NA).

(4-Ethylphenyl)(4-hydroxyphenyl)methanone

[83888-76-0] $C_{15}H_{14}O_2$ mol.wt. 226.27



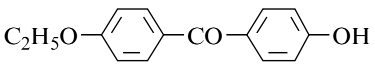
Synthesis

- Preparation by reaction of p-ethylbenzoyl chloride with phenol in the presence of aluminium chloride [165].

m.p. 99–100° [165]; Spectra (NA).

(4-Ethoxyphenyl)(4-hydroxyphenyl)methanone

[13380-65-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



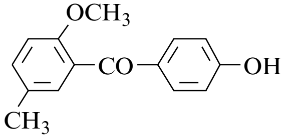
Synthesis

- Obtained (by-product) by action of ethyl iodide with 4,4′-dihydroxybenzophenone in the presence of potassium hydroxide in refluxing ethanol for 3 h [323].
- Also refer to: [1006–1009].

m.p. 146–147° [323]; Spectra (NA).

(4-Hydroxyphenyl)(2-methoxy-5-methylphenyl)methanone

$C_{15}H_{14}O_3$ mol.wt. 242.27



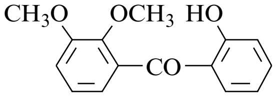
Synthesis

- Refer to: [683].

m.p. 160° [683]; Spectra (NA).

(2,3-Dimethoxyphenyl)(2-hydroxyphenyl)methanone

[129103-87-3] $C_{15}H_{14}O_4$ mol.wt. 258.27



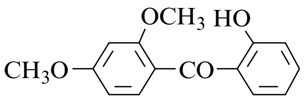
Synthesis

- Preparation from 2-iodophenyl 2,3-dimethoxybenzoate by rearrangement with n-butyllithium in a mixture of ethyl ether, hexane and tetrahydrofuran at −70° for 2 h, then treatment with saturated aqueous ammonium chloride (quantitative yield) [58].

m.p. 75–77° [58]; ^{1}H NMR [58], MS [58].

(2,4-Dimethoxyphenyl)(2-hydroxyphenyl)methanone

[108475-95-2] $C_{15}H_{14}O_4$ mol.wt. 258.27



Synthesis

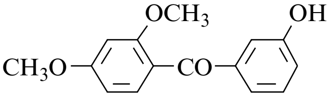
- Obtained, in mixture with 2-hydroxy-2′,4-dimethoxy-benzophenone, by reaction of

2-methoxybenzoyl chloride with resorcinol dimethyl ether in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (total yield: 65%) [1010,1011].

– Also refer to: [684].

m.p. (NA); ^{1}H NMR [1010,1011].

(2,4-Dimethoxyphenyl)(3-hydroxyphenyl)methanone



$C_{15}H_{14}O_4$ mol.wt. 258.27

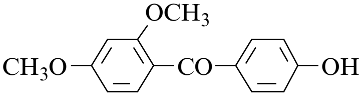
Synthesis

– Preparation by saponification of 3′-(acetyloxy)-2,4-di-methoxybenzophenone (SM) with sodium hydroxide in refluxing dilute ethanol for 30 min [990]. SM was obtained by Friedel–Crafts acylation of resorcinol dimethyl ether with m-acetoxybenzoyl chloride in the presence of aluminium chloride in carbon disulfide at 0° for 20 h (10%).

m.p. 163° [990]; Spectra (NA).

(2,4-Dimethoxyphenyl)(4-hydroxyphenyl)methanone

[41351-30-8]



$C_{15}H_{14}O_4$ mol.wt. 258.27

Syntheses

– Preparation by Friedel–Crafts acylation of resorcinol dimethyl ether with p-hydroxybenzoic acid,

- in the presence of zinc chloride and phosphorous oxychloride at 60–65° for 1.5 h (71%) [1012] or in nitrobenzene at 60° for 2–3 h [1013], (64%) [1014], according to the method [626];
- in the presence of polyphosphoric acid [429].

– Also obtained [610] according to the method of [1015].

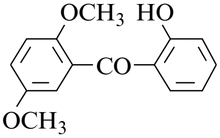
– Also refer to: [592,625,1016,1017].

N.B.: Cs salt [1013,1017].

m.p. 138–139° [1014], 135–137° [1013], 134–136° [1012];
^{1}H NMR [1012–1014], ^{13}C NMR [1014],
IR [1012–1014]; TLC [1012].

(2,5-Dimethoxyphenyl)(2-hydroxyphenyl)methanone

[183106-14-1]



$C_{15}H_{14}O_4$ mol.wt. 258.27

Synthesis

– Obtained by partial demethylation of 2,2′,5-trimethoxy-benzophenone,

- with boron tribromide in methylene chloride at 0° for 1.5 h (50%) [395];

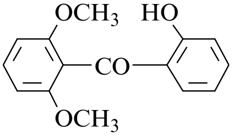
- with boron trichloride in methylene chloride at 0° for 3 h (50%) [395];
- with boron trifluoride-etherate in refluxing benzene for 6 h (40%) or in refluxing toluene for 4 h (40%) [395];
- with beryllium chloride in refluxing benzene for 8–10 h (60%) or in refluxing toluene for 5 h (60%) [395].

N.B.: In these experiments, only the reactions using boron halides were carried out under nitrogen atmosphere.

m.p. 98–100° [395]; ^{1}H NMR [395], IR [395], UV [395], MS [395].

(2,6-Dimethoxyphenyl)(2-hydroxyphenyl)methanone

[129103-86-2] $C_{15}H_{14}O_4$ mol.wt. 258.27



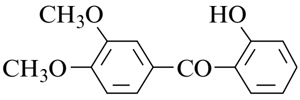
Synthesis

– Preparation from 2-iodophenyl 2,6-dimethoxybenzoate by rearrangement with n-butyllithium in a mixture of ethyl ether, hexane and tetrahydrofuran at −70° for 2 h, then treatment with saturated aqueous ammonium chloride (quantitative yield) [58].

m.p. 120°5–121°5 [58]; ^{1}H NMR [58], IR [58], MS [58].

(3,4-Dimethoxyphenyl)(2-hydroxyphenyl)methanone

[183106-12-9] $C_{15}H_{14}O_4$ mol.wt. 258.27



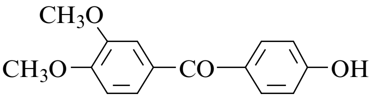
Synthesis

– Preparation by selective demethylation of 2′,3,4-tri-methoxybenzophenone with excess beryllium chloride in refluxing toluene for 3.5 h (92%) [395].

m.p. 76–78° [395]; ^{1}H NMR [395], IR [395], UV [395], MS [395]; TLC [395].

(3,4-Dimethoxyphenyl)(4-hydroxyphenyl)methanone

[26955-00-0] $C_{15}H_{14}O_4$ mol.wt. 258.27



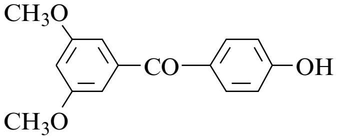
Synthesis

– Obtained by condensation of veratric acid with phenol [1018].

– Also refer to: [1019].

m.p. 166–167° [1018]; Spectra (NA).

(3,5-Dimethoxyphenyl)(4-hydroxyphenyl)methanone



$C_{15}H_{14}O_4$ mol.wt. 258.27

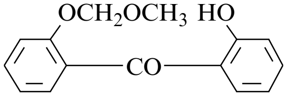
Synthesis

- Preparation by Friedel–Crafts acylation of phenol with 3,5-dimethoxybenzoyl chloride [429].

m.p. and Spectra (NA).

(2-Hydroxyphenyl)[2-(methoxymethoxy)phenyl]methanone

[59410-99-0]



$C_{15}H_{14}O_4$ mol.wt. 258.27

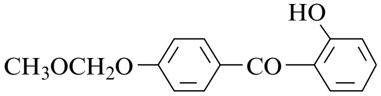
Synthesis

- Preparation by reaction of dimethoxymethane with 2,2′-di-hydroxybenzophenone in the presence of p-toluenesulfonic acid and a “Linde” type 3 Å molecular sieve in refluxing methylene chloride overnight under nitrogen (69%) [1020].

m.p. 43°5 [1020]; ^{1}H NMR [1020].

(2-Hydroxyphenyl)[4-(methoxymethoxy)phenyl]methanone

[31772-30-2]



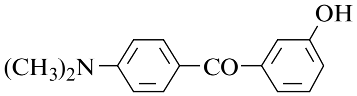
$C_{15}H_{14}O_4$ mol.wt. 258.27

Synthesis

- Preparation by reaction of chloromethyl methyl ether with 2,4′-dihydroxybenzophenone monosodium salt (4′) in toluene at r.t. overnight (64%) [1021].

oil [1021]; b.p. (NA); IR [1021].

[4-(Dimethylamino)phenyl](3-hydroxyphenyl)methanone



$C_{15}H_{15}NO_2$ mol.wt. 241.29

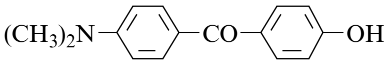
Synthesis

- Preparation by heating 3-methoxycarbonyloxybenzoic acid anilide and N,N-dimethylaniline in the presence of phosphorous oxychloride on a water bath for 4 h (50%) [1022].

m.p. 185–187° [1022]; Spectra (NA).

[4-(Dimethylamino)phenyl](4-hydroxyphenyl)methanone

[102827-03-2]



$C_{15}H_{15}NO_2$ mol.wt. 241.29

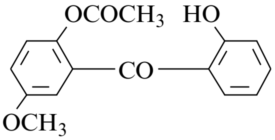
Syntheses

- Preparation by demethylation of 4-(dimethylamino)-4′-methoxybenzophenone (SM) with aluminiumbromide in refluxing benzene for 4 h (95%) [18]. SM was obtained by condensation of N-(p-anisoyl)aniline with dimethylaniline [1023].
- Preparation by heating p-methylcarbonatobenzanilide and N,N-dimethylaniline in the presence of phosphorous oxychloride on a water bath for 4 h (60%) [1022].
- Also obtained by treating benzophenone-4,4′-bis(trimethylammonium chloride) with sodium methoxide in refluxing methanol for 3 h [1024].

m.p. 200° [18], 199–200° [1022,1023], 198–200° [1024]; Spectra (NA).

[2-(Acetyloxy)-5-methoxyphenyl](2-hydroxyphenyl)methanone

[83570-59-6]



$C_{16}H_{14}O_5$ mol.wt. 286.28

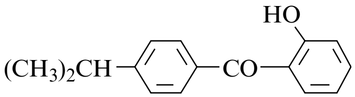
Synthesis

- Obtained by adding silica gel to a solution of 2′-acetoxy-2-hydroxy-5-methoxybenzophenone in ethyl ether, then elimination of the solvent, and the resulting powder allowed to stand at r.t. during 42 h (75%) [1025]. There is a transacylation on silica gel.

m.p. 70–73° [1025]; ^{1}H NMR [1025], IR [1025].

(2-Hydroxyphenyl)[4-(1-methylethyl)phenyl]methanone

[35839-45-3]



$C_{16}H_{16}O_2$ mol.wt. 240.30

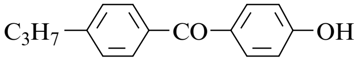
Synthesis

- Preparation by reaction of salicyloyl chloride with cumene in nitrobenzene in the presence of aluminium chloride, first between 4° and 8° for 50 min, then at 40° for 3 h and at r.t. overnight (60%) [889,891].
- Also refer to: [890].

b.p.$_{2.25}$ 165–167° [889,891]; Spectra (NA).

(4-Hydroxyphenyl)(4-propylphenyl)methanone

[64357-90-0] $C_{16}H_{16}O_2$ mol.wt. 240.30



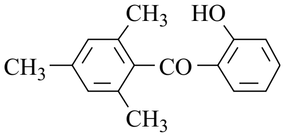
Synthesis

- Preparation by Friedel–Crafts acylation of phenol with p-propylbenzoyl chloride [175].

m.p. (NA); IR [174,175].

(2-Hydroxyphenyl)(2,4,6-trimethylphenyl)methanone

[46863-20-1] $C_{16}H_{16}O_2$ mol.wt. 240.30



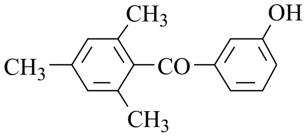
Syntheses

- Obtained by photo-Fries rearrangement of phenyl mesitoate(phenyl 2,4,6-trimethylbenzoate),
  - in methanol (39%) or in methanol in the presence of β-cyclodextrin (99%) [65];
  - in pentane (19%) [64];
  - in benzene at 40° for 332 h (9%) or in hexane at 40° for 109 h (6%) [72].
- Also obtained from mesityl o-methoxyphenyl ketone (2′-methoxy-2,4,6-trimethylbenzophenone) by cleavage of methoxy group with hydriodic acid or with a binary mixture prepared from magnesium and iodine in refluxing toluene/butyl ether solution (poor yield) [1026].
- Preparation by reaction of 2-methoxybenzoyl chloride with mesitylene in the presence of aluminium chloride in benzene (65%) [102].

m.p. 94–95° [102], 82° [94,95], 81–83° [72], 81–82° [1026];
^{1}H NMR [94,95,101,102], ^{13}C NMR [101],
IR [94,95,102,1026]; thermal behaviour [94,95].

(3-Hydroxyphenyl)(2,4,6-trimethylphenyl)methanone

[76981-50-5] $C_{16}H_{16}O_2$ mol.wt. 240.30



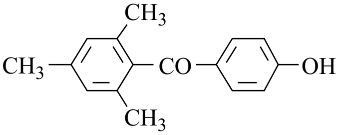
Syntheses

- Preparation by reaction of m-anisoyl chloride with mesitylene (1,3,5-trimethylbenzene) in the presence of an excess of aluminium chloride: first at 20°, then at reflux for 2 h (82%) [119].
- Preparation by Friedel–Crafts acylation of mesitylene with m-anisoyl chloride, followed by demethylation of the 3′-methoxy-2,4,6-trimethylbenzophenone so obtained with pyridinium bromide [429].

m.p. 130° [119]; Spectra (NA).

(4-Hydroxyphenyl)(2,4,6-trimethylphenyl)methanone

[2004-55-9] $C_{16}H_{16}O_2$ mol.wt. 240.30



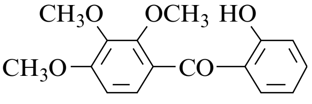
Syntheses

- Preparation by Friedel–Crafts acylation of phenol with mesitoyl chloride in the presence of aluminium chloride in carbon disulfide [174,175].
- Also obtained by photo-Fries rearrangement of phenyl mesitoate (phenyl 2,4,6-trimethylbenzoate),
  - in methanol (46%) [65];
  - in pentane (24%) [64];
  - in benzene at 40° for 332 h (12%) [72];
  - in hexane at 40° for 109 h (11%) [72].

m.p. 167–168° [72]; ^{1}H NMR [72], IR [72,174,175], UV [72].

(2-Hydroxyphenyl)(2,3,4-trimethoxyphenyl)methanone

[147188-09-8] $C_{16}H_{16}O_5$ mol.wt. 288.30



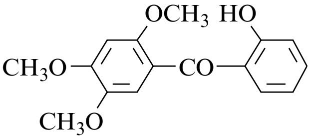
Synthesis

- Obtained, in mixture with 2-hydroxy-2′,3,4-trimethoxy-benzophenone, by reaction of 2-methoxybenzoyl chloride with pyrogallol trimethyl ether in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (total yield: 56–59%) [1010,1011].
- Also refer to: [1027].

m.p. (NA); ^{1}H NMR [1010].

(2-Hydroxyphenyl)(2,4,5-trimethoxyphenyl)methanone

[147188-07-6] $C_{16}H_{16}O_5$ mol.wt. 288.30



Synthesis

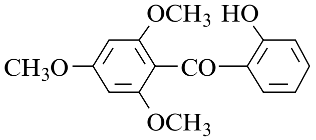
- Obtained, in mixture with 2-hydroxy-2′,4,5-trimethoxy-benzophenone, by reaction of 2-methoxybenzoyl chloride with 1,2,4-trimethoxybenzene in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (total yield: 56%) [1010].
- Also refer to: [1011].

m.p. (NA); ^{1}H NMR [1010].

(2-Hydroxyphenyl)(2,4,6-trimethoxyphenyl)methanone

[147188-05-4]

$C_{16}H_{16}O_5$ mol.wt. 288.30



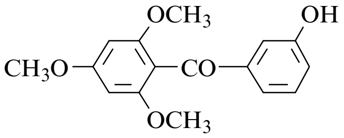
Synthesis

- Obtained, in mixture with 2-hydroxy-2′,4,6-trimethoxy-benzophenone, by reaction of 2-methoxybenzoyl chloride with phloroglucinol trimethyl ether in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (total yield: 59%) [1010].
- Also refer to: [1011].

m.p. (NA); ^{1}H NMR [1010].

(3-Hydroxyphenyl)(2,4,6-trimethoxyphenyl)methanone

$C_{16}H_{16}O_5$ mol.wt. 288.30



Synthesis

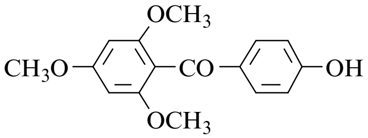
- Preparation by reaction of m-hydroxybenzonitrile with phloroglucinol trimethyl ether (Hoesch reaction) [429].

m.p. and Spectra (NA).

(4-Hydroxyphenyl)(2,4,6-trimethoxyphenyl)methanone

[41351-32-0]

$C_{16}H_{16}O_5$ mol.wt. 288.30



Synthesis

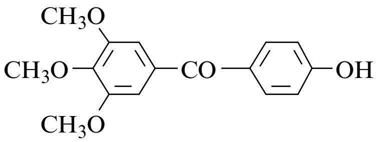
- Refer to: [429].

m.p. and Spectra (NA).

(4-Hydroxyphenyl)(3,4,5-trimethoxyphenyl)methanone

[14938-63-7]

$C_{16}H_{16}O_5$ mol.wt. 288.30



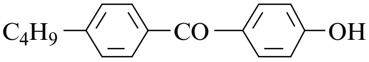
Synthesis

- Preparation by Friedel–Crafts acylation of phenol with 3,4,5-trimethoxybenzoyl chloride in nitrobenzene in the presence of aluminium chloride, first at 10°, then for 24 h at r.t. (45%) [1028].

m.p. 120° [1028]; Spectra (NA).

(4-Butylphenyl)(4-hydroxyphenyl)methanone

[190728-33-7]



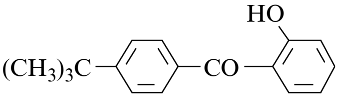
$C_{17}H_{18}O_2$ mol.wt. 254.33

Synthesis

- Refer to: Chem. Abstr., **127**, 34137f (1997).

m.p. and Spectra (NA).

[4-(1,1-Dimethylethyl)phenyl](2-hydroxyphenyl)methanone



$C_{17}H_{18}O_2$ mol.wt. 254.33

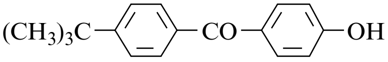
Synthesis

- Refer to: [84].

m.p. and Spectra (NA).

[4-(1,1-Dimethylethyl)phenyl](4-hydroxyphenyl)methanone

[55044-96-7]



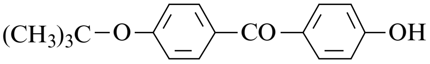
$C_{17}H_{18}O_2$ mol.wt. 254.33

Synthesis

- Preparation by reaction of p-tert-butylbenzoyl chloride with phenol in the presence of aluminium chloride [165].

m.p. 130–131° [165]; Spectra (NA).

[4-(1,1-Dimethylethoxy)phenyl](4-hydroxyphenyl)methanone



$C_{17}H_{18}O_3$ mol.wt. 270.33

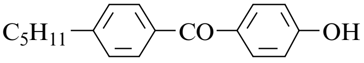
Synthesis

- Obtained by decomposition of the benzophenone-4,4′-bis(trimethylammonium chloride) with sodium tert-butoxide in refluxing alcohol for 3 h [1024].

m.p. 85° [1024]; Spectra (NA).

(4-Hydroxyphenyl)(4-pentylphenyl)methanone

[64357-91-1]



$C_{18}H_{20}O_2$ mol.wt. 268.36

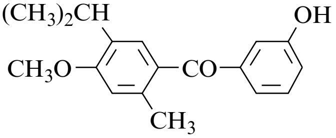
Synthesis

- Preparation by reaction of 4-pentylbenzoyl chloride with phenol in the presence of aluminium chloride in carbon disulfide [174,175].

m.p. (NA); IR [174,175].

(3-Hydroxyphenyl)[4-methoxy-2-methyl-5-(1-methylethyl)phenyl]methanone

[109250-48-8] $C_{18}H_{20}O_3$ mol.wt. 284.36



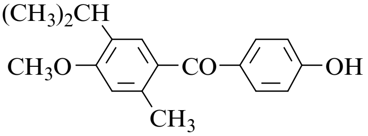
Synthesis

– Obtained by saponification of 3′-acetoxy-4-methoxy-2-methyl-5-isopropylbenzophenone (SM) with sodium hydroxide in dilute ethanol at reflux for 30 min (73%). SM was prepared by Friedel–Crafts acylation of thymol methyl ether with m-acetoxybenzoyl chloride in the presence of aluminium chloride in carbon disulfide at 0° for 20 h (12%) [990].

m.p. 106° [990]; Spectra (NA).

(4-Hydroxyphenyl)[4-methoxy-2-methyl-5-(1-methylethyl)phenyl]methanone

[109250-49-9] $C_{18}H_{20}O_3$ mol.wt. 284.36



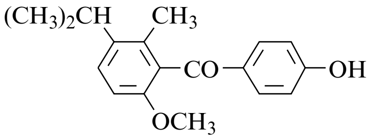
Synthesis

– Obtained by saponification of 4′-acetoxy-4-methoxy-2-methyl-5-isopropylbenzophenone (SM) with sodium hydroxide in dilute ethanol at reflux for 30 min (80%). SM was prepared by Friedel–Crafts acylation of thymol methyl ether with p-acetoxy-benzoyl chloride in the presence of aluminium chloride in carbon disulfide at 15° for 3 h (17%) [990].

b.p.$_{16}$ 265–268° [990]; m.p. 65° [990]; Spectra (NA).

(4-Hydroxyphenyl)[6-methoxy-2-methyl-3-(1-methylethyl)phenyl]methanone

[109250-50-2] $C_{18}H_{20}O_3$ mol.wt. 284.36



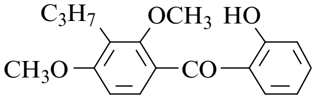
Synthesis

– Obtained by saponification of 4′-acetoxy-6-methoxy-2-methyl-3-isopropylbenzophenone (SM) with sodium hydroxide in dilute ethanol at reflux for 30 min (79%). SM was prepared by Friedel–Crafts acylation of p-thymol methyl ether (3-methyl-4-isopropylanisole) with p-acetoxybenzoyl chloride in the presence of aluminium chloride in carbon disulfide at 15° for 3 h (7%) [990].

b.p.$_{0.3}$ 212–213° [990]; Spectra (NA).

(2,4-Dimethoxy-3-propylphenyl)(2-hydroxyphenyl)methanone

[115296-03-2] $C_{18}H_{20}O_4$ mol.wt. 300.35



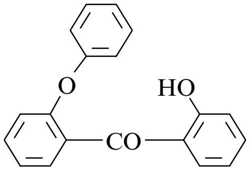
Synthesis

– Obtained (poor yield) by reaction of o-anisoyl chloride with 1,3-dimethoxy-2-propylbenzene in methylene chloride in the presence of aluminium chloride, first at 0° for 2 h then at r.t. for 1 h (13%) [1029].

oil [1029]; b.p. and Spectra (NA).

(2-Hydroxyphenyl)(2-phenoxyphenyl)methanone

[194548-68-0] $C_{19}H_{14}O_3$ mol.wt. 290.32



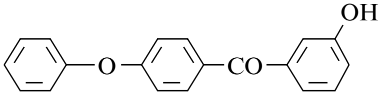
Synthesis

– Refer to: Chem. Abstr., **127**, 205428h (1997).

m.p. and Spectra (NA).

(3-Hydroxyphenyl)(4-phenoxyphenyl)methanone

[76981-53-8] $C_{19}H_{14}O_3$ mol.wt. 290.32



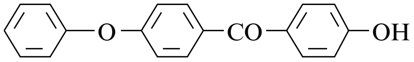
Synthesis

– Preparation by reaction of m-anisoyl chloride with diphenyl oxide in the presence of an excess of aluminium chloride: first at 20°, then at reflux for 2 h (46%) [119].

m.p. 142° [119]; Spectra (NA).

(4-Hydroxyphenyl)(4-phenoxyphenyl)methanone

[78930-16-2] $C_{19}H_{14}O_3$ mol.wt. 290.32



Synthesis

– Preparation from 4-fluoro-4′-phenoxy-benzophenone by reaction under nitrogen with potassium hydroxide in aqueous dimethyl sulfoxide at 60° for 18 h [170].

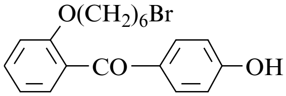
– Also refer to: [943].

N.B.: K salt [170].

m.p. 143° [170]; Spectra (NA).

[2-[(6-Bromohexyl)oxy]phenyl](4-hydroxyphenyl)methanone

[31772-32-4] $C_{19}H_{21}BrO_3$ mol.wt. 377.28

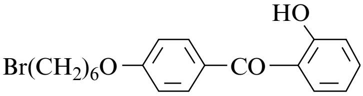


Synthesis

– Preparation by heating 2-(6-bromohexyloxy)-4′-methoxy-benzophenone in refluxing aqueous acetic acid in the presence of a few concentrated sulfuric acid for 15 min (95%) [1021].

oil [1021]; b.p. (NA); IR [1021].

[4-[(6-Bromohexyl)oxy]phenyl](2-hydroxyphenyl)methanone



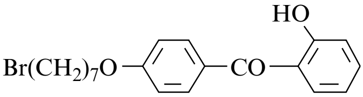
$C_{19}H_{21}BrO_3$ mol.wt. 377.28

Synthesis

- Preparation by reaction of 1,6-dibromohexane with 2,4′-dihydroxybenzophenone in ethanol in the presence of potassium hydroxide, first at r.t. for 1 h then at reflux for 40 min (63%) [1021].

yellow oil [1021]; b.p. (NA); IR [1021].

[4-[(7-Bromoheptyl)oxy]phenyl](2-hydroxyphenyl)methanone



$C_{20}H_{23}BrO_3$ mol.wt. 391.30

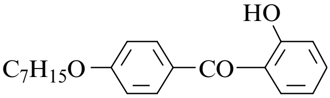
Synthesis

- Preparation by reaction of 1,7-dibromoheptane with 2,4′-dihydroxybenzophenone in ethanol in the presence of potassium hydroxide, first at r.t. for 1 h then at reflux for 40 min [1021].

yellow oil [1021]; b.p. (NA); IR [1021].

[4-(Heptyloxy)phenyl](2-hydroxyphenyl)methanone

[33213-89-7]



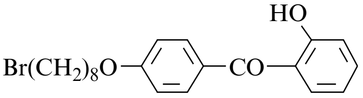
$C_{20}H_{24}O_3$ mol.wt. 312.41

Synthesis

- Refer to: [852].

m.p. and Spectra (NA).

[4-[(8-Bromooctyl)oxy]phenyl](2-hydroxyphenyl)methanone



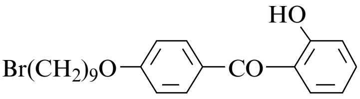
$C_{21}H_{25}BrO_3$ mol.wt. 405.33

Synthesis

- Preparation by reaction of 1,8-dibromooctane with 2,4′-dihydroxybenzophenone in ethanol in the presence of potassium hydroxide, first at r.t. for 1 h, then at reflux for 40 min (57%) [1021].

yellow oil [1021]; b.p. (NA); IR [1021].

[4-[(9-Bromononyl)oxy]phenyl](2-hydroxyphenyl)methanone



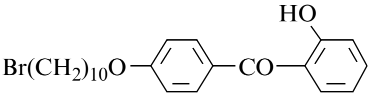
$C_{22}H_{27}BrO_3$ mol.wt. 419.36

Synthesis

- Preparation by reaction of 1,9-dibromononane with 2,4′-dihydroxybenzophenone in ethanol in the presence of potassium hydroxide, first at r.t. for 1 h, then at reflux for 40 min (74%) [1021].

yellow oil [1021]; b.p. (NA); IR [1021].

[4-[(10-Bromodecyl)oxy]phenyl](2-hydroxyphenyl)methanone



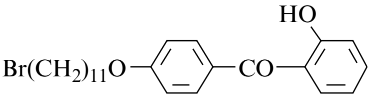
$C_{23}H_{29}BrO_3$ mol.wt. 433.39

Synthesis

- Preparation by reaction of 1,10-dibromodecane with 2,4′-dihydroxybenzophenone in ethanol in the presence of potassium hydroxide, first at r.t. for 1 h, then at reflux for 40 min (68%) [1021].

yellow oil [1021]; b.p.$_5$ 138–141° [1021]; IR [1021].

[4-[(11-Bromoundecyl)oxy]phenyl](2-hydroxyphenyl)methanone



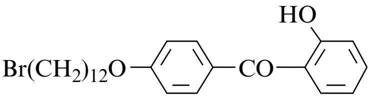
$C_{24}H_{31}BrO_3$ mol.wt. 447.41

Synthesis

- Preparation by reaction of 1,11-dibromoundecane with 2,4′-dihydroxybenzophenone in ethanol in the presence of potassium hydroxide, first at r.t. for 1 h, then at reflux for 40 min (quantitative yield) [1021].

yellow oil [1021]; b.p. (NA); IR [1021].

[4-[(12-Bromododecyl)oxy]phenyl](2-hydroxyphenyl)methanone



$C_{25}H_{33}BrO_3$ mol.wt. 461.44

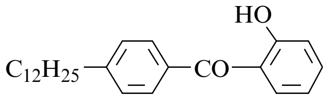
Synthesis

- Preparation by reaction of 1,12-dibromododecane with 2,4′-dihydroxybenzophenone in ethanol in the presence of potassium hydroxide, first at r.t. for 1 h, then at reflux for 40 min (46%) [1021].

yellow crystals [1021]; m.p. (NA); IR [1021].

(4-Dodecylphenyl)(2-hydroxyphenyl)methanone

[35698-22-7] $C_{25}H_{34}O_2$ mol.wt. 366.54



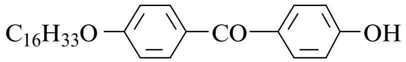
Synthesis

- Obtained by reaction of 2-hydroxybenzoyl chloride with dodecylbenzene in nitrobenzene in the presence of aluminium chloride for 4 h at 40° then 16 h at r.t. (16%) [889,891].

b.p.$_{0.1}$ 175–195° [889,891]; Spectra (NA).

[4-(Hexadecyloxy)phenyl](4-hydroxyphenyl)methanone

[129727-61-3] $C_{29}H_{42}O_3$ mol.wt. 438.65



Synthesis

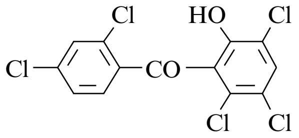
- Refer to: [1030] (Japanese patent).

m.p. and Spectra (NA).

2.1.3 Substituents Located on Both Rings

(2,4-Dichlorophenyl)(2,3,5-trichloro-6-hydroxyphenyl)methanone

[34171-61-4] $C_{13}H_5Cl_5O_2$ mol.wt. 370.45



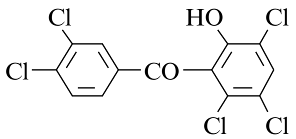
Synthesis

- Preparation by Fries rearrangement of 2,4,5-trichlorophenyl 2,4-dichlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 81–82° [458]; Spectra (NA).

(3,4-Dichlorophenyl)(2,3,5-trichloro-6-hydroxyphenyl)methanone

[34171-60-3] $C_{13}H_5Cl_5O_2$ mol.wt. 370.45



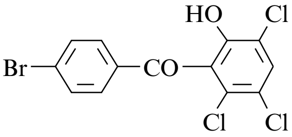
Synthesis

- Preparation by Fries rearrangement of 2,4,5-trichlorophenyl 3,4-dichlorobenzoate with aluminium chloride for
- 30 min at 150–160° [458].

m.p. 217–218° [458]; Spectra (NA).

(4-Bromophenyl)(2,3,5-trichloro-6-hydroxyphenyl)methanone

[34171-63-6] $C_{13}H_6BrCl_3O_2$ mol.wt. 380.45



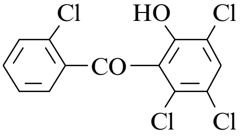
Synthesis

- Preparation by Fries rearrangement of 2,4,5-trichlorophenyl p-bromobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 185–186° [458]; Spectra (NA).

(2-Chlorophenyl)(2,3,5-trichloro-6-hydroxyphenyl)methanone

[34174-12-4] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



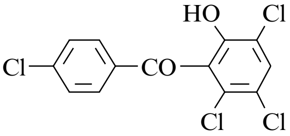
Synthesis

- Preparation by Fries rearrangement of 2,4,5-trichlorophenyl o-chlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 94–95° [458]; Spectra (NA).

(4-Chlorophenyl)(2,3,5-trichloro-6-hydroxyphenyl)methanone

[34171-59-0] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



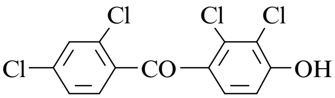
Synthesis

- Preparation by Fries rearrangement of 2,4,5-trichlorophenyl p-chlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 176–177° [458]; Spectra (NA).

(2,3-Dichloro-4-hydroxyphenyl)(2,4-dichlorophenyl)methanone

[34183-05-6] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



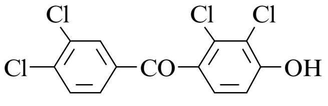
Synthesis

- Obtained by Fries rearrangement of 2,3-dichlorophenyl 2,4-dichlorobenzoate with aluminium chloride in chlorobenzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 164–165° [480]; Spectra (NA).

(2,3-Dichloro-4-hydroxyphenyl)(3,4-dichlorophenyl)methanone

[72482-75-8] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



Synthesis

– Preparation by demethylation of 2,3,3′,4′-tetrachloro-4-methoxybenzophenone (SM) with aluminium chloride,

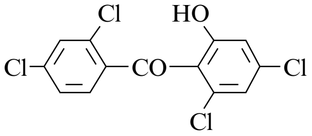
- in refluxing methylene chloride for overnight [475];
- in refluxing benzene for 5 h, then at r.t. for 18 h [476].

SM was obtained by Friedel–Crafts acylation of 2,3-dichloroanisole with 3,4-dichlorobenzoyl chloride in the presence of aluminium chloride in refluxing methylene chloride [475] or in ethylene dichloride at 60° for 1 h [476].

m.p. 179–180° [476]; Spectra (NA).

(2,4-Dichloro-6-hydroxyphenyl)(2,4-dichlorophenyl)methanone

[34174-05-5] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



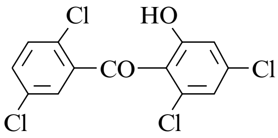
Synthesis

– Preparation by Fries rearrangement of 3,5-dichlorophenyl 2,4-dichlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 105–106° [458]; Spectra (NA).

(2,4-Dichloro-6-hydroxyphenyl)(2,5-dichlorophenyl)methanone

[34174-09-9] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



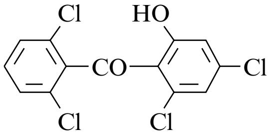
Synthesis

– Preparation by Fries rearrangement of 3,5-dichlorophenyl 2,5-dichlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 107–108° [458]; Spectra (NA).

(2,4-Dichloro-6-hydroxyphenyl)(2,6-dichlorophenyl)methanone

[34786-96-4] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



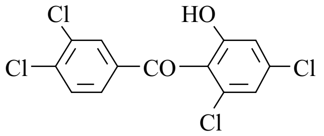
Synthesis

– Preparation by Fries rearrangement of 3,5-dichlorophenyl 2,6-dichlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 110–111° [458]; Spectra (NA).

(2,4-Dichloro-6-hydroxyphenyl)(3,4-dichlorophenyl)methanone

[31656-23-2] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



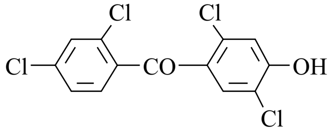
Synthesis

- Preparation by Fries rearrangement of 3,5-dichlorophenyl 3,4-dichlorobenzoate with aluminium chloride first 10 min at 140–150°, then for 30 min at 150–160° (80%) [458].

m.p. 138–139° [458]; Spectra (NA).

(2,5-Dichloro-4-hydroxyphenyl)(2,4-dichlorophenyl)methanone

[34183-04-5] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



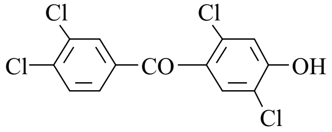
Synthesis

- Obtained by Fries rearrangement of 2,5-dichlorophenyl 2,4-dichlorobenzoate with aluminium chloride in chloro-benzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 147–148° [480]; Spectra (NA).

(2,5-Dichloro-4-hydroxyphenyl)(3,4-dichlorophenyl)methanone

[34183-03-4] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



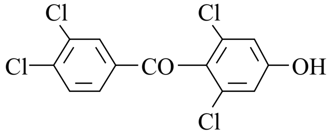
Synthesis

- Obtained by Fries rearrangement of 2,5-dichlorophenyl 3,4-dichlorobenzoate with aluminium chloride for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 160–161° [480]; Spectra (NA).

(2,6-Dichloro-4-hydroxyphenyl)(3,4-dichlorophenyl)methanone

[34183-00-1] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



Synthesis

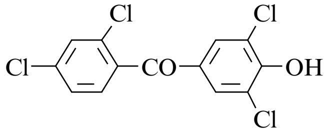
- Obtained by Fries rearrangement of 3,5-dichlorophenyl 3,4-dichlorobenzoate with aluminium chloride,

- in chlorobenzene for 20 min at 140–150° or in nitro-benzene for 24 h at 75° [480];
- without solvent, first for 10 min at 140–150°, then for 30 min at 150–160° (by-product) [458].

m.p. 180–181° [458,480]; Spectra (NA).

(3,5-Dichloro-4-hydroxyphenyl)(2,4-dichlorophenyl)methanone

[34182-98-4] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



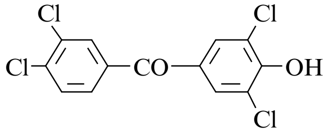
Synthesis

- Preparation by Fries rearrangement of 2,6-dichlorophenyl 2,4-dichlorobenzoate with aluminium chloride in chloro-benzene at 140–150° for 20 min or in nitrobenzene at 75° for 24 h [480].
- Also refer to: [468] (Japanese patent).

m.p. 183°5–184°5 [480]; Spectra (NA).

(3,5-Dichloro-4-hydroxyphenyl)(3,4-dichlorophenyl)methanone

[34189-57-6] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



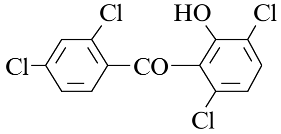
Synthesis

- Preparation by Fries rearrangement of 2,6-dichlorophenyl 3,4-dichlorobenzoate with aluminium chloride,
  - in chlorobenzene for 20 min at 140–150° (89%) [480];
  - in nitrobenzene for 24 h at 75° (89%) [480].

m.p. 202–203° [480]; Spectra (NA).

(3,6-Dichloro-2-hydroxyphenyl)(2,4-dichlorophenyl)methanone

[34171-57-8] $C_{13}H_6Cl_4O_2$ mol.wt. 336.00



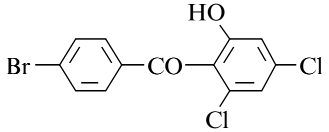
Synthesis

- Preparation by Fries rearrangement of 2,5-dichlorophenyl 2,4-dichlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 115–116° [458]; Spectra (NA).

(4-Bromophenyl)(2,4-dichloro-6-hydroxyphenyl)methanone

[34174-06-6] $C_{13}H_7BrCl_2O_2$ mol.wt. 346.01



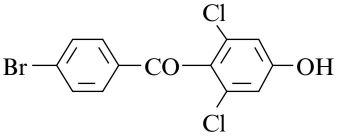
Synthesis

- Preparation by Fries rearrangement of 3,5-dichlorophenyl p-bromobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 157–158° [458]; Spectra (NA).

(4-Bromophenyl)(2,6-dichloro-4-hydroxyphenyl)methanone

[34183-12-5] $C_{13}H_7BrCl_2O_2$ mol.wt. 346.01



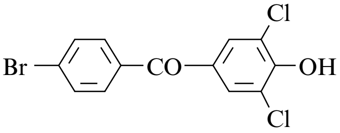
Synthesis

- Obtained by Fries rearrangement of 3,5-dichlorophenyl p-bromobenzoate with aluminium chloride in chloro-benzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 147–148° [480]; Spectra (NA).

(4-Bromophenyl)(3,5-dichloro-4-hydroxyphenyl)methanone

[34183-07-8] $C_{13}H_7BrCl_2O_2$ mol.wt. 346.01



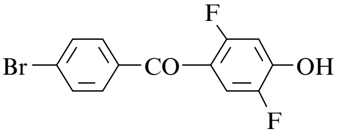
Synthesis

- Preparation by Fries rearrangement of 2,6-dichlorophenyl p-bromobenzoate with aluminium chloride in chloro-benzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 173–174° [480]; Spectra (NA).

(4-Bromophenyl)(2,5-difluoro-4-hydroxyphenyl)methanone

[192437-36-8] $C_{13}H_7BrF_2O_2$ mol.wt. 313.10



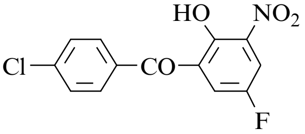
Synthesis

- Preparation by demethylation of 4′-bromo-2,5-difluoro-4-methoxybenzophenone (SM) with 62% aqueous hydrobromic acid in refluxing acetic acid for 13 h (95%). SM was obtained by Friedel–Crafts acylation of 2,5-di-fluoroanisole with 4-bromobenzoyl chloride in nitrobenzene in the presence of aluminium chloride, first at temperature <6°, then at r.t. overnight (80%).
- Refer to: Chem. Abstr., **127**, 108766j (1997).
- Also refer to: Chem. Abstr., **127**, 108921f (1997).

m.p. and Spectra (NA).

(4-Chlorophenyl)(5-fluoro-2-hydroxy-3-nitrophenyl)methanone

[85052-27-3] $C_{13}H_7ClFNO_4$ mol.wt. 295.65



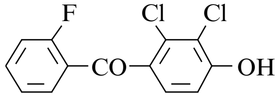
Synthesis

- Preparation by reaction of 60% nitric acid with 4′-chloro-5-fluoro-2-hydroxybenzophenone in acetic acid at r.t. for 30 min (84%) [472].

m.p. (NA); yellow crystals [472]; ^{1}H NMR [472], IR [472], MS [472]; TLC [472].

(2,3-Dichloro-4-hydroxyphenyl)(2-fluorophenyl)methanone

[72498-54-5] $C_{13}H_7Cl_2FO_2$ mol.wt. 285.10



Synthesis

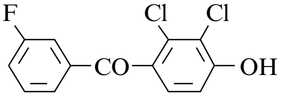
– Preparation by demethylation of 2,3-dichloro-2′-fluoro-4-methoxybenzophenone (SM) with aluminium chloride in refluxing benzene for 5 h [476], (89%) [1031] or in refluxing methylene chloride overnight [475]. SM was obtained by Friedel–Crafts acylation of 2,3-dichloroanisole with o-fluorobenzoyl chloride in the presence of aluminium chloride in refluxing methylene chloride [475] or in ethylene dichloride for 2 h [476].

– Also refer to: [1032].

m.p. 128–131° [476,1031]; Spectra (NA).

(2,3-Dichloro-4-hydroxyphenyl)(3-fluorophenyl)methanone

[72482-40-7] $C_{13}H_7Cl_2FO_2$ mol.wt. 285.10



Syntheses

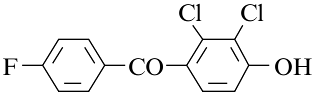
– Preparation by demethylation of 2,3-dichloro-3′-fluoro-4-methoxybenzophenone (SM) with aluminium chloride in refluxing methylene chloride overnight. SM was obtained by Friedel–Crafts acylation of 2,3-dichloroanisole with m-fluorobenzoyl chloride in the presence of aluminium chloride in refluxing methylene chloride [475].

– Preparation by reaction of m-fluorobenzoyl chloride with 2,3-dichloroanisole in the presence of aluminium chloride [476]. Here, in this process, their is simultaneously acylation and demethylation in one step.

m.p. and Spectra (NA).

(2,3-Dichloro-4-hydroxyphenyl)(4-fluorophenyl)methanone

[62967-10-6] $C_{13}H_7Cl_2FO_2$ mol.wt. 285.10



Synthesis

– Preparation by demethylation of 2,3-dichloro-4′-fluoro-4-methoxybenzophenone (SM),

- with aluminium chloride in refluxing methylene chloride overnight (72%) [475];
- with pyridinium chloride at 200° for 1 h [476].

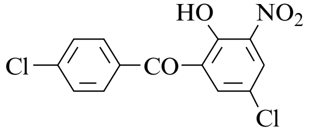
SM was obtained by Friedel–Crafts acylation of 2,3-dichloroanisole with p-fluorobenzoyl chloride in the presence of aluminium chloride in methylene chloride first at 5°, then at reflux for 2 h (50%) [475] or in ethylene dichloride at 60° for 1 h [476].

– Also refer to: [1031].

m.p. 155–159° [475]; Spectra (NA).

(5-Chloro-2-hydroxy-3-nitrophenyl)(4-chlorophenyl)methanone

[85052-24-0] $C_{13}H_7Cl_2NO_4$ mol.wt. 312.11



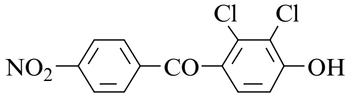
Synthesis

– Preparation by reaction of 60% nitric acid with 4′,5-di-chloro-2-hydroxybenzophenone in the presence of one drop of concentrated sulfuric acid at r.t. for 25 min (92%) [472].

m.p. (NA); crystals [472]; ^{1}H NMR [472], IR [472], MS [472]; TLC [472].

(2,3-Dichloro-4-hydroxyphenyl)(4-nitrophenyl)methanone

[92285-28-4] $C_{13}H_7Cl_2NO_4$ mol.wt. 312.11



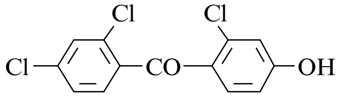
Synthesis

– Preparation by demethylation of 2,3-dichloro-4-methoxy-4′-nitrobenzophenone (SM) with aluminium chloride in refluxing ethylene dichloride for 1.5 h (95%) [979] or in refluxing methylene chloride overnight [475]. SM was obtained by Friedel–Crafts acylation of 2,3-dichloroanisole with p-nitrobenzoyl chloride in the presence of aluminium chloride in refluxing methylene chloride [475].

m.p. 201–204° [979]; Spectra (NA).

(2-Chloro-4-hydroxyphenyl)(2,4-dichlorophenyl)methanone

[34294-62-7] $C_{13}H_7Cl_3O_2$ mol.wt. 301.56



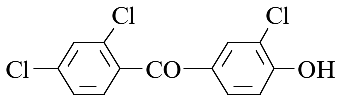
Synthesis

– Obtained by Fries rearrangement of m-chlorophenyl 2,4-dichlorobenzoate with aluminium chloride in chloro-benzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 163–164° [480]; Spectra (NA).

(3-Chloro-4-hydroxyphenyl)(2,4-dichlorophenyl)methanone

[34182-96-2] $C_{13}H_7Cl_3O_2$ mol.wt. 301.56



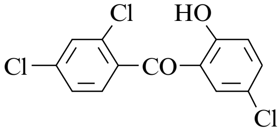
Synthesis

– Preparation by Fries rearrangement of o-chlorophenyl 2,4-dichlorobenzoate with aluminium chloride in chloro-benzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 144–145° [480]; Spectra (NA).

(5-Chloro-2-hydroxyphenyl)(2,4-dichlorophenyl)methanone

[72089-86-2] $C_{13}H_7Cl_3O_2$ mol.wt. 301.56



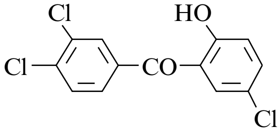
Synthesis

- Preparation by Fries rearrangement of p-chlorophenyl 2,4-dichlorobenzoate with aluminium chloride at 190° for 15 min [569].

m.p. 96–97° [569]; Spectra (NA).

(5-Chloro-2-hydroxyphenyl)(3,4-dichlorophenyl)methanone

$C_{13}H_7Cl_3O_2$ mol.wt. 301.56



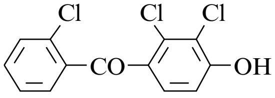
Synthesis

- Preparation by reaction of 3,4-dichlorobenzoyl chloride with p-chlorophenol in the presence of aluminium chloride, first at 100° for 3 min, then at 178–180° for 23 min (53%) [484].

m.p. 92–92°5 [484]; Spectra (NA).

(2-Chlorophenyl)(2,3-dichloro-4-hydroxyphenyl)methanone

[72482-80-5] $C_{13}H_7Cl_3O_2$ mol.wt. 301.56



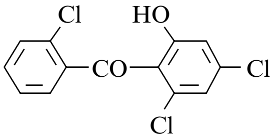
Synthesis

- Preparation by demethylation of 2,2′,3-trichloro-4-methoxybenzophenone (SM) with aluminium chloride in refluxing benzene for 5 h, then at r.t. for 18 h.
- SM was obtained by reaction of o-chlorobenzoyl chloride with 2,3-dichloroanisole in ethylene dichloride in the presence of aluminium chloride at 60° for 1 h [476].

m.p. 74–77° [476]; Spectra (NA).

(2-Chlorophenyl)(2,4-dichloro-6-hydroxyphenyl)methanone

[34174-11-3] $C_{13}H_7Cl_3O_2$ mol.wt. 301.56

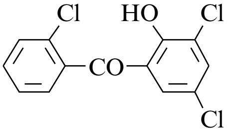


Synthesis

- Preparation by Fries rearrangement of 3,5-dichlorophenyl o-chlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 72–73° [458]; Spectra (NA).

(2-Chlorophenyl)(3,5-dichloro-2-hydroxyphenyl)methanone



$C_{13}H_7Cl_3O_2$ mol.wt. 301.56

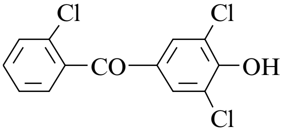
Synthesis

- Preparation by reaction of o-chlorobenzoyl chloride with 2,4-dichlorophenol in the presence of aluminium chloride at 180° [484].

m.p. 92° [484]; Spectra (NA).

(2-Chlorophenyl)(3,5-dichloro-4-hydroxyphenyl)methanone

[34183-18-1]



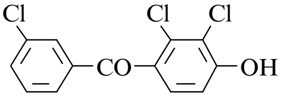
$C_{13}H_7Cl_3O_2$ mol.wt. 301.56

Synthesis

- Preparation by Fries rearrangement of 2,6-dichlorophenyl o-chlorobenzoate with aluminium chloride in chlorobenzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 162–163° [480]; Spectra (NA).

(3-Chlorophenyl)(2,3-dichloro-4-hydroxyphenyl)methanone



$C_{13}H_7Cl_3O_2$ mol.wt. 301.56

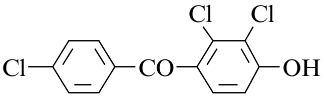
Synthesis

- Preparation by demethylation of 2,3,3′-trichloro-4-methoxy-benzophenone (SM) with aluminium chloride in refluxing methylene chloride overnight. SM was obtained by Friedel–Crafts acylation of 2,3-dichloroanisole with m-chloro-benzoyl chloride in the presence of aluminium chloride in refluxing methylene chloride [475].

m.p. and Spectra (NA).

(4-Chlorophenyl)(2,3-dichloro-4-hydroxyphenyl)methanone

[72498-76-1]



$C_{13}H_7Cl_3O_2$ mol.wt. 301.56

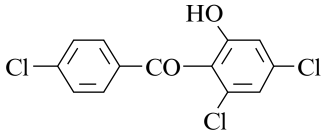
Synthesis

- Preparation by demethylation of 2,3,4′-trichloro-4-methoxybenzophenone (SM) with aluminium chloride in refluxing methylene chloride overnight. SM was obtained by Friedel–Crafts acylation of 2,3-dichloroanisole with p-chlorobenzoyl chloride in the presence of aluminium chloride in refluxing methylene chloride [475].
- Also refer to: [476].

m.p. and Spectra (NA).

(4-Chlorophenyl)(2,4-dichloro-6-hydroxyphenyl)methanone

[34171-58-9] $C_{13}H_7Cl_3O_2$ mol.wt. 301.56



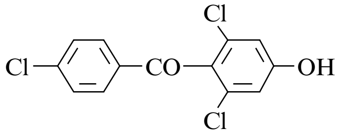
Synthesis

– Preparation by Fries rearrangement of 3,5-dichlorophenyl p-chlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 161–162° [458]; Spectra (NA).

(4-Chlorophenyl)(2,6-dichloro-4-hydroxyphenyl)methanone

[34183-11-4] $C_{13}H_7Cl_3O_2$ mol.wt. 301.56



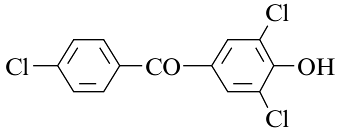
Synthesis

– Obtained by Fries rearrangement of 3,5-dichlorophenyl p-chlorobenzoate with aluminium chloride in chloro-benzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 137°5–138° [480]; Spectra (NA).

(4-Chlorophenyl)(3,5-dichloro-4-hydroxyphenyl)methanone

[34182-97-3] $C_{13}H_7Cl_3O_2$ mol.wt. 301.56



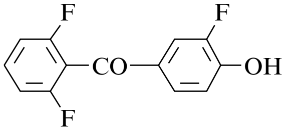
Synthesis

– Preparation by Fries rearrangement of 2,6-dichlorophenyl p-chlorobenzoate with aluminium chloride in chloro-benzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 179–180° [480]; Spectra (NA).

(2,6-Difluorophenyl)(3-fluoro-4-hydroxyphenyl)methanone

[161581-97-1] $C_{13}H_7F_3O_2$ mol.wt. 252.19



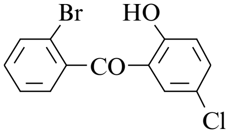
Synthesis

– Preparation by demethylation of 4-methoxy-2,3′,6-tri-fluorobenzophenone (SM) with 62% aqueous hydrobromic acid in acetic acid at 125° (97%). SM (m.p. 79–83°) was obtained by Friedel–Crafts acylation of o-fluoroanisole with 2,6-difluorobenzoyl chloride in nitrobenzene in the presence of aluminium chloride [512].

m.p. (NA); MS [512].

(2-Bromophenyl)(5-chloro-2-hydroxyphenyl)methanone

[92739-90-7] $C_{13}H_8BrClO_2$ mol.wt. 311.56



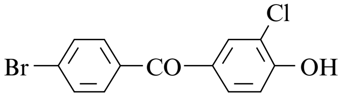
Synthesis

- Refer to: [1033].

m.p. and Spectra (NA).

(4-Bromophenyl)(3-chloro-4-hydroxyphenyl)methanone

[161582-04-3] $C_{13}H_8BrClO_2$ mol.wt. 311.56



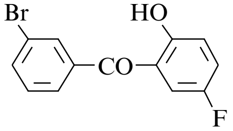
Synthesis

- Refer to: [512].

m.p. and Spectra (NA).

(3-Bromophenyl)(5-fluoro-2-hydroxyphenyl)methanone

[62433-28-7] $C_{13}H_8BrFO_2$ mol.wt. 295.11



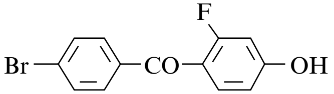
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl m-bromobenzoate with aluminium chloride at 130° for 2 h (91%) [1034].

m.p. 159–160° [1034]; Spectra (NA).

(4-Bromophenyl)(2-fluoro-4-hydroxyphenyl)methanone

[161581-99-3] $C_{13}H_8BrFO_2$ mol.wt. 295.11



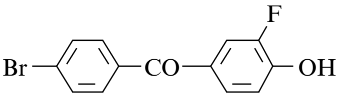
Synthesis

- Preparation by demethylation of 4′-bromo-2-fluoro-4-methoxybenzophenone (SM) with 62% aqueous hydrobromic acid in acetic acid at 125°. SM was obtained by Friedel–Crafts acylation of m-fluoroanisole with p-bromobenzoyl chloride in nitrobenzene in the presence of aluminium chloride [512].
- Also refer to: [1035] and Chem. Abstr., **127**, 108766j (1997).

m.p. (NA); MS [512].

(4-Bromophenyl)(3-fluoro-4-hydroxyphenyl)methanone

[161581-98-2] $C_{13}H_8BrFO_2$ mol.wt. 295.11



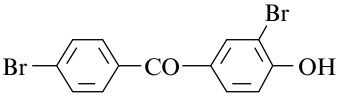
Synthesis

- Preparation by demethylation of 4′-bromo-3-fluoro-4-methoxybenzophenone (SM) with 62% aqueous hydrobromic acid in acetic acid at 125°. SM was obtained by Friedel–Crafts acylation of o-fluoroanisole with p-bromobenzoyl chloride in nitrobenzene in the presence of aluminium chloride [512].
- Also refer to: Chem. Abstr., **127**, 108766j (1997).

m.p. 183–184° [512]; Spectra (NA).

(3-Bromo-4-hydroxyphenyl)(4-bromophenyl)methanone

[161582-02-1] $C_{13}H_8Br_2O_2$ mol.wt. 356.01



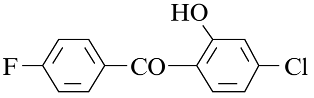
Synthesis

- Refer to: [512].

m.p. and Spectra (NA).

(4-Chloro-2-hydroxyphenyl)(4-fluorophenyl)methanone

[169781-85-5] $C_{13}H_8ClFO_2$ mol.wt. 250.66



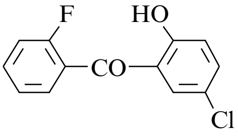
Syntheses

- Obtained by Fries rearrangement of m-chlorophenyl p-fluorobenzoate with aluminium chloride at 200° for 20 min [20].
- Also obtained by demethylation of 4-chloro-4′-fluoro-2-methoxybenzophenone with boron tribromide in methylene chloride at r.t. for 12 h [20], according to [21].

m.p. and Spectra (NA).

(5-Chloro-2-hydroxyphenyl)(2-fluorophenyl)methanone

[65185-33-3] $C_{13}H_8ClFO_2$ mol.wt. 250.66



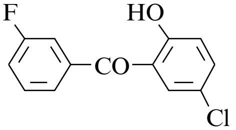
Syntheses

- Preparation by Friedel–Crafts acylation of p-chlorophenol with o-fluorobenzoyl chloride in the presence of aluminium chloride at 195° for 25 min (70%) [523].
- Preparation by Fries rearrangement of p-chlorophenyl o-fluorobenzoate with aluminium chloride [23].
- Also refer to: [1036].

m.p. 76–80°5 [523]; Spectra (NA).

(5-Chloro-2-hydroxyphenyl)(3-fluorophenyl)methanone

[62433-31-2] $C_{13}H_8ClFO_2$ mol.wt. 250.66



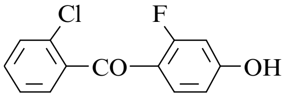
Synthesis

- Preparation by Fries rearrangement of p-chlorophenyl m-fluorobenzoate with aluminium chloride at 130° for 2 h (88%) [1034].

m.p. 153° [1034]; Spectra (NA).

(2-Chlorophenyl)(2-fluoro-4-hydroxyphenyl)methanone

[87750-64-9] $C_{13}H_8ClFO_2$ mol.wt. 250.66



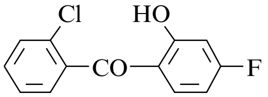
Synthesis

- Preparation by reaction of o-chlorobenzoyl chloride with m-fluorophenol in an hydrofluoric acid solution in the presence of boron trifluoride at 0° for 1 h [1037].

m.p. and Spectra (NA).

(2-Chlorophenyl)(4-fluoro-2-hydroxyphenyl)methanone

[87750-63-8] $C_{13}H_8ClFO_2$ mol.wt. 250.66



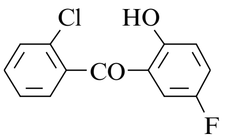
Synthesis

- Preparation by reaction of o-chlorobenzoyl chloride with m-fluorophenol in an hydrofluoric acid solution in the presence of boron trifluoride at 0° for 1 h [1037].

m.p. and Spectra (NA).

(2-Chlorophenyl)(5-fluoro-2-hydroxyphenyl)methanone

[2341-94-8] $C_{13}H_8ClFO_2$ mol.wt. 250.66



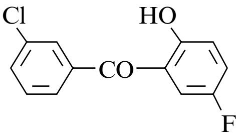
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl o-chlorobenzoate with aluminium chloride [23], at 130° for 2 h (95%) [1038].
- Also refer to: [1039].

m.p. 65° [1038]; Spectra (NA).

(3-Chlorophenyl)(5-fluoro-2-hydroxyphenyl)methanone

[62666-38-0] $C_{13}H_8ClFO_2$ mol.wt. 250.66



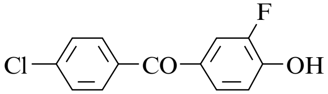
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl m-chlorobenzoate with aluminium chloride [23].
- Also refer to: [1039].

m.p. and Spectra (NA).

(4-Chlorophenyl)(3-fluoro-4-hydroxyphenyl)methanone

[83885-18-1] $C_{13}H_8ClFO_2$ mol.wt. 250.66



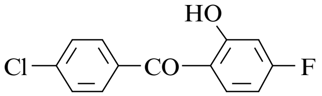
Synthesis

- Preparation by reaction of p-chlorobenzoyl chloride with o-fluorophenol in the presence of aluminium chloride [165].

m.p. 173–176°5 [165]; Spectra (NA).

(4-Chlorophenyl)(4-fluoro-2-hydroxyphenyl)methanone

[169781-86-6] $C_{13}H_8ClFO_2$ mol.wt. 250.66



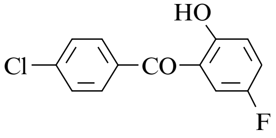
Syntheses

- Obtained by Fries rearrangement of m-fluorophenyl p-chlorobenzoate with aluminium chloride at 200° for 20 min [20].
- Also obtained by demethylation of 4′-chloro-4-fluoro-2-methoxybenzophenone with boron tribromide in methylene chloride at r.t. for 12 h [20], according to [21].

m.p. and Spectra (NA).

(4-Chlorophenyl)(5-fluoro-2-hydroxyphenyl)methanone

[62433-26-5] $C_{13}H_8ClFO_2$ mol.wt. 250.66



Syntheses

- Preparation by Fries rearrangement of p-fluorophenyl p-chlorobenzoate,
  - with titanium tetrachloride at 150° for 18 h (81%) [472];
  - with aluminium chloride [23,1040], at 130° for 2 h (98%) [1034] or at 200° for 5 min [1041] or for 15 min (65%) [1042].
- Preparation by reaction of p-chlorobenzoyl chloride with p-fluorophenol in hydrofluoric acid in the presence of boron trifluoride at 80° for 20 h (74%) [1037].

N.B.: In the patent, this compound was erroneously named 3-fluoro-4′-chloro-2-hydroxybenzophenone (assay 21, page 13) [1037].

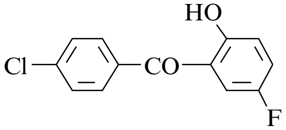
- Also refer to: [1033,1039,1043–1055].

m.p. 174° [1034], 67°6 [1042]. One of the reported melting points is obviously wrong.

^{1}H NMR [472], ^{13}C NMR [1042], IR [472,1042], MS [472]; TLC [472]; HPLC [1056]; pK_a [1046].

(4-Chlorophenyl)(5-fluoro-2-hydroxyphenyl)methanone-^{14}C

[82589-26-2] $C_{13}H_8ClFO_2$ mol.wt. 252.66



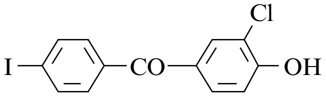
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl p-chloro-[carboxyl-^{14}C]benzoate with aluminium chloride at 200° for 5 min (76%) (51.8 mCi/mmol) [1041].

m.p. and Spectra (NA).

(3-Chloro-4-hydroxyphenyl)(4-iodophenyl)methanone

[161582-03-2] $C_{13}H_8ClIO_2$ mol.wt. 358.56



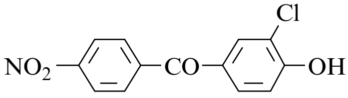
Synthesis

- Refer to: [512].

m.p. and Spectra (NA).

(3-Chloro-4-hydroxyphenyl)(4-nitrophenyl)methanone

$C_{13}H_8ClNO_4$ mol.wt. 277.66



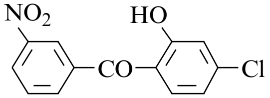
Synthesis

- Obtained by Fries rearrangement of o-chlorophenyl p-nitrobenzoate with aluminium chloride at 120° for 2 h [1057].

m.p. 196° [1057]; Spectra (NA).

(4-Chloro-2-hydroxyphenyl)(3-nitrophenyl)methanone

[22293-33-0] $C_{13}H_8ClNO_4$ mol.wt. 277.66



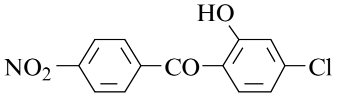
Synthesis

- Obtained by photo-Fries rearrangement of m-chlorophenyl m-nitrobenzoate in ethanol for 60–75 h (35%) [641].

m.p. 126° [641]; IR [641], UV [641].

(4-Chloro-2-hydroxyphenyl)(4-nitrophenyl)methanone

[22359-51-9] $C_{13}H_8ClNO_4$ mol.wt. 277.66



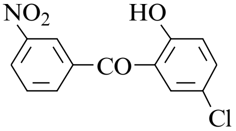
Syntheses

- Preparation by Fries rearrangement of m-chlorophenyl p-nitrobenzoate with aluminium chloride at 120° for 2 h [1057].
- Also obtained by photo-Fries rearrangement of m-chlorophenyl p-nitrobenzoate in ethanol for 60–75 h (13%) [641].

m.p. 160° [1057], 117° [641]; IR [641], UV [641].

(5-Chloro-2-hydroxyphenyl)(3-nitrophenyl)methanone

[126260-47-7] $C_{13}H_8ClNO_4$ mol.wt. 277.66



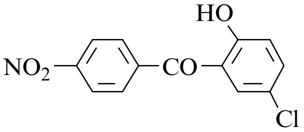
Syntheses

- Obtained (poor yield) by Fries rearrangement of p-chloro-phenyl m-nitrobenzoate with aluminium chloride in refluxing chlorobenzene for 4 h (11%) [520].
- Obtained by photo-Fries rearrangement of p-chlorophenyl m-nitrobenzoate in ethanol for 60–75 h (35%) [641].

m.p. 126° [641], 125–126° [520]; IR [641], UV [641].

(5-Chloro-2-hydroxyphenyl)(4-nitrophenyl)methanone

[84443-36-7] $C_{13}H_8ClNO_4$ mol.wt. 277.66



Syntheses

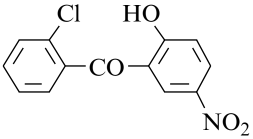
- Obtained by Fries rearrangement of p-chlorophenyl p-nitrobenzoate,
  - with aluminium chloride at 120° for 2 h [1057];
  - with UV light irradiation in ethanol during 60–75 h (13%) [641].

- Also refer to: [1058].

m.p. 120° [1057], 117° [641]; Spectra (NA).

(2-Chlorophenyl)(2-hydroxy-5-nitrophenyl)methanone

[95263-98-2] $C_{13}H_8ClNO_4$ mol.wt. 277.66



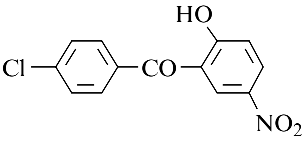
Synthesis

- Obtained by alkaline degradation of nizofenone fumarate [1059].
- Also refer to: [545].

m.p. and Spectra (NA).

(4-Chlorophenyl)(2-hydroxy-5-nitrophenyl)methanone

[124071-26-7] $C_{13}H_8ClNO_4$ mol.wt. 277.66



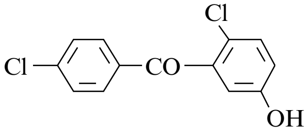
Synthesis

- Preparation according to the method [539], (23%) [1060].

m.p. 170–172° [1060]; Spectra (NA).

(2-Chloro-5-hydroxyphenyl)(4-chlorophenyl)methanone

[62810-45-1] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



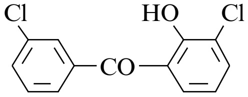
Syntheses

- Preparation by diazotization of 5-amino-2,4′-dichloro-benzophenone followed by hydrolysis of the diazonium salt so obtained [900].
- Also obtained by demethylation of 2,4′-dichloro-5-methoxy-benzophenone with aluminium chloride in refluxing chlorobenzene [900].

m.p. 172° [900]; Spectra (NA).

(3-Chloro-2-hydroxyphenyl)(3-chlorophenyl)methanone

[41796-26-3] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



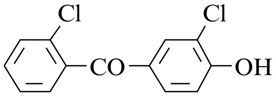
Synthesis

- Refer to: [1061].

m.p. and Spectra (NA).

(3-Chloro-4-hydroxyphenyl)(2-chlorophenyl)methanone

$C_{13}H_8Cl_2O_2$ mol.wt. 267.11



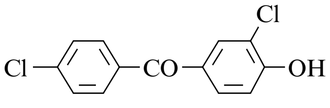
Synthesis

- Preparation by demethylation of 2′,3-dichloro-4-methoxy-benzophenone with pyridinium chloride at reflux for 30 min [461].

m.p. 156° [461]; Spectra (NA).

(3-Chloro-4-hydroxyphenyl)(4-chlorophenyl)methanone

[34189-58-7] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



Syntheses

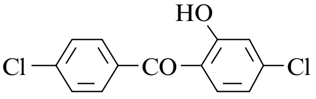
- Preparation by reaction of p-chlorobenzoyl chloride with o-chlorophenol in the presence of aluminium chloride [165].
- Preparation by demethylation of 3,4′-dichloro-4-methoxybenzophenone in refluxing pyridinium chloride for 30 min [461].
- Preparation by Friedel–Crafts acylation of chlorobenzene with 3-chloro-4-methoxy-benzoyl chloride in the presence of aluminium chloride at 120° for 3 h (50–70%) [931].
- Preparation by Fries rearrangement of o-chlorophenyl p-chlorobenzoate with aluminium chloride in chlorobenzene at 140–150° for 20 min or in nitrobenzene at 75° for 24 h [480].

m.p. 178–179° [931], 176–177°5 [480], 168° [461], 52°5–53°5 [165]. One of the reported melting points is obviously wrong.

^{1}H NMR [931], MS [931]; TLC [931].

(4-Chloro-2-hydroxyphenyl)(4-chlorophenyl)methanone

[60805-31-4] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



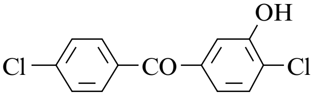
Syntheses

- Preparation by Friedel–Crafts acylation of chlorobenzene with 4-chloro-2-methoxybenzoyl chloride in the presence of aluminium chloride at 120° for 3 h (50–70%) [931].
- Also obtained by Fries rearrangement of m-chlorophenyl p-chlorobenzoate with aluminium chloride at 200° for 20 min [20].
- Also obtained by demethylation of 4,4′-dichloro-2-methoxybenzophenone with boron tribromide in methylene chloride at r.t. for 12 h [20], according to [21].

m.p. 78–79° [931]; ^{1}H NMR [931], MS [931]; TLC [931].

(4-Chloro-3-hydroxyphenyl)(4-chlorophenyl)methanone

[60805-30-3] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



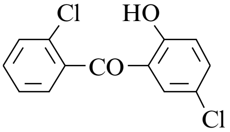
Synthesis

- Preparation by Friedel–Crafts acylation of chlorobenzene with 4-chloro-3-methoxybenzoyl chloride in the presence of aluminium chloride at 120° for 3 h (50–70%) [931].

m.p. 160–162° [931]; ^{1}H NMR [931], MS [931]; TLC [931].

(5-Chloro-2-hydroxyphenyl)(2-chlorophenyl)methanone

[61785-35-1] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



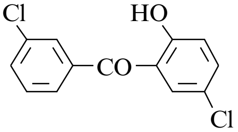
Syntheses

- Preparation by reaction of o-chlorobenzoyl chloride with p-chlorophenol in the presence of aluminium chloride at 180° [484].
- Preparation by Fries rearrangement of p-chlorophenyl o-chlorobenzoate with aluminium chloride [1062].
- Also refer to: [1033,1036,1063–1065].

m.p. 107°9 [1062], 106°5–107°5 [484]; Spectra (NA).

(5-Chloro-2-hydroxyphenyl)(3-chlorophenyl)methanone

[61785-36-2] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



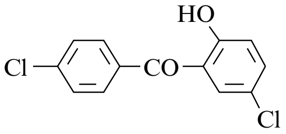
Synthesis

- Preparation by Fries rearrangement of p-chlorophenyl m-chlorobenzoate with aluminium chloride for 30 min at 160° (64%) [528].

m.p. 72° [528]; Spectra (NA).

(5-Chloro-2-hydroxyphenyl)(4-chlorophenyl)methanone

[61785-37-3] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



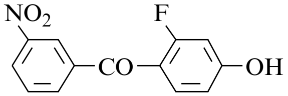
Synthesis

- Obtained by Fries rearrangement of p-chlorophenyl p-chlorobenzoate with titanium tetrachloride at 150° for 18 h (23%) [472].
- Also refer to: [484,523,1066].

m.p. (NA); yellow crystals [472]; ^{1}H NMR [472], IR [472], MS [472]; TLC [472]; HPLC [1056].

(2-Fluoro-4-hydroxyphenyl)(3-nitrophenyl)methanone

[194290-75-0] $C_{13}H_8FNO_4$ mol.wt. 247.20



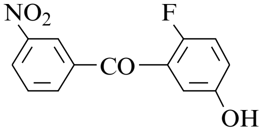
Synthesis

- Obtained by Friedel–Crafts acylation of m-fluorophenol with m-nitrobenzoyl chloride in ethylene dichloride in the presence of aluminium chloride at r.t. for 4 h (general procedure C; compound **33 i**).
- Refer to: Chem. Abstr., **127**, 190681j (1997).

m.p. and Spectra (NA).

(2-Fluoro-5-hydroxyphenyl)(3-nitrophenyl)methanone

[194290-73-8] $C_{13}H_8FNO_4$ mol.wt. 247.20



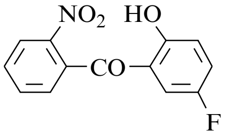
Synthesis

- Obtained by Friedel–Crafts acylation of p-fluorophenol with m-nitrobenzoyl chloride in ethylene dichloride in the presence of aluminium chloride at r.t. for 4 h (General Procedure C; compound **33 h**).
- Refer to: Chem. Abstr., **127**, 190681j (1997).

m.p. and Spectra (NA).

(5-Fluoro-2-hydroxyphenyl)(2-nitrophenyl)methanone

[62433-27-6] $C_{13}H_8FNO_4$ mol.wt. 247.20



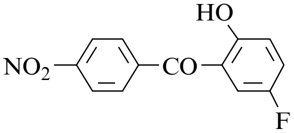
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl o-nitrobenzoate with aluminium chloride at 130° for 2 h (86%) [1034].

m.p. 123° [1034]; Spectra (NA).

(5-Fluoro-2-hydroxyphenyl)(4-nitrophenyl)methanone

[62433-30-1] $C_{13}H_8FNO_4$ mol.wt. 247.20



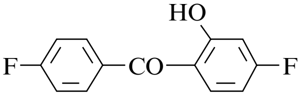
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl p-nitrobenzoate with aluminium chloride at 130° for 2 h (93%) [1034].

m.p. 141° [1034]; IR [1034].

(4-Fluoro-2-hydroxyphenyl)(4-fluorophenyl)methanone

[153411-29-1] $C_{13}H_8F_2O_2$ mol.wt. 234.20



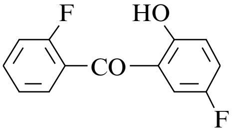
Syntheses

- Obtained by Fries rearrangement of m-fluorophenyl p-fluorobenzoate with aluminium chloride at 200° for 20 min [20].
- Also obtained by demethylation of 4,4′-difluoro-2-methoxy benzophenone with boron tribromide in methylene chloride at r.t. for 12 h [20], according to [21].
- Also refer to: [1067].

m.p. and Spectra (NA).

(5-Fluoro-2-hydroxyphenyl)(2-fluorophenyl)methanone

$C_{13}H_8F_2O_2$ mol.wt. 234.20



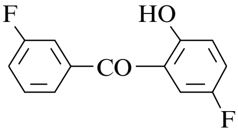
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl o-fluorobenzoate with aluminium chloride [23].

m.p. and Spectra (NA).

(5-Fluoro-2-hydroxyphenyl)(3-fluorophenyl)methanone

$C_{13}H_8F_2O_2$ mol.wt. 234.20



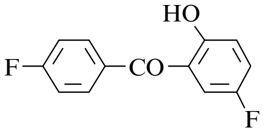
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl m-fluorobenzoate with aluminium chloride [23].

m.p. and Spectra (NA).

(5-Fluoro-2-hydroxyphenyl)(4-fluorophenyl)methanone

[2559-64-0] $C_{13}H_8F_2O_2$ mol.wt. 234.20



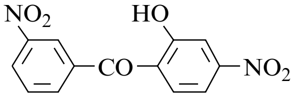
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl p-fluorobenzoate with aluminium chloride [23], without solvent at 130° for 2 h (83%) [1038].
- Also refer to: [1068,1069].

b.p.$_{30}$ 205° [1038]; Spectra (NA).

(2-Hydroxy-4-nitrophenyl)(3-nitrophenyl)methanone

[1834-89-5] $C_{13}H_8N_2O_6$ mol.wt. 288.22



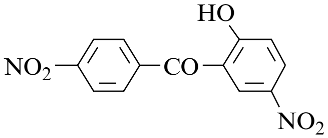
Syntheses

- Obtained (trace) by reaction of m-nitrobenzoyl chloride with m-nitrophenol in the presence of aluminium chloride without solvent at 175–180° for 2.5 h (0.5%) [537].
- Also obtained (poor yield) by Fries rearrangement of m-nitrophenyl m-nitrobenzoate with aluminium chloride without solvent at 175° for 2 h (2%) [1070].

m.p. 158–159° [1070], 157–158° [537]; Spectra (NA).

(2-Hydroxy-5-nitrophenyl)(4-nitrophenyl)methanone

$C_{13}H_8N_2O_6$ mol.wt. 288.22



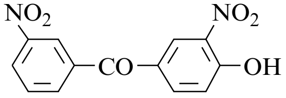
Synthesis

- Obtained by photo-Fries rearrangement of p-nitrophenyl p-nitrobenzoate in benzene at 50° for 35 h (11%) [72].

m.p. 189° [72]; Spectra (NA).

(4-Hydroxy-3-nitrophenyl)(3-nitrophenyl)methanone

[37567-45-6] $C_{13}H_8N_2O_6$ mol.wt. 288.22



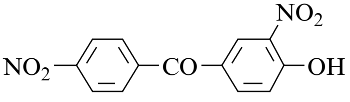
Syntheses

- Preparation by reaction of 48% hydrobromic acid with 3,3′-dinitro-4-methoxybenzophenone (SM) in refluxing acetic acid. SM was obtained from 3′-nitro-4-methoxy-benzophenone by a two-step synthesis [140].
- Preparation by hydrolysis of 4-chloro-3,3′-dinitrobenzophenone with 5–15% sodium hydroxide at 155–160° for 1–1.5 h (97–98%) [1071].

m.p. 165° [140], 149°8–150°6 [1071]; Spectra (NA).

(4-Hydroxy-3-nitrophenyl)(4-nitrophenyl)methanone

[37567-41-2] $C_{13}H_8N_2O_6$ mol.wt. 288.22



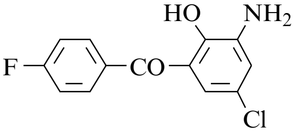
Synthesis

– Preparation by hydrolysis of 4-chloro-3,4′-dinitro-benzophenone with 5–15% aqueous sodium hydroxide at 155–160° for 1–1.5 h (97–98%) [1071].

m.p. 154°5–154°8 [1071]; Spectra (NA).

(3-Amino-5-chloro-2-hydroxyphenyl)(4-fluorophenyl)methanone

$C_{13}H_9ClFNO_2$ mol.wt. 265.67



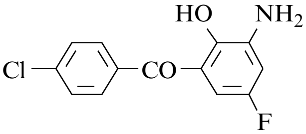
Synthesis

– Refer to: [472].

m.p. and Spectra (NA).

(3-Amino-5-fluoro-2-hydroxyphenyl)(4-chlorophenyl)methanone

[85052-42-2] $C_{13}H_9ClFNO_2$ mol.wt. 265.67



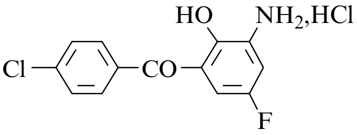
Synthesis

– Preparation by hydrogenation of 4′-chloro-5-fluoro-2-hydroxy-3-nitrobenzophenone in the presence of 10% Pd/C in a chloroform/ethanol mixture for 2 h (82%) [472].

m.p. 124–127° [472]; ^{1}H NMR [472], IR [472], MS [472]; TLC [472].

(3-Amino-5-fluoro-2-hydroxyphenyl)(4-chlorophenyl)methanone *(Hydrochloride)*

[85052-69-3] $C_{13}H_9ClFNO_2,HCl$ mol.wt. 302.14



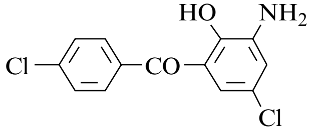
Synthesis

– Obtained by reaction of hydrochloric acid with 3-amino-4′-chloro-5-fluoro-2-hydroxybenzophenone [472].

m.p. and Spectra (NA).

(3-Amino-5-chloro-2-hydroxyphenyl)(4-chlorophenyl)methanone

[85052-41-1] $C_{13}H_9Cl_2NO_2$ mol.wt. 282.13



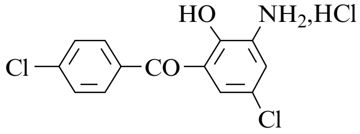
Synthesis

- Preparation by hydrogenation of 4′,5-dichloro-2-hydroxy-3-nitrobenzophenone in the presence of 10% Pd/C in a chloroform/ethanol mixture for 2 h (21%) [472].

m.p. 91–94° (d) [472]; ^{1}H NMR [472], IR [472], MS [472]; TLC [472].

(3-Amino-5-chloro-2-hydroxyphenyl)(4-chlorophenyl)methanone *(Hydrochloride)*

[85052-68-2] $C_{13}H_9Cl_2NO_2,HCl$ mol.wt. 318.60



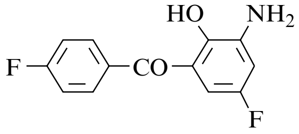
Synthesis

- Obtained by reaction of hydrochloric acid with 3-amino-4′,5-dichloro-2-hydroxybenzophenone [472].

m.p. and Spectra (NA).

(3-Amino-5-fluoro-2-hydroxyphenyl)(4-fluorophenyl)methanone

$C_{13}H_9F_2NO_2$ mol.wt. 249.22



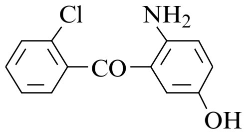
Synthesis

- Refer to: [472].

m.p. and Spectra (NA).

(2-Amino-5-hydroxyphenyl)(2-chlorophenyl)methanone

[61871-78-1] $C_{13}H_{10}ClNO_2$ mol.wt. 247.68



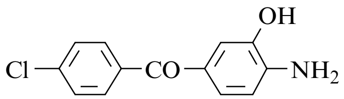
Synthesis

- Obtained by heating N-(o-chlorobenzoyl)-p-methoxyaniline with bismuth chloride (5 mol excess) at 180–200° for 3 min (80%) [558].

m.p. (NA); UV [558], MS [558]; TLC [558].

(4-Amino-3-hydroxyphenyl)(4-chlorophenyl)methanone

[123172-45-2] $C_{13}H_{10}ClNO_2$ mol.wt. 247.68

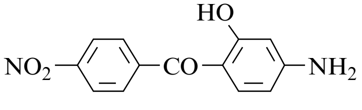


Synthesis

- Preparation from 6-(p-chlorobenzoyl)benzoxazolinone by heating with sodium hydroxide (80%) [566].

m.p. 196–198° [566]; Spectra (NA).

(4-Amino-2-hydroxyphenyl)(4-nitrophenyl)methanone



$C_{13}H_{10}N_2O_4$ mol.wt. 258.23

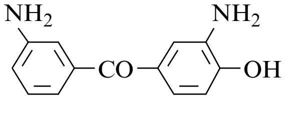
Synthesis

- Preparation by hydrolysis of 4-acetamido-2-hydroxy-4′-nitrobenzophenone with boiling 50% hydrochloric acid [562].

m.p. 228° [562]; Spectra (NA).

(3-Amino-4-hydroxyphenyl)(3-aminophenyl)methanone

[37567-47-8]



$C_{13}H_{12}N_2O_2$ mol.wt. 228.25

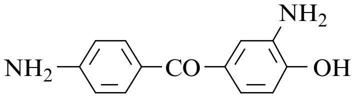
Synthesis

- Preparation by hydrogenation of 4-hydroxy-3,3′-dinitro-benzophenone in the presence of Raney nickel in water at 90° for 1 h under 90 atmospheres (70%) [1071].

m.p. >178° (d) [1071]; Spectra (NA).

(3-Amino-4-hydroxyphenyl)(4-aminophenyl)methanone

[37567-42-3]



$C_{13}H_{12}N_2O_2$ mol.wt. 228.25

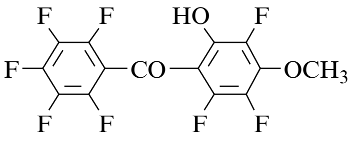
Synthesis

- Preparation by hydrogenation of 4-hydroxy-3,4′-di-nitrobenzophenone in the presence of Raney nickel in water at 90° for 1 h under 100 atmospheres (71%) [1071].

m.p. 191°5–191°8 [1071]; Spectra (NA).

(2,3,4,5,6-Pentafluorophenyl)(2,3,5-trifluoro-6-hydroxy-4-methoxyphenyl) methanone

[32541-22-3]



$C_{14}H_4F_8O_3$ mol.wt. 372.17

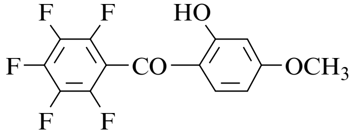
Syntheses

- Preparation by partial demethylation of 2,4-dimethoxy-2′,3,3′,4′,5,5′,6,6′-octa-fluorobenzophenone (SM) in methylene chloride in the presence of aluminium chloride at 0–5° for 3–6 h (63%) [570]. SM was obtained by condensation of methyl 2,4-dimethoxy-3,5,6-trifluorobenzoate with pentafluorophenylmagnesium bromide in ethyl ether at 20° for 2 h (28%).
- Preparation by partial methylation of 2,4-dihydroxy-2′,3,3′,4′,5,5′,6,6′-octafluorobenzophenone with diazomethane in ethyl ether between −10° and 0° (70%) [570].

m.p. 68–69° [570]; ^{1}H NMR [570], IR [570], UV [570].

(2-Hydroxy-4-methoxyphenyl)(2,3,4,5,6-pentafluorophenyl)methanone

[32541-23-4]



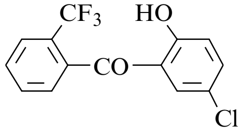
$C_{14}H_7F_5O_3$ mol.wt. 318.20

Synthesis

– Preparation by partial demethylation of 2′,4′-dimethoxy-2,3,4,5,6-pentafluorobenzophenone (SM) in methylene chloride in the presence of aluminium chloride at 0–5° for 3–6 h (69%) [570]. SM was obtained in two steps: first, preparation of 2,4-dimethoxy-phenylbis(pentafluorophenyl)carbinol by condensation of methyl 2,4-dimethoxybenzoate with pentafluorophenylmagnesium bromide in ethyl ether at 20° for 2 h (37%). Then, this carbinol was treated with potassium fluoride in boiling acetone for 4 h (98%) [570].

m.p. 152°5–154° [570]; ^{1}H NMR [570], IR [570], UV [570].

(5-Chloro-2-hydroxyphenyl)[2-(trifluoromethyl)phenyl]methanone



$C_{14}H_8ClF_3O_2$ mol.wt. 300.66

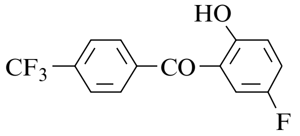
Synthesis

– Preparation from 2-bromo-4-chloro-(2-methoxyethoxy)-methoxybenzene and o-(trifluoromethyl)benzaldehyde as the starting materials [624].

m.p. 71–72° [624]; Spectra (NA).

(5-Fluoro-2-hydroxyphenyl)[4-(trifluoromethyl)phenyl]methanone

[183280-21-9]



$C_{14}H_8F_4O_2$ mol.wt. 284.21

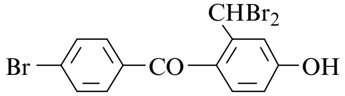
Synthesis

– Preparation by demethylation of 5-fluoro-2-methoxy-4′-(trifluoromethyl)benzophenone with boron tribromide in methylene chloride under argon: first at 0°, then at r.t. for 12 h (98%) [492].

m.p. 58°5 [492]; ^{1}H NMR [492], UV [492], MS [492].

(4-Bromophenyl)[2-(dibromomethyl)-4-hydroxyphenyl]methanone

[192443-53-1]



$C_{14}H_9Br_3O_2$ mol.wt. 448.94

Synthesis

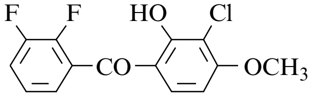
– Preparation by demethylation of 4′-bromo-2-dibromo-methyl-4-methoxybenzophenone (SM) with boron tribromide in methylene chloride at −78° (91%). SM was obtained by bromination of 4′-bromo-4-methoxy-2-methylbenzophenone with N-bromosuccinimide in carbon tetrachloride in the presence of few dibenzoyl peroxide under irradiation at r.t. (90%; MS).

– Refer to: Chem. Abstr., **127**, 108921f (1997)^T.

m.p. (NA); MS^T.

(3-Chloro-2-hydroxy-4-methoxyphenyl)(2,3-difluorophenyl)methanone

[72482-00-9] $C_{14}H_9ClF_2O_3$ mol.wt. 298.67



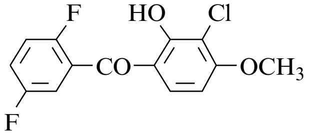
Synthesis

- Preparation by reaction of 2,3-difluorobenzoyl chloride with 2-chlororesorcinol dimethyl ether in ethylene dichloride in the presence of aluminium chloride: first at 5°, then at r.t. and at reflux for 30 min [476].

m.p. 161–162° [476]; Spectra (NA).

(3-Chloro-2-hydroxy-4-methoxyphenyl)(2,5-difluorophenyl)methanone

[72482-10-1] $C_{14}H_9ClF_2O_3$ mol.wt. 298.67



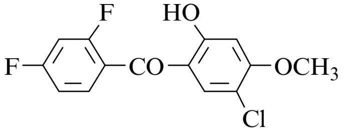
Synthesis

- Preparation by reaction of 2,5-difluorobenzoyl chloride with 2-chlororesorcinol dimethyl ether in ethylene dichloride in the presence of aluminium chloride: first at 5–10°, then at r.t. and at reflux for 30 min [476].

m.p. 178–180° [476]; Spectra (NA).

(5-Chloro-2-hydroxy-4-methoxyphenyl)(2,4-difluorophenyl)methanone

[136741-46-3] $C_{14}H_9ClF_2O_3$ mol.wt. 298.67



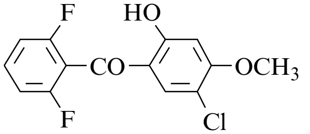
Synthesis

- Preparation by reaction of 2,4-difluorobenzoyl chloride with 4-chlororesorcinol dimethyl ether in the presence of aluminium chloride in ethylene dichloride (99%) [589].

m.p. 136–137° [589]; Spectra (NA).

(5-Chloro-2-hydroxy-4-methoxyphenyl)(2,6-difluorophenyl)methanone

[136741-45-2] $C_{14}H_9ClF_2O_3$ mol.wt. 298.67



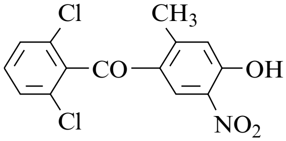
Synthesis

- Preparation by reaction of 2,6-difluorobenzoyl chloride with 4-chlororesorcinol dimethyl ether in the presence of aluminium chloride in ethylene dichloride (92%) [589].

m.p. 132–133° [589]; Spectra (NA).

(2,6-Dichlorophenyl)(4-hydroxy-2-methyl-5-nitrophenyl)methanone

[183725-86-2] $C_{14}H_9Cl_2NO_4$ mol.wt. 326.14



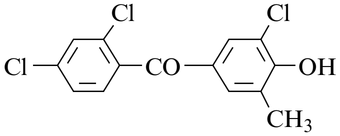
Synthesis

– Preparation by demethylation of 2′,6′-dichloro-4-methoxy-2-methyl-5-nitrobenzophenone with aluminium chloride in methylene chloride, first for 30 min at 20° and then for 1 h at 45° (73%) [1072].

m.p. 170° [1072]; Spectra (NA).

(3-Chloro-4-hydroxy-5-methylphenyl)(2,4-dichlorophenyl)methanone

[34182-99-5] $C_{14}H_9Cl_3O_2$ mol.wt. 315.58



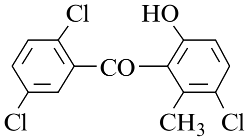
Synthesis

– Preparation by Fries rearrangement of 2-chloro-6-methyl-phenyl 2,4-dichlorobenzoate with aluminium chloride in chlorobenzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 168–170° [480]; Spectra (NA).

(3-Chloro-6-hydroxy-2-methylphenyl)(2,5-dichlorophenyl)methanone

[34174-13-5] $C_{14}H_9Cl_3O_2$ mol.wt. 315.58



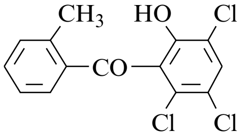
Synthesis

– Preparation by Fries rearrangement of 4-chloro-3-methyl-phenyl 2,5-dichlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 122–123° [458]; Spectra (NA).

(2-Methylphenyl)(2,3,5-trichloro-6-hydroxyphenyl)methanone

[34171-64-7] $C_{14}H_9Cl_3O_2$ mol.wt. 315.58



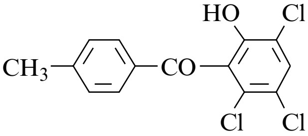
Synthesis

– Preparation by Fries rearrangement of 2,4,5-trichlorophenyl o-toluate with aluminium chloride for 30 min at 150–160° [458].

m.p. 125–126° [458]; Spectra (NA).

(4-Methylphenyl)(2,3,5-trichloro-6-hydroxyphenyl)methanone

[34171-62-5] $C_{14}H_9Cl_3O_2$ mol.wt. 315.58



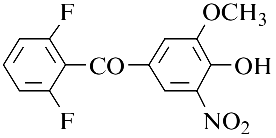
Synthesis

- Preparation by Fries rearrangement of 2,4,5-trichlorophenyl p-toluate with aluminium chloride for 30 min at 150–160° [458].

m.p. 141–142°5 [458]; Spectra (NA).

(2,6-Difluorophenyl)(4-hydroxy-3-methoxy-5-nitrophenyl)methanone

[134612-75-2] $C_{14}H_9F_2NO_5$ mol.wt. 309.23



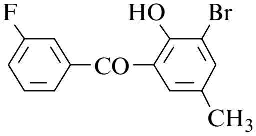
Synthesis

- Preparation by nitration of 2′,6′-difluoro-4-hydroxy-3-methoxybenzophenone with 65% nitric acid in acetic acid at 20° [1019].

m.p. 147–149° [1019]; Spectra (NA).

(3-Bromo-2-hydroxy-5-methylphenyl)(3-fluorophenyl)methanone

$C_{14}H_{10}BrFO_2$ mol.wt. 309.13



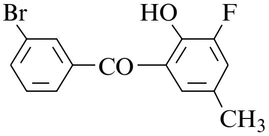
Synthesis

- Preparation by treatment of 3′-fluoro-2-hydroxy-5-methyl-benzophenone sodium salt with bromine in aqueous potassium bromide solution [919].

m.p. 130–131° [919]; Spectra (NA).

(3-Bromophenyl)(3-fluoro-2-hydroxy-5-methylphenyl)methanone

[55270-81-0] $C_{14}H_{10}BrFO_2$ mol.wt. 309.13



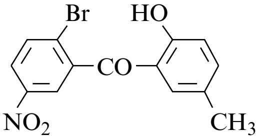
Synthesis

- Not yet described. This compound was mentioned in the Chem. Abstr., **82**, 170585e (1975). Nevertheless, it is not described in the original paper [919].

m.p. and Spectra (NA).

(2-Bromo-5-nitrophenyl)(2-hydroxy-5-methylphenyl)methanone

$C_{14}H_{10}BrNO_4$ mol.wt. 336.14

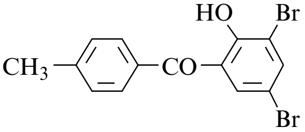


Synthesis

- Preparation by reaction of 2-bromo-5-nitrobenzoyl chloride with p-cresol methyl ether in the presence of aluminium chloride in carbon disulfide at r.t. for 1 h, then in a boiling water bath for 2–4 h (78%) [719].

m.p. 151–152° [719]; Spectra (NA).

(3,5-Dibromo-2-hydroxyphenyl)(4-methylphenyl)methanone



$C_{14}H_{10}Br_2O_2$ mol.wt. 370.04

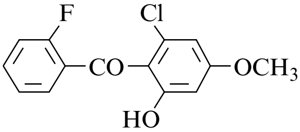
Synthesis

- Preparation by reaction of bromine with 2-hydroxy-4′-methylbenzophenone in chloroform [13].

m.p. 132°5 [13]; Spectra (NA).

(2-Chloro-6-hydroxy-4-methoxyphenyl)(2-fluorophenyl)methanone

[72482-27-0]



$C_{14}H_{10}ClFO_3$ mol.wt. 280.68

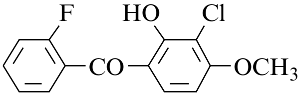
Syntheses

- Preparation by reaction of 2-fluorobenzoyl chloride with 5-chlororesorcinol dimethyl ether in the presence of aluminium chloride in refluxing ethylene dichloride (70%) [589], 96% [1031].
- Preparation by partial demethylation of 2-chloro-2′-fluoro-4,6-dimethoxybenzophenone (SM) in ethylene dichloride in the presence of aluminium chloride at reflux (90°) for 3 h [476]. SM was obtained by reaction of o-fluorobenzoyl chloride with 5-chlororesorcinol dimethyl ether in methylene chloride in the presence of aluminium chloride at r.t. for 4 h [476].

m.p. 111–113° [589], 108–110° [1031], 85–90° [476]; Spectra (NA).

(3-Chloro-2-hydroxy-4-methoxyphenyl)(2-fluorophenyl)methanone

[72482-07-6]



$C_{14}H_{10}ClFO_3$ mol.wt. 280.68

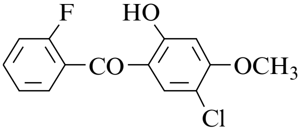
Synthesis

- Preparation by reaction of o-fluorobenzoyl chloride with 2-chlororesorcinol dimethyl ether in the presence of aluminium chloride in refluxing ethylene dichloride for 30 min [476], (67%) [1031].

m.p. 132–133° [476,1031]; Spectra (NA).

(5-Chloro-2-hydroxy-4-methoxyphenyl)(2-fluorophenyl)methanone

[130556-06-8]



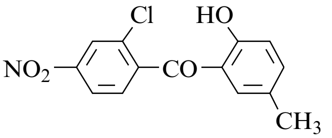
$C_{14}H_{10}ClFO_3$ mol.wt. 280.68

Synthesis

- Preparation by reaction of 2-fluorobenzoyl chloride with the 4-chlororesorcinol dimethyl ether in the presence of aluminium chloride in ethylene dichloride (64%) [589].

m.p. 133–134° [589]; Spectra (NA).

(2-Chloro-4-nitrophenyl)(2-hydroxy-5-methylphenyl)methanone



$C_{14}H_{10}ClNO_4$ mol.wt. 291.69

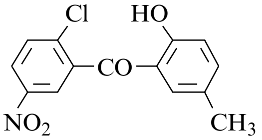
Synthesis

- Preparation by demethylation of 2-chloro-2′-methoxy-5′-methyl-4-nitrobenzophenone with excess boiling pyridinium chloride for 30 min (57%) [1073].

m.p. 130° [1073]; Spectra (NA).

(2-Chloro-5-nitrophenyl)(2-hydroxy-5-methylphenyl)methanone

[37883-98-0]



$C_{14}H_{10}ClNO_4$ mol.wt. 291.69

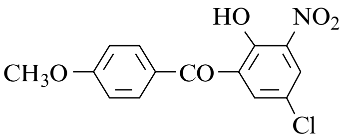
Synthesis

- Refer to: [1073].

m.p. and Spectra (NA).

(5-Chloro-2-hydroxy-3-nitrophenyl)(4-methoxyphenyl)methanone

[85052-28-4]



$C_{14}H_{10}ClNO_5$ mol.wt. 307.69

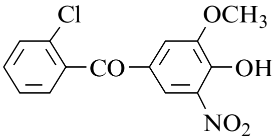
Synthesis

- Preparation by reaction of 60% nitric acid with 5-chloro-2-hydroxy-4′-methoxybenzophenone in acetic acid at r.t. for 30 min (79%) [472].

m.p. (NA); ^{1}H NMR [472], IR [472], MS [472]; TLC [472].

(2-Chlorophenyl)(4-hydroxy-3-methoxy-5-nitrophenyl)methanone

[134612-76-3]



$C_{14}H_{10}ClNO_5$ mol.wt. 307.69

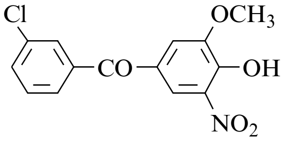
Synthesis

- Preparation by nitration of 2′-chloro-4-hydroxy-3-methoxy-benzophenone with 65% nitric acid in acetic acid at 20° [1019].

m.p. 123–125° [1019]; Spectra (NA).

(3-Chlorophenyl)(4-hydroxy-3-methoxy-5-nitrophenyl)methanone

[134612-77-4]



$C_{14}H_{10}ClNO_5$ mol.wt. 307.69

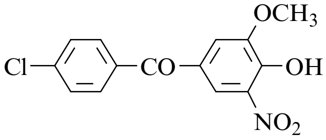
Synthesis

- Preparation by nitration of 3′-chloro-4-hydroxy-3-methoxy-benzophenone with 65% nitric acid in acetic acid at 20° [1019].

m.p. 152–154° [1019]; Spectra (NA).

(4-Chlorophenyl)(4-hydroxy-3-methoxy-5-nitrophenyl)methanone

[134612-78-5] $C_{14}H_{10}ClNO_5$ mol.wt. 307.69



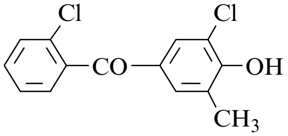
Synthesis

– Preparation by nitration of 4′-chloro-4-hydroxy-3-methoxybenzophenone with 65% nitric acid in acetic acid at 20° [1019].

m.p. 129–131° [1019]; Spectra (NA).

(3-Chloro-4-hydroxy-5-methylphenyl)(2-chlorophenyl)methanone

[34183-19-2] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



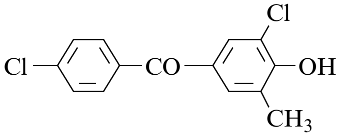
Synthesis

– Preparation by Fries rearrangement of 2-chloro-6-methyl-phenyl o-chlorobenzoate with aluminium chloride in chloro-benzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 164–165° [480]; Spectra (NA).

(3-Chloro-4-hydroxy-5-methylphenyl)(4-chlorophenyl)methanone

[34183-09-0] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



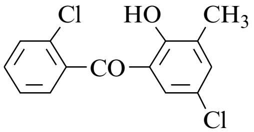
Synthesis

– Preparation by Fries rearrangement of 2-chloro-6-methyl-phenyl p-chlorobenzoate with aluminium chloride in chlorobenzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 138–139° [480]; Spectra (NA).

(5-Chloro-2-hydroxy-3-methylphenyl)(2-chlorophenyl)methanone

[86914-82-1] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



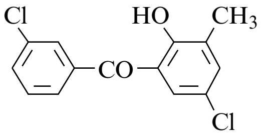
Synthesis

– Preparation by Fries rearrangement of 4-chloro-2-methyl-phenyl o-chlorobenzoate with aluminium chloride at 180° for 10 min [1074].

m.p. 87–88° [1074]; Spectra (NA).

(5-Chloro-2-hydroxy-3-methylphenyl)(3-chlorophenyl)methanone

[86914-87-6] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



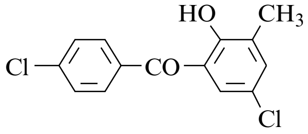
Synthesis

– Preparation by Fries rearrangement of 4-chloro-2-methyl-phenyl m-chlorobenzoate with aluminium chloride at 180° for 10 min [1074].

m.p. 130–131° [1074]; Spectra (NA).

(5-Chloro-2-hydroxy-3-methylphenyl)(4-chlorophenyl)methanone

[86914-72-9] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



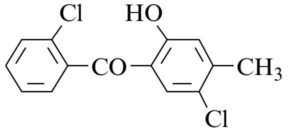
Synthesis

- Preparation by Fries rearrangement of 4-chloro-2-methyl-phenyl p-chlorobenzoate with aluminium chloride at 160° [1074].
- Also refer to: [1033,1075–1077].

m.p. 41–42° [1074]; Spectra (NA).

(5-Chloro-2-hydroxy-4-methylphenyl)(2-chlorophenyl)methanone

$C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



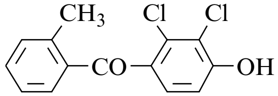
Synthesis

- Refer to: [461].

m.p. 105° [461]; Spectra (NA).

(2,3-Dichloro-4-hydroxyphenyl)(2-methylphenyl)methanone

[72482-84-9] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



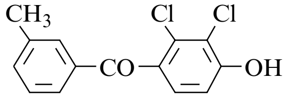
Synthesis

- Preparation by demethylation of 2,3-dichloro-4-methoxy-2′-methylbenzophenone (SM) with aluminium chloride in refluxing benzene for 5 h, then at r.t. for 18 h. SM was obtained by reaction of o-toluoyl chloride with 2,3-di-chloroanisole in ethylene dichloride in the presence of aluminium chloride at 60° for 1 h [476].

m.p. and Spectra (NA).

(2,3-Dichloro-4-hydroxyphenyl)(3-methylphenyl)methanone

$C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



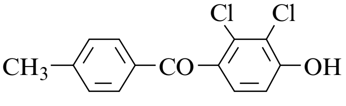
Synthesis

- Preparation by demethylation of 2,3-dichloro-4-methoxy-3′-methylbenzophenone (SM) with aluminium chloride in refluxing methylene chloride overnight. SM was obtained by Friedel–Crafts acylation of 2,3-dichloroanisole with m-toluoyl chloride in the presence of aluminium chloride in refluxing methylene chloride [475].

m.p. and Spectra (NA).

(2,3-Dichloro-4-hydroxyphenyl)(4-methylphenyl)methanone

[72498-72-7] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



Synthesis

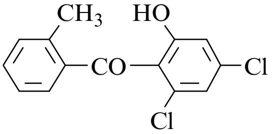
– Preparation by demethylation of 2,3-dichloro-4-methoxy-4′-methylbenzophenone (SM) with aluminium chloride in refluxing methylene chloride overnight. SM was obtained by Friedel–Crafts acylation of 2,3-dichloroanisole with p-methylbenzoyl chloride in the presence of aluminium chloride in refluxing methylene chloride [475].

– Also refer to: [476].

m.p. and Spectra (NA).

(2,4-Dichloro-6-hydroxyphenyl)(2-methylphenyl)methanone

[34174-08-8] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



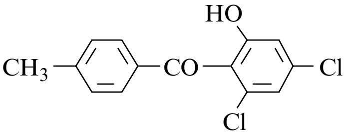
Synthesis

– Preparation by Fries rearrangement of 3,5-dichlorophenyl o-toluate with aluminium chloride for 30 min at 150–160° [458].

m.p. 117–118° [458]; Spectra (NA).

(2,4-Dichloro-6-hydroxyphenyl)(4-methylphenyl)methanone

[34174-07-7] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



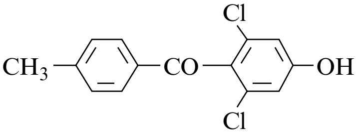
Synthesis

– Preparation by Fries rearrangement of 3,5-dichloro-phenyl p-toluate with aluminium chloride for 30 min at 150–160° [458].

m.p. 177–178° [458]; Spectra (NA).

(2,6-Dichloro-4-hydroxyphenyl)(4-methylphenyl)methanone

[34183-14-7] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



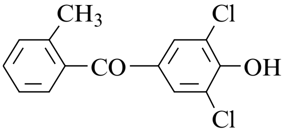
Synthesis

– Obtained by Fries rearrangement of 3,5-dichlorophenyl p-toluate with aluminium chloride in chlorobenzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 165–166° [480]; Spectra (NA).

(3,5-Dichloro-4-hydroxyphenyl)(2-methylphenyl)methanone

[34183-17-0] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



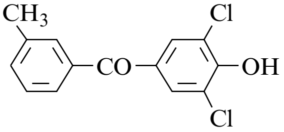
Synthesis

- Preparation by Fries rearrangement of 2,6-dichlorophenyl o-toluate with aluminium chloride in chlorobenzene at 140–150° for 20 min or in nitrobenzene at 75° for 24 h [480].

m.p. 163–164° [480]; Spectra (NA).

(3,5-Dichloro-4-hydroxyphenyl)(3-methylphenyl)methanone

[70036-75-8] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



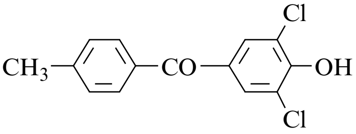
Synthesis

- Refer to: [468] (Japanese patent).

m.p. and Spectra (NA).

(3,5-Dichloro-4-hydroxyphenyl)(4-methylphenyl)methanone

[34183-10-3] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



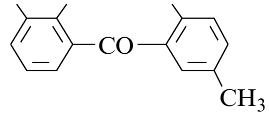
Synthesis

- Preparation by Fries rearrangement of 2,6-dichloro-phenyl p-toluate with aluminium chloride in chloro-benzene at 140–150° for 20 min or in nitrobenzene at 75° for 24 h [480].

m.p. 163–164° [480]; Spectra (NA).

(2,3-Dichlorophenyl)(2-hydroxy-5-methylphenyl)methanone

[77151-84-9] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



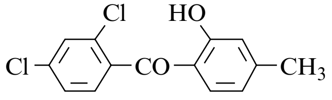
Synthesis

- Preparation by reaction of 2,3-dichlorobenzoyl chloride with p-cresol in the presence of aluminium chloride for 8 h at 190° [77].

m.p. and Spectra (NA).

(2,4-Dichlorophenyl)(2-hydroxy-4-methylphenyl)methanone

[59746-93-9] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



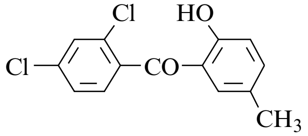
Synthesis

- Preparation by reaction of 2,4-dichlorobenzotrichloride with m-cresol in hydrofluoric acid in the presence of water at −10°, then at 15° overnight and at 80° for 30 min (87%) [213].

m.p. 83° [213]; Spectra (NA).

(2,4-Dichlorophenyl)(2-hydroxy-5-methylphenyl)methanone

$C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



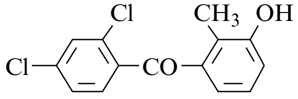
Syntheses

- Preparation by demethylation of 2,4-dichloro-2′-methoxy-5′-methylbenzophenone with excess boiling pyridinium chloride for 30 min (72%) [1073].
- Preparation by Fries rearrangement of p-cresyl 2,4-dichloro-benzoate with aluminium chloride without solvent at 120° for 2 h [1078].
- Preparation by reaction of 2,4-dichlorobenzotrichloride with p-cresol in the presence of aluminium chloride in carbon disulfide at 0° (22–32% yields) [1079].
- Also obtained by sulfuric acid hydrolysis of 6,12-di(2,4-dichlorophenyl)-2,8-dimethyl-6,12-epoxy-*6H,12H*-dibenzo[b,f][1,5]dioxocin (56%) [1079].

m.p. 92°1–92°8 [1079], 92–93° [1078], 91–92° [1073]; Spectra (NA).

(2,4-Dichlorophenyl)(3-hydroxy-2-methylphenyl)methanone

[74167-88-7] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14



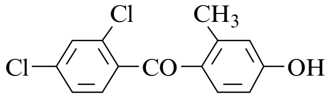
Synthesis

- Preparation by diazotization of 3-amino-2′,4′-dichloro-2-methylbenzophenone, followed by hydrolysis of the resulting diazonium salt (70%) [119], according to [634].

m.p. 98° [119]; Spectra (NA).

(2,4-Dichlorophenyl)(4-hydroxy-2-methylphenyl)methanone

$C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14

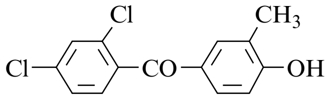


Synthesis

- Obtained by Fries rearrangement of m-cresyl 2,4-di-chlorobenzoate with aluminium chloride without solvent at 120° for 2 h [1078].
- Also refer to: [901] (compound **11**).

m.p. 155° [1078]; Spectra (NA).

(2,4-Dichlorophenyl)(4-hydroxy-3-methylphenyl)methanone



$C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14

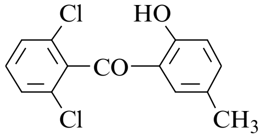
Synthesis

- Preparation by Fries rearrangement of o-cresyl 2,4-di-chlorobenzoate with aluminium chloride without solvent at 120° for 2 h [1078].

m.p. 165° [1078]; Spectra (NA).

(2,6-Dichlorophenyl)(2-hydroxy-5-methylphenyl)methanone

[174186-21-1]



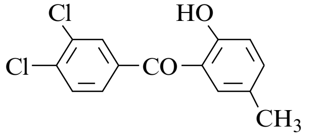
$C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14

Syntheses

- Preparation by reaction of 2,6-dichlorobenzoyl chloride with p-cresol in the presence of aluminium chloride for 8 h at 190° [77].
- Preparation by Fries rearrangement of 4-methylphenyl 2,6-dichlorobenzoate with aluminium chloride in refluxing ethylene dichloride for 3 h (68%) [1080].

m.p. 139–141° [1080]; ^{1}H NMR [1080].

(3,4-Dichlorophenyl)(2-hydroxy-5-methylphenyl)methanone



$C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14

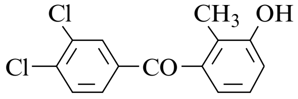
Syntheses

- Preparation by Fries rearrangement of p-cresyl 3,4-dichloro-benzoate with aluminium chloride without solvent at 120° for 2 h [1078].
- Preparation by reaction of 3,4-dichlorobenzotrichloride in the presence of aluminium chloride in carbon disulfide at 0° (56%) [1079].
- Also obtained by sulfuric acid hydrolysis of the 6,12-di(3,4-dichlorophenyl)-2,8-dimethyl-6,12-epoxy-*6H,12H*-dibenzo[b,f][1,5]dioxocin (76%) [1079].

m.p. 90°2–90°6 [1079], 90–91° [1078]; Spectra (NA).

(3,4-Dichlorophenyl)(3-hydroxy-2-methylphenyl)methanone

[76981-65-2]



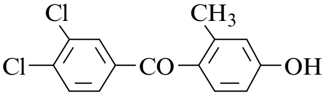
$C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14

Synthesis

- Preparation by diazotization of 3-amino-3′,4′-dichloro-2-methylbenzophenone, followed by hydrolysis of the resulting diazonium salt (48%) [119], according to [634].

m.p. 140° [119]; Spectra (NA).

(3,4-Dichlorophenyl)(4-hydroxy-2-methylphenyl)methanone



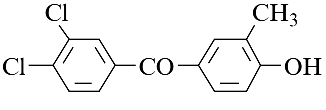
$C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14

Synthesis

- Obtained by Fries rearrangement of m-cresyl 3,4-dichloro-benzoate with aluminium chloride without solvent at 120° for 2 h [1078].

m.p. 95° [1078]; Spectra (NA).

(3,4-Dichlorophenyl)(4-hydroxy-3-methylphenyl)methanone



$C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14

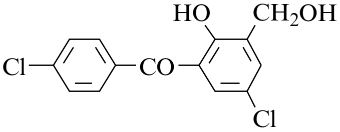
Synthesis

- Preparation by Fries rearrangement of o-cresyl 3,4-dichlorobenzoate with aluminium chloride without solvent at 120° for 2 h [1078].

m.p. 209° [1078]; Spectra (NA).

[5-Chloro-2-hydroxy-3-(hydroxymethyl)phenyl](4-chlorophenyl)methanone

[95304-56-6]



$C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14

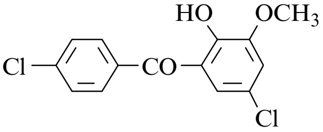
Synthesis

- Refer to: [1081].

m.p. and Spectra (NA).

(5-Chloro-2-hydroxy-3-methoxyphenyl)(4-chlorophenyl)methanone

[95304-54-4]



$C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14

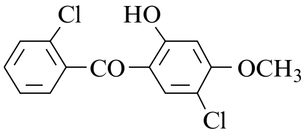
Synthesis

- Refer to: [1081].

m.p. and Spectra (NA).

(5-Chloro-2-hydroxy-4-methoxyphenyl)(2-chlorophenyl)methanone

[136741-43-0]



$C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14

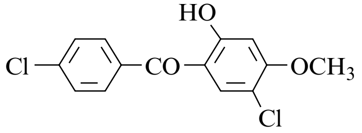
Synthesis

- Preparation by reaction of 2-chlorobenzoyl chloride with 1-chloro-2,4-dimethoxybenzene in the presence of aluminium chloride in ethylene dichloride (77%) [589].

m.p. 98–99° [589]; Spectra (NA).

(5-Chloro-2-hydroxy-4-methoxyphenyl)(4-chlorophenyl)methanone

[87118-99-8] $C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14



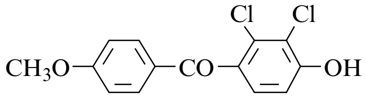
Synthesis

- Preparation by reaction of p-chlorobenzoyl chloride with 1-chloro-2,4-dimethoxybenzene in the presence of aluminium chloride in methylene chloride at r.t. for 20 h (66%) [1082].

crystals [1082]; m.p. (NA); ^{1}H NMR [1082], IR [1082], MS [1082]; TLC [1082].

(2,3-Dichloro-4-hydroxyphenyl)(4-methoxyphenyl)methanone

[78235-18-4] $C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14



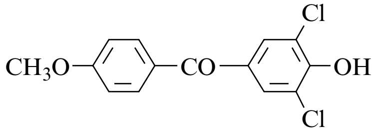
Synthesis

- Preparation by selective demethylation of 2,3-di-chloro-4,4′-dimethoxybenzophenone (SM) with aluminium chloride in refluxing methylene chloride overnight. SM was obtained by Friedel–Crafts acylation of 2,3-dichloroanisole with p-methoxybenzoyl chloride in the presence of aluminium chloride in refluxing methylene chloride [475,979].
- Also refer to: [967,1083].

m.p. and Spectra (NA).

(3,5-Dichloro-4-hydroxyphenyl)(4-methoxyphenyl)methanone

[34183-20-5] $C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14



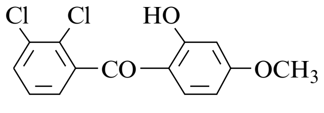
Synthesis

- Preparation by Fries rearrangement of 2,6-dichloro-phenyl p-anisate with aluminium chloride in chloro-benzene at 140–150° for 20 min or in nitrobenzene at 75° for 24 h [480].

m.p. 239–240° [480]; Spectra (NA).

(2,3-Dichlorophenyl)(2-hydroxy-4-methoxyphenyl)methanone

$C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14

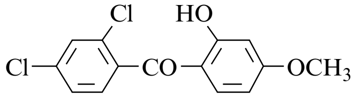


Synthesis

- Preparation by reaction of 2,3-dichlorobenzoyl chloride with m-methoxyphenol in the presence of aluminium chloride for 8 h at 190° [77].

m.p. and Spectra (NA).

(2,4-Dichlorophenyl)(2-hydroxy-4-methoxyphenyl)methanone



$C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14

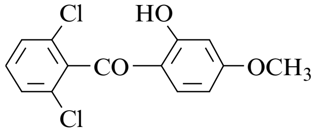
Synthesis

- Preparation by partial demethylation of 2′,4′-dichloro-2,4-dimethoxybenzophenone with aluminium chloride (or aluminium bromide) in chlorobenzene at 90–95° (good yields) [655].

m.p. and Spectra (NA).

(2,6-Dichlorophenyl)(2-hydroxy-4-methoxyphenyl)methanone

[77156-44-6]



$C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14

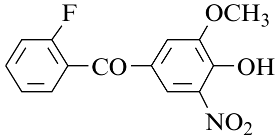
Synthesis

- Preparation by reaction of 2,6-dichlorobenzoyl chloride with m-methoxyphenol in the presence of aluminium chloride for 8 h at 190° [77].

m.p. and Spectra (NA).

(2-Fluorophenyl)(4-hydroxy-3-methoxy-5-nitrophenyl)methanone

[125629-31-4]



$C_{14}H_{10}FNO_5$ mol.wt. 291.24

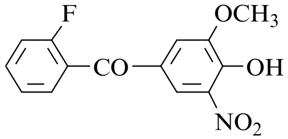
Synthesis

- Preparation by nitration of 2′-fluoro-4-hydroxy-3-methoxy-benzophenone with 65% nitric acid at 20° for 90 min (94%) [1019,1084].

m.p. 150–152° [1084], 127–129° [1019]; ^{1}H NMR [1084], MS [1084].

[2-(Fluoro-^{18}F)phenyl](4-hydroxy-3-methoxy-5-nitrophenyl)methanone

[190585-66-1]



$C_{14}H_{10}FNO_5$ mol.wt. 290.24

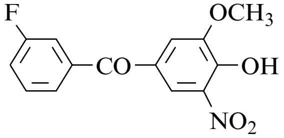
Synthesis

- Obtained by adding methyl iodide to a solution of 2′-[^{18}F] fluoro-3,4-dihydroxy-5-nitrobenzophenone in N,N-di-methylformamide first treated with sodium hydride at 0° (90%). -Refer to: Chem. Abstr., **127**, 17465u (1997).

m.p. and Spectra (NA).

(3-Fluorophenyl)(4-hydroxy-3-methoxy-5-nitrophenyl)methanone

[134612-73-0] $C_{14}H_{10}FNO_5$ mol.wt. 291.24



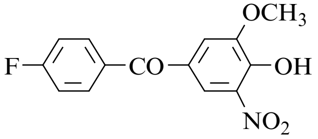
Synthesis

- Preparation by nitration of 3′-fluoro-4-hydroxy-3-methoxy-benzophenone with 65% nitric acid in acetic acid at 20° [1019].

m.p. 168–170° [1019]; Spectra (NA).

(4-Fluorophenyl)(4-hydroxy-3-methoxy-5-nitrophenyl)methanone

[134612-74-1] $C_{14}H_{10}FNO_5$ mol.wt. 291.24



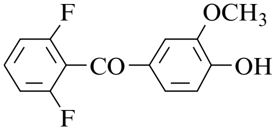
Synthesis

- Preparation by nitration of 4′-fluoro-4-hydroxy-3-methoxybenzophenone with 65% nitric acid in acetic acid at 20° [1019].

m.p. 126–128° [1019]; Spectra (NA).

(2,6-Difluorophenyl)(4-hydroxy-3-methoxyphenyl)methanone

[134612-34-3] $C_{14}H_{10}F_2O_3$ mol.wt. 264.23



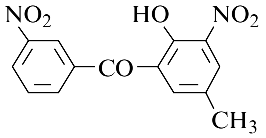
Synthesis

- Preparation by reaction of 33% hydrobromic acid in acetic acid with 4-(benzyloxy)-2′,6′-difluoro-3-methoxy-benzophenone in methylene chloride at 20–25° [1019].

m.p. 130–132° [1019]; Spectra (NA).

(2-Hydroxy-5-methyl-3-nitrophenyl)(3-nitrophenyl)methanone

[67246-06-4] $C_{14}H_{10}N_2O_6$ mol.wt. 302.24



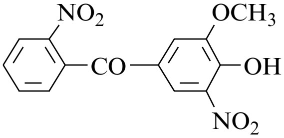
Synthesis

- Obtained by reaction of nitric acid (d = 1.42) with 2-hydroxy-5-methylbenzophenone in acetic acid at 100° [592].

m.p. 118° [592]; ^{1}H NMR [592].

(4-Hydroxy-3-methoxy-5-nitrophenyl)(2-nitrophenyl)methanone

[190522-98-6] $C_{14}H_{10}N_2O_7$ mol.wt. 318.24



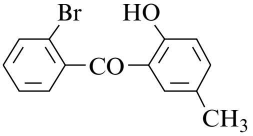
Synthesis

- Preparation by reaction of 85% nitric acid with 4-hydroxy-3-methoxy-2′-nitrobenzophenone in acetic acid, first 15 min at 0°, then 1.5 h at 20° (77%).
- Refer to: Chem. Abstr., **127**, 17465u (1997)^T.

m.p. (NA); ^{1}H NMR^T, MS^T.

(2-Bromophenyl)(2-hydroxy-5-methylphenyl)methanone

[55270-73-0] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



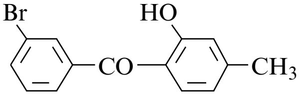
Syntheses

- Preparation by reaction of 2-bromobenzoyl chloride with 4-methoxytoluene in the presence of aluminium chloride,
  - in refluxing carbon disulfide for 3 h (80%) [719];
  - without solvent for 1 h at 150° (68%) [919].
- Preparation by Fries rearrangement of 4-methylphenyl 2-bromobenzoate with aluminium chloride without solvent for 10 min at 140° (quantitative yield) [132].
- Preparation by demethylation of 2′-bromo-2-methoxy-5-methylbenzophenone with excess boiling pyridinium chloride for 1 h (87%) [1073].

m.p. 78°5 [719], 76–77° [132], 76° [1073], 75°5 [919]; Spectra (NA).

(3-Bromophenyl)(2-hydroxy-4-methylphenyl)methanone

$C_{14}H_{11}BrO_2$ mol.wt. 291.14



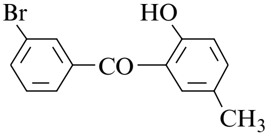
Syntheses

- Preparation by reaction of m-bromobenzoic acid with m-cresol in the presence of alumina in methanesulfonic acid for 1 h at 140° (85%).
- Preparation by Fries rearrangement of m-cresyl m-bromo-benzoate with alumina in methanesulfonic acid for 2 h at 160° (70%). -Refer to: Chem. Abstr., **130**, 81248q (1999)^T.

m.p. 88°^{T}; ^{1}H NMR^T, IR^T, UV^T, MS^T.

(3-Bromophenyl)(2-hydroxy-5-methylphenyl)methanone

[55270-77-4] $C_{14}H_{11}BrO_2$ mol.wt. 291.14

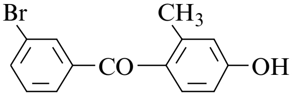


Synthesis

- Preparation by reaction of 3-bromobenzoyl chloride with 4-methoxytoluene in the presence of aluminium chloride without solvent for 1 h at 150° (20%) [919].

m.p. 88–89° [919]; Spectra (NA).

(3-Bromophenyl)(4-hydroxy-2-methylphenyl)methanone



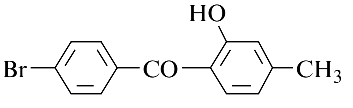
$C_{14}H_{11}BrO_2$ mol.wt. 291.14

Synthesis

- Obtained by reaction of m-bromobenzoic acid with m-cresol in methanesulfonic acid with or without phosphorous pentoxide, for 12 h at 100° (30%).
- Refer to: Chem. Abstr., **130**, 81248q (1999).

m.p. and Spectra (NA).

(4-Bromophenyl)(2-hydroxy-4-methylphenyl)methanone



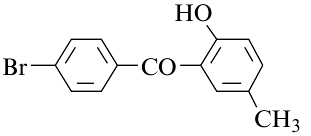
$C_{14}H_{11}BrO_2$ mol.wt. 291.14

Syntheses

- Preparation by acylation of m-cresol with p-bromo-benzoic acid in methanesulfonic acid in the presence of alumina for 40 min at 140° (80%).
- Preparation by Fries rearrangement of m-cresyl p-bromobenzoate with alumina in methanesulfonic acid for 2 h at 160° (83%). -Refer to: Chem. Abstr., **130**, 81248q (1999)^{T}.

m.p. 66°^{T}; ^{1}H NMR^{T}, IR^{T}, UV^{T}, MS^{T}.

(4-Bromophenyl)(2-hydroxy-5-methylphenyl)methanone



$C_{14}H_{11}BrO_2$ mol.wt. 291.14

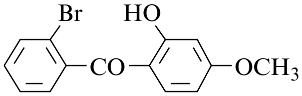
Synthesis

- Preparation by Fries rearrangement of p-cresyl p-bromo-benzoate with aluminium chloride without solvent at 140° for 10 min (90%) [132].

m.p. 79° [132]; b.p.$_{11}$ 210° [132]; Spectra (NA).

(2-Bromophenyl)(2-hydroxy-4-methoxyphenyl)methanone

[183106-15-2]



$C_{14}H_{11}BrO_3$ mol.wt. 307.14

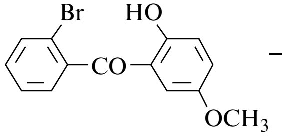
Synthesis

- Preparation by selective demethylation of 2′-bromo-2,4-di-methoxybenzophenone with excess beryllium chloride in refluxing toluene for 3.5 h (90%) [395].

m.p. 96–98° [395]; ^{1}H NMR [395], IR [395], UV [395], MS [395]; TLC [395].

(2-Bromophenyl)(2-hydroxy-5-methoxyphenyl)methanone

[183106-23-2] $C_{14}H_{11}BrO_3$ mol.wt. 307.14



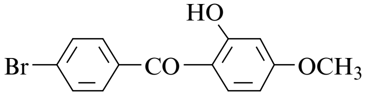
Synthesis

- Preparation by selective demethylation of 2′-bromo-2,5-di-methoxybenzophenone with excess beryllium chloride in refluxing toluene for 3.5 h (90%) [395].

pale yellow oil [395]; b.p. (NA); ^{1}H NMR [395], IR [395], UV [395], MS [395]; TLC [395].

(4-Bromophenyl)(2-hydroxy-4-methoxyphenyl)methanone

$C_{14}H_{11}BrO_3$ mol.wt. 307.14



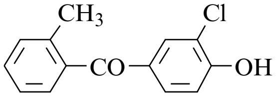
Synthesis

- Refer to: [222,655].

m.p. and Spectra (NA).

(3-Chloro-4-hydroxyphenyl)(2-methylphenyl)methanone

$C_{14}H_{11}ClO_2$ mol.wt. 246.69



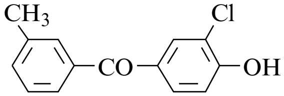
Synthesis

- Obtained by Friedel–Crafts acylation of o-chloroanisole with o-toluoyl chloride in the presence of aluminium chloride in tetrachloroethane at 120–130° for 3 h (25%) [508].

m.p. 128–129° [508]; Spectra (NA).

(3-Chloro-4-hydroxyphenyl)(3-methylphenyl)methanone

$C_{14}H_{11}ClO_2$ mol.wt. 246.69



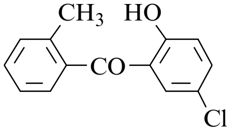
Syntheses

- Obtained (poor yield) by Friedel–Crafts acylation of o-chloroanisole with m-toluoyl chloride in the presence of aluminium chloride in tetrachloroethane at 120–130° for 1 h (14%) [508].
- Preparation by demethylation of 3-chloro-4-methoxy-3′-methylbenzophenone with aluminium chloride in tetrachloroethane at 100–110° for 1 h (quantitative yield) [508].

m.p. 145–146° [508]; Spectra (NA).

(5-Chloro-2-hydroxyphenyl)(2-methylphenyl)methanone

[52980-94-6] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



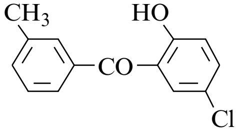
Syntheses

- Preparation by Friedel–Crafts acylation of p-chloroanisole with o-toluoyl chloride in the presence of aluminium chloride in tetrachloroethane at 120–130° for 1 h (49%) [508] or at 150° for 7 h [624].
- Also obtained by reaction of o-toluoyl chloride with p-chlorophenol in the presence of aluminium chloride in tetrachloroethane at 120–130° for 2 h (19%) [508].
- Preparation by Fries rearrangement of p-chlorophenyl o-toluate with aluminium chloride at 100–150° for 0.5–3 h (71%) [29].
- Also refer to: [1085,1086].

m.p. 67°5–68° [508], 67–68° [29], 65–66° [624]; Spectra (NA).

(5-Chloro-2-hydroxyphenyl)(3-methylphenyl)methanone

[52980-95-7] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



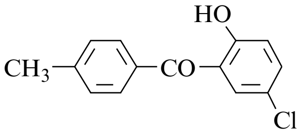
Synthesis

- Preparation by Friedel–Crafts acylation of p-chloroanisole with m-toluoyl chloride in the presence of aluminium chloride in tetrachloroethane at 120–130° for 1 h (63%) [508].
- Also refer to: [1086].

m.p. 106–106°5 [508]; Spectra (NA).

(5-Chloro-2-hydroxyphenyl)(4-methylphenyl)methanone

[116544-78-6] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



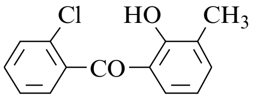
Synthesis

- Preparation by Fries rearrangement of p-chlorophenyl p-toluate with aluminium chloride at 180° for 10 min (75%) [518].

m.p. 90° [518]; ^{1}H NMR [518], IR [518].

(2-Chlorophenyl)(2-hydroxy-3-methylphenyl)methanone

$C_{14}H_{11}ClO_2$ mol.wt. 246.69



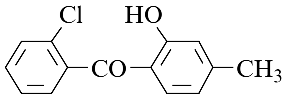
Synthesis

- Obtained (by-product) by Fries rearrangement of o-cresyl o-chlorobenzoate in the presence of aluminium chloride without solvent according to [722], (12%) [1087].

m.p. 72°3–72°8 [1087]; Spectra (NA).

(2-Chlorophenyl)(2-hydroxy-4-methylphenyl)methanone

[107623-97-2] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



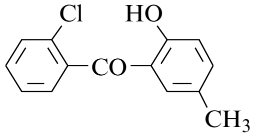
Syntheses

- Preparation by Fries rearrangement of m-cresyl o-chloro-benzoate,
  - with polyphosphoric acid at 130° for 40 min (87%) [1088];
  - with aluminium chloride between 120° and 160° for 2 h [920].
- Preparation by Friedel–Crafts acylation of m-cresol with o-chlorobenzoic acid in the presence of polyphosphoric acid at 110° for 4 h (77%) [1088].
- Also obtained by isomerization of 2′-chloro-4-hydroxy-2-methylbenzophenone (para isomer) with polyphosphoric acid at 130° for 4 h (60%) [1088].

m.p. 106° [920,1088]; ^{1}H NMR [1088], IR [1088], UV [1088].

(2-Chlorophenyl)(2-hydroxy-5-methylphenyl)methanone

[6280-52-0] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



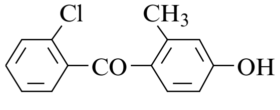
Syntheses

- Preparation by Fries rearrangement of p-cresyl o-chloro-benzoate in the presence of aluminium chloride [23] without solvent at 120° and 160° [132,919,920], (71%) [1087].
- Preparation by reaction of 2-chlorobenzotrichloride with p-cresol in the presence of aluminium chloride in carbon disulfide at 0° (57%) [1079].
- Also obtained by sulfuric acid hydrolysis of 6,12-di(2-chlorophenyl)-2,8-dimethyl-6,12-epoxy-*6H*,*12H*-dibenzo[b,f][1,5]dioxocin (74%) [1079].
- Preparation by reaction of o-chlorobenzoyl chloride with p-cresol in the presence of aluminium chloride at 100° for 4 min, then at 175° for 6 min (61%) [484].
- Preparation by reaction of o-chlorobenzoic acid with p-cresol in the presence of 80% polyphosphoric acid at 190° for 3 h (46%) [923].
- Preparation by demethylation of 2′-chloro-2-methoxy-5-methylbenzophenone with excess boiling pyridinium chloride for 1 h (91%) [1073].
- Also refer to: [1063].

m.p. 80° [920], 78° [132,923], 77–78° [484], 77° [1073], 76°3–77°2 [1087], 76–77° [1079], 75–77° [919]; b.p.$_{15}$ 195° [132], b.p.$_{1-2}$ 141–145° [1079]; IR [923].

(2-Chlorophenyl)(4-hydroxy-2-methylphenyl)methanone

[92103-15-6] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



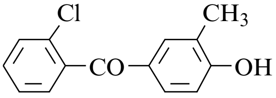
Syntheses

- Preparation by Fries rearrangement of m-cresyl o-chloro-benzoate without solvent in the presence of,
  - aluminium chloride at 160° for 2 h [920];
  - polyphosphoric acid at 70° for 6 h (42%) [1089].

- Also obtained by Friedel–Crafts acylation of m-cresol with o-chlorobenzoic acid in the presence of polyphosphoric acid at 90° for 1 h (31%) [1089].
- Preparation by reaction of o-chlorobenzoyl chloride with m-tolyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (31%) [55].

m.p. 159–160° [920], 153° [55,1089]; ^{1}H NMR [1089], IR [1089], UV [1089].

(2-Chlorophenyl)(4-hydroxy-3-methylphenyl)methanone



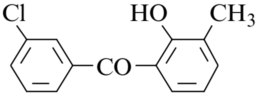
$C_{14}H_{11}ClO_2$ mol.wt. 246.69

Syntheses

- Preparation by action of o-chlorobenzoyl chloride with o-tolyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (72%) [55].
- Preparation by Fries rearrangement of o-cresyl o-chloro-benzoate in the presence of aluminium chloride without solvent (35%) [1087], at 120° or 160° for 2 h [920].

m.p. 167°9–168°6 [1087], 167° [55], 162° [920]; Spectra (NA).

(3-Chlorophenyl)(2-hydroxy-3-methylphenyl)methanone



$C_{14}H_{11}ClO_2$ mol.wt. 246.69

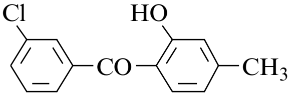
Synthesis

- Obtained (by-product) by Fries rearrangement of o-cresyl m-chlorobenzoate in the presence of aluminium chloride without solvent according to [722], (17%) [1087].

m.p. 69°5–70°3 [1087]; Spectra (NA).

(3-Chlorophenyl)(2-hydroxy-4-methylphenyl)methanone

[67548-59-8]



$C_{14}H_{11}ClO_2$ mol.wt. 246.69

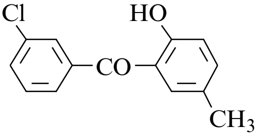
Synthesis

- Preparation by Fries rearrangement of m-cresyl m-chloro-benzoate in the presence of aluminium chloride,
  - without solvent at 120° and 160° [924];
  - in nitrobenzene at 62–63° for 72 h (28%) [31].
- Also refer to: [1090].

m.p. 89°5–90°5 [31], 89° [924]; Spectra (NA).

(3-Chlorophenyl)(2-hydroxy-5-methylphenyl)methanone

[6280-54-2] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



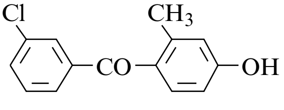
Synthesis

- Preparation by Fries rearrangement of p-cresyl m-chloro-benzoate in the presence of,
  - aluminium chloride without solvent at 120° and 160° [919,924], (45%) [1087];
  - Nafion-H, a polymeric perfluorinated resin sulfonic acid, in refluxing nitrobenzene for 12 h (71%) [38].

m.p. 72° [924], 70°5–71°5 [1087], 70–71°5 [919], 69–70° [629];
^{1}H NMR [629], IR [629], UV [629]; pK_a [104,629]; polarographic study [117].

(3-Chlorophenyl)(4-hydroxy-2-methylphenyl)methanone

$C_{14}H_{11}ClO_2$ mol.wt. 246.69



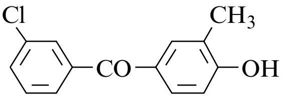
Synthesis

- Obtained by Fries rearrangement of m-cresyl m-chloro-benzoate with aluminium chloride in nitrobenzene at 62–63° for 72 h (40%) [31] or without solvent at 120° or 160° [924].

m.p. 125–125°5 [31], 108° [924]. One of the reported melting points is obviously wrong.
Spectra (NA).

(3-Chlorophenyl)(4-hydroxy-3-methylphenyl)methanone

$C_{14}H_{11}ClO_2$ mol.wt. 246.69



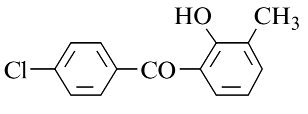
Synthesis

- Preparation by Fries rearrangement of o-cresyl m-chloro-benzoate with aluminium chloride without solvent according to [722], (55%) [1087] and at 120° or 160° [924].

m.p. 151°2–151°8 [1087], 149–150° [924]; Spectra (NA).

(4-Chlorophenyl)(2-hydroxy-3-methylphenyl)methanone

[6279-04-5] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



Syntheses

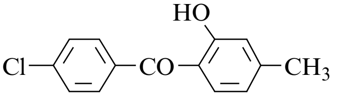
- Obtained (by-product) by Fries rearrangement of o-cresyl p-chlorobenzoate in the presence of aluminium chloride without solvent (12%) [1087].

- Preparation by reaction of 3-methylsalicylic acid chloride (2-hydroxy-3-methylbenzoyl chloride) with chlorobenzene in the presence of aluminium chloride at 100° overnight (47%) [92,709–711,1091].
- Also refer to: [1092,1093].

m.p. 61°5–62° [1087], 61–63° [92,711], 55–58° [709,710,1091];
b.p.$_{0.5}$ 148–152° [709,710,711,1091]; Spectra (NA).

(4-Chlorophenyl)(2-hydroxy-4-methylphenyl)methanone

[107622-28-6] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



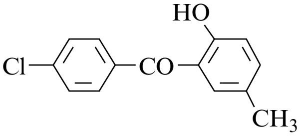
Syntheses

- Preparation by Fries rearrangement of m-cresyl p-chloro-benzoate with aluminium chloride between 120° and 160° (good yield) [926].
- Preparation by reaction of p-chlorobenzoic acid with m-cresol in the presence of boron trifluoride at 160° for 2 h (70%) [150].
- Also obtained by reaction of p-chlorobenzoyl chloride,
  - with m-tolyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (26%) [55];
  - with m-cresol in the presence of aluminium chloride at 105° for 22 h (22%) [92].

m.p. 83–84° [926], 81°5 [150], 80–81° [92]; Spectra (NA).

(4-Chlorophenyl)(2-hydroxy-5-methylphenyl)methanone

[6279-05-6] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



Syntheses

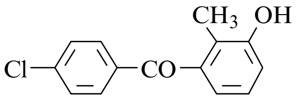
- Preparation by Friedel–Crafts acylation of p-cresol,
  - with p-chlorobenzoic acid in the presence of boron trifluoride at 160° for 5 h (89%) [150];
  - with p-chlorobenzoyl chloride in the presence of aluminium chloride without solvent (78%) [92,711], or in o-dichlorobenzene at 135° for 2 h (quantitative yield) [1094].
- Preparation by Fries rearrangement of p-cresyl p-chlorobenzoate with aluminium chloride without solvent at 120° and 160° [926], (51%) [1087].
- Also obtained by reaction of 4-chlorobenzotrichloride with p-cresol in the presence of aluminium chloride in carbon disulfide at 0° (28%) [1079].
- Also obtained by photo-Fries rearrangement of p-cresyl p-chlorobenzoate in methylene chloride (major product) [1095].
- Also obtained by sulfuric acid hydrolysis of 6,12-di(4-chlorophenyl)-2,8-dimethyl-6,12-epoxy-*6H,12H*-dibenzo[b,f][1,5]dioxocin (93%) [1079].

- Also obtained (poor yield) by diazotization of 2-amino-4′-chloro-5-methylbenzophenone and decomposition of the resulting diazonium salt in 0.1 M sulfuric acid at 60° (10%) [1096].
- Also refer to: [709,710,1091,1093,1097,1098].

m.p. 71° [926], 69° [629], 68° [92], 66°9–67°6 [1087], 66°4–67°2 [1079], 66–67° [150]; b.p.$_{0.1}$ 168–174° [711], b.p.$_{0.25}$ 160–170° [150], b.p.$_{1-2}$ 143–145° [1079]; ^{1}H NMR [629,1094], IR [104,629,1094], UV [629]; pK_a [104,629]; polarographic study [117].

(4-Chlorophenyl)(3-hydroxy-2-methylphenyl)methanone

[74167-86-5] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



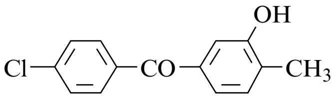
Synthesis

- Preparation by diazotization of 3-amino-4′-chloro-2-methylbenzophenone, followed by hydrolysis of the diazonium salt so obtained (66%) [119], according to [634].
- Also refer to: [123,912].

m.p. 100° [119]; Spectra (NA).

(4-Chlorophenyl)(3-hydroxy-4-methylphenyl)methanone

[74177-55-2] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



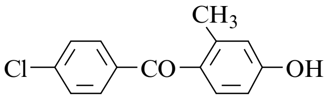
Synthesis

- Refer to: [119,912].

m.p. and Spectra (NA).

(4-Chlorophenyl)(4-hydroxy-2-methylphenyl)methanone

[61002-51-5] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



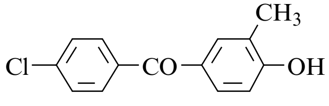
Syntheses

- Preparation by Fries rearrangement of m-cresyl p-chloro-benzoate in the presence of aluminium chloride without solvent at 120° and 160° (good yield) [926].
- Also obtained by reaction of p-chlorobenzoyl chloride,
  - with m-tolyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (11%) [55];
  - with m-cresol in the presence of aluminium chloride in nitrobenzene, first at 15° and at r.t. for 28 h [904].
- Also refer to: [902,1099].

m.p. 116–117° [926]; Spectra (NA).

(4-Chlorophenyl)(4-hydroxy-3-methylphenyl)methanone

[6279-06-7] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



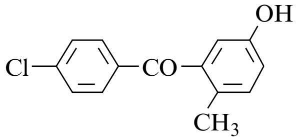
Syntheses

- Preparation by Fries rearrangement of o-cresyl p-chloro-benzoate with aluminium chloride without solvent at 120°and 160° (good yield) [926], (52%) [1087].
- Preparation by reaction of p-chlorobenzoyl chloride with o-tolyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (47%) [55].
- Preparation by reaction of p-chlorobenzoyl chloride with o-cresol in the presence of aluminium chloride [165].

m.p. 211–211°5 [165], 210°5–211°5 [1087], 209–210° [926]; Spectra (NA).

(4-Chlorophenyl)(5-hydroxy-2-methylphenyl)methanone

$C_{14}H_{11}ClO_2$ mol.wt. 246.69



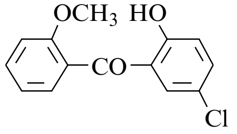
Synthesis

- Preparation by diazotization of 5-amino-4′-chloro-2-methyl-benzophenone, followed by hydrolysis of the resulting diazonium salt (82%) [119], according to [634].

m.p. 188° [119]; Spectra (NA).

(5-Chloro-2-hydroxyphenyl)(2-methoxyphenyl)methanone

[159819-70-2] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



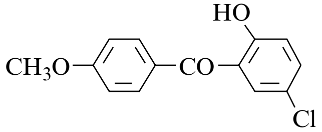
Synthesis

- Preparationfrom2-bromo-4-chloro-(2-methoxyethoxy)-methoxybenzene and o-anisaldehyde as the starting materials [624].

m.p. 94–95° [624]; Spectra (NA).

(5-Chloro-2-hydroxyphenyl)(4-methoxyphenyl)methanone

[85052-20-6] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



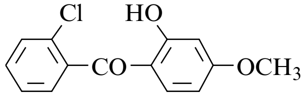
Synthesis

- Preparation by reaction of p-anisoyl chloride with p-chloro-anisole in the presence of aluminium chloride in methylene chloride at r.t. for 18 h under nitrogen (41%) [472].

m.p. (NA); crystals [472]; ^1H NMR [472], IR [472], MS [472]; TLC [472].

(2-Chlorophenyl)(2-hydroxy-4-methoxyphenyl)methanone

[107517-49-7] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



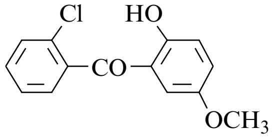
Syntheses

- Preparation by selective demethylation of 2′-chloro-2,4-di-methoxybenzophenone with excess beryllium chloride in refluxing toluene for 3 h (90%) [395].
- Preparation by reaction of o-chlorobenzoyl chloride with resorcinol dimethyl ether in tetrachloroethane in the presence of aluminium chloride at 90° [222].
- Also refer to: [78,666].

m.p. 85–88° [222], 74–75° [395]; ^{1}H NMR [395], IR [395], UV [235,395]; TLC [395].

(2-Chlorophenyl)(2-hydroxy-5-methoxyphenyl)methanone

[183106-21-0] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



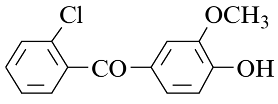
Synthesis

- Preparation by selective demethylation of 2′-chloro-2,5-di-methoxybenzophenone with excess beryllium chloride in refluxing toluene for 3 h (90%) [395].

pale yellow oil [395]; b.p. (NA); ^{1}H NMR [395], IR [395], UV [395], MS [395]; TLC [395].

(2-Chlorophenyl)(4-hydroxy-3-methoxyphenyl)methanone

[134612-35-4] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



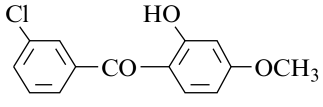
Synthesis

- Preparation by debenzylation of 4-(benzyloxy)-2′-chloro-3-methoxybenzophenone with 33% hydrobromic acid/acetic acid in methylene chloride at 20–25° [1019].

m.p. and Spectra (NA).

(3-Chlorophenyl)(2-hydroxy-4-methoxyphenyl)methanone

[96410-70-7] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



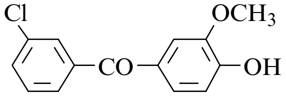
Synthesis

- Preparation by reaction of m-chlorobenzonitrile with m-methoxyphenol in the presence of aluminium chloride for 8 h at 190°, followed by hydrolysis of the ketimine formed [77].
- Also refer to: [1100–1103] (Japanese patents).

m.p. and Spectra (NA).

(3-Chlorophenyl)(4-hydroxy-3-methoxyphenyl)methanone

[134612-36-5] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



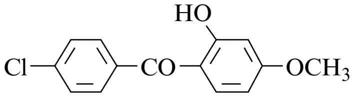
Synthesis

- Preparation by debenzylation of 4-(benzyloxy)-3′-chloro-3-methoxybenzophenone with 33% hydrobromic acid/acetic acid in methylene chloride at 20–25° [1019].

m.p. 136–138° [1019]; Spectra (NA).

(4-Chlorophenyl)(2-hydroxy-4-methoxyphenyl)methanone

[85-28-9] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



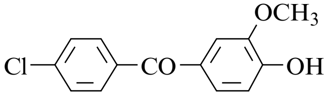
Syntheses

- Preparation by oxidation of 6-methoxy-2-phenyl-3-(4-chlorophenyl)benzofuran with chromium trioxide in boiling acetic acid for 40 min, followed by saponification of the resulting keto ester—2-(benzoyloxy)-4′-chloro-4-methoxybenzophenone—with 4 N sodium hydroxide in refluxing ethanol for 1 h [44].
- Preparation by reaction of p-chlorobenzoyl chloride with resorcinol dimethyl ether,
  - in the presence of aluminium chloride;
    in a chlorobenzene/N,N-dimethylformamide mixture (22:1) at 115° [235,657];
    in tetrachloroethane first at r.t., then at 90° [222];
  - in the presence of titanium tetrachloride in chlorobenzene for 1 h at 120° (74%) [662];
  - in the presence of ferric chloride, without solvent or in o-dichlorobenzene, at 180–200° for 6–7 h (by-product) [663].
- Preparation by partial demethylation of 4′-chloro-2,4-dimethoxybenzophenone with aluminium chloride or aluminium bromide in chlorobenzene at 90–95° (good yield) [655].
- Preparation by Fries rearrangement of m-methoxyphenyl p-chlorobenzoate with aluminium chloride [23].
- Also refer to: [837] (Japanese patent) and [75,77,666,668,669,1104].

m.p. 115° [662], 113° [663], 111° [44], 109–112° [222]; IR [44], UV [235].

(4-Chlorophenyl)(4-hydroxy-3-methoxyphenyl)methanone

[134612-37-6] $C_{14}H_{11}ClO_3$ mol.wt. 262.69

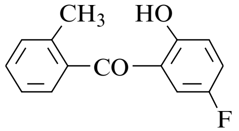


Synthesis

- Preparation by debenzylation of 4-(benzyloxy)-4′-chloro-3-methoxybenzophenone with 33% hydrobromic acid/acetic acid in methylene chloride at 20–25° [1019].

m.p. 114–116° [1019]; Spectra (NA).

(5-Fluoro-2-hydroxyphenyl)(2-methylphenyl)methanone



$C_{14}H_{11}FO_2$ mol.wt. 230.24

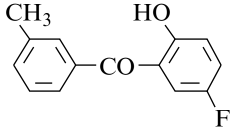
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl o-toluate with aluminium chloride [23].

m.p. and Spectra (NA).

(5-Fluoro-2-hydroxyphenyl)(3-methylphenyl)methanone

[342-18-7]



$C_{14}H_{11}FO_2$ mol.wt. 230.24

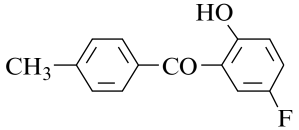
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl m-toluate with aluminium chloride [23] without solvent at 130° for 2 h (92%) [1038].

b.p.$_{15}$ 185° [1038]; Spectra (NA).

(5-Fluoro-2-hydroxyphenyl)(4-methylphenyl)methanone

[62433-29-8]



$C_{14}H_{11}FO_2$ mol.wt. 230.24

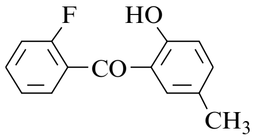
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl p-toluate with aluminium chloride at 150–180° for 20 min (92%) [492] or at 130° for 2 h (82%) [1034].

m.p. 89° [1034], 75–76° [492]; Spectra (NA).

(2-Fluorophenyl)(2-hydroxy-5-methylphenyl)methanone

[55270-76-3]



$C_{14}H_{11}FO_2$ mol.wt. 230.24

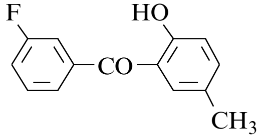
Synthesis

- Preparation by Fries rearrangement of p-cresyl 2-fluoro-benzoate with aluminium chloride [23] without solvent at 160° (36%) [919].

m.p. 72–73° [919]; Spectra (NA).

(3-Fluorophenyl)(2-hydroxy-5-methylphenyl)methanone

[55270-80-9]



$C_{14}H_{11}FO_2$ mol.wt. 230.24

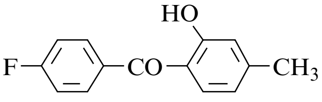
Synthesis

- Preparation by reaction of 3-fluorobenzoyl chloride with 4-methoxytoluene in the presence of aluminium chloride at 150° for 1.5 h (21%) [919].

m.p. 31–34° [919]; b.p.$_{15}$ 70–74° [919]; $n_D^{27.4}$ = 1.5946 [919]; Spectra (NA).

(4-Fluorophenyl)(2-hydroxy-4-methylphenyl)methanone

[108294-71-9] $C_{14}H_{11}FO_2$ mol.wt. 230.24



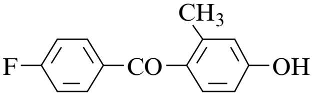
Synthesis

- Obtained (poor yield) by reaction of p-fluorobenzoyl chloride with m-cresol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (9%) [92].

m.p. 78° [92]; Spectra (NA).

(4-Fluorophenyl)(4-hydroxy-2-methylphenyl)methanone

[32192-52-2] $C_{14}H_{11}FO_2$ mol.wt. 230.24



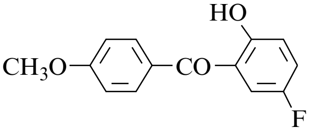
Synthesis

- Preparation by reaction of p-fluorobenzoyl chloride with m-cresol in nitrobenzene in the presence of aluminium chloride first at 0°, then at 60° for 20 h (33%) [1105].

m.p. 110–112° [1105]; ^{1}H NMR [1105].

(5-Fluoro-2-hydroxyphenyl)(4-methoxyphenyl)methanone

[727-93-5] $C_{14}H_{11}FO_3$ mol.wt. 246.24



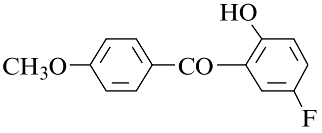
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl p-anisate without solvent,
  - with aluminium chloride at 130° for 2 h (60%) [1038];
  - with titanium tetrachloride at 160° for 20 min (49%) [1106], according to [1107].

m.p. 79°5 [1106]; b.p.$_{30}$ 260° [1038]; ^{1}H NMR [1106,1108], MS [1106,1108]; HPLC [1108].

(5-Fluoro-2-hydroxyphenyl)[4-(methoxy- ^{11}C)phenyl]methanone

[161585-22-4] $C_{14}H_{11}FO_3$ mol.wt. 245.24



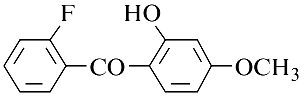
Synthesis

- Preparation by partial methylation of 2,4′-dihydroxy-5-fluorobenzophenone with [^{11}C]methyliodide in N,N-dimethylformamide at –45° [1106].

m.p. (NA); ^{1}H NMR [1106,1108], MS [1106,1108]; HPLC [1108].

(2-Fluorophenyl)(2-hydroxy-4-methoxyphenyl)methanone

[3119-88-8] $C_{14}H_{11}FO_3$ mol.wt. 246.24



Syntheses

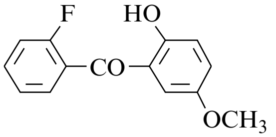
- Preparation by reaction of 2-fluorobenzoyl chloride with resorcinol dimethyl ether in the presence of aluminium chloride in refluxing ethylene dichloride for 30 min (78%) [1031] or refluxing hexane for 8 h [239]. Titanium tetrachloride can also be used instead of aluminium chloride [239].
- Preparation by selective demethylation of 2′-fluoro-2,4-dimethoxybenzophenone with excess beryllium chloride in refluxing toluene for 3 h (92%) [395].

m.p. 149–150° (d) [395], 53–54° [1031], 49–50° [239]. One of the reported melting points is obviously wrong.

^{1}H NMR [395], IR [395], UV [395]; TLC [395]; vapour pressure [248].

(2-Fluorophenyl)(2-hydroxy-5-methoxyphenyl)methanone

[183106-19-6] $C_{14}H_{11}FO_3$ mol.wt. 246.24



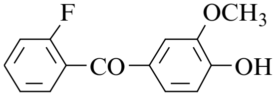
Synthesis

- Preparation by selective demethylation of 2′-fluoro-2,5-di-methoxybenzophenone with excess beryllium chloride in refluxing toluene for 3.5 h (90%) [395].

pale yellow oil [395]; b.p. (NA); ^{1}H NMR [395], IR [395], UV [395], MS [395]; TLC [395].

(2-Fluorophenyl)(4-hydroxy-3-methoxyphenyl)methanone

[125629-30-3] $C_{14}H_{11}FO_3$ mol.wt. 246.24



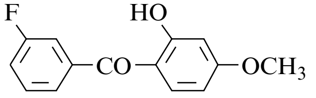
Synthesis

- Preparation by debenzylation of 4-(benzyloxy)-2′-fluoro-3-methoxybenzophenone with 33% hydrobromic acid/acetic acid in methylene chloride at r.t. for 2 h [1019], (92%) [1084].

m.p. 84–86° [1084]; ^{1}H NMR [1084], MS [1084].

(3-Fluorophenyl)(2-hydroxy-4-methoxyphenyl)methanone

$C_{14}H_{11}FO_3$ mol.wt. 246.24

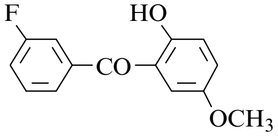


Synthesis

- Preparation by reaction of m-fluorobenzoyl chloride with resorcinol dimethyl ether in the presence of aluminium chloride or titanium tetrachloride in refluxing n-hexane for 8 h [239].

m.p. 88°5–89°5 [239]; Spectra (NA); vapour pressure [248].

(3-Fluorophenyl)(2-hydroxy-5-methoxyphenyl)methanone



$C_{14}H_{11}FO_3$ mol.wt. 246.24

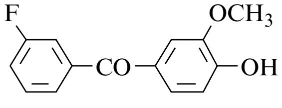
Synthesis

- Obtained by partial demethylation of 3′-fluoro-2,5-di-methoxybenzophenone with aluminium chloride in benzene at 80° [258].

m.p. and Spectra (NA).

(3-Fluorophenyl)(4-hydroxy-3-methoxyphenyl)methanone

[134612-32-1]



$C_{14}H_{11}FO_3$ mol.wt. 246.24

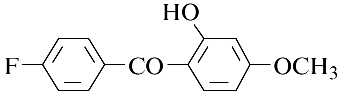
Synthesis

- Preparation by debenzylation of 4-(benzyloxy)-3′-fluoro-3-methoxybenzophenone with 33% hydrobromic acid/acetic acid in methylene chloride at r.t. [1019].

m.p. 133–135° [1019]; Spectra (NA).

(4-Fluorophenyl)(2-hydroxy-4-methoxyphenyl)methanone

[3602-47-9]



$C_{14}H_{11}FO_3$ mol.wt. 246.24

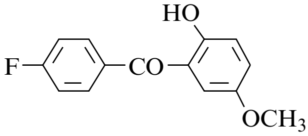
Syntheses

- Preparation by reaction of p-fluorobenzoyl chloride with resorcinol dimethyl ether in the presence of aluminium chloride or titanium tetrachloride in refluxing hexane for 8 h [239].
- Preparation by reaction of dimethyl sulfate with 4′-fluoro-2,4-dihydroxybenzophenone in the presence of 10% potassium hydroxide first at 35°, then at reflux for 30 min (79%) [1109].

m.p. 88–89° [239], 86–88° [1109]; Spectra (NA); vapour pressure [248].

(4-Fluorophenyl)(2-hydroxy-5-methoxyphenyl)methanone

[162657-93-4]



$C_{14}H_{11}FO_3$ mol.wt. 246.24

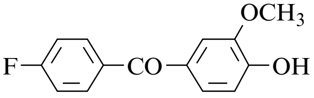
Synthesis

- Preparation by partial demethylation of 4′-fluoro-2,5-di-methoxybenzophenone with aluminium chloride in benzene under nitrogen at 80° for 12 h (66%) [258,680].

m.p. 93° [680]; ^{1}H NMR [680], ^{13}C NMR [680], MS [680].

(4-Fluorophenyl)(4-hydroxy-3-methoxyphenyl)methanone

[134612-33-2] $C_{14}H_{11}FO_3$ mol.wt. 246.24



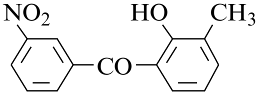
Synthesis

– Preparation by debenzylation of 4-(benzyloxy)-4′-fluoro-3-methoxybenzophenone with 33% hydrobromic acid/acetic acid in methylene chloride at r.t. [1019].

m.p. 139–141° [1019]; Spectra (NA).

(2-Hydroxy-3-methylphenyl)(3-nitrophenyl)methanone

$C_{14}H_{11}NO_4$ mol.wt. 257.25



Syntheses

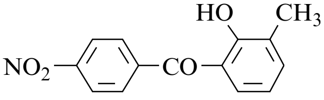
– Obtained (poor yield) by Fries rearrangement of o-tolyl m-nitrobenzoate with aluminium chloride at 120 or at 160° for 2 h (<8%) [958].

– Also obtained (poor yield) by reaction of m-nitrobenzoyl chloride with o-tolyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (3%) [55].

m.p. 115° [958]; Spectra (NA).

(2-Hydroxy-3-methylphenyl)(4-nitrophenyl)methanone

[65611-78-1] $C_{14}H_{11}NO_4$ mol.wt. 257.25



Syntheses

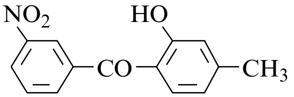
– Preparation by Fries rearrangement of o-tolyl p-nitro-benzoate with aluminium chloride at 120° [961].

– Preparation by reaction between (o-tolyloxy)magnesium bromide complexed with HMPT and p-nitrobenzaldehyde in refluxing benzene for 48 h (68%) [50].

m.p. 118° [961], 113° [50];
^{1}H NMR [50], IR [50], MS [50].

(2-Hydroxy-4-methylphenyl)(3-nitrophenyl)methanone

$C_{14}H_{11}NO_4$ mol.wt. 257.25



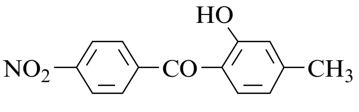
Syntheses

– Obtained by reaction of m-nitrobenzoyl chloride with m-tolyl borate in the presence of aluminium chloride in tetrachloro-ethane at 100° (25%) [55].

– Also obtained by Fries rearrangement of m-tolyl m-nitro-benzoate with aluminium chloride at 120° or at 160° for 2 h (15%) [958].

m.p. 132° [958]; Spectra (NA).

(2-Hydroxy-4-methylphenyl)(4-nitrophenyl)methanone



$C_{14}H_{11}NO_4$ mol.wt. 257.25

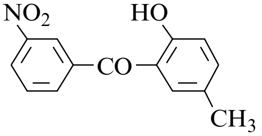
Synthesis

- Preparation by Fries rearrangement of m-tolyl p-nitro-benzoate with aluminium chloride at 120° for 2 h (major product) (<26%) [961].

m.p. 134° [961]; Spectra (NA).

(2-Hydroxy-5-methylphenyl)(3-nitrophenyl)methanone

[53669-31-1] $C_{14}H_{11}NO_4$ mol.wt. 257.25



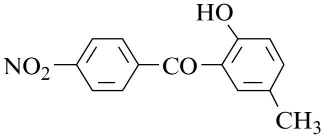
Syntheses

- Preparation by Fries rearrangement of p-cresyl m-nitro-benzoate with aluminium chloride without solvent at 120–160° for 2 h [629], (62%) [958] or in refluxing o-dichlorobenzene for 2 h (30%) [520] or in refluxing chlorobenzene for 3 h (22%) [520].
- Also obtained (by-product) by reaction of m-nitrobenzoyl chloride with p-methylanisole in the presence of aluminium chloride without solvent at 140° for 30 min (11%) [919] or in refluxing carbon disulfide [959].

m.p. 104–105° [958], 102–103° [629], 102° [959], 99–100° [520], 98–100° [919]; ^{1}H NMR [629], IR [104,629,959], UV [629,959]; pK_a [104,629].

(2-Hydroxy-5-methylphenyl)(4-nitrophenyl)methanone

[53669-32-2] $C_{14}H_{11}NO_4$ mol.wt. 257.25

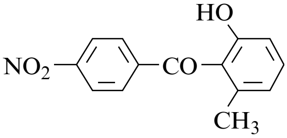


Syntheses

- Preparation by deethylation of 2-ethoxy-5-methyl-4′-nitrobenzophenone (SM) in the presence of aluminium chloride in carbon disulfide at 60–70° for 8 h [141,619] according to [139]. SM was obtained by reaction of p-nitrobenzoyl chloride with p-methylphenetole in the presence of aluminium chloride.
- Preparation by Fries rearrangement of p-cresyl p-nitrobenzoate with aluminium chloride at 160° for 2 h (60%) [961] or at 140° for 30 min (52%) [485]. In this case, the starting ester was obtained by heating p-nitrobenzoyl chloride with aluminium tris(p-methylphenoxide) in a boiling water bath for 30 min [485].
- Also obtained by reaction of p-nitrobenzoyl chloride with p-methylanisole in the presence of aluminium chloride in boiling carbon disulfide during several hours (by-product) [683] or in ethylene dichloride first at 0–5°, then at 50° for 1 h [471].
- Also refer to: [471].

m.p. 149–150° [471], 143° [961], 142–143° [619,683], 142° [485]; IR [104]; pK_a [104]; cryoscopic study [141].

(2-Hydroxy-6-methylphenyl)(4-nitrophenyl)methanone



$C_{14}H_{11}NO_4$ mol.wt. 257.25

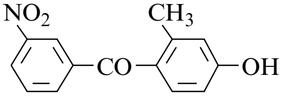
Synthesis

- Obtained (by-product) by Fries rearrangement of m-tolyl p-nitrobenzoate with aluminium chloride at 120° for 2 h (poor yield) [961].

m.p. 143° [961]; Spectra (NA).

(4-Hydroxy-2-methylphenyl)(3-nitrophenyl)methanone

[107558-23-6]



$C_{14}H_{11}NO_4$ mol.wt. 257.25

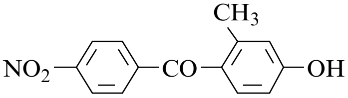
Syntheses

- Obtained by reaction of m-nitrobenzoyl chloride with m-tolyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (17%) [55].
- Also obtained by Fries rearrangement of m-tolyl m-nitro-benzoate with aluminium chloride at 120 or at 160° for 2 h (10%) [958].

m.p. 200° [958]; Spectra (NA).

(4-Hydroxy-2-methylphenyl)(4-nitrophenyl)methanone

[203060-34-8]



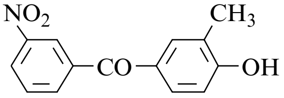
$C_{14}H_{11}NO_4$ mol.wt. 257.25

Syntheses

- Preparation by Fries rearrangement of m-tolyl p-nitro-benzoate with aluminium-chloride at 120° or at 160° for 2 h (21%) [961].
- Preparation by reaction of p-nitrobenzoyl chloride with m-methylanisole in the presence of aluminium chloride in carbon disulfide at 25° for 4 h, followed by demethylation of the keto ether so formed, that is to say 4-methoxy-2-methyl-4′-nitro-benzophenone (68%) [1110].

m.p. 194° [961], 191–192° [1110]; Spectra (NA).

(4-Hydroxy-3-methylphenyl)(3-nitrophenyl)methanone



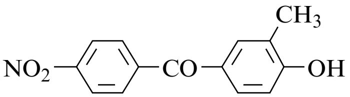
$C_{14}H_{11}NO_4$ mol.wt. 257.25

Syntheses

- Preparation by reaction of m-nitrobenzoyl chloride with o-tolyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (41%) [55].
- Also obtained by Fries rearrangement of o-tolyl m-nitro-benzoate with aluminium chloride at 120° or at 160° for 2 h (15%) [958].

m.p. 182–183° [958]; Spectra (NA).

(4-Hydroxy-3-methylphenyl)(4-nitrophenyl)methanone



$C_{14}H_{11}NO_4$ mol.wt. 257.25

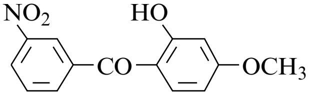
Syntheses

- Preparation by Fries rearrangement of o-tolyl p-nitro-benzoate with aluminium chloride at 120° (25%) [961].
- Preparation by reaction of p-nitrobenzoyl chloride with o-methylanisole in the presence of aluminium chloride in carbon disulfide at 25° for 4 h, followed by demethylation of the resulting keto ether, that is to say 4-methoxy-3-methyl-4′-nitro-benzophenone (66%) [1110].

m.p. 215–216° [1110], 215° [961]; Spectra (NA).

(2-Hydroxy-4-methoxyphenyl)(3-nitrophenyl)methanone

[126077-53-0]



$C_{14}H_{11}NO_5$ mol.wt. 273.25

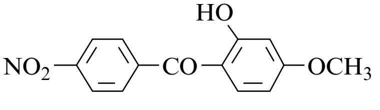
Synthesis

- Refer to: [1111].
- Also refer to: [1112,1113].

m.p. and Spectra (NA).

(2-Hydroxy-4-methoxyphenyl)(4-nitrophenyl)methanone

[6994-36-1]



$C_{14}H_{11}NO_5$ mol.wt. 273.25

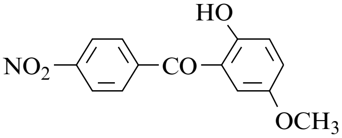
Synthesis

- Preparation by reaction of p-nitrobenzoyl chloride with m-methoxyphenol in the presence of aluminium chloride in carbon disulfide at 25° for 4 h (55%) [1110].
- Also refer to: [627].

m.p. 149° [1110]; Spectra (NA).

(2-Hydroxy-5-methoxyphenyl)(4-nitrophenyl)methanone

[80427-39-0]



$C_{14}H_{11}NO_5$ mol.wt. 273.25

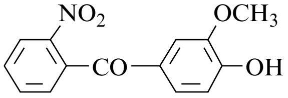
Syntheses

- Preparation by Friedel–Crafts acylation of hydroquinone dimethyl ether with p-nitrobenzoyl chloride in the presence of stannic chloride in nitromethane at 20° for 1 h, followed by demethylation of the resulting ketone (75%) with aluminium chloride in nitromethane at 20° for 24 h (64%) [679].
- Also obtained by Fries rearrangement of p-methoxyphenyl p-nitrobenzoate with titanium tetrachloride without solvent at 120° for 1 h (20–35%) [679].

m.p. 127° [679]; ^{1}H NMR (Sadtler: standard n° 35276 M) [679],
IR (Sadtler: standard n° 62644 K) [679], UV [679].

(4-Hydroxy-3-methoxyphenyl)(2-nitrophenyl)methanone

[190522-97-5] $C_{14}H_{11}NO_5$ mol.wt. 273.25



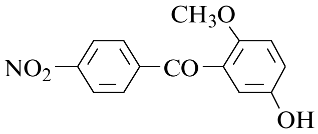
Synthesis

- Preparation by adding a solution of 30% hydrobromic acid to a solution of 4-(benzyloxy)-3-methoxy-2′-nitro-benzophenone in acetic acid, within 10 min at 0° and stirring for 1 h at 20° (98%). -Refer to: Chem. Abstr., **127**, 17465u (1997)^T.

m.p. (NA); ^{1}H NMR^T, MS^T.

(5-Hydroxy-2-methoxyphenyl)(4-nitrophenyl)methanone

[80427-35-6] $C_{14}H_{11}NO_5$ mol.wt. 273.25



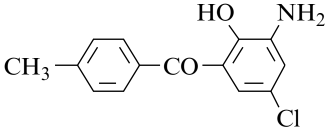
Syntheses

- Preparation by reaction of p-nitrobenzoyl chloride with hydroquinone monomethyl ether in the presence of aluminium chloride in carbon disulfide at 25° for 4 h (50%) [1110].
- Preparation by reaction of p-nitrobenzoyl chloride with p-methoxyphenyl p-nitrobenzoate in the presence of stannic chloride in nitromethane at 20° for 2 days (49%). The m-keto ester formed, the 4-methoxy-3-(4-nitrobenzoyl)phenyl 4-nitrobenzoate, (49%), gave the expected ketone by saponification with sodium hydroxide in refluxing methanol for 1 h (quantitative yield) [679].

m.p. 129° [679], 117° [1110]; ^{1}H NMR (Sadtler: standard n° 35277 M) [679], IR (Sadtler: standard n° 62645 K) [679], UV [679].

(3-Amino-5-chloro-2-hydroxyphenyl)(4-methylphenyl)methanone

$C_{14}H_{12}ClNO_2$ mol.wt. 261.71



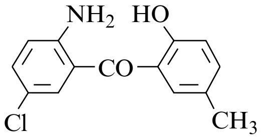
Synthesis

- Refer to: [472].

m.p. and Spectra (NA).

(2-Amino-5-chlorophenyl)(2-hydroxy-5-methylphenyl)methanone

$C_{14}H_{12}ClNO_2$ mol.wt. 261.71



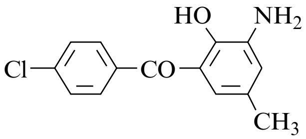
Synthesis

- Obtained from 5-chloro-3-(2-hydroxy-5-methylphenyl)-anthranil (other name: 5-chloro-3-(2-hydroxy-5-methyl-phenyl)-2,1-benzisoxazole) (SM) by reaction with concentrated hydrochloric acid and an excess of tin in boiling acetic acid. SM (m.p. 210°) was prepared by condensation of o-nitrobenzaldehyde with p-cresol in the presence of hydrogen chloride and phosphorous oxychloride in acetic acid [925].

N.B.: Na salt [925].

m.p. 115° [925]; Spectra (NA).

(3-Amino-2-hydroxy-5-methylphenyl)(4-chlorophenyl)methanone



$C_{14}H_{12}ClNO_2$ mol.wt. 261.71

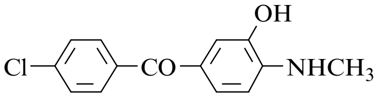
Synthesis

- Refer to: [472].

m.p. and Spectra (NA).

(4-Chlorophenyl)[3-hydroxy-4-(methylamino)phenyl]methanone

[123172-46-3]



$C_{14}H_{12}ClNO_2$ mol.wt. 261.71

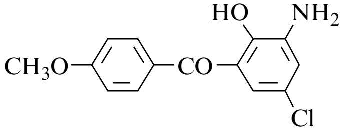
Synthesis

- Preparation by alkaline degradation of 6-(4-chloro-benzoyl)-3-methylbenzoxazolinone (85%) [566].

m.p. 144° [566]; Spectra (NA).

(3-Amino-5-chloro-2-hydroxyphenyl)(4-methoxyphenyl)methanone

[85052-70-6]



$C_{14}H_{12}ClNO_3$ mol.wt. 277.71

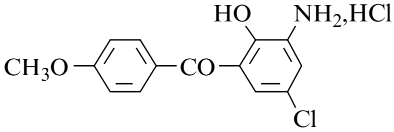
Synthesis

- Preparation by reduction of 5-chloro-2-hydroxy-4′-methoxy-3-nitrobenzophenone with sodium hydrosulfite in a aqueous ammonia/tetrahydrofuran mixture for 15 min (71%) [472].

m.p. 125–135° [472]; ^{1}H NMR [472], IR [472], MS [472]; TLC [472].

(3-Amino-5-chloro-2-hydroxyphenyl)(4-methoxyphenyl)methanone *(Hydrochloride)*

[85052-38-6]



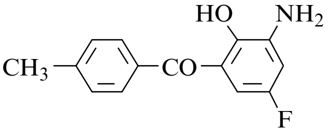
$C_{14}H_{12}ClNO_3,HCl$ mol.wt. 314.18

Synthesis

- Obtained by treatment of 3-amino-5-chloro-2-hydroxy-4′-methoxybenzophenone with concentrated hydrochloric acid [472].

m.p. and Spectra (NA).

(3-Amino-5-fluoro-2-hydroxyphenyl)(4-methylphenyl)methanone



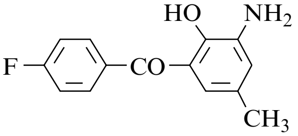
$C_{14}H_{12}FNO_2$ mol.wt. 245.25

Synthesis

- Refer to: [472].

m.p. and Spectra (NA).

(3-Amino-2-hydroxy-5-methylphenyl)(4-fluorophenyl)methanone



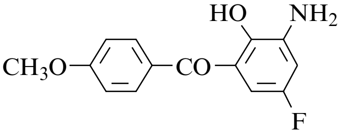
$C_{14}H_{12}FNO_2$ mol.wt. 245.25

Synthesis

- Refer to: [472].

m.p. and Spectra (NA).

(3-Amino-5-fluoro-2-hydroxyphenyl)(4-methoxyphenyl)methanone



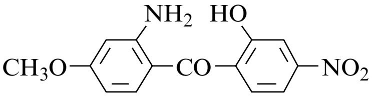
$C_{14}H_{12}FNO_3$ mol.wt. 261.25

Synthesis

- Refer to: [472].

m.p. and Spectra (NA).

(2-Amino-4-methoxyphenyl)(2-hydroxy-4-nitrophenyl)methanone



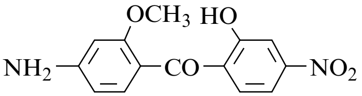
$C_{14}H_{12}N_2O_5$ mol.wt. 288.26

Synthesis

- Preparation by treatment of 2-acetamido-2′-hydroxy-4-methoxy-4′-nitrobenzophenone with refluxing 20% hydrochloric acid for 3 h (91%) [286].

m.p. 224–226° [286]; Spectra (NA).

(4-Amino-2-methoxyphenyl)(2-hydroxy-4-nitrophenyl)methanone



$C_{14}H_{12}N_2O_5$ mol.wt. 288.26

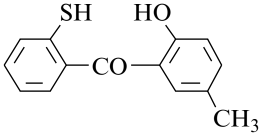
Synthesis

- Preparation by treatment of 4-acetamido-2′-hydroxy-2-methoxy-4′-nitrobenzophenone with refluxing 20% hydrochloric acid for 3 h (78%) [286].

m.p. 176–178° [286]; Spectra (NA).

(2-Hydroxy-5-methylphenyl)(2-mercaptophenyl)methanone

[127024-47-9] $C_{14}H_{12}O_2S$ mol.wt. 244.31



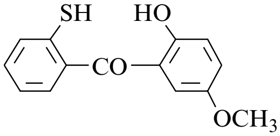
Synthesis

– Obtained by reduction of 2-(2-hydroxy-5-methylbenzoyl)-phenyl disulfide (SM) in the presence of zinc powder in refluxing acetic acid for 2 h (40%) [1114]. SM was obtained (poor yields) by UV light irradiation of p-cresyl 2-mercaptobenzoate or of p-cresyl 2-(acetylthio)benzoate in benzene for 1 h (14% and 7% yields, respectively).

m.p. 164–167° [1114];
^{1}H NMR [1114], IR [1114], MS [1114].

(2-Hydroxy-5-methoxyphenyl)(2-mercaptophenyl)methanone

[127024-46-8] $C_{14}H_{12}O_3S$ mol.wt. 260.31



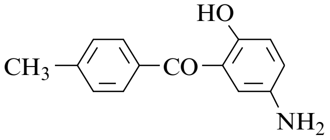
Synthesis

– Obtained by treatment of 2′-(ethoxycarbonylthio)-2-hydroxy-5-methoxybenzophenone (SM) in methanol with potassium carbonate at r.t. for 70 min (33%) [1114]. SM was prepared by UV light irradiation of p-methoxyphenyl 2-(ethoxycarbonylthio)benzoate in benzene for 2 h (39%).

m.p. (NA);
^{1}H NMR [1114], IR [1114], UV [1114], MS [1114].

(5-Amino-2-hydroxyphenyl)(4-methylphenyl)methanone

$C_{14}H_{13}NO_2$ mol.wt. 227.26



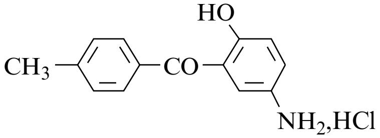
Synthesis

– Obtained by electrolytic reduction of 4-methyl-3′-nitro-benzophenone in concentrated sulfuric acid [567].

m.p. 93° [567]; Spectra (NA).

(5-Amino-2-hydroxyphenyl)(4-methylphenyl)methanone *(Hydrochloride)*

$C_{14}H_{13}NO_2,HCl$ mol.wt. 263.72



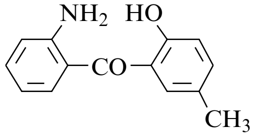
Synthesis

– Preparation from the corresponding amino ketone (see above) [567].

m.p. and Spectra (NA).

(2-Aminophenyl)(2-hydroxy-5-methylphenyl)methanone

[131946-77-5] $C_{14}H_{13}NO_2$ mol.wt. 227.26



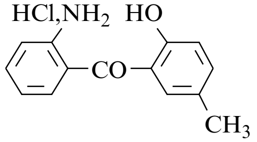
Synthesis

- Obtained (poor yield) by photo-Fries rearrangement of p-cresyl anthranilate (p-cresyl 2-aminobenzoate) in benzene for 10 h (4%) [1115].

m.p. and Spectra (NA).

(2-Aminophenyl)(2-hydroxy-5-methylphenyl)methanone *(Hydrochloride)*

[55270-74-1] $C_{14}H_{13}NO_2,HCl$ mol.wt. 263.72



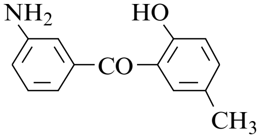
Synthesis

- Obtained by adding 2′-bromo-2-hydroxy-5-methylbenzophenone or 2′-chloro-2-hydroxy-5-methylbenzophenone to a solution of potassium amide in liquid ammonia, isolation of the amino compound, then treatment with 2 N hydrochloric acid (19% and 12% yields, respectively) [919].

m.p. 175–179° [919]; Spectra (NA).

(3-Aminophenyl)(2-hydroxy-5-methylphenyl)methanone

[35486-63-6] $C_{14}H_{13}NO_2$ mol.wt. 227.26



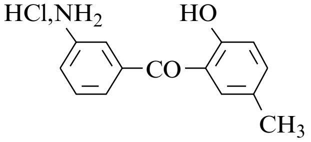
Synthesis

- Preparation by reduction of 2-hydroxy-5-methyl-3′-nitro-benzophenone with ammonium ferrous sulfate (84%) [959].
- Also refer to: [974].

m.p. 115–120° [959]; IR [959], UV [959].

(3-Aminophenyl)(2-hydroxy-5-methylphenyl)methanone *(Hydrochloride)*

[55270-78-5] $C_{14}H_{13}NO_2,HCl$ mol.wt. 263.72



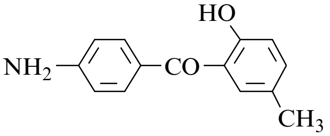
Syntheses

- Obtained by adding 3′-chloro-2-hydroxy-5-methyl-benzophenone to a solution of potassium amide in liquid ammonia, isolation of the amino compound, then treatment with 2 N hydrochloric acid (20%) [919].
- Also obtained by adding aqueous ammonia to a mixture of 2-hydroxy-5-methyl-3′-nitrobenzophenone and ferrous sulfate in aqueous ethanol at 80–85°, isolation of the amino compound, then treatment with 2 N hydrochloric acid (33%) [919].

m.p. 165–167° [919]; Spectra (NA).

(4-Aminophenyl)(2-hydroxy-5-methylphenyl)methanone

[106612-60-6] $C_{14}H_{13}NO_2$ mol.wt. 227.26



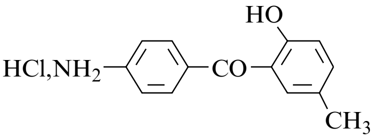
Syntheses

- Preparation by hydrogenation of 2-hydroxy-5-methyl-4′-nitrobenzophenone in the presence of 10% Pd/C as a catalyst in ethanol under pressure (0.14 MPa) for 1 h [471].
- Also obtained by reduction of 2-methoxy-5-methyl-4′-nitrobenzophenone (m.p. 101–102°) [683],
  - with stannous chloride/hydrochloric acid (poor yield);
  - with ammonium sulfide in refluxing ethanol (good yield).

m.p. 137–138° [471], 137° [683]; Spectra (NA).

(4-Aminophenyl)(2-hydroxy-5-methylphenyl)methanone *(Hydrochloride)*

$C_{14}H_{13}NO_2,HCl$ mol.wt. 263.72



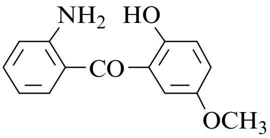
Synthesis

- Preparation by action of hydrochloric acid with the corresponding amino ketone (see above) in ethyl ether [683].

m.p. and Spectra (NA).

(2-Aminophenyl)(2-hydroxy-5-methoxyphenyl)methanone

[131946-76-4] $C_{14}H_{13}NO_3$ mol.wt. 243.26



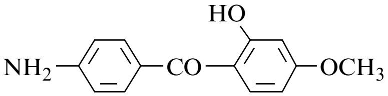
Syntheses

- Preparation by saponification of 2′-acetylamino-2-hydroxy-5-methoxybenzophenone with 10% sodium hydroxide (25%) [1115].
- Also obtained (poor yield) by photo-Fries rearrangement of p-methoxyphenyl o-aminobenzoate in benzene for 5 h (7%) [1115].

m.p. (NA); ^{1}H NMR [1115], IR [1115], UV [1115], MS [1115].

(4-Aminophenyl)(2-hydroxy-4-methoxyphenyl)methanone

[6994-37-2] $C_{14}H_{13}NO_3$ mol.wt. 243.26

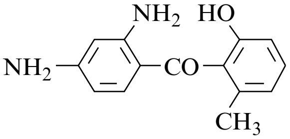


Synthesis

- Refer to: [228].

m.p. and Spectra (NA).

(2,4-Diaminophenyl)(2-hydroxy-6-methylphenyl)methanone



$C_{14}H_{14}N_2O_2$ mol.wt. 242.28

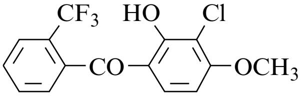
Synthesis

- Preparation by reaction of 2,4-diaminobenzoyl chloride with trimethylsilyl derivative of m-cresol in the presence of stannic chloride (or titanium tetrachloride or aluminium chloride) in refluxing methylene chloride for 2 h [921].

m.p. and Spectra (NA).

(3-Chloro-2-hydroxy-4-methoxyphenyl)[2-(trifluoromethyl)phenyl] methanone

[72482-16-7]



$C_{15}H_{10}ClF_3O_3$ mol.wt. 330.69

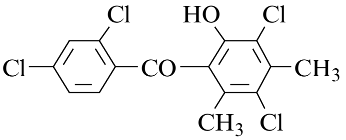
Synthesis

- Preparation by reaction of o-(trifluoromethyl) benzoyl chloride with 2-chlororesorcinol dimethyl ether in ethylene dichloride in the presence of ferric chloride first at 5–7°, then at r.t. for 18 h and at reflux for 30 min [476].

m.p. 101–102° [476]; Spectra (NA).

(3,5-Dichloro-2-hydroxy-4,6-dimethylphenyl)(2,4-dichlorophenyl)methanone

[34174-15-7]



$C_{15}H_{10}Cl_4O_2$ mol.wt. 364.05

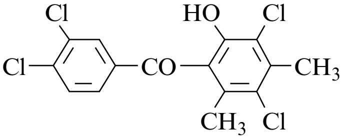
Synthesis

- Preparation by Fries rearrangement of 2, 4-dichloro-3,5-dimethylphenyl 2,4-dichlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 129–130° [458]; Spectra (NA).

(3,5-Dichloro-2-hydroxy-4,6-dimethylphenyl)(3,4-dichlorophenyl)methanone

[34174-14-6]



$C_{15}H_{10}Cl_4O_2$ mol.wt. 364.05

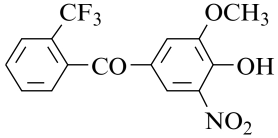
Synthesis

- Preparation by Fries rearrangement of 2, 4-dichloro-3,5-dimethylphenyl 3,4-dichlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 136–137° [458]; Spectra (NA).

(4-Hydroxy-3-methoxy-5-nitrophenyl)[2-(trifluoromethyl)phenyl]methanone

[134612-82-1] $C_{15}H_{10}F_3NO_5$ mol.wt. 341.24



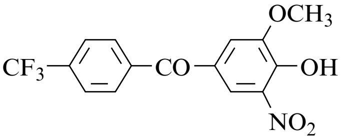
Synthesis

- Preparation by nitration of 4-hydroxy-3-methoxy-2′-(tri-fluoromethyl)benzophenone with 65% nitric acid in acetic acid at 20° [1019].

m.p. 138–140° [1019]; Spectra (NA).

(4-Hydroxy-3-methoxy-5-nitrophenyl)[4-(trifluoromethyl)phenyl]methanone

[134611-75-9] $C_{15}H_{10}F_3NO_5$ mol.wt. 341.24



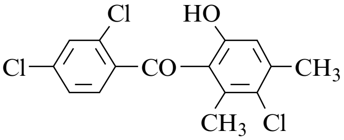
Synthesis

- Preparation by nitration of 4-hydroxy-3-methoxy-4′-(trifluoromethyl)benzophenone with 65% nitric acid in acetic acid at 20° for 90 min [1019].

m.p. 172° [1019]; Spectra (NA).

(3-Chloro-6-hydroxy-2,4-dimethylphenyl)(2,4-dichlorophenyl)methanone

[34171-56-7] $C_{15}H_{11}Cl_3O_2$ mol.wt. 329.61



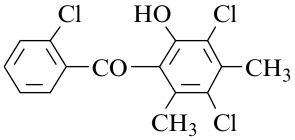
Synthesis

- Preparation by Fries rearrangement of 4-chloro-3,5-di-methylphenyl 2,4-dichlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 103–104° [458]; Spectra (NA).

(2-Chlorophenyl)(3,5-dichloro-2-hydroxy-4,6-dimethylphenyl)methanone

[34174-16-8] $C_{15}H_{11}Cl_3O_2$ mol.wt. 329.61



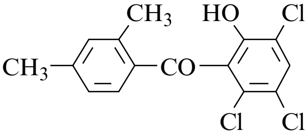
Synthesis

- Preparation by Fries rearrangement of 2,4-dichloro-3,5-di-methylphenyl o-chlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 96–97° [458]; Spectra (NA).

(2,4-Dimethylphenyl)(2,3,5-trichloro-6-hydroxyphenyl)methanone

[34174-01-1] $C_{15}H_{11}Cl_3O_2$ mol.wt. 329.61



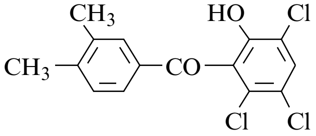
Synthesis

- Preparation by Fries rearrangement of 2,4,5-trichlorophenyl 2,4-dimethylbenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 103–104° [458]; Spectra (NA).

(3,4-Dimethylphenyl)(2,3,5-trichloro-6-hydroxyphenyl)methanone

[34174-00-0] $C_{15}H_{11}Cl_3O_2$ mol.wt. 329.61



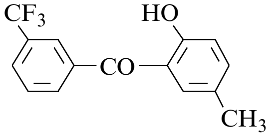
Synthesis

- Preparation by Fries rearrangement of 2,4,5-trichloro-phenyl 3,4-dimethylbenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 163–164° [458]; Spectra (NA).

(2-Hydroxy-5-methylphenyl)[3-(trifluoromethyl)phenyl]methanone

$C_{15}H_{11}F_3O_2$ mol.wt. 280.25



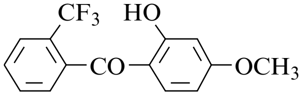
Synthesis

- Preparation by reaction of m-(trifluoromethyl) benzoyl chloride with p-cresol in the presence of aluminium chloride for 8 h at 190° [77].

m.p. and Spectra (NA).

(2-Hydroxy-4-methoxyphenyl)[2-(trifluoromethyl)phenyl]methanone

[3119-86-6] $C_{15}H_{11}F_3O_3$ mol.wt. 296.21



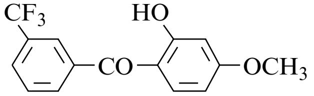
Synthesis

- Preparation by reaction of o-(trifluoromethyl) benzoyl chloride with resorcinol dimethyl ether in the presence of aluminium chloride or titanium tetrachloride in refluxing n-hexane for 8 h [239].
- Also refer to: [248].

m.p. 95–95°5 [239]; b.p. 358–362° [239];
Spectra (NA); vapour pressure [248].

(2-Hydroxy-4-methoxyphenyl)[3-(trifluoromethyl)phenyl]methanone

[7396-89-6] $C_{15}H_{11}F_3O_3$ mol.wt. 296.21



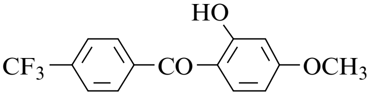
Syntheses

- Preparation by reaction of m-(trifluoromethyl) benzoyl chloride with resorcinol dimethyl ether in the presence of aluminium chloride or titanium tetrachloride in refluxing n-hexane for 8 h [239].
- Preparation by reaction of m-(trifluoromethyl)benzonitrile with m-methoxyphenol in the presence of aluminium chloride for 8 h at 190°, followed by hydrolysis of the ketimine so formed [77].

m.p. 65°5–66° [239]; b.p. 360–362° [239];
Spectra (NA); vapour pressure [248].

(2-Hydroxy-4-methoxyphenyl)[4-(trifluoromethyl)phenyl]methanone

[7396-90-9] $C_{15}H_{11}F_3O_3$ mol.wt. 296.21



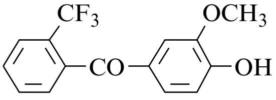
Synthesis

– Preparation by reaction of p-(trifluoromethyl)benzoyl chloride with resorcinol dimethyl ether in the presence of aluminium chloride or titanium tetrachloride in refluxing n-hexane for 8 h [239].
– Also refer to: [248].

m.p. 66°5–67° [239]; b.p. 380–385° [239]; Spectra (NA); vapour pressure [248].

(4-Hydroxy-3-methoxyphenyl)[2-(trifluoromethyl)phenyl]methanone

[134612-41-2] $C_{15}H_{11}F_3O_3$ mol.wt. 296.21



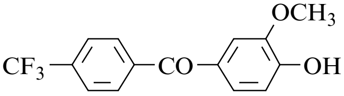
Synthesis

– Preparation by reaction of 33% hydrobromic acid in acetic acid with 4-(benzyloxy)-3-methoxy-2′-(trifluoromethyl)- benzophenone in methylene chloride at 20–25° [1019].

m.p. 115–117° [1019]; Spectra (NA).

(4-Hydroxy-3-methoxyphenyl)[4-(trifluoromethyl)phenyl]methanone

[134611-74-8] $C_{15}H_{11}F_3O_3$ mol.wt. 296.21



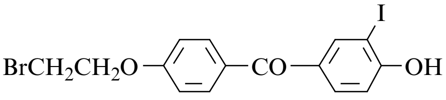
Synthesis

– Preparation by reaction of 33% hydrobromic acid in acetic acid with 4-(benzyloxy)-3-methoxy-4′-(trifluoromethyl)-benzophenone in methylene chloride at 20° for 90 min [1019].

m.p. 97° [1019]; Spectra (NA).

[4-(2-Bromoethoxy)phenyl](4-hydroxy-3-iodophenyl)methanone

[79578-67-9] $C_{15}H_{12}BrIO_3$ mol.wt. 447.07



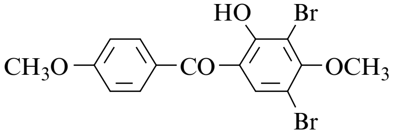
Synthesis

– Obtained (poor yield) by reaction of 3-iodo-4-hydroxybenzoic acid with β-bromophenetole in solution of a polyphosphoric acid/85% phosphoric acid/zinc chloride mixture. The solution was heated at 50°, phosphorous trichloride was added during 1 h and the mixture was heated for 2.5 h at 70° (11%) [1002].

m.p. 175–176°5 [1002]; ^{1}H NMR [1002], IR [1002], MS [1002].

(3,5-Dibromo-2-hydroxy-4-methoxyphenyl)(4-methoxyphenyl)methanone

[66666-25-9] $C_{15}H_{12}Br_2O_4$ mol.wt. 416.07



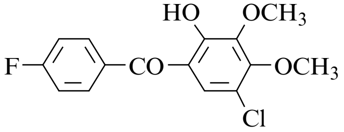
Synthesis

– Preparation by saponification of 2-(p-anisoyloxy)-3,5-dibromo-4,4′-dimethoxybenzophenone (SM) with potassium hydroxide in refluxing ethanol for 1 h (66%). SM was obtained by oxidation of 5,7-dibromo-6-methoxy-2,3-bis(p-methoxyphenyl)-benzofuran with chromium trioxide in refluxing acetic acid for 45 min (70%) [1116].

m.p. 200° [1116]; Spectra (NA).

(5-Chloro-2-hydroxy-3,4-dimethoxyphenyl)(4-fluorophenyl)methanone

[140665-40-3] $C_{15}H_{12}ClFO_4$ mol.wt. 310.71



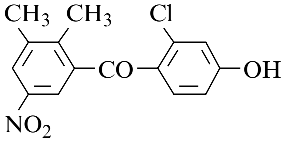
Synthesis

– Preparation by reaction of sulfuryl chloride with 4′-fluoro-2-hydroxy-3,4-dimethoxybenzophenone in methylene chloride at r.t. overnight (69%) [704].

m.p. 130–131° [704]; Spectra (NA).

(2-Chloro-4-hydroxyphenyl)(2,3-dimethyl-5-nitrophenyl)methanone

[110969-66-9] $C_{15}H_{12}ClNO_4$ mol.wt. 305.72



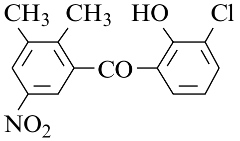
Synthesis

– Obtained (by-product) by Fries rearrangement of m-chlorophenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (11%) [1003].

m.p. 214° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(3-Chloro-2-hydroxyphenyl)(2,3-dimethyl-5-nitrophenyl)methanone

[110969-62-5] $C_{15}H_{12}ClNO_4$ mol.wt. 305.72



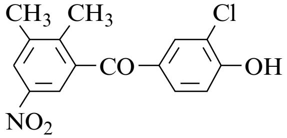
Synthesis

– Obtained (by-product) by Fries rearrangement of o-chlorophenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (6%) [1003].

m.p. 130° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(3-Chloro-4-hydroxyphenyl)(2,3-dimethyl-5-nitrophenyl)methanone

[110969-63-6] $C_{15}H_{12}ClNO_4$ mol.wt. 305.72



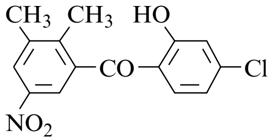
Synthesis

- Preparation by Fries rearrangement of o-chlorophenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (60%) [1003].

m.p. 195° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(4-Chloro-2-hydroxyphenyl)(2,3-dimethyl-5-nitrophenyl)methanone

[110969-65-8] $C_{15}H_{12}ClNO_4$ mol.wt. 305.72



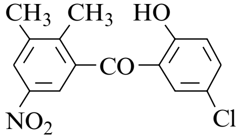
Synthesis

- Preparation by Fries rearrangement of m-chlorophenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (40%) [1003].

m.p. 154° [1003]; ^{1}H NMR [1003], IR [1003], UV[1003].

(5-Chloro-2-hydroxyphenyl)(2,3-dimethyl-5-nitrophenyl)methanone

[110969-68-1] $C_{15}H_{12}ClNO_4$ mol.wt. 305.72



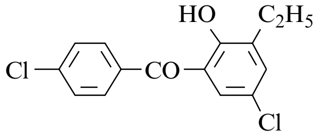
Synthesis

- Preparation by Fries rearrangement of p-chlorophenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (70%) [1003].

m.p. 147° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(5-Chloro-3-ethyl-2-hydroxyphenyl)(4-chlorophenyl)methanone

[93575-71-4] $C_{15}H_{12}Cl_2O_2$ mol.wt. 295.16



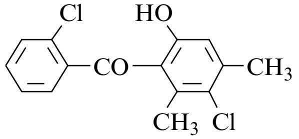
Synthesis

- Preparation by Fries rearrangement of 4-chloro-2-ethyl-phenyl p-chlorobenzoate with aluminium chloride at 160° for 15 min [1107].

m.p. 47–49° [1107]; Spectra (NA).

(3-Chloro-6-hydroxy-2,4-dimethylphenyl)(2-chlorophenyl)methanone

[34174-03-3] $C_{15}H_{12}Cl_2O_2$ mol.wt. 295.16



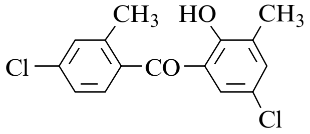
Synthesis

- Preparation by Fries rearrangement of 4-chloro-3,5-di-methylphenyl o-chlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 91–92° [458]; Spectra (NA).

(5-Chloro-2-hydroxy-3-methylphenyl)(4-chloro-2-methylphenyl)methanone

[86914-83-2] $C_{15}H_{12}Cl_2O_2$ mol.wt. 295.16



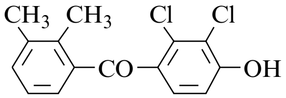
Synthesis

– Preparation by Fries rearrangement of 4-chloro-2-methyl-phenyl 4-chloro-2-methylbenzoate with aluminium chloride at 180° for 10 min [1074].

m.p. 53–54° [1074]; Spectra (NA).

(2,3-Dichloro-4-hydroxyphenyl)(2,3-dimethylphenyl)methanone

[72482-89-4] $C_{15}H_{12}Cl_2O_2$ mol.wt. 295.16



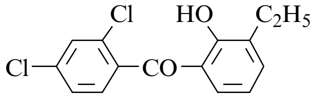
Synthesis

– Preparation by demethylation of 2,3-dichloro-4-methoxy-2′,3′-dimethylbenzophenone (SM) with aluminium chloride in refluxing benzene for 5 h, then at r.t. for 18 h. SM was obtained by reaction of 2,3-dimethylbenzoyl chloride with 2,3-dichloroanisole in ethylene dichloride in the presence of aluminium chloride at 60° for 1 h [476].

m.p. and Spectra (NA).

(2,4-Dichlorophenyl)(3-ethyl-2-hydroxyphenyl)methanone

[61466-78-2] $C_{15}H_{12}Cl_2O_2$ mol.wt. 295.16



Synthesis

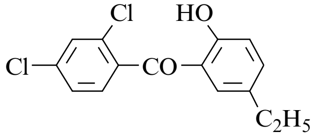
– Obtained by reaction of 2,4-dichlorobenzoyl chloride with o-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 21–22 h (86–91%) [710,1091], (6%) [92].

oil [92]; b.p.$_{0.06}$ 156–172° [710,1091]. A typing error probably occurred in the published data.

n_D^{22} = 1.6163 [710,1091]; Spectra (NA).

(2,4-Dichlorophenyl)(5-ethyl-2-hydroxyphenyl)methanone

[61466-83-9] $C_{15}H_{12}Cl_2O_2$ mol.wt. 295.16



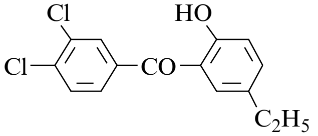
Synthesis

– Preparation by reaction of 2,4-dichlorobenzoyl chloride with p-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (34%) [92,1091].

m.p. 44°6 [1091], 44–46° [92]; b.p.$_{0.45}$ 148–151° [1091]; Spectra (NA).

(3,4-Dichlorophenyl)(5-ethyl-2-hydroxyphenyl)methanone

[61466-87-3] $C_{15}H_{12}Cl_2O_2$ mol.wt. 295.16



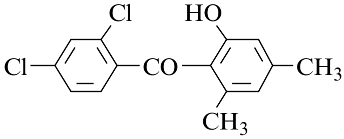
Synthesis

- Preparation by reaction of 3,4-dichlorobenzoyl chloride with p-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (30%) [92], (17%) [1091].

m.p. 50° [1091]; b.p.$_{0.35}$ 175–177° [92,1091]; Spectra (NA).

(2,4-Dichlorophenyl)(2-hydroxy-4,6-dimethylphenyl)methanone

[34203-52-6] $C_{15}H_{12}Cl_2O_2$ mol.wt. 295.16



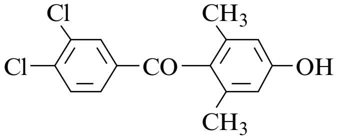
Synthesis

- Preparation by Fries rearrangement of 3,5-dimethyl-phenyl 2,4-dichlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 94–95° [458]; Spectra (NA).

(3,4-Dichlorophenyl)(4-hydroxy-2,6-dimethylphenyl)methanone

[34183-02-3] $C_{15}H_{12}Cl_2O_2$ mol.wt. 295.16



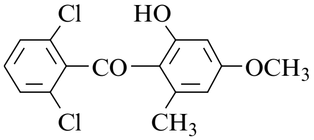
Synthesis

- Obtained by Fries rearrangement of 3,5-dimethylphenyl 3,4-dichlorobenzoate with aluminium chloride in chloro-benzene for 20 min at 140–150° or in nitrobenzene for 24 h at 75° [480].

m.p. 138–139° [480]; Spectra (NA).

(2,6-Dichlorophenyl)(2-hydroxy-4-methoxy-6-methylphenyl)methanone

[183726-73-0] $C_{15}H_{12}Cl_2O_3$ mol.wt. 311.16



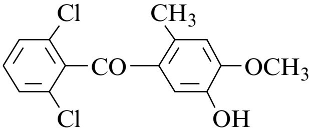
Synthesis

- Refer to: [1072].

m.p. and Spectra (NA).

(2,6-Dichlorophenyl)(5-hydroxy-4-methoxy-2-methylphenyl)methanone

[183724-10-9] $C_{15}H_{12}Cl_2O_3$ mol.wt. 311.16



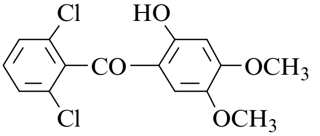
Synthesis

- Preparation by partial demethylation of 2′,6′-dichloro-4,5-dimethoxy-2-methylbenzophenone with 33% hydrobromic acid in acetic acid for 1.5 h at 75° (45%) [1072].

m.p. 152° [1072]; Spectra (NA).

(2,6-Dichlorophenyl)(2-hydroxy-4,5-dimethoxyphenyl)methanone

[183725-20-4] $C_{15}H_{12}Cl_2O_4$ mol.wt. 327.16



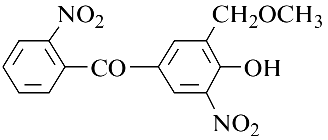
Syntheses

- Preparation by Friedel–Crafts acylation of 3,4-dimethoxy-phenol with 2,6-dichlorobenzoyl chloride [1072].
- Preparation by partial demethylation of 2′,6′-dichloro-2,4,5-trimethoxybenzophenone [1072].
- Preparation by diazotization of 2-amino-2′,6′-dichloro-4,5-dimethoxybenzophenone (compound **3**) [1072].

m.p. 80° [1072]; Spectra (NA).

[4-Hydroxy-3-(methoxymethyl)-5-nitrophenyl](2-nitrophenyl)methanone

$C_{15}H_{12}N_2O_7$ mol.wt. 332.27



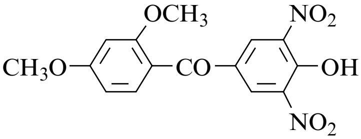
Synthesis

- Obtained by reaction of chloromethyl methyl ether in methylene chloride with 3,4-dihydroxy-2′,5-dinitro-benzophenone in tetrahydrofuran in the presence of N,N-diisopropylethylamine (Huenig′s base) for 40 min at 0° (41%). -Refer to: Chem. Abstr., **127**, 17465u (1997)^T.

m.p. (NA); ^{1}H NMR^T.

(2,4-Dimethoxyphenyl)(4-hydroxy-3,5-dinitrophenyl)methanone

[67246-02-0] $C_{15}H_{12}N_2O_8$ mol.wt. 348.27



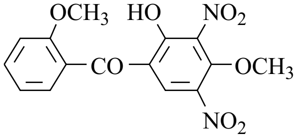
Synthesis

- Obtained by reaction of nitric acid (d = 1.42) with 4-hydroxy-2′,4′-dimethoxybenzophenone in acetic acid at 32° [592].

m.p. 157° [592]; ^{1}H NMR [592].

(2-Hydroxy-4-methoxy-3,5-dinitrophenyl)(2-methoxyphenyl)methanone

[79204-71-0] $C_{15}H_{12}N_2O_8$ mol.wt. 348.27



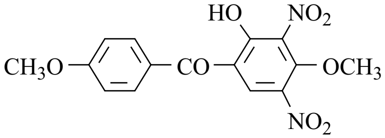
Synthesis

– Preparation by oxidation of 2,3-bis(2-methoxyphenyl)-5,7-dinitro-6-methoxybenzofuran with chromium trioxide in acetic acid, followed by saponification of the keto ester so formed with potassium hydroxide in ethanol (60%) [1117].

m.p. 120° [1117]; IR [1117].

(2-Hydroxy-4-methoxy-3,5-dinitrophenyl)(4-methoxyphenyl)methanone

[66666-08-8] $C_{15}H_{12}N_2O_8$ mol.wt. 348.27



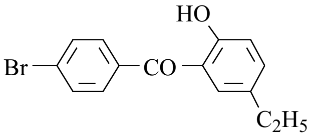
Synthesis

– Preparation by saponification of 2-(p-anisoyloxy)-4,4′-dimethoxy-3,5-dinitrobenzophenone (SM) with potassium hydroxide in refluxing ethanol for 1 h (70%). SM was obtained by oxidation of 5,7-di-nitro-6-methoxy-2,3-bis(p-methoxyphenyl)-benzofuran with chromium trioxide in refluxing acetic acid for 45 min (72%) [1116].

m.p. 140° [1116]; Spectra (NA).

(4-Bromophenyl)(5-ethyl-2-hydroxyphenyl)methanone

[108294-74-2] $C_{15}H_{13}BrO_2$ mol.wt. 305.17



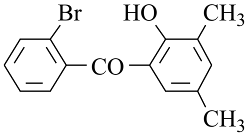
Synthesis

– Preparation by reaction of p-bromobenzoyl chloride with p-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (45%) [92].

b.p.$_{0.95}$ 186° [92]; Spectra (NA).

(2-Bromophenyl)(2-hydroxy-3,5-dimethylphenyl)methanone

[86914-81-0] $C_{15}H_{13}BrO_2$ mol.wt. 305.17



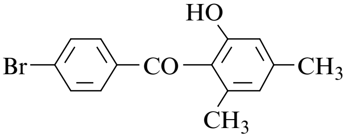
Synthesis

– Preparation by Fries rearrangement of 2,4-dimethylphenyl o-bromobenzoate with aluminium chloride at 180° for 10 min [1074].

m.p. 86–89° [1074]; Spectra (NA).

(4-Bromophenyl)(2-hydroxy-4,6-dimethylphenyl)methanone

[34174-04-4] $C_{15}H_{13}BrO_2$ mol.wt. 305.17



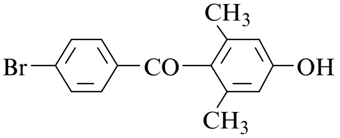
Synthesis

– Preparation by Fries rearrangement of 3,5-dimethyl-phenyl p-bromobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 147–148° [458]; Spectra (NA).

(4-Bromophenyl)(4-hydroxy-2,6-dimethylphenyl)methanone

[34183-16-9] $C_{15}H_{13}BrO_2$ mol.wt. 305.17



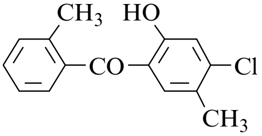
Synthesis

– Obtained by Fries rearrangement of 3,5-dimethylphenyl p-bromobenzoate with aluminium chloride in chloro-benzene at 140–150° for 20 min or in nitrobenzene at 75° for 24 h [480].

m.p. 125–126° [480]; Spectra (NA).

(4-Chloro-2-hydroxy-5-methylphenyl)(2-methylphenyl)methanone

[170799-18-5] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



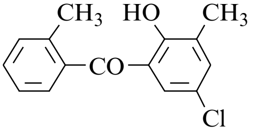
Synthesis

– Preparation by Fries rearrangement of 3-chloro-4-methyl-phenyl o-toluate with aluminium chloride at 100–150° for 0.5–3 h (31%) [29].

m.p. 72–73° [29]; Spectra (NA).

(5-Chloro-2-hydroxy-3-methylphenyl)(2-methylphenyl)methanone

[86914-77-4] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



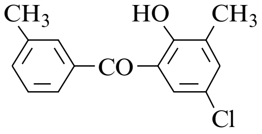
Synthesis

– Preparation by Fries rearrangement of 4-chloro-2-methyl-phenyl o-toluate with aluminium chloride at 180° for 10 min [1074].

m.p. 38° [1074]; Spectra (NA).

(5-Chloro-2-hydroxy-3-methylphenyl)(3-methylphenyl)methanone

[86914-90-1] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



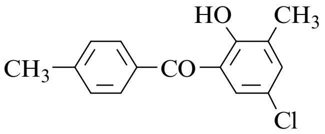
Synthesis

- Preparation by Fries rearrangement of 4-chloro-2-methyl-phenyl m-toluate with aluminium chloride at 180° for 10 min [1074].

m.p. 108–109° [1074]; Spectra (NA).

(5-Chloro-2-hydroxy-3-methylphenyl)(4-methylphenyl)methanone

[86914-86-5] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



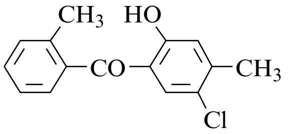
Synthesis

- Preparation by Fries rearrangement of 4-chloro-2-methyl-phenyl p-toluate with aluminium chloride at 180° for 10 min [1074].

m.p. 76–77° [1074]; Spectra (NA).

(5-Chloro-2-hydroxy-4-methylphenyl)(2-methylphenyl)methanone

[170799-04-9] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



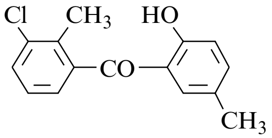
Synthesis

- Preparation by Fries rearrangement of 4-chloro-3-methyl-phenyl o-toluate with aluminium chloride at 100–150° for 0.5–3 h (76%) [29].

m.p. 57–58° [29]; Spectra (NA).

(3-Chloro-2-methylphenyl)(2-hydroxy-5-methylphenyl)methanone

$C_{15}H_{13}ClO_2$ mol.wt. 260.72



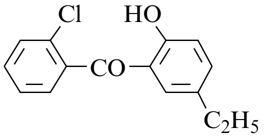
Synthesis

- Preparation by reaction of 3-chloro-2-methylbenzoyl chloride with p-cresol in the presence of aluminium chloride for 8 h at 190° [77].

m.p. and Spectra (NA).

(2-Chlorophenyl)(5-ethyl-2-hydroxyphenyl)methanone

[108294-72-0] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



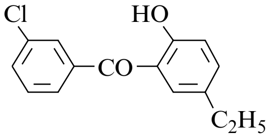
Synthesis

- Preparation by reaction of o-chlorobenzoyl chloride with p-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (60%) [92].

b.p.$_{0.45}$ 138–140° [92]; Spectra (NA).

(3-Chlorophenyl)(5-ethyl-2-hydroxyphenyl)methanone

[61466-85-1] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



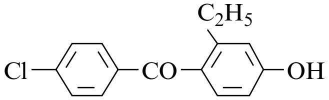
Synthesis

- Preparation by reaction of m-chlorobenzoyl chloride with p-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (66%) [92,1091].

b.p.$_{0.25}$ 153–156° [92,1091]; Spectra (NA).

(4-Chlorophenyl)(2-ethyl-4-hydroxyphenyl)methanone

[61466-73-7] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



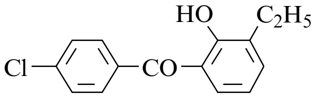
Synthesis

- Obtained (by-product) by reaction of p-chlorobenzoyl chloride with m-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (12%) [1091].
- Also refer to: [1093].

m.p. and Spectra (NA).

(4-Chlorophenyl)(3-ethyl-2-hydroxyphenyl)methanone

[61466-80-6] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



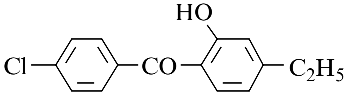
Synthesis

- Preparation by reaction of 3-ethylsalicylic acid chloride (3-ethyl-2-hydroxybenzoyl chloride) with chlorobenzene in the presence of aluminium chloride at 100° overnight (31%) [92,711], (23%) [1091].

m.p. 72–73° [92,711,1091]; Spectra (NA).

(4-Chlorophenyl)(4-ethyl-2-hydroxyphenyl)methanone

[56394-72-0] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



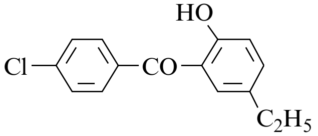
Syntheses

- Preparation by reaction of p-chlorobenzoyl chloride with m-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (83%) [92,711], (48%) [709,1091].
- Obtained from the corresponding oxime (m.p. 159–161°) by treatment with sodium metabisulfite in refluxing ethanol for 40 h [1091].

m.p. 52° [92,711,1091]; b.p.$_{0.09}$ 155–160° [709]; Spectra (NA).

(4-Chlorophenyl)(5-ethyl-2-hydroxyphenyl)methanone

[56394-67-3] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



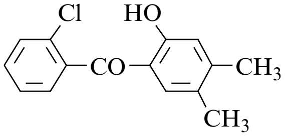
Syntheses

- Preparation by reaction of p-chlorobenzoyl chloride with p-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (72%) [92,711], (43–49%) [709,710,1091].
- Preparation by Fries rearrangement of p-ethylphenyl p-chlorobenzoate with aluminium chloride in tetrachloroethane at 125° for 6 h [710].
- Also refer to: [714].

m.p. 41°5–43°5 [92,711], 35–38° [710,1091];
b.p.$_{0.09}$ 147–150° [711], b.p.$_{3}$ 150–160° [92], b.p.$_{0.3}$ 160–168° [709,710,1091]; Spectra (NA).

(2-Chlorophenyl)(2-hydroxy-4,5-dimethylphenyl)methanone

[170799-17-4] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



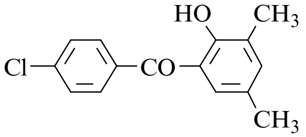
Synthesis

- Preparation by Fries rearrangement of 3,4-dimethylphenyl o-chlorobenzoate with aluminium chloride at 100–150° for 0.5–3 h (58%) [29].

m.p. 81–82° [29]; Spectra (NA).

(4-Chlorophenyl)(2-hydroxy-3,5-dimethylphenyl)methanone

[86914-84-3] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



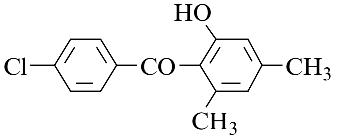
Synthesis

- Preparation by Fries rearrangement of 2,4-dimethylphenyl p-chlorobenzoate with aluminium chloride at 180° for 10 min [1074].

m.p. 45–46° [1074]; Spectra (NA).

(4-Chlorophenyl)(2-hydroxy-4,6-dimethylphenyl)methanone

[34199-74-1] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



Syntheses

- Preparation by reaction of p-chlorobenzoyl chloride with 3,5-dimethylanisole in the presence of stannic chloride in benzene, first between 0° and 5°, then at r.t. for 3 h.

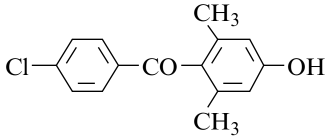
The 4′-chloro-4,6-dimethyl-2-methoxybenzophenone obtained (27%) by demethylation with 48% hydrobromic acid gave the expected ketone [904].

- Preparation by Fries rearrangement of 3,5-dimethylphenyl p-chlorobenzoate with aluminium chloride for 30 min at 150–160° [458].

m.p. 132–133° [458]; Spectra (NA).

(4-Chlorophenyl)(4-hydroxy-2,6-dimethylphenyl)methanone

[34183-15-8] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



Syntheses

- Preparation by reaction of p-chlorobenzoyl chloride with 3,5-dimethylanisole in the presence of stannic chloride in benzene, first between 0° and −5°, then at r.t. for 3 h.

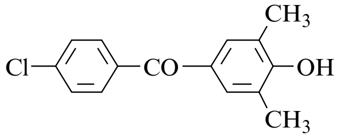
The 4-(p-chlorobenzoyl)-3,5-dimethylanisole obtained (40%) by demethylation with 48% hydrobromic acid gave the expected ketone [904].

- Also obtained by Fries rearrangement of 3,5-dimethylphenyl p-chlorobenzoate with aluminium chloride in chlorobenzene at 140–150° for 20 min or in nitrobenzene at 75° for 24 h [480].

m.p. 139–140° [480]; Spectra (NA).

(4-Chlorophenyl)(4-hydroxy-3,5-dimethylphenyl)methanone

[61002-59-3] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



Synthesis

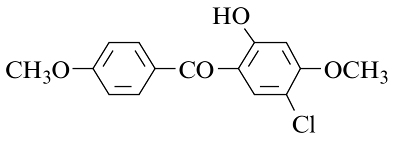
- Preparation by Friedel–Crafts acylation of 2,6-dimethyl-anisole with p-chlorobenzoyl chloride in the presence of aluminium chloride [902].

N.B.: Na and K salts [1118].

m.p. 98° [902]; Spectra (NA).

(5-Chloro-2-hydroxy-4-methoxyphenyl)(4-methoxyphenyl)methanone

[136741-44-1] $C_{15}H_{13}ClO_4$ mol.wt. 292.72



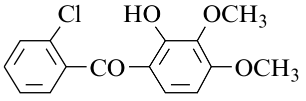
Synthesis

- Preparation by reaction of 4-methoxybenzoyl chloride with 4-chlororesorcinol dimethyl ether in the presence of aluminium chloride in ethylene dichloride (85%) [589].

m.p. 130–132° [589]; Spectra (NA).

(2-Chlorophenyl)(2-hydroxy-3,4-dimethoxyphenyl)methanone

[140665-36-7] $C_{15}H_{13}ClO_4$ mol.wt. 292.72



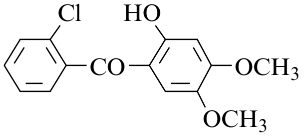
Synthesis

– Preparation by reaction of o-chlorobenzoyl chloride with 1,2,3-trimethoxybenzene in the presence of aluminium chloride in ethylene dichloride between 0° and 5°, then at r.t. for 3 h and at reflux for 1 h (86%) [704].

m.p. 115–117° [704]; Spectra (NA).

(2-Chlorophenyl)(2-hydroxy-4,5-dimethoxyphenyl)methanone

[140665-22-1] $C_{15}H_{13}ClO_4$ mol.wt. 292.72



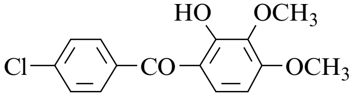
Synthesis

– Preparation by reaction of o-chlorobenzoyl chloride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride in ethylene dichloride between 0° and 5°, then at r.t. for 4 h and at reflux for 2 h (60%) [704].

m.p. 75–77° [704]; Spectra (NA).

(4-Chlorophenyl)(2-hydroxy-3,4-dimethoxyphenyl)methanone

[7508-29-4] $C_{15}H_{13}ClO_4$ mol.wt. 292.72



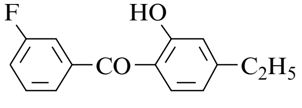
Synthesis

– Preparation by reaction of p-chlorobenzoyl chloride with 1,2,3-trimethoxybenzene in the presence of aluminium chloride in ethylene dichloride between 0° and 5°, then at r.t. for 3 h and at reflux for 1 h (54%) [704].

m.p. 148–149° [704]; Spectra (NA).

(4-Ethyl-2-hydroxyphenyl)(3-fluorophenyl)methanone

[61466-88-4] $C_{15}H_{13}FO_2$ mol.wt. 244.27



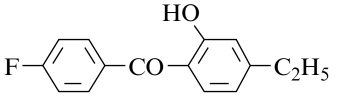
Synthesis

– Obtained (poor yield) by reaction of m-fluorobenzoyl chloride with m-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h [710,1091], (5%) [92].

oil [92]; m.p. <20° [710,1091]; $n_D^{22} = 1.5962$ [710,1091]; b.p. and Spectra (NA).

(4-Ethyl-2-hydroxyphenyl)(4-fluorophenyl)methanone

[56394-78-6] $C_{15}H_{13}FO_2$ mol.wt. 244.27



Synthesis

– Obtained by reaction of p-fluorobenzoyl chloride with m-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (64%) [709], (41%) [710,1091], (5%) [92,711].

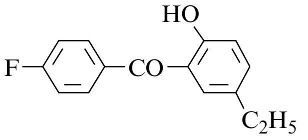
– Also refer to: [714].

m.p. 44–48° [92,709–711,1091];

b.p.$_{0.06}$ 129–132° [709,710,1091]; Spectra (NA).

(5-Ethyl-2-hydroxyphenyl)(4-fluorophenyl)methanone

[108294-75-3] $C_{15}H_{13}FO_2$ mol.wt. 244.27



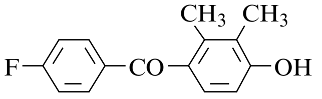
Synthesis

– Preparation by reaction of p-fluorobenzoyl chloride with p-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (38%) [92].

m.p. 44–46° [92]; Spectra (NA).

(4-Fluorophenyl)(4-hydroxy-2,3-dimethylphenyl)methanone

$C_{15}H_{13}FO_2$ mol.wt. 244.27



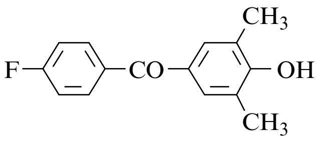
Synthesis

– Preparation by demethylation of 4′-fluoro-4-methoxy-2,3-dimethylbenzophenone (SM) with aluminium chloride in refluxing methylene chloride overnight. SM was obtained by Friedel–Crafts acylation of 2,3-dimethylanisole with p-fluorobenzoyl chloride in the presence of aluminium chloride in refluxing methylene chloride [475].

m.p. and Spectra (NA).

(4-Fluorophenyl)(4-hydroxy-3,5-dimethylphenyl)methanone

[102331-06-6] $C_{15}H_{13}FO_2$ mol.wt. 244.27



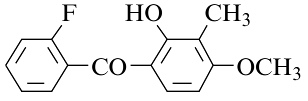
Synthesis

– Preparation by Fries rearrangement of 2,6-dimethylphenyl p-fluorobenzoate in the presence of aluminium chloride in o-dichlorobenzene at 150° (93%) [1119].

m.p. and Spectra (NA).

(2-Fluorophenyl)(2-hydroxy-4-methoxy-3-methylphenyl)methanone

[72483-03-5] $C_{15}H_{13}FO_3$ mol.wt. 260.26



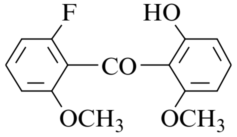
Synthesis

– Preparation by reaction of o-fluorobenzoyl chloride with 2,6-dimethoxytoluene in the presence of aluminium chloride in refluxing ethylene dichloride for 30 min (93%) [476,1031].

m.p. 119–120° [1031], 118–120° [476]; Spectra (NA).

(2-Fluoro-6-methoxyphenyl)(2-hydroxy-6-methoxyphenyl)methanone

[129103-94-2] $C_{15}H_{13}FO_4$ mol.wt. 276.26



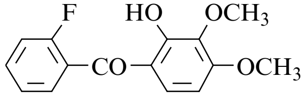
Synthesis

– Preparation from 2-iodo-3-methoxyphenyl 2-fluoro-6-methoxybenzoate by rearrangement on treatment with n-butyllithium in a mixture of ethyl ether, hexane and tetrahydrofuran at −100° followed by heating to −70°, then treatment with saturated aqueous ammonium chloride (88%) [58].

m.p. 79°5–80° [58]; ^{1}H NMR [58], IR [58], MS [58].

(2-Fluorophenyl)(2-hydroxy-3,4-dimethoxyphenyl)methanone

[140665-37-8] $C_{15}H_{13}FO_4$ mol.wt. 276.26



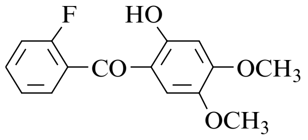
Synthesis

– Preparation by reaction of o-fluorobenzoyl chloride with 1,2,3-trimethoxybenzene in the presence of aluminium chloride in ethylene dichloride between 0° and 5°, then at r.t. for 3 h and at reflux for 1 h (95%) [704].

m.p. 97–99° [704]; Spectra (NA).

(2-Fluorophenyl)(2-hydroxy-4,5-dimethoxyphenyl)methanone

[140665-23-2] $C_{15}H_{13}FO_4$ mol.wt. 276.26



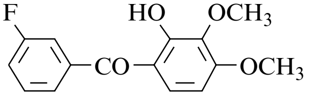
Synthesis

– Preparation by reaction of o-fluorobenzoyl chloride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride in ethylene dichloride between 0° and 5°, then at r.t. for 4 h and at reflux for 2 h (95%) [704].

m.p. 89–90° [704]; Spectra (NA).

(3-Fluorophenyl)(2-hydroxy-3,4-dimethoxyphenyl)methanone

[140665-38-9] $C_{15}H_{13}FO_4$ mol.wt. 276.26



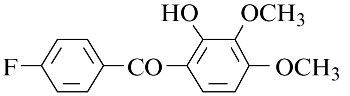
Synthesis

– Preparation by reaction of m-fluorobenzoyl chloride with 1,2,3-trimethoxybenzene in the presence of aluminium chloride in ethylene dichloride between 0° and 5°, then at r.t. for 3 h and at reflux for 1 h (80%) [704].

m.p. 84–85° [704]; Spectra (NA).

(4-Fluorophenyl)(2-hydroxy-3,4-dimethoxyphenyl)methanone

[140665-39-0] $C_{15}H_{13}FO_4$ mol.wt. 276.26



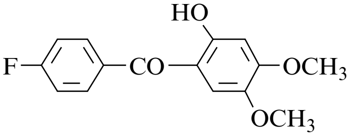
Synthesis

– Preparation by reaction of p-fluorobenzoyl chloride with 1,2,3-trimethoxybenzene in the presence of aluminium chloride in ethylene dichloride between 0° and 5°, then at r.t. for 3 h and at reflux for 1 h (79%) [704].

m.p. 144–146° [704]; Spectra (NA).

(4-Fluorophenyl)(2-hydroxy-4,5-dimethoxyphenyl)methanone

$C_{15}H_{13}FO_4$ mol.wt. 276.26



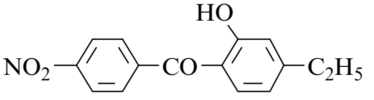
Synthesis

– Preparation by reaction of p-fluorobenzoyl chloride with 3,4-dimethoxyphenol in the presence of boron trichloride in a benzene/ methylene chloride mixture at r.t. for 2 h [598].

m.p. (NA); orange solid [598]; ^1H NMR [598].

(4-Ethyl-2-hydroxyphenyl)(4-nitrophenyl)methanone

[78473-49-1] $C_{15}H_{13}NO_4$ mol.wt. 271.27



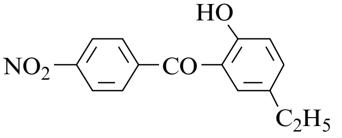
Synthesis

– Refer to: [714].

m.p. and Spectra (NA).

(5-Ethyl-2-hydroxyphenyl)(4-nitrophenyl)methanone

[108294-76-4] $C_{15}H_{13}NO_4$ mol.wt. 271.27



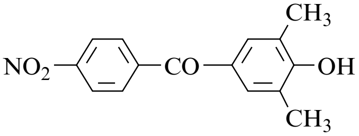
Synthesis

– Preparation by reaction of p-nitrobenzoyl chloride with p-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (63%) [92].

m.p. 100–101°5 [92]; Spectra (NA).

(4-Hydroxy-3,5-dimethylphenyl)(4-nitrophenyl)methanone

[85916-09-2]



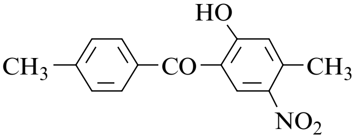
$C_{15}H_{13}NO_4$ mol.wt. 271.27

Synthesis

- Refer to: [1120].

m.p. and Spectra (NA).

(2-Hydroxy-4-methyl-5-nitrophenyl)(4-methylphenyl)methanone



$C_{15}H_{13}NO_4$ mol.wt. 271.27

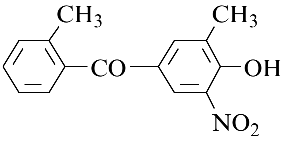
Synthesis

- Obtained by diazotization of 2-amino-4,4′-dimethyl-5-nitrobenzophenone, followed by hydrolysis of the diazonium salt so formed (24%) [1121].

m.p. 144° [1121]; Spectra (NA).

(4-Hydroxy-3-methyl-5-nitrophenyl)(2-methylphenyl)methanone

[103555-90-4]



$C_{15}H_{13}NO_4$ mol.wt. 271.27

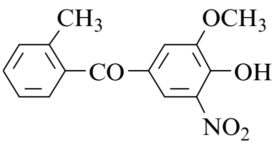
Synthesis

- Preparation from o-methylanisole in three steps: first, acylation of o-methylanisole with o-toluoyl chloride in the presence of ferric chloride during 3 h at 20°. Then, demethylation of 4-methoxy-2′,3-dimethylbenzophenone so formed by treatment with refluxing 55% hydriodic acid for 10 h and subsequent nitration of the obtained 4-hydroxy-2′,3-dimethylbenzophenone with a concentrated nitric acid/concentrated sulfuric acid mixture under cooling [594].

m.p. and Spectra (NA).

(4-Hydroxy-3-methoxy-5-nitrophenyl)(2-methylphenyl)methanone

[134612-79-6]



$C_{15}H_{13}NO_5$ mol.wt. 287.27

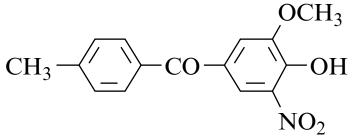
Synthesis

- Preparation by nitration of 4-hydroxy-3-methoxy-2′-methyl-benzophenone with 65% nitric acid in acetic acid at 20° [1019].

m.p. 125–127° [1019]; Spectra (NA).

(4-Hydroxy-3-methoxy-5-nitrophenyl)(4-methylphenyl)methanone

[134612-80-9] $C_{15}H_{13}NO_5$ mol.wt. 287.27



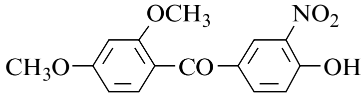
Synthesis

– Preparation by nitration of 4-hydroxy-3-methoxy-4′-methylbenzophenone with 65% nitric acid in acetic acid at 20° [1019].

m.p. 137–139° [1019]; Spectra (NA).

(2,4-Dimethoxyphenyl)(4-hydroxy-3-nitrophenyl)methanone

[67286-44-6] $C_{15}H_{13}NO_6$ mol.wt. 303.27



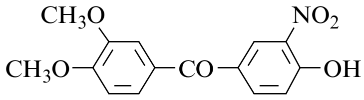
Synthesis

– Obtained by reaction of nitric acid (d = 1.42) with 4-hydroxy-2′,4′-dimethoxybenzophenone in acetic acid at 12° (major product) [592].

m.p. 105° [592]; ^{1}H NMR [592].

(3,4-Dimethoxyphenyl)(4-hydroxy-3-nitrophenyl)methanone

[134612-83-2] $C_{15}H_{13}NO_6$ mol.wt. 303.27



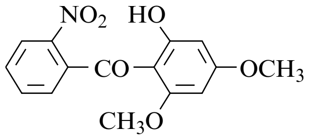
Synthesis

– Refer to: [1019].

m.p. and Spectra (NA).

(2-Hydroxy-4,6-dimethoxyphenyl)(2-nitrophenyl)methanone

[61736-75-2] $C_{15}H_{13}NO_6$ mol.wt. 303.27



Syntheses

– Obtained by partial demethylation of 2,4,6-trimethoxy-2′-nitrobenzophenone with boron tribromide in methylene chloride at 0° for 10 min and at r.t. overnight (97%) [1122].

– Also obtained (poor yield) by reaction of o-nitrobenzoyl

– chloride with 3,5-dimethoxyphenol in the presence of aluminium chloride in ethyl ether, first at 0° for 3 h, then at 20° for 3 h (15%) [1123].

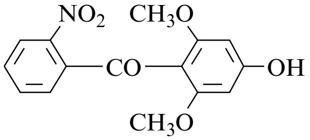
– Preparation by photo-Fries rearrangement of 3,5-dimethoxyphenyl o-nitrobenzoate in benzene for 1.5 h (quantitative yield) [1123].

m.p. 198–199° [1123], 133–135° [1122]. One of the reported melting points is obviously wrong.

^{1}H NMR [1122,1123], IR [1122,1123], UV [1122,1123], MS [1122,1123].

(4-Hydroxy-2,6-dimethoxyphenyl)(2-nitrophenyl)methanone

[59190-66-8] $C_{15}H_{13}NO_6$ mol.wt. 303.27



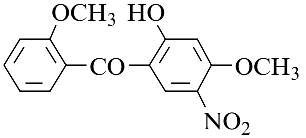
Synthesis

– Obtained (poor yield) by reaction of o-nitrobenzoyl chloride with 3,5-dimethoxyphenol in the presence of aluminium chloride in ethyl ether, first at 0° for 3 h, then at 20° for 3 h (5%) [1123].

m.p. 175–177° [1123]; ^{1}H NMR [1123], IR [1123], UV [1123], MS [1123].

(2-Hydroxy-4-methoxy-5-nitrophenyl)(2-methoxyphenyl)methanone

[79204-64-1] $C_{15}H_{13}NO_6$ mol.wt. 303.27



Syntheses

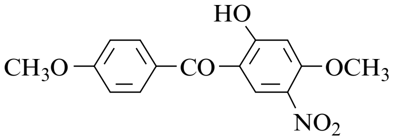
– Obtained by oxidation of 6-methoxy-2-(2-methoxy-4-nitrophenyl)-3-(2-methoxyphenyl)-5-nitrobenzofuran with chromium trioxide in acetic acid, followed by saponification of the keto ester so formed (70%) with potassium hydroxide in boiling ethanol (75%) [1117].

– Also obtained by oxidation of 2,3-bis(2-methoxyphenyl)-6-methoxy-5-nitrobenzofuran with chromium trioxide in acetic acid, followed by saponification of the keto ester so formed (65%) with potassium hydroxide in boiling ethanol (60%) [1117].

m.p. 155° [1117]; IR [1117], MS [1117].

(2-Hydroxy-4-methoxy-5-nitrophenyl)(4-methoxyphenyl)methanone

[66666-07-7] $C_{15}H_{13}NO_6$ mol.wt. 303.27



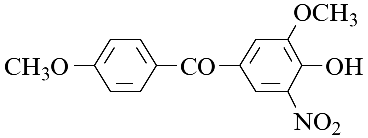
Syntheses

– Obtained by reaction of nitric acid (d = 1.42) with 2-hydroxy-4,4′-dimethoxybenzophenone in acetic acid at 24° (major product) [592].

– Preparation by saponification of 2-(4-anisoyloxy)-4,4′-dimethoxy-5-nitrobenzophenone (SM) with potassium hydroxide in refluxing ethanol for 1 h (70%). SM was obtained by oxidation of 2,3-bis(4-methoxyphenyl)-6-methoxy-5-nitrobenzofuran with chromium trioxide in refluxing acetic acid for 45 min (75%) [1116].

m.p. 162° [592], 140° [1116]. One of the reported melting points is obviously wrong. Spectra (NA).

(4-Hydroxy-3-methoxy-5-nitrophenyl)(4-methoxyphenyl)methanone



$C_{15}H_{13}NO_6$ mol.wt. 303.27

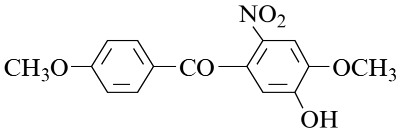
Synthesis

- Obtained by reaction of 65% nitric acid with 3,4′-di-methoxy-4-hydroxybenzophenone in acetic acid at 20° [1019].

m.p. 134–136° [1019]; Spectra (NA).

(5-Hydroxy-4-methoxy-2-nitrophenyl)(4-methoxyphenyl)methanone

[2898-51-3]



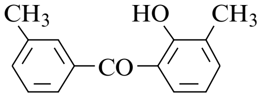
$C_{15}H_{13}NO_6$ mol.wt. 303.27

Synthesis

- Obtained by heating 2-nitro-4,4′,5-trimethoxy-benzophenone with potassium hydroxide and dilute methanol in an autoclave at 140° for 8 h [1124].

m.p. 178° [1124]; Spectra (NA).

(2-Hydroxy-3-methylphenyl)(3-methylphenyl)methanone



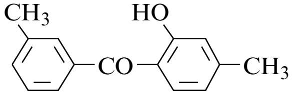
$C_{15}H_{14}O_2$ mol.wt. 226.27

Synthesis

- Preparation (poor yield) by action of m-toluoyl chloride with o-tolyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (8%) [55].

m.p. 42° [55]; Spectra (NA).

(2-Hydroxy-4-methylphenyl)(3-methylphenyl)methanone



$C_{15}H_{14}O_2$ mol.wt. 226.27

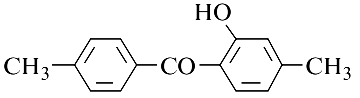
Synthesis

- Preparation by reaction of m-toluoyl chloride with m-tolyl borate in the presence of aluminium chloride in tetrachloro-ethane at 100° (37%) [55].

b.p.$_{8}$ 194–195° [55]; Spectra (NA).

(2-Hydroxy-4-methylphenyl)(4-methylphenyl)methanone

[81652-53-1]



$C_{15}H_{14}O_2$ mol.wt. 226.27

Syntheses

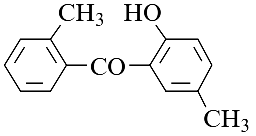
- Obtained (by-product) by Fries rearrangement of m-cresyl p-toluate in the presence of aluminium chloride in nitrobenzene at 62–63° for 18 h (16%) [31].

- Also obtained (poor yield) by diazotization of 2-amino-4,4′-dimethylbenzophenone, followed by hydrolysis of the diazonium salt so obtained (9%) [1121]. In this reaction, the major product was 3,6-dimethylfluorenone (70%).
- Also refer to: [1125].

m.p. 73–74° [1121]; Spectra (NA).

(2-Hydroxy-5-methylphenyl)(2-methylphenyl)methanone

[147029-79-6] $C_{15}H_{14}O_2$ mol.wt. 226.27



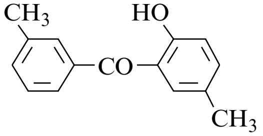
Syntheses

- Preparation by Fries rearrangement of p-tolyl o-toluate with aluminium chloride at 100–150° for 0.5–3 h (73%) [29].
- Preparation by condensation of the Grignard reagent of p-methylanisole with o-toluoyl chloride, followed by demethylation in acid medium of the resultant methyl ether (excellent yield) [964].

m.p. 95° [964], 94–95° [29]; Spectra (NA).

(2-Hydroxy-5-methylphenyl)(3-methylphenyl)methanone

[26880-98-8] $C_{15}H_{14}O_2$ mol.wt. 226.27



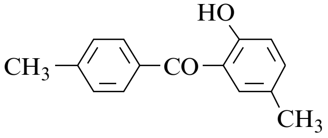
Synthesis

- Preparation by Fries rearrangement of p-tolyl m-toluate with aluminium chloride at 130° for 5 h (54%) [629].

m.p. 54–55° [629];
^{1}H NMR [629], IR [104,629], UV [629];
pK_a [104,629]; polarographic study [117].

(2-Hydroxy-5-methylphenyl)(4-methylphenyl)methanone

[26880-95-5] $C_{15}H_{14}O_2$ mol.wt. 226.27



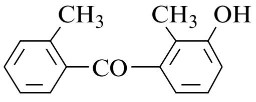
Synthesis

- Preparation by Fries rearrangement of p-tolyl p-toluate in the presence of,
  - aluminium chloride [117], (64%) [97], at 120° (82%) [1126] or at 180° for 10 min (85%) [518];
  - Nafion-H, a polymeric perfluorinated resin sulfonic acid, in refluxing nitrobenzene for 12 h (72%) [38].
- Also refer to: [1127].

m.p. 89°5–90° [1126], 88–88°5 [629], 88° [518], 63°5–65° [97]; ^{1}H NMR [38,97,629], ^{13}C NMR [38,97],
IR [97,104,629], UV [629];
pK_a [104,629]; polarographic study [117].

(3-Hydroxy-2-methylphenyl)(2-methylphenyl)methanone

[50454-58-5] $C_{15}H_{14}O_2$ mol.wt. 226.27



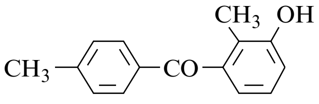
Synthesis

- Refer to: [1128] (Japanese patent).

m.p. and Spectra (NA).

(3-Hydroxy-2-methylphenyl)(4-methylphenyl)methanone

[74167-89-8] $C_{15}H_{14}O_2$ mol.wt. 226.27



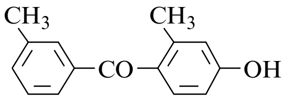
Synthesis

- Preparation by diazotization of 3-amino-2,4′-dimethyl-benzophenone, followed by hydrolysis of the diazonium salt so obtained (85%) [119], according to [634].
- Also refer to: [912].

m.p. 102° [119]; Spectra (NA).

(4-Hydroxy-2-methylphenyl)(3-methylphenyl)methanone

$C_{15}H_{14}O_2$ mol.wt. 226.27



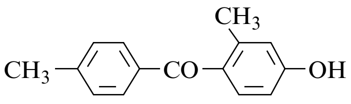
Synthesis

- Obtained by action of m-toluoyl chloride with m-tolyl borate in the presence of aluminium chloride in tetrachloroethane at 100° (30%) [55].

m.p. 110° [55]; Spectra (NA).

(4-Hydroxy-2-methylphenyl)(4-methylphenyl)methanone

$C_{15}H_{14}O_2$ mol.wt. 226.27



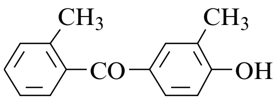
Synthesis

- Preparation by Fries rearrangement of m-cresyl p-toluate with aluminium chloride in nitrobenzene at 62–63° for 18 h (50%) [31].

m.p. 108–109° [31]; Spectra (NA).

(4-Hydroxy-3-methylphenyl)(2-methylphenyl)methanone

[147029-76-3] $C_{15}H_{14}O_2$ mol.wt. 226.27



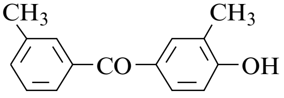
Synthesis

- Preparation by condensation of the Grignard reagent of o-methylanisole with o-toluoyl chloride, followed by demethylation in acid medium of the resultant methyl ether (excellent yield) [964].

m.p. 142° [964]; Spectra (NA).

(4-Hydroxy-3-methylphenyl)(3-methylphenyl)methanone

[62064-85-1] $C_{15}H_{14}O_2$ mol.wt. 226.27



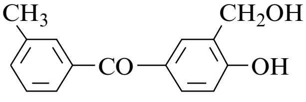
Synthesis

– Obtained by reaction of m-toluoyl chloride with o-cresyl borate in the presence of aluminium chloride in tetra-chloroethane at 100° (21%) [55].

m.p. 158° [55]; Spectra (NA).

[4-Hydroxy-3-(hydroxymethyl)phenyl](3-methylphenyl)methanone

[62064-89-5] $C_{15}H_{14}O_3$ mol.wt. 242.27



Synthesis

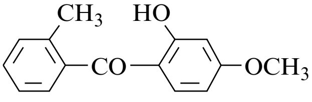
– Not yet described.

– **N.B.**: A metabolite of 3,3′-dimethyl-4-methoxy benzophenone (NK-049) in the rat [1129] (Japanese paper).

m.p. and Spectra (NA).

(2-Hydroxy-4-methoxyphenyl)(2-methylphenyl)methanone

[27847-83-2] $C_{15}H_{14}O_3$ mol.wt. 242.27



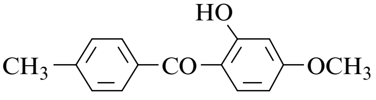
Synthesis

– Refer to: [405].

m.p. and Spectra (NA).

(2-Hydroxy-4-methoxyphenyl)(4-methylphenyl)methanone *(Mexenone)*

[1641-17-4] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

– Preparation by Friedel–Crafts acylation of m-methoxy-phenol with p-toluoyl chloride,

- in the presence of boron trichloride in benzene first at −10°, then at reflux for 10 h under nitrogen (80%) [660];
- in the presence of titanium tetrachloride in benzene first at −10°, then at reflux for 14 h under nitrogen (73%) [660] or in chlorobenzene for 1 h at 120° (79%) [662].

– Preparation by reaction of p-toluoyl chloride with 1,3-dimethoxybenzene in the presence of aluminium chloride in a chlorobenzene/N,N-dimethylformamide mixture (22:1) at 115° [235,657].

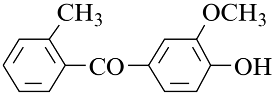
– Also obtained by oxidation of 6-methoxy-3-(4-methylphenyl)-2-phenylbenzofuran with chromium trioxide in boiling acetic acid for 30–40 min, followed by saponification of the keto ester so formed [44,46].

– Also refer to: [77,222,241,655].

m.p. 98° [44,46], 95–96° [662], 95° [660]; IR [44], UV [235,240,241].

(4-Hydroxy-3-methoxyphenyl)(2-methylphenyl)methanone

[134612-38-7] $C_{15}H_{14}O_3$ mol.wt. 242.27



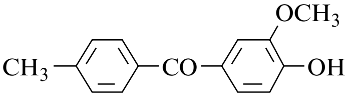
Synthesis

– Preparation by reaction of 33% hydrobromic acid in acetic acid with 4-(benzyloxy)-3-methoxy-2′-methylbenzophenone in methylene chloride at 20–25° [1019].

m.p. 103–105° [1019]; Spectra (NA).

(4-Hydroxy-3-methoxyphenyl)(4-methylphenyl)methanone

[134612-39-8] $C_{15}H_{14}O_3$ mol.wt. 242.27



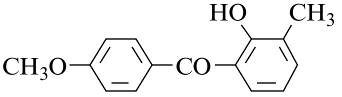
Synthesis

– Preparation by reaction of 33% hydrobromic acid in acetic acid with 4-(benzyloxy)-3-methoxy-4′-methyl-benzophenone in methylene chloride at 20–25° [1019].

m.p. 103–105° [1019]; Spectra (NA).

(2-Hydroxy-3-methylphenyl)(4-methoxyphenyl)methanone

[65611-79-2] $C_{15}H_{14}O_3$ mol.wt. 242.27



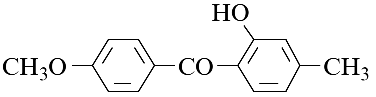
Synthesis

– Preparation by reaction between (o-tolyloxy)magnesium bromide complexed with HMPT and p-methoxy-benzaldehyde in refluxing benzene for 48 h (47%) [50].

m.p. 52° [50]; ^{1}H NMR [50], IR [50], MS [50].

(2-Hydroxy-4-methylphenyl)(4-methoxyphenyl)methanone

[108478-27-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

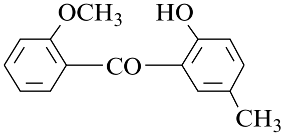
– Preparation by reaction of p-anisic acid with m-cresol in the presence of boron trifluoride at 160° for 1 h (81%) [150].

– Obtained by Fries rearrangement of m-cresyl p-anisate with aluminium chloride without solvent at 120° or at 160° [315].

m.p. 96–97° [150,315]; Spectra (NA).

(2-Hydroxy-5-methylphenyl)(2-methoxyphenyl)methanone

[53271-51-5] $C_{15}H_{14}O_3$ mol.wt. 242.27



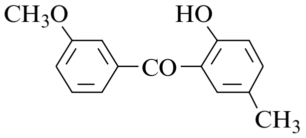
Syntheses

- Obtained (poor yield) by Fries rearrangement of p-cresyl o-anisate with polyphosphoric acid at 100° for 20 min (4%) [991].
- Also obtained (poor yield) by Friedel–Crafts acylation of p-cresol with o-anisic acid in the presence of methane-sulfonic acid at 120° for 20 min (5%) [991].
- Also obtained by photo-Fries rearrangement of p-cresyl o-anisate in benzene during 72 h (53%) [1130].

yellow oil, very viscous [1130]; m.p. 60° [991];
^{1}H NMR (Sadtler: standard n° 38494 M) [1130],
IR (Sadtler: standard n° 65532 K) [991,1130], UV [991], MS [1130,1131].

(2-Hydroxy-5-methylphenyl)(3-methoxyphenyl)methanone

[26880-99-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



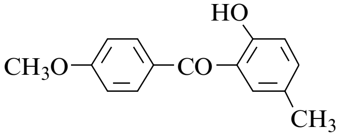
Syntheses

- Preparation by photo-Fries rearrangement of p-tolyl m-anisate in methanol at 20° for 10 h (34%) [629].
- Preparation by acylating p-methylanisole with m-anisoyl chloride in the presence of aluminium chloride in carbon disulfide [1132].

m.p. 130–132° [629]; ^{1}H NMR [629], IR [104,629], UV [629];
pK_a [104,629]; polarographic study [117].

(2-Hydroxy-5-methylphenyl)(4-methoxyphenyl)methanone

[26880-96-6] $C_{15}H_{14}O_3$ mol.wt. 242.27



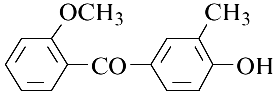
Syntheses

- Preparation by Fries rearrangement of p-tolyl p-anisate (without solvent),
  - with aluminium chloride at 120° for 1 h (26%) [991], at 120° or at 160° [315,629];
  - with titanium tetrachloride at 120° for 1 h (89%) [991].
- Preparation by oxidation of 2-phenyl-3-(4-methoxyphenyl)-5-methylbenzofuran with chromium trioxide in boiling acetic acid for 2 h, followed by saponification of the keto ester so formed with 10% sodium hydroxide in boiling ethanol for 15 min [1133].
- Obtained (by-product) by reaction of p-anisoyl chloride with p-methylanisole in the presence of aluminium chloride in boiling carbon disulfide [683].

m.p. 108–109° [315,629,683,1133], 107° [991];
^{1}H NMR (Sadtler: standard n° 38493 M) [629,1133],
IR (Sadtler: standard n° 65531 K) [104,629,991,1133],
UV [629,991], MS [1131]; pK_a [104,629]; polarographic study [117].

(4-Hydroxy-3-methylphenyl)(2-methoxyphenyl)methanone

[72324-24-4] $C_{15}H_{14}O_3$ mol.wt. 242.27



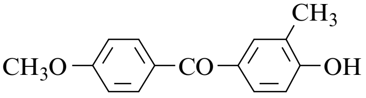
Syntheses

- Obtained by Fries rearrangement of o-cresyl o-anisate with polyphosphoric acid at 100° for 20 min (20%) [991].
- Preparation by reaction of o-anisic acid with o-cresol in the presence of methanesulfonic acid at 120° for 20 min (48%) [991].

m.p. 137° [991]; ^{1}H NMR (Sadtler: standard n° 38491 M),
IR (Sadtler: standard n° 65529 K) [991], UV [991], MS [1131].

(4-Hydroxy-3-methylphenyl)(4-methoxyphenyl)methanone

[72324-23-3] $C_{15}H_{14}O_3$ mol.wt. 242.27



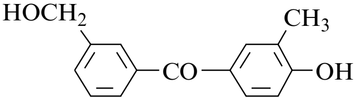
Syntheses

- Preparation by Fries rearrangement of o-cresyl p-anisate,
  - with titanium tetrachloride in nitromethane at 20° for 170 h (84%) [991];
  - with aluminium chloride in nitromethane at 20° for 170 h (42%) [991] or at 75° for 6 h (60%) [991]. The same reaction carried out without solvent at 120° or at 160° gave the same product [315].
- Also obtained (via o-cresyl p-anisate) by heating a mixture of p-anisic acid and o-cresol in the presence of Tonsil [645].
- Also refer to: [995,996,999].

m.p. 188° [991], 186° [315], 143° [645]. One of the reported melting points is obviously wrong. 1H NMR (Sadtler: standard n° 38499 M),
IR (Sadtler: standard n° 65537 K) [991], UV [991], MS [1131].

[3-(Hydroxymethyl)phenyl](4-Hydroxy-3-methylphenyl)methanone

[62064-88-4] $C_{15}H_{14}O_3$ mol.wt. 242.27



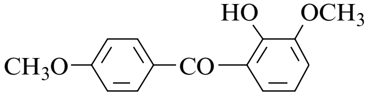
Synthesis

- Not yet described.
- **N.B.**: A metabolite of 3,3′-dimethyl-4-methoxy-benzophenone (NK-049) in the rat [1129] (Japanese paper).

m.p. and Spectra (NA).

(2-Hydroxy-3-methoxyphenyl)(4-methoxyphenyl)methanone

[155645-18-4] $C_{15}H_{14}O_4$ mol.wt. 258.27



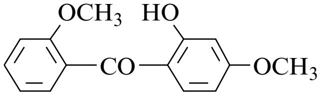
Synthesis

- Preparation by reaction of 2,3-dimethoxybenzoyl chloride with anisole (74%) [1134], according to the method [756].

m.p. 96° [1134]; Spectra (NA).

(2-Hydroxy-4-methoxyphenyl)(2-methoxyphenyl)methanone

[62495-36-7] $C_{15}H_{14}O_4$ mol.wt. 258.27



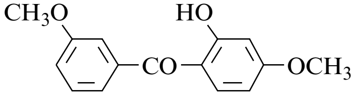
Syntheses

- Preparation by reaction of o-anisic acid with m-methoxy-phenol [1135] in the presence of zinc chloride and phosphorous oxychloride at 65–70° for 2 h (68%) [1136].
- Preparation by oxidation of 2,3-bis(2-methoxyphenyl)-6-methoxybenzofuran with chromium trioxide in refluxing acetic acid for 30 min, followed by saponification of the keto ester so formed (55%) with potassium hydroxide in refluxing ethanol for 1 h (50%) [1117].
- Preparation by selective demethylation of 2,2′,4-trimethoxybenzophenone with excess beryllium chloride in refluxing toluene for 3 h (90%) [395].
- Also obtained, in mixture with 2,4-dimethoxy-2′-hydroxybenzophenone, by reaction of 2-methoxybenzoyl chloride with resorcinol dimethyl ether in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (total yield: 65%) [1010,1011].

m.p. 92–93° [395], 91° [1135], 88°5–89° [1136], 80° [1117]; ^{1}H NMR [395,1010,1011,1135,1136],
IR [395,1117,1136], UV [395,1136], MS [1135]; TLC [395].

(2-Hydroxy-4-methoxyphenyl)(3-methoxyphenyl)methanone

[62495-37-8] $C_{15}H_{14}O_4$ mol.wt. 258.27



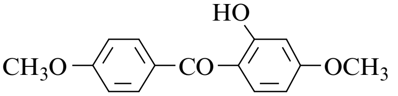
Syntheses

- Preparation by reaction of m-anisic acid with 3-methoxyphenol in the presence of zinc chloride and phosphorous oxychloride at 65–70° for 2 h (54%) [1136].
- Preparation by partial methylation of 2,4-dihydroxy-3′-methoxybenzophenone with methyl iodide in the presence of potassium carbonate in boiling acetone (69%) [1137].
- Preparation by selective demethylation of 2,3′,4-trimethoxybenzophenone with excess beryllium chloride in refluxing toluene for 3 h (92%) [395].

clear oil [395]; b.p.$_{0.3}$ 165–170° [1136]; m.p. 66° [1137]; ^{1}H NMR [395, 1136], IR [395,1136], UV [395,1136]; TLC [395].

(2-Hydroxy-4-methoxyphenyl)(4-methoxyphenyl)methanone

[6131-38-0] $C_{15}H_{14}O_4$ mol.wt. 258.27



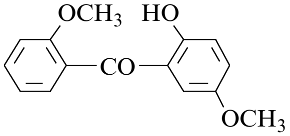
Syntheses

- Preparation by reaction of p-methoxybenzoic acid (p-anisic acid) with resorcinol monomethyl ether in the presence of boron trifluoride [650].
- Preparation by reaction of 2-hydroxy-4-methoxy-benzoic acid with anisole in the presence of stannic chloride at 115–120° for 3–4 h [190,237,671].
- Preparation by oxidation of 6-methoxy-3-(4-methoxyphenyl)-2-phenylbenzofuran with chromium trioxide, followed by saponification of the keto ester so formed—the 2-(benzoyloxy)-4,4′-di-methoxybenzophenone—with potassium hydroxide [44,46].
- Preparation by partial methylation,
  - of 2,4-dihydroxy-4′-methoxybenzophenone;
    with methyl iodide in the presence of potassium carbonate in boiling acetone (88%) [1137];
    with methyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [738];
  - of 2,4′-dihydroxy-4-methoxybenzophenone [610], with methyl iodide in the presence of potassium carbonate in acetone at r.t. [592];
  - of 2,4,4′-trihydroxybenzophenone with dimethyl sulfate in alkaline solution (40%) [380].
- Preparation by selective demethylation of 2,4,4′-trimethoxybenzophenone with excess beryllium chloride in refluxing toluene for 3 h (90%) [395].
- Preparation by reaction of p-anisoyl chloride with resorcinol dimethyl ether,
  - in chlorobenzene in the presence of titanium tetrachloride for 1 h at 120° (86%) [662];
  - in o-dichlorobenzene or without solvent, in the presence of ferric chloride between 180° and 200° for 6–7 h (by-product) [663].
- Also refer to: [78,194,222,223,625,655,657,668,1016,1125,1138–1144].

m.p. 144° [1139], 130° [113], 129–131° [190,237], 118° [592,610,1137], 117–118° [380,650], 115° [44,46], 111–112° [662], 110–112° [738], 110° [395]. There is a discrepancy between the various melting points.
^{1}H NMR [98,395,610], IR [44,395,610,650],
UV [113,190,237,380,395,650,830], EPR [98];
TLC [395]; paper chromatography [383].

(2-Hydroxy-5-methoxyphenyl)(2-methoxyphenyl)methanone

[42833-51-2] $C_{15}H_{14}O_4$ mol.wt. 258.27



Syntheses

- Obtained by reaction of o-anisoyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride in ethyl ether for 19 h [416].
- Obtained by partial demethylation of 2,2′,5-trimethoxy-benzophenone,

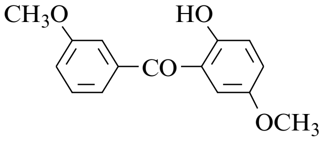
- with boron tribromide in methylene chloride at 0° for 1.5 h (50%) [395];
- with boron trichloride in methylene chloride at 0° for 3 h (50%) [395];
- with boron trifluoride-etherate in refluxing benzene for 6 h (60%) or in refluxing toluene for 4 h (60%) [395];
- with beryllium chloride in refluxing benzene for 8–10 h (40%) or in refluxing toluene for 5 h (40%) [395].

N.B.: In these experiments, only the reactions using boron halides were carried out under nitrogen atmosphere.

m.p. 100–101° [395]; ^{1}H NMR [395,416], IR [395]; TLC [395].

(2-Hydroxy-5-methoxyphenyl)(3-methoxyphenyl)methanone

[183106-25-4] $C_{15}H_{14}O_4$ mol.wt. 258.27



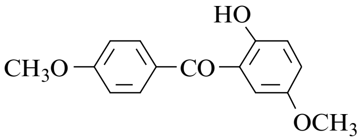
Synthesis

- Preparation by selective demethylation of 2,3′,5-tri-methoxybenzophenone with excess beryllium chloride in refluxing toluene for 4 h (90%) [395].

pale yellow oil [395]; b.p. (NA);
^{1}H NMR [395], IR [395], UV [395], MS [395]; TLC [395].

(2-Hydroxy-5-methoxyphenyl)(4-methoxyphenyl)methanone

[16762-04-2] $C_{15}H_{14}O_4$ mol.wt. 258.27



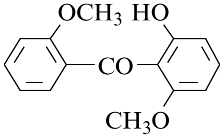
Syntheses

- Obtained by Fries rearrangement of p-methoxyphenyl p-anisate with titanium tetrachloride without solvent at 120° for 1 h (20–35% yields) [679].
- Also obtained by partial demethylation of 2,4′,5-tri-methoxybenzophenone (SM),
- with aluminium chloride in nitromethane at 20° for 24 h (27%). SM was prepared by Friedel–Crafts acylation of 1,4-dimethoxybenzene with p-anisoyl chloride in the presence of stannic chloride in nitromethane at 20° for 1 h (78–94%) [679];
- with excess beryllium chloride in refluxing toluene for 3 h (95%) [395].
- Also refer to: [46,1145].

m.p. 71° [679], 66–68° [395]; ^{1}H NMR [395], IR [395,679], UV [395,679], MS [395]; TLC [395].

(2-Hydroxy-6-methoxyphenyl)(2-methoxyphenyl)methanone

[129103-90-8] $C_{15}H_{14}O_4$ mol.wt. 258.27



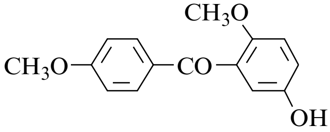
Synthesis

– Preparation from 2-iodo-3-methoxyphenyl 2-methoxybenzoate by rearrangement on treatment with n-butyllithium in a mixture of ethyl ether, hexane and tetrahydrofuran at –70° for 2 h, then treatment with saturated aqueous ammonium chloride (81%) [58].

m.p. 83–83°5 [58]; ^{1}H NMR [58], IR [58], MS [58].

(5-Hydroxy-2-methoxyphenyl)(4-methoxyphenyl)methanone

[80427-36-7] $C_{15}H_{14}O_4$ mol.wt. 258.27



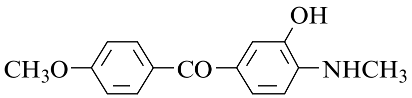
Synthesis

– Preparation by reaction of p-methoxybenzoyl chloride with p-methoxyphenyl benzoate in the presence of stannic chloride in nitromethane at 20° for 2 days, followed by saponification of the m-keto ester so obtained—the 2,4′-dimethoxy-5-(4-methoxybenzoyloxy)benzophenone (73%)—with sodium hydroxide in refluxing methanol for 1 h (quantitative yield) [679].

oil [679]; b.p. (NA); ^{1}H NMR [679], IR [679], UV [679].

[3-Hydroxy-4-(methylamino)phenyl](4-methoxyphenyl)methanone

[136134-37-7] $C_{15}H_{15}NO_3$ mol.wt. 257.29



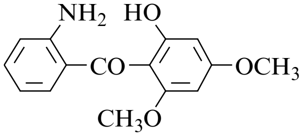
Synthesis

– Preparation from 6-(4-methoxybenzoyl)-3-methyl-benzoxazolinone by hydrolysis with 10% aqueous sodium hydroxide solution [564].

m.p. and Spectra (NA).

(2-Aminophenyl)(2-hydroxy-4,6-dimethoxyphenyl)methanone

[61736-72-9] $C_{15}H_{15}NO_4$ mol.wt. 273.29



Syntheses

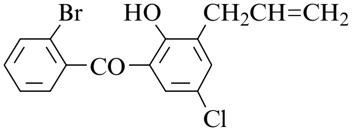
– Preparation by treatment of 2-hydroxy-4,6-dimethoxy-2′-nitrobenzophenone with a mixture of ammonium chloride and zinc moss in dilute ethanol at r.t. for 8 h (quantitative yield) [1123], (77%) [1122].

– Preparation by photo-Fries rearrangement of 3,5-dimethoxyphenyl 2-aminobenzoate in benzene for 12.5 h (45%) [1123].

m.p. 71–75° [1123], 61–63° [1122]; ^{1}H NMR [1122,1123], IR [1122,1123], UV [1122,1123], MS [1122,1123].

(2-Bromophenyl)[5-chloro-2-hydroxy-3-(2-propenyl)phenyl]methanone

[93575-78-1] $C_{16}H_{12}BrClO_2$ mol.wt. 351.63



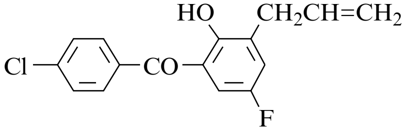
Synthesis

- Preparation by Fries rearrangement of 2-allyl-4-chloro-phenyl o-bromobenzoate with aluminium chloride at 160° for 15 min [1107].

oil [1107]; b.p. and Spectra (NA).

(4-Chlorophenyl)[5-fluoro-2-hydroxy-3-(2-propenyl)phenyl]methanone

[93575-77-0] $C_{16}H_{12}ClFO_2$ mol.wt. 290.72



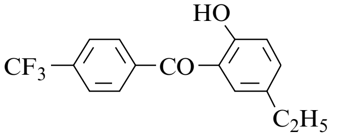
Synthesis

- Preparation by Fries rearrangement of 2-allyl-4-fluorophenyl 4-chlorobenzoate with aluminium chloride at 160° for 15 min [1107].

m.p. 44–46° [1107]; Spectra (NA).

(5-Ethyl-2-hydroxyphenyl)[4-(trifluoromethyl)phenyl]methanone

[61750-29-6] $C_{16}H_{13}F_3O_2$ mol.wt. 294.27



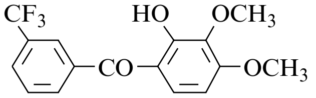
Synthesis

- Preparation by demethylation of 5-ethyl-2-methoxy-4′-(trifluoromethyl)benzophenone (SM) by heating with 55% hydrobromic acid at 110–120° for 5 h. SM was obtained by reaction of p-(trifluoromethyl)benzoyl chloride with p-ethylanisole in the presence of aluminium chloride in methylene chloride at r.t. overnight (92%) [710].

m.p. and Spectra (NA).

(2-Hydroxy-3,4-dimethoxyphenyl)[3-(trifluoromethyl)phenyl]methanone

[140665-42-5] $C_{16}H_{13}F_3O_4$ mol.wt. 326.27



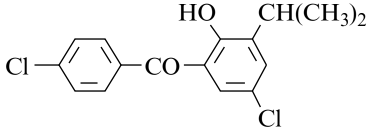
Synthesis

- Obtained by reaction of m-(trifluoromethyl) benzoyl chloride with 1,2,3-trimethoxybenzene in the presence of aluminium chloride in ethylene dichloride between 0° and 5°, then at r.t. for 3 h and at reflux for 1 h (33%) [704].

m.p. 124–126° [704]; Spectra (NA).

[5-Chloro-2-hydroxy-3-(1-methylethyl)phenyl](4-chlorophenyl)methanone

[93575-39-4] $C_{16}H_{14}Cl_2O_2$ mol.wt. 309.19



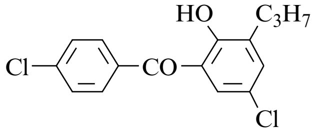
Synthesis

- Preparation by Fries rearrangement of 4-chloro-2-isopropylphenyl 4-chlorobenzoate with aluminium chloride at 160° for 15 min [1107].

m.p. 54–55° [1107]; Spectra (NA).

(5-Chloro-2-hydroxy-3-propylphenyl)(4-chlorophenyl)methanone

[93575-68-9] $C_{16}H_{14}Cl_2O_2$ mol.wt. 309.19



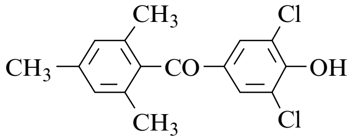
Synthesis

- Preparation by Fries rearrangement of 4-chloro-2-propyl-phenyl p-chlorobenzoate with aluminium chloride at 160° for 15 min [1107].
- Also refer to: [1146].

m.p. 55–56° [1107]; Spectra (NA).

(3,5-Dichloro-4-hydroxyphenyl)(2,4,6-trimethylphenyl)methanone

$C_{16}H_{14}Cl_2O_2$ mol.wt. 309.19



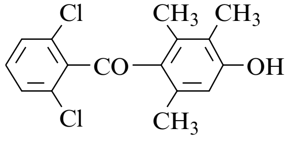
Synthesis

- Preparation by Fries rearrangement of 2,6-dichloro-phenyl mesitylenecarboxylate (2,6-dichlorophenyl 2,4,6-trimethylbenzoate) with aluminium chloride at 155° for 1 h (79%) [489].

m.p. 201°5–203° [489]; Spectra (NA).

(2,6-Dichlorophenyl)(4-hydroxy-2,3,6-trimethylphenyl)methanone

[183724-89-2] $C_{16}H_{14}Cl_2O_2$ mol.wt. 309.19



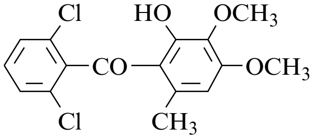
Synthesis

- Preparation by demethylation of 2′,6′-dichloro-4-methoxy-2,3,6-trimethylbenzophenone with hydrobromic acid in acetic acid for 1.5 h at 75° [1072].

m.p. 122° [1072]; Spectra (NA).

(2,6-Dichlorophenyl)(2-hydroxy-3,4-dimethoxy-6-methylphenyl)methanone

[183726-43-4] $C_{16}H_{14}Cl_2O_4$ mol.wt. 341.19



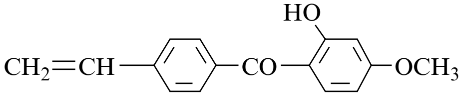
Syntheses

- Preparation by acylation of 3,4,5-trimethoxytoluene with 2,6-dichlorobenzoyl chloride in methylene chloride in the presence of aluminium chloride for 1 h at 0° and for 16 h at r.t. (30%) [1072].
- Also obtained by partial demethylation of 2′,6′-dichloro-6-methyl-2,3,4-trimethoxybenzophenone with hydrobromic acid in acetic acid for 1.5 h at 75° [1072].

N.B.: K salt [1072]. m.p. 161° [1072]; Spectra (NA).

(4-Ethenylphenyl)(2-hydroxy-4-methoxyphenyl)methanone

[48177-42-0] $C_{16}H_{14}O_3$ mol.wt. 254.29



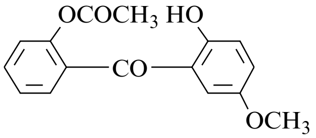
Synthesis

- Preparation by reaction of aqueous potassium hydroxide with 2-hydroxy-4-methoxy-4′-(2-bromoethyl)benzophenone in the presence of hydroquinone in refluxing methanol for 1.5 h under nitrogen bubbling (30%) [1147].
- Also refer to: [85].

m.p. 69–71° [1147]; ^{1}H NMR [1147], IR [1147], UV [1147], MS [1147].

[2-(Acetyloxy)phenyl](2-hydroxy-5-methoxyphenyl)methanone

[83570-58-5] $C_{16}H_{14}O_5$ mol.wt. 286.28



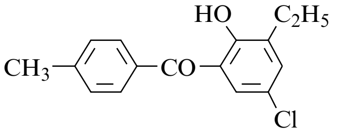
Synthesis

- Obtained by UV light irradiation of p-methoxyphenyl o-acetoxybenzoate (p-methoxyphenyl acetylsalicylate) in benzene [1148], for 11 h (40%) [1025].

yellow oil [1025]; b.p. (NA); ^{1}H NMR [1025], IR [1025], UV [1025], MS [1025].

(5-Chloro-3-ethyl-2-hydroxyphenyl)(4-methylphenyl)methanone

[93575-40-7] $C_{16}H_{15}ClO_2$ mol.wt. 274.75



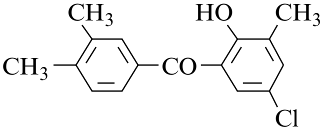
Synthesis

- Preparation by Fries rearrangement of 4-chloro-2-ethyl-phenyl p-toluate with aluminium chloride at 160° for 15 min [1107].

m.p. 53–55° [1107]; Spectra (NA).

(5-Chloro-2-hydroxy-3-methylphenyl)(3,4-dimethylphenyl)methanone

[86914-89-8] $C_{16}H_{15}ClO_2$ mol.wt. 274.75



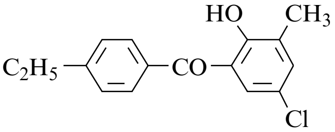
Synthesis

- Preparation by Fries rearrangement of 4-chloro-2-methyl-phenyl 3,4-dimethylbenzoate with aluminium chloride at 180° for 10 min [1074].

m.p. 108° [1074]; Spectra (NA).

(5-Chloro-2-hydroxy-3-methylphenyl)(4-ethylphenyl)methanone

[86914-74-1] $C_{16}H_{15}ClO_2$ mol.wt. 274.75



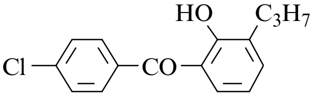
Synthesis

- Preparation by Fries rearrangement of 4-chloro-2-methyl-phenyl 4-ethylbenzoate with aluminium chloride at 180–185° for 10 min [1074,1149].

m.p. 152–155° [1074,1149]; Spectra (NA).

(4-Chlorophenyl)(2-hydroxy-3-propylphenyl)methanone

[108294-79-7] $C_{16}H_{15}ClO_2$ mol.wt. 274.75



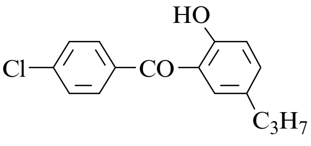
Synthesis

- Preparation by reaction of 2-hydroxy-3-propylbenzoyl chloride with chlorobenzene in the presence of aluminium chloride in refluxing carbon disulfide (35%) [92].

m.p. 68–70° [92]; Spectra (NA).

(4-Chlorophenyl)(2-hydroxy-5-propylphenyl)methanone

[61466-81-7] $C_{16}H_{15}ClO_2$ mol.wt. 274.75

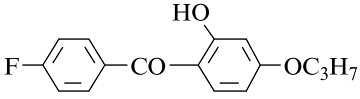


Synthesis

- Preparation by reaction of p-chlorobenzoyl chloride with p-propylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (27–28%) [92,1091].

b.p.$_{0.02}$ 151–153° [92,1091]; Spectra (NA).

(4-Fluorophenyl)(2-hydroxy-4-propoxyphenyl)methanone



$C_{16}H_{15}FO_3$ mol.wt. 274.29

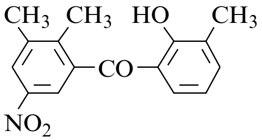
Synthesis

- Preparation by partial alkylation of 4′-fluoro-2,4-di-hydroxybenzophenone with a propyl halide in the presence of an alkali [1109].

m.p. and Spectra (NA).

(2,3-Dimethyl-5-nitrophenyl)(2-hydroxy-3-methylphenyl)methanone

[110969-54-5]



$C_{16}H_{15}NO_4$ mol.wt. 285.30

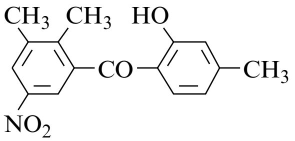
Synthesis

- Obtained (by-product) by Fries rearrangement of o-cresyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (<4%) [1003].

m.p. 104° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2,3-Dimethyl-5-nitrophenyl)(2-hydroxy-4-methylphenyl)methanone

[110969-57-8]



$C_{16}H_{15}NO_4$ mol.wt. 285.30

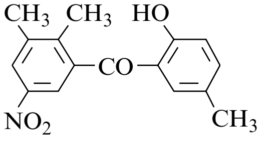
Synthesis

- Preparation by Fries rearrangement of m-cresyl 2,3-di-methyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (43%) [1003].

m.p. 158° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2,3-Dimethyl-5-nitrophenyl)(2-hydroxy-5-methylphenyl)methanone

[110969-60-3]



$C_{16}H_{15}NO_4$ mol.wt. 285.30

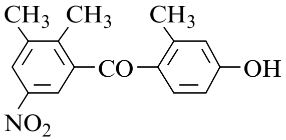
Synthesis

- Preparation by Fries rearrangement of p-cresyl 2,3-di-methyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (70%) [1003].

m.p. 165° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2,3-Dimethyl-5-nitrophenyl)(4-hydroxy-2-methylphenyl)methanone

[110969-58-9]



$C_{16}H_{15}NO_4$ mol.wt. 285.30

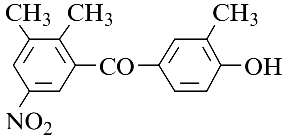
Synthesis

- Obtained (by-product) by Fries rearrangement of m-cresyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (12%) [1003].

m.p. 222° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2,3-Dimethyl-5-nitrophenyl)(4-hydroxy-3-methylphenyl)methanone

[110969-55-6] $C_{16}H_{15}NO_4$ mol.wt. 285.30



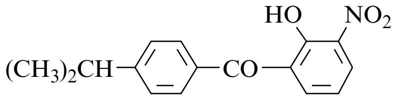
Synthesis

– Preparation by Fries rearrangement of o-cresyl 2,3-di-methyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (56%) [1003].

m.p. 210° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2-Hydroxy-3-nitrophenyl)[4-(1-methylethyl)phenyl]methanone

[35698-18-1] $C_{16}H_{15}NO_4$ mol.wt. 285.30



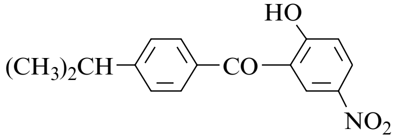
Synthesis

– Obtained by reaction of fuming nitric acid with 2-hydroxy-4′-isopropylben zophenone in an acetic acid/acetic anhydride mixture (4:3), first at 10° for 50 min, then at 20° for 40 min (19%) [889,891].

m.p. and Spectra (NA).

(2-Hydroxy-5-nitrophenyl)[4-(1-methylethyl)phenyl]methanone

[35698-19-2] $C_{16}H_{15}NO_4$ mol.wt. 285.30



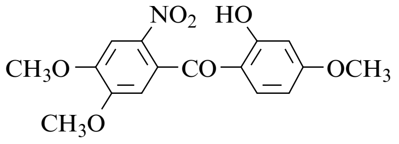
Synthesis

– Obtained by reaction of fuming nitric acid with 2-hydroxy-4′-isopropyl-benzophenone in an acetic acid/acetic anhydride mixture (4:3), first at 10° for 50 min, then at 20° for 40 min (10%) [889,891].

m.p. and Spectra (NA).

(4,5-Dimethoxy-2-nitrophenyl)(2-hydroxy-4-methoxyphenyl)methanone

$C_{16}H_{15}NO_7$ mol.wt. 333.30



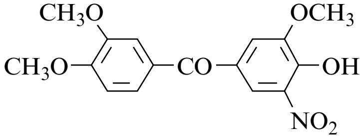
Synthesis

– Obtained by action of a mixture of nitric acid (d = 1.42)/concentrated sulfuric acid with 2-hydroxy-3′,4,4′-trimethoxybenzophenone in acetic acid at 48–52° for 16 min [1150].

m.p. 211° [1150]; Spectra (NA).

(3,4-Dimethoxyphenyl)(4-hydroxy-3-methoxy-5-nitrophenyl)methanone

[134612-42-3]



$C_{16}H_{15}NO_7$ mol.wt. 333.30

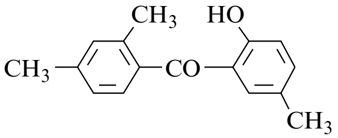
Synthesis

- Preparation by reaction of 65% nitric acid with 4-hydroxy-3,3′,4′-trimethoxy-benzophenone in acetic acid at 20° [1019].

m.p. 170–180° [1019]; Spectra (NA).

(2,4-Dimethylphenyl)(2-hydroxy-5-methylphenyl)methanone

[80018-48-0]



$C_{16}H_{16}O_2$ mol.wt. 240.30

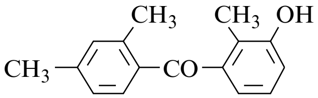
Synthesis

- Preparation by Fries rearrangement of p-cresyl 2,4-di-methylbenzoate with aluminium chloride [1062].

m.p. and Spectra (NA).

(2,4-Dimethylphenyl)(3-hydroxy-2-methylphenyl)methanone

[76981-57-2]



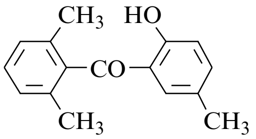
$C_{16}H_{16}O_2$ mol.wt. 240.30

Synthesis

- Preparation by diazotization of 3-amino-2,2′,4′-trimethyl-benzophenone, followed by hydrolysis of the diazonium salt so obtained (54%) [119], according to [634].

m.p. 124° [119]; Spectra (NA).

(2,6-Dimethylphenyl)(2-hydroxy-5-methylphenyl)methanone



$C_{16}H_{16}O_2$ mol.wt. 240.30

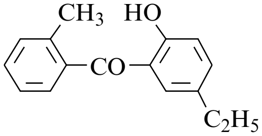
Synthesis

- Obtained by Fries rearrangement of p-cresyl 2,6-dimethyl-benzoate in the presence of aluminium chloride, first in refluxing carbon disulfide for 2 h, then at 150° for 1 h after elimination of solvent (22%) [1151].

m.p. 89°7–90°7 [1151]; Spectra (NA).

(5-Ethyl-2-hydroxyphenyl)(2-methylphenyl)methanone

[170799-15-2] $C_{16}H_{16}O_2$ mol.wt. 240.30



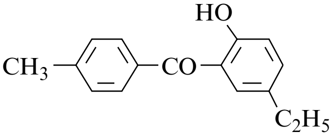
Synthesis

– Preparation by Fries rearrangement of p-ethylphenyl o-toluate with aluminium chloride at 100–150° for 0.5–3 h (95%) [29].

oil [29]; b.p. and Spectra (NA).

(5-Ethyl-2-hydroxyphenyl)(4-methylphenyl)methanone

[61750-25-2] $C_{16}H_{16}O_2$ mol.wt. 240.30



Syntheses

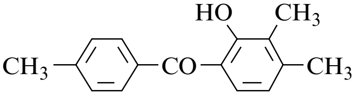
– Preparation by reaction of p-methylbenzoyl chloride with p-ethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (38%) [92].

– Preparation by Fries rearrangement of p-ethylphenyl p-toluate with aluminium chloride in tetrachloroethane at 125° for 6 h [710].

m.p. 49–51° [92,710]; Spectra (NA).

(2-Hydroxy-3,4-dimethylphenyl)(4-methylphenyl)methanone

[16762-05-3] $C_{16}H_{16}O_2$ mol.wt. 240.30



Syntheses

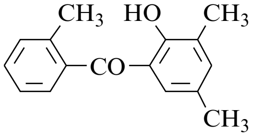
– Preparation by Fries rearrangement of 2,3-dimethyl-phenyl p-toluate with aluminium chloride at 180° for 10 min (55%) [518].

– Also obtained by degradation of 6,7-dimethyl-3-(4-methylphenyl)-2-phenylb enzofuran with chromium trioxide in boiling acetic acid, followed by saponification of the keto ester so obtained (2-benzoyloxy-3,4-dimethyl-4′-methyl benzophenone) [44,46].

m.p. 84° [44,46,518,718]; IR [44].

(2-Hydroxy-3,5-dimethylphenyl)(2-methylphenyl)methanone

[86914-79-6] $C_{16}H_{16}O_2$ mol.wt. 240.30



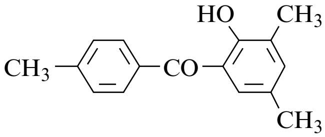
Synthesis

– Preparation by Fries rearrangement of 2,4-dimethyl phenyl o-toluate with aluminium chloride at 180° for 10 min [1074].

oil [1074]; b.p. and Spectra (NA).

(2-Hydroxy-3,5-dimethylphenyl)(4-methylphenyl)methanone

[86914-85-4] $C_{16}H_{16}O_2$ mol.wt. 240.30



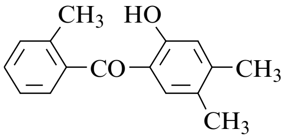
Syntheses

- Preparation by Fries rearrangement of 2,4-dimethylphenyl p-toluate with aluminium chloride at 180° for 10 min [1074], (60%) [518].
- Obtained by degradation of 5,7-dimethyl-3-(4-methyl-phenyl)-2-phenylbenzofuran in boiling acetic acid, followed by saponification of the keto ester so obtained (2-benzoyloxy-3,4′,5-trimethyl-benzophenone) [44].

oil [44]; m.p. 55–56° [1074], 54° [518]; ^{1}H NMR [518], IR [44,518].

(2-Hydroxy-4,5-dimethylphenyl)(2-methylphenyl)methanone

[93433-88-6] $C_{16}H_{16}O_2$ mol.wt. 240.30



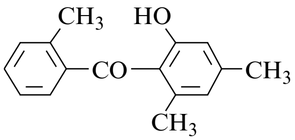
Synthesis

- Preparation by Fries rearrangement of 3,4-dimethylphenyl o-toluate with aluminium chloride at 100–150° for 0.5–3 h (80%) [29].

m.p. 56–57° [29]; Spectra (NA).

(2-Hydroxy-4,6-dimethylphenyl)(2-methylphenyl)methanone

[62261-96-5] $C_{16}H_{16}O_2$ mol.wt. 240.30



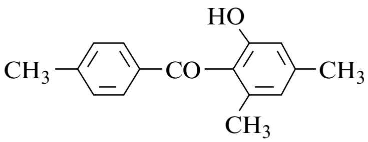
Synthesis

- Refer to: [734].

m.p. and Spectra (NA).

(2-Hydroxy-4,6-dimethylphenyl)(4-methylphenyl)methanone

[62261-95-4] $C_{16}H_{16}O_2$ mol.wt. 240.30

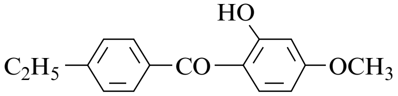


Synthesis

- Refer to: [734].

m.p. and Spectra (NA).

(4-Ethylphenyl)(2-hydroxy-4-methoxyphenyl)methanone



$C_{16}H_{16}O_3$ mol.wt. 256.30

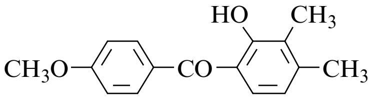
Synthesis

- Preparation by partial demethylation of 2,4-di-methoxy-4′-ethylbenzophenone with aluminium chloride in chlorobenzene at 80–100° [655].
- Also refer to: [222].

m.p. and Spectra (NA).

(2-Hydroxy-3,4-dimethylphenyl)(4-methoxyphenyl)methanone

[16762-06-4]



$C_{16}H_{16}O_3$ mol.wt. 256.30

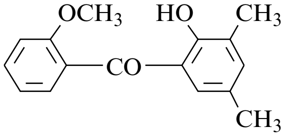
Synthesis

- Preparation by oxidation of 6,7-dimethyl-2-phenyl-3-(4-methoxyphenyl) benzofuran with chromium trioxide in boiling acetic acid, followed by saponification of the keto ester so formed with sodium hydroxide [44] or potassium hydroxide [46] in refluxing ethanol.

m.p. 86° [44], 85° [46]; IR [44,46].

(2-Hydroxy-3,5-dimethylphenyl)(2-methoxyphenyl)methanone

[72324-22-2]



$C_{16}H_{16}O_3$ mol.wt. 256.30

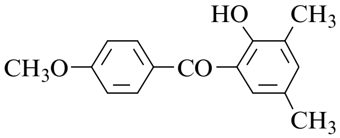
Synthesis

- Obtained (poor yields) by reaction of o-anisic acid with 2,4-dimethylphenol,
  - in the presence of methanesulfonic acid at 100° for 1 h (6%) [991];
  - in the presence of butanesulfonic acid at 140° for 1 h (4%) [991].

m.p. 75° [991]; ^{1}H NMR (Sadtler: standard n° 38495 M),
IR (Sadtler: standard n° 65533 K) [991], UV [991], MS [1131].

(2-Hydroxy-3,5-dimethylphenyl)(4-methoxyphenyl)methanone

[72324-20-0]



$C_{16}H_{16}O_3$ mol.wt. 256.30

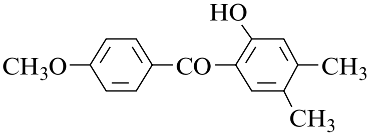
Synthesis

- Preparation by Fries rearrangement of 2,4-dimethylphenyl p-anisate with titanium tetrachloride at 120° for 1 h (82%) [991].

m.p. 66° [991]; ^{1}H NMR (Sadtler: standard n° 38496 M),
IR (Sadtler: standard n° 65534 K) [991], UV [991], MS [1131].

(2-Hydroxy-4,5-dimethylphenyl)(4-methoxyphenyl)methanone

[54921-19-6] $C_{16}H_{16}O_3$ mol.wt. 256.30



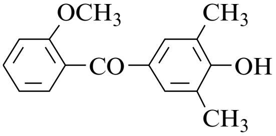
Synthesis

– Preparation by oxidation of 2-phenyl-3-(4-methoxy-phenyl)-5,6-dimethylbenzofuran with chromium trioxide in boiling acetic acid for 2 h, followed by saponification of the keto ester so formed with 10% sodium hydroxide in boiling ethanol for 15 min [1133].

m.p. 92–93° [1133]; ^{1}H NMR [1133], IR [1133].

(4-Hydroxy-3,5-dimethylphenyl)(2-methoxyphenyl)methanone

[72324-21-1] $C_{16}H_{16}O_3$ mol.wt. 256.30



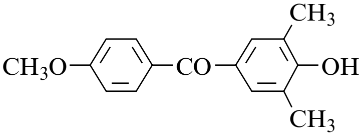
Synthesis

– Preparation by reaction of o-anisic acid with 2,6-dimethyl-phenol in the presence of methanesulfonic acid at 120° for 20 min (46%) [991].

m.p. 145° [991]; ^{1}H NMR (Sadtler: standard n° 38492 M), IR (Sadtler: standard n° 65530 K) [991], UV [991], MS [1131].

(4-Hydroxy-3,5-dimethylphenyl)(4-methoxyphenyl)methanone

[72324-19-7] $C_{16}H_{16}O_3$ mol.wt. 256.30



Synthesis

– Preparation by Fries rearrangement of 2,6-dimethyl-phenyl p-anisate in nitromethane,

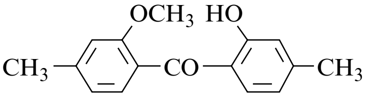
- in the presence of titanium tetrachloride at 20° for 170 h (82%) [991];
- in the presence of antimony pentachloride at 20° for 25 h (40%) [991].

– Also refer to: [996].

m.p. 125° [991]; ^{1}H NMR (Sadtler: standard n° 38500 M), IR (Sadtler: standard n° 65538 K) [991], UV [991], MS [1131].

(2-Hydroxy-4-methylphenyl)(2-methoxy-4-methylphenyl)methanone

[54468-79-0] $C_{16}H_{16}O_3$ mol.wt. 256.30



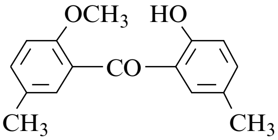
Synthesis

– Obtained by oxidation of 6-methyl-2-phenyl-3-(2-methoxy-4-methylphenyl) benzofuran with chromium trioxide, then saponification of the keto ester so formed, the 2-(benzoyloxy)-2′-methoxy-4,4′-dimethylbenzophenone [1152].

oil [1152]; b.p. (NA); IR [1152].

(2-Hydroxy-5-methylphenyl)(2-methoxy-5-methylphenyl)methanone

[32229-35-9] $C_{16}H_{16}O_3$ mol.wt. 256.30



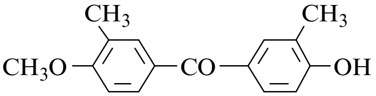
Syntheses

- Obtained by Friedel–Crafts acylation of excess p-methoxy-toluene with 2-methoxy-5-methylbenzoyl chloride in the presence of aluminium chloride, first at 0° for 2 h, then at r.t. overnight and at reflux for 3 h (23%) [1153].
- Also obtained (by-product) by Friedel–Crafts acylation of p-methoxytoluene with 2-methoxy-5-methylbenzoyl chloride in carbon disulfide in the presence of aluminium chloride, first at 0° for 5 h, then at r.t. overnight and at reflux for 2 h (10%) [1153].
- Also refer to: [1154].

m.p. 68–69° [1153,1154]; IR [1154], UV [1154].

(4-Hydroxy-3-methylphenyl)(4-methoxy-3-methylphenyl)methanone

[79002-05-4] $C_{16}H_{16}O_3$ mol.wt. 256.30



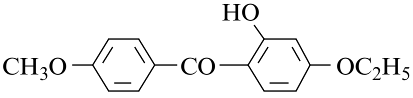
Synthesis

- Obtained by rapid degradation of 4-methoxy-3,3′-di-methylbenzophenone [41295-28-7] (an herbicide) in reductive flooded soils (main metabolite) [1155].

m.p. and Spectra (NA).

(4-Ethoxy-2-hydroxyphenyl)(4-methoxyphenyl)methanone

$C_{16}H_{16}O_4$ mol.wt. 272.30



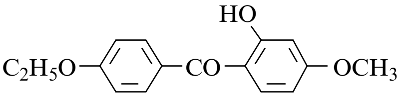
Synthesis

- Preparation by partial demethylation of 2,4′-di-methoxy-4-ethoxybenzophenone with aluminium chloride in chlorobenzene at 80–100° [655].
- Also refer to: [222].

m.p. and Spectra (NA).

(4-Ethoxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone

$C_{16}H_{16}O_4$ mol.wt. 272.30



Syntheses

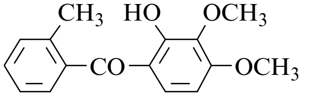
- Preparation by reaction of 2,4-dimethoxybenzoyl chloride with phenetole in the presence of aluminium chloride in a chlorobenzene/N,N-di-methylformamide mixture (22:1) at 115° [235,657].

- Preparation by partial demethylation of 4′-ethoxy-2,4-dimethoxybenzophenone with aluminium chloride or aluminium bromide in chlorobenzene at 90–95° (good yield) [655].

m.p. (NA); UV [235].

(2-Hydroxy-3,4-dimethoxyphenyl)(2-methylphenyl)methanone

[140665-41-4] $C_{16}H_{16}O_4$ mol.wt. 272.30



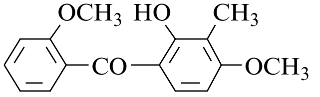
Synthesis

- Preparation by reaction of o-toluoyl chloride with 1,2,3-tri-methoxybenzene in the presence of aluminium chloride in ethylene dichloride between 0° and 5°, then at r.t. for 3 h and at reflux for 1 h (63%) [704].

m.p. 93–95° [704]; Spectra (NA).

(2-Hydroxy-4-methoxy-3-methylphenyl)(2-methoxyphenyl)methanone

[115296-09-8] $C_{16}H_{16}O_4$ mol.wt. 272.30



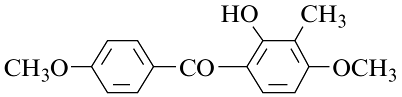
Synthesis

- Preparation by reaction of o-anisoyl chloride with 3-methoxy-2-methylphenol in methylene chloride in the presence of aluminium chloride first at 0° for 2 h and at r.t. for 1 h (31%) [1029].

m.p. 111–115° [1029]; Spectra (NA).

(2-Hydroxy-4-methoxy-3-methylphenyl)(4-methoxyphenyl)methanone

$C_{16}H_{16}O_4$ mol.wt. 272.30



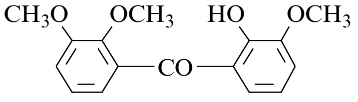
Synthesis

- Preparation by partial demethylation of 2,4,4′-tri-methoxy-3-methylbenzophenone with aluminium chloride in chlorobenzene at 80–100° [655].
- Also refer to: [222].

m.p. and Spectra (NA).

(2,3-Dimethoxyphenyl)(2-hydroxy-3-methoxyphenyl)methanone

[35040-42-7] $C_{16}H_{16}O_5$ mol.wt. 288.30



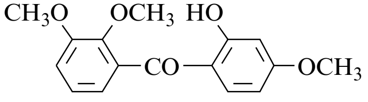
Synthesis

- Obtained by photo-Fries rearrangement of o-methoxy-phenyl 2,3-dimethoxybenzoate in ethanol at r.t. for 22 h (16%) [397].

m.p. 110–110°5 [397]; ^{1}H NMR [397], IR [397], UV [397].

(2,3-Dimethoxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone

[147188-10-1] $C_{16}H_{16}O_5$ mol.wt. 288.30



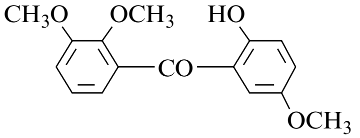
Synthesis

- Obtained, in mixture with 2′-hydroxy-2,3′,4-tri-methoxybenzophenone, by reaction of 2,3-di-methoxybenzoyl chloride with resorcinol dimethyl ether in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (total yield: 54%) [1010].
- Also refer to: [1011,1027].

m.p. (NA); ^{1}H NMR [1010].

(2,3-Dimethoxyphenyl)(2-hydroxy-5-methoxyphenyl)methanone

[35040-36-9] $C_{16}H_{16}O_5$ mol.wt. 288.30



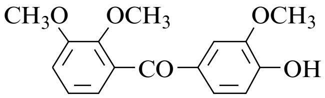
Synthesis

- Obtained by photo-Fries rearrangement of p-methoxy-phenyl 2,3-dimethoxybenzoate in ethanol at 30–40° for 9 h (42%) [397].

m.p. 74–76° [397]; ^{1}H NMR [397], IR [397], UV [397].

(2,3-Dimethoxyphenyl)(4-hydroxy-3-methoxyphenyl)methanone

[35042-49-0] $C_{16}H_{16}O_5$ mol.wt. 288.30



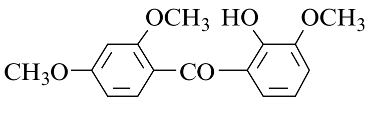
Synthesis

- Obtained (poor yield) by photo-Fries rearrangement of o-methoxyphenyl 2,3-dimethoxybenzoate in ethanol at r.t. for 22 h (<7%) [397].

m.p. 143–143°5 [397]; ^{1}H NMR [397], IR [397], UV [397].

(2,4-Dimethoxyphenyl)(2-hydroxy-3-methoxyphenyl)methanone

$C_{16}H_{16}O_5$ mol.wt. 288.30



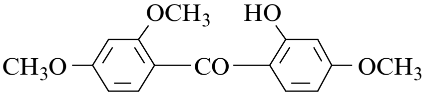
Synthesis

- Obtained, in mixture with 2-hydroxy-2′,3′,4-tri-methoxybenzophenone, by reaction of 2,3-di-methoxybenzoyl chloride with resorcinol dimethyl ether in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (total yield: 54%) [1010].
- Also refer to: [1011].

m.p. (NA); ^{1}H NMR [1010].

(2,4-Dimethoxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone

[4142-51-2] $C_{16}H_{16}O_5$ mol.wt. 288.30



Syntheses

- Preparation by reaction of 2,4-dimethoxybenzoic acid with m-methoxyphenol in the presence of boron trifluoride [650].
- Preparation by partial methylation of 2,2′-di-hydroxy-4,4′-dimethoxybenzophenone with methyl iodide in the presence of potassium carbonate in refluxing acetone for 12 h (68%) [1156].
- Preparation by treatment of a difluoroboron chelate (SM) in methanol at 50° for 10 min (95%) [1157,1158]. SM was obtained by reaction of 2,2′,4,4′-tetramethoxybenzophenone with boron trifluoride-etherate in refluxing toluene (86%, m.p. 160–161°) [1157,1158].

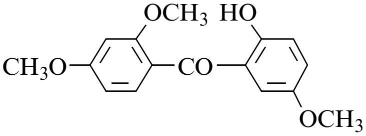
N.B.: A mixture of products is obtained from aluminium chloride, induced cleavage of 2,2′,4,4′-tetramethoxy-benzophenone [1159].

- Also obtained by reaction of 2,4-dimethoxybenzoyl chloride with resorcinol dimethyl ether in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (52%) [1010].
- Also refer to: [222,1011,1136].

m.p. 110–111°5 [1156], 108–109° [1157,1158], 104–105° [650];
^{1}H NMR [1156,1158], IR [650,1010,1156,1158], UV [650], MS [1156].

(2,4-Dimethoxyphenyl)(2-hydroxy-5-methoxyphenyl)methanone

[169455-12-3] $C_{16}H_{16}O_5$ mol.wt. 288.30



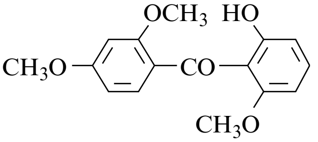
Synthesis

- Obtained, in mixture with 2′-hydroxy-2,4′,5-trimethoxybenzophenone, by reaction of 2,4-dimethoxybenzoyl chloride with hydroquinone dimethyl ether in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (total yield: 54%) [1010].
- Also refer to: [1011,1027].

m.p. (NA); ^{1}H NMR [1010].

(2,4-Dimethoxyphenyl)(2-hydroxy-6-methoxyphenyl)methanone

[147188-11-2] $C_{16}H_{16}O_5$ mol.wt. 288.30



Synthesis

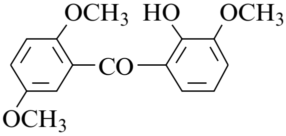
- Obtained, in mixture with 2′-hydroxy-2,4′,6-trimethoxybenzophenone, by reaction of 2,4-dimethoxy-benzoyl chloride with resorcinol dimethyl ether in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (total yield: 52%) [1010].

- Also refer to: [1011,1027].

m.p. (NA); ^{1}H NMR [1010].

(2,5-Dimethoxyphenyl)(2-hydroxy-3-methoxyphenyl)methanone

[129168-52-1] $C_{16}H_{16}O_5$ mol.wt. 288.30



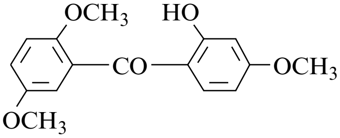
Synthesis

- Preparation by Friedel–Crafts acylation of hydroquinone dimethyl ether with 2,3-dimethoxybenzoyl chloride in ethyl ether in the presence of aluminium chloride for 1 h at 45–50° (72%) [1160].

m.p. 100–100°5 [1160]; Spectra (NA).

(2,5-Dimethoxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone

[62495-96-9] $C_{16}H_{16}O_5$ mol.wt. 288.30



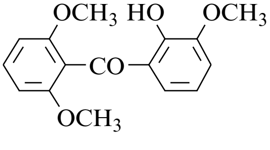
Syntheses

- Preparation by reaction of 2,5-dimethoxybenzoic acid with 3-methoxyphenol in the presence of zinc chloride and phosphorous oxychloride at 65–70° for 2 h (21%) [1136].
- Also obtained, in mixture with 2-hydroxy-2′,4′,5-trimethoxybenzophenone, by reaction of 2,4-dimethoxybenzoyl chloride with hydroquinone dimethyl ether in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (total yield: 54%) [1010].
- Also refer to: [1011].

m.p. 110° [1136]; ^{1}H NMR [1010,1136], IR [1136], UV [1136].

(2,6-Dimethoxyphenyl)(2-hydroxy-3-methoxyphenyl)methanone

[37570-57-3] $C_{16}H_{16}O_5$ mol.wt. 288.30



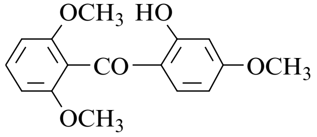
Synthesis

- Obtained by demethylation of 2,2′,3,6′-tetramethoxy-benzophenone,
  - in the presence of boron trichloride in methylene chloride for 30 min (88%) (high selectivity) [1161];
  - in the presence of 40% aqueous hydrobromic acid in refluxing acetic acid for 2 h (17%) [1161].

m.p. 146–148° [1161]; ^{1}H NMR [1161], IR [1161], MS [1161].

(2,6-Dimethoxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone

[147188-12-3] $C_{16}H_{16}O_5$ mol.wt. 288.30



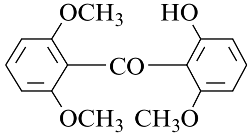
Synthesis

- Obtained, in mixture with 2-hydroxy-2′,4′,6-trimethoxy-benzophenone, by reaction of 2,4-dimethoxybenzoyl chloride with resorcinol dimethyl ether in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (total yield: 52%) [1010].
- Also refer to: [1011].

m.p. (NA); ^{1}H NMR [1010].

(2,6-Dimethoxyphenyl)(2-hydroxy-6-methoxyphenyl)methanone

[129103-93-1] $C_{16}H_{16}O_5$ mol.wt. 288.30



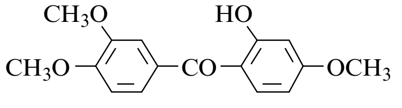
Synthesis

- Obtained from 2-iodo-3-methoxyphenyl 2,6-dimethoxy-benzoate by rearrangement on treatment with n-butyllithium in a mixture of ethyl ether, hexane and tetrahydrofuran at −100° followed by heating to −70°, then treatment with saturated aqueous ammonium chloride (9%) [58].

m.p. and Spectra (NA).

(3,4-Dimethoxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone

[42045-60-3] $C_{16}H_{16}O_5$ mol.wt. 288.30



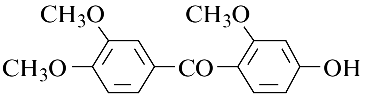
Syntheses

- Obtained by reaction of methyl iodide with 2,4-di-hydroxy-3′,4′-dimethoxy-benzophenone in the presence of potassium carbonate in refluxing acetone [218], (89%) [1137].
- Also obtained from β-(5-methoxy-2-veratroylphenoxy)propionic acid by heating with aqueous sodium hydroxide solution [218].
- Preparation by reaction of 3,4-dimethoxybenzoic acid with m-methoxyphenol in the presence of boron trifluoride [650].
- Preparation by reaction of veratroyl chloride,
  - with resorcinol monomethyl ether in the presence of aluminium chloride in nitrobenzene, first at 0° for 3 h, then at 10–16° for 18 h [1150];
  - with resorcinol dimethyl ether in the presence of aluminium chloride in refluxing carbon disulfide for 1 h (61%) [1162].

- Also obtained by condensation of ethyl m-methoxyphenoxypropionate with veratroyl chloride in the presence of aluminium chloride in nitrobenzene at 0° for 12 h [1150].
- Also refer to: [193,222,235,1136].

m.p. 141° [218,1150], 140–141° [1162], 140° [1137], 135–136° [650];
IR [650], UV [650].

(3,4-Dimethoxyphenyl)(4-hydroxy-2-methoxyphenyl)methanone



$C_{16}H_{16}O_5$ mol.wt. 288.30

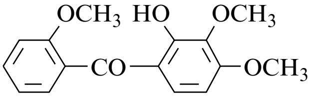
Syntheses

- Obtained by reaction of veratroyl chloride with resorcinol monomethyl ether in the presence of aluminium chloride in nitrobenzene, first at 0° for 3 h, then at 10–16° for 18 h [1150].
- Also obtained from β-(3-methoxy-4-veratroylphenoxy)propionic acid on heating with an aqueous sodium hydroxide solution [218].

m.p. 179° [218], 175° [1150]; Spectra (NA).

(2-Hydroxy-3,4-dimethoxyphenyl)(2-methoxyphenyl)methanone

[147188-08-7]



$C_{16}H_{16}O_5$ mol.wt. 288.30

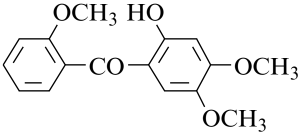
Synthesis

- Obtained, in mixture with 2′-hydroxy-2,3,4-trimethoxy-benzophenone, by reaction of 2-methoxybenzoyl chloride with pyrogallol trimethyl ether in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (total yield: 56–59%) [1010,1011].
- Also refer to: [1027].

m.p. (NA); ^{1}H NMR [1010].

(2-Hydroxy-4,5-dimethoxyphenyl)(2-methoxyphenyl)methanone

[42833-48-7]



$C_{16}H_{16}O_5$ mol.wt. 288.30

Syntheses

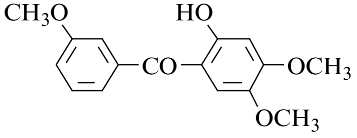
- Obtained, in mixture with 2,4,5-trimethoxy-2′-hydroxy-benzophenone, by reaction of o-anisoyl chloride with 1,2,4-trimethoxybenzene in ethyl ether in the presence of aluminium chloride for 20 h at r.t. (total yield: 56%) [416,1010] or for 48 h at r.t. [1163].

- Also obtained from 2,5-dihydroxy-2′,4-dimethoxybenzophenone on treatment with ethereal methanolic diazomethane for 4.5 h [416].
- Also refer to: [1011].

m.p. 104–105° [416], 98° [1163]; ^{1}H NMR [416,1010,1163], UV [416], MS [416].

(2-Hydroxy-4,5-dimethoxyphenyl)(3-methoxyphenyl)methanone

[51106-90-2] $C_{16}H_{16}O_5$ mol.wt. 288.30



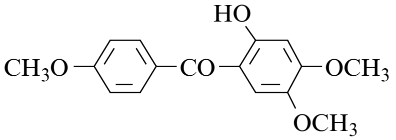
Synthesis

- Preparation by acylation of 1,2,4-trimethoxybenzene with m-anisoyl chloride in ethyl ether in the presence of aluminium chloride at r.t. for 48 h (35%) [1163].

m.p. 94° [1163]; ^{1}H NMR [1163].

(2-Hydroxy-4,5-dimethoxyphenyl)(4-methoxyphenyl)methanone

[58115-11-0] $C_{16}H_{16}O_5$ mol.wt. 288.30



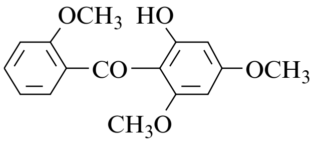
Synthesis

- Obtained (by-product) by condensation of p-anisoyl chloride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride in refluxing carbon disulfide for 6 h [428,761] or at r.t. several days [762].

m.p. 127–128° [761,762], 124–125° [428]; Spectra (NA).

(2-Hydroxy-4,6-dimethoxyphenyl)(2-methoxyphenyl)methanone

[147188-04-3] $C_{16}H_{16}O_5$ mol.wt. 288.30



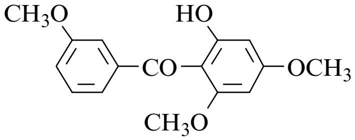
Synthesis

- Obtained, in mixture with 2′-hydroxy-2,4,6-trimethoxy-benzophenone, by reaction of 2-methoxybenzoyl chloride with phloroglucinol trimethyl ether in ethyl ether in the presence of aluminium chloride for 8 h at r.t. (total yield: 59%) [1010].
- Also refer to: [1011,1027].

m.p. (NA); ^{1}H NMR [1010].

(2-Hydroxy-4,6-dimethoxyphenyl)(3-methoxyphenyl)methanone

[21382-23-0]



$C_{16}H_{16}O_5$ mol.wt. 288.30

Synthesis

- Preparation by partial methylation of 2,3′,4,6-tetra-hydroxybenzophenone or of 2,4,6-trihydroxy-3′-methoxybenzophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 6 h (45–50%) [422].

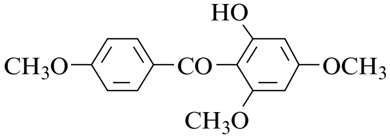
Isolation from natural source

- From fresh *Gentiana lutea* rhizome (Gentianaceae) [423].

m.p. 123–125° [422]; b.p.$_{0.1}$ 180–188° [422]; ^{1}H NMR [422], IR [422], UV [422].

(2-Hydroxy-4,6-dimethoxyphenyl)(4-methoxyphenyl)methanone

[97746-14-0]



$C_{16}H_{16}O_5$ mol.wt. 288.30

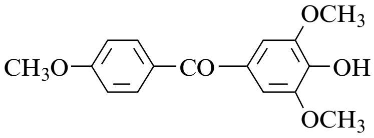
Synthesis

- Preparation by Friedel–Crafts acylation of 1,3,5-tri-methoxybenzene with p-methoxybenzoyl chloride in the presence of aluminium chloride in ethyl ether for 4 h (70%) [653].

m.p. 150–151° [653]; ^{1}H NMR [653].

(4-Hydroxy-3,5-dimethoxyphenyl)(4-methoxyphenyl)methanone

[54808-44-5]



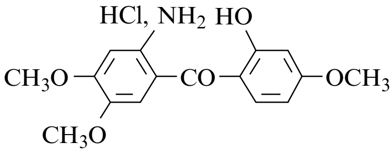
$C_{16}H_{16}O_5$ mol.wt. 288.30

Synthesis

- Refer to: [334].

m.p. and Spectra (NA).

(2-Amino-4,5-dimethoxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone *(Hydrochloride)*



$C_{16}H_{17}NO_5$,HCl mol.wt. 339.78

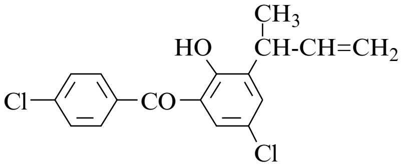
Synthesis

- Preparation by reduction of 2-hydroxy-2′-nitro-4,4′,5′-trimethoxybenzophenone in ethanol with stannous chloride, tin foil and concentrated hydrochloric acid on a steam bath for 15 min [1150].

m.p. 240° [1150]; Spectra (NA).

[5-Chloro-2-hydroxy-3-(1-methyl-2-propenyl)phenyl](4-chlorophenyl) methanone

[93575-41-8]



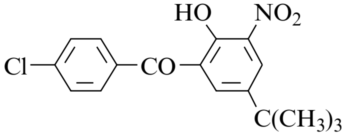
$C_{17}H_{14}Cl_2O_2$ mol.wt. 321.20

Synthesis

- Preparation by Fries rearrangement of 4-chloro-2-(1-methylallyl)phenyl p-chlorobenzoate with aluminium chloride at 160° for 15 min [1107].

m.p. 45–47° [1107]; Spectra (NA).

[5-(1,1-Dimethylethyl)-2-hydroxy-3-nitrophenyl](4-chlorophenyl)methanone



$C_{17}H_{16}ClNO_4$ mol.wt. 333.77

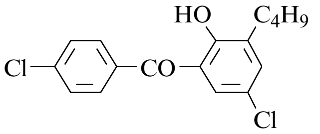
Synthesis

- Preparation by reaction of a concentrated nitric acid/concentrated sulfuric acid mixture with 5-tert-butyl-4′-chloro-2-hydroxybenzophenone in methylene chloride at 5° for 30 min (43%) [472].

m.p. 96–98° [472]; ^{1}H NMR [472]; TLC [472].

(3-Butyl-5-chloro-2-hydroxyphenyl)(4-chlorophenyl)methanone

[93575-74-7]



$C_{17}H_{16}Cl_2O_2$ mol.wt. 323.22

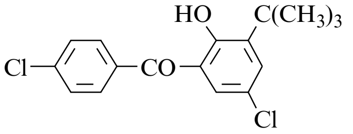
Synthesis

- Preparation by Fries rearrangement of 2-butyl-4-chloro-phenyl p-chlorobenzoate with aluminium chloride at 160° for 15 min [1107].

oil [1107]; b.p.$_{0.04}$ 168–170° [1107]; Spectra (NA).

[5-Chloro-3-(1,1-dimethylethyl)-2-hydroxyphenyl](4-chlorophenyl)methanone

[93575-72-5]



$C_{17}H_{16}Cl_2O_2$ mol.wt. 323.22

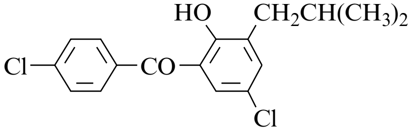
Synthesis

- Preparation by photo-Fries rearrangement of 2-tert-butyl-4-chlorophenyl p-chlorobenzoate in benzene under nitrogen for 32 h [1107].

m.p. 86°5–87° [1107]; Spectra (NA).

[5-Chloro-2-hydroxy-3-(2-methylpropyl)phenyl](4-chlorophenyl)methanone

[93575-75-8] $C_{17}H_{16}Cl_2O_2$ mol.wt. 323.22



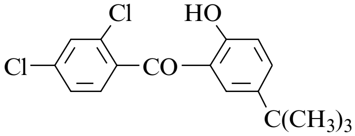
Synthesis

- Preparation by Fries rearrangement of 2-iso-butyl-4-chlorophenyl 4-chlorobenzoate with aluminium chloride at 160° for 15 min [1107].

m.p. 65–66° [1107]; Spectra (NA).

(2,4-Dichlorophenyl)[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone

[61709-37-3] $C_{17}H_{16}Cl_2O_2$ mol.wt. 323.22



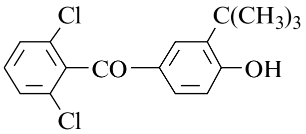
Synthesis

- Preparation by demethylation of 5-tert-butyl-2′,4′-di-chloro-2-methoxybenzophenone (SM) with aluminium chloride in methylene chloride at 10°. SM was obtained by Friedel–Crafts acylation of p-tert-butylanisole with 2,4-dichlorobenzoyl chloride in methylene chloride in the presence of aluminium chloride at 10° [1164].
- Also refer to: [1165].

m.p. 42–43° [1164]; Spectra (NA).

(2,6-Dichlorophenyl)[3-(1,1-dimethylethyl)-4-hydroxyphenyl]methanone

[124979-18-6] $C_{17}H_{16}Cl_2O_2$ mol.wt. 323.22



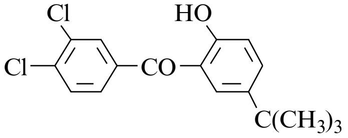
Synthesis

- Preparation by Friedel–Crafts acylation of o-tert-butyl-phenol with 2,6-dichlorobenzoyl chloride in ethylene dichloride in the presence of titanium tetrachloride, first at 0°, then at r.t. [816].

m.p. 229–230° [816]; Spectra (NA).

(3,4-Dichlorophenyl)[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone

$C_{17}H_{16}Cl_2O_2$ mol.wt. 323.22



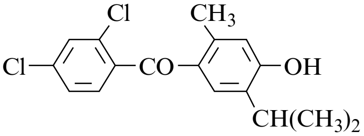
Synthesis

- Obtained by photo-Fries rearrangement of p-tert-butylphenyl 3,4-dichlorobenzoate in benzene (37%) [154].

m.p. 110–111° [154]; Spectra (NA).

(2,4-Dichlorophenyl)[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]methanone

[72236-97-6] $C_{17}H_{16}Cl_2O_2$ mol.wt. 323.22



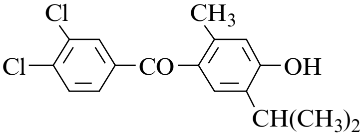
Synthesis

- Preparation by Fries rearrangement of the intermediate 5-methyl-2-isopropylphenyl 2,4-dichlorobenzoate formed in situ by reaction of 2,4-dichlorobenzoyl chloride with thymol in the presence of aluminium chloride in nitrobenzene at r.t. for 24 h (70%) [1166].

m.p. 132° [1166]; ^{1}H NMR [1166].

(3,4-Dichlorophenyl)[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]methanone

[72236-99-8] $C_{17}H_{16}Cl_2O_2$ mol.wt. 323.22



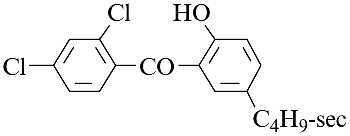
Synthesis

- Preparation by demethylation of 4-(3,4-dichloro-benzoyl)-5-methyl-2-isopropylanisole with boiling pyridinium chloride for 15 min (80%) [1166].

m.p. 133° [1166]; ^{1}H NMR [1166].

(2,4-Dichlorophenyl)[2-hydroxy-5-(1-methylpropyl)phenyl]methanone

[59746-94-0] $C_{17}H_{16}Cl_2O_2$ mol.wt. 323.22



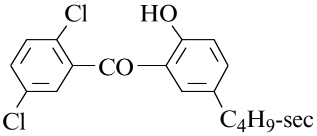
Synthesis

- Preparation by reaction of 2,4-dichlorobenzotrichloride with 4-sec-butylphenol in hydrofluoric acid in the presence of water at −10°, then between 0° and −10° for 2 h, at r.t. for 7 h and at 80° for 30 min into an autoclave (68%) [213].
- Also refer to: [1165].

yellow oil [213]; b.p.$_{0.05}$ 170° [213]; Spectra (NA).

(2,5-Dichlorophenyl)[2-hydroxy-5-(1-methylpropyl)phenyl]methanone

$C_{17}H_{16}Cl_2O_2$ mol.wt. 323.22

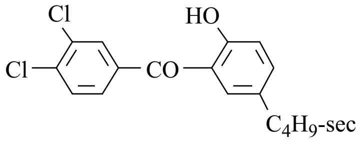


Synthesis

- Preparation by reaction of 2,5-dichlorobenzotrichloride with 4-sec-butylphenol in hydrofluoric acid in the presence of water at −10°, then between 0° and −10° for 2 h, at r.t. for 7 h and at 80° for 30 min into an autoclave [213].

m.p. and Spectra (NA).

(3,4-Dichlorophenyl)[2-hydroxy-5-(1-methylpropyl)phenyl]methanone



$C_{17}H_{16}Cl_2O_2$ mol.wt. 323.22

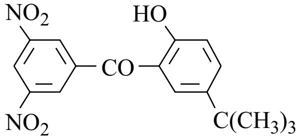
Synthesis

- Preparation by reaction of 3,4-dichlorobenzo-trichloride with 4-sec-butylphenol in hydrofluoric acid in the presence of water at −10°, then between 0° and −10° for 2 h, at r.t. for 7 h and at 80° for 30 min into an autoclave [213].

m.p. and Spectra (NA).

(3,5-Dinitrophenyl)[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone

[93332-04-8]



$C_{17}H_{16}N_2O_6$ mol.wt. 344.32

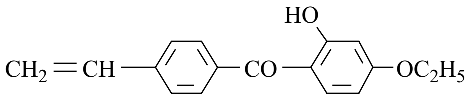
Synthesis

- Obtained (poor yield) by photo-Fries rearrangement of p-tert-butylphenyl 3,5-dinitrobenzoate in benzene (7%) [154].

m.p. 133–134° [154]; Spectra (NA).

(4-Ethenylphenyl)(4-ethoxy-2-hydroxyphenyl)methanone

[80167-00-6]



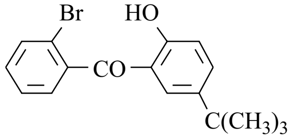
$C_{17}H_{16}O_3$ mol.wt. 268.31

Synthesis

- Preparation by reaction of aqueous potassium hydroxide with 4′-(2-bromo-ethyl)-4-ethoxy-2-hydroxybenzophenone in the presence of hydro quinone in refluxing methanol for 1.5 h with nitrogen bubbling (51%) [1147].

m.p. 76–79° [1147]; ^{1}H NMR [1147], IR [1147], UV [1147], MS [1147].

(2-Bromophenyl)[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone



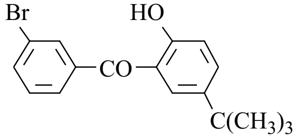
$C_{17}H_{17}BrO_2$ mol.wt. 333.22

Synthesis

- Preparation by demethylation of 2′-bromo-5-tert-butyl-2-methoxybenzophenone with aluminium chloride in benzene at 50–60° for 5 h (80%) [9].

b.p.$_{12}$ 208–212° [9]; Spectra (NA).

(3-Bromophenyl)[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone



$C_{17}H_{17}BrO_2$ mol.wt. 333.22

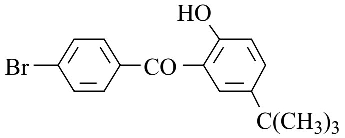
Synthesis

- Preparation by demethylation of 5-tert-butyl-3′-bromo-2-methoxybenzophenone with aluminium chloride in benzene at 50–60° for 5 h (75%) [9].

b.p.$_{12}$ 225–227° [9]; Spectra (NA).

(4-Bromophenyl)[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone

[75060-50-3]



$C_{17}H_{17}BrO_2$ mol.wt. 333.22

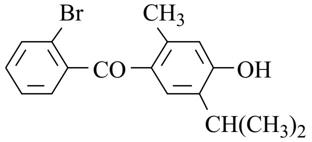
Synthesis

- Preparation by demethylation of 2-(4-bromobenzoyl)-4-tert-butylanisole with a mixture of 57% hydriodic acid and 47% hydrobromic acid in refluxing acetic acid (87%) [817].

m.p. 83–85° [817]; Spectra (NA).

(2-Bromophenyl)[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]methanone

[72237-01-5]



$C_{17}H_{17}BrO_2$ mol.wt. 333.22

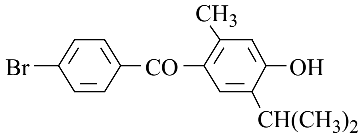
Synthesis

- Preparation by demethylation of 4-(2-bromobenzoyl)-5-methyl-2-isopropylanisole with boiling pyridinium chloride for 15 min (85%) [1166].

m.p. 126° [1166]; ^{1}H NMR [1166].

(4-Bromophenyl)[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]methanone

[72237-03-7]



$C_{17}H_{17}BrO_2$ mol.wt. 333.22

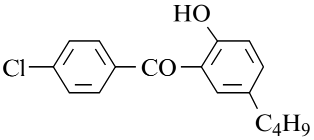
Synthesis

- Preparation by demethylation of 4-(4-bromobenzoyl)-5-methyl-2-isopropylanisole with boiling pyridinium chloride for 15 min (85%) [1166].

m.p. 150° [1166]; ^{1}H NMR [1166].

(5-Butyl-2-hydroxyphenyl)(4-chlorophenyl)methanone

[108294-80-0] $C_{17}H_{17}ClO_2$ mol.wt. 288.77



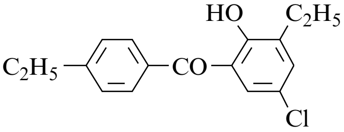
Synthesis

– Preparation by reaction of p-chlorobenzoyl chloride with p-butylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (54%) [92].

b.p.$_{0.75}$ 174° [92]; Spectra (NA).

(5-Chloro-3-ethyl-2-hydroxyphenyl)(4-ethylphenyl)methanone

[93575-37-2] $C_{17}H_{17}ClO_2$ mol.wt. 288.77



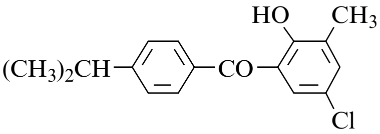
Synthesis

– Preparation by Fries rearrangement of 4-chloro-2-ethylphenyl 4-ethylbenzoate with aluminium chloride at 160° for 15 min [1107].

oil [1107]; b.p. (NA); $n_D^{21} = 1.6060$ [1107]; Spectra (NA).

(5-Chloro-2-hydroxy-3-methylphenyl)[4-(1-methylethyl)phenyl]methanone

[86914-75-2] $C_{17}H_{17}ClO_2$ mol.wt. 288.77



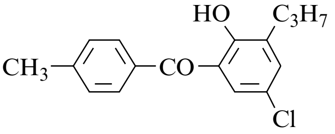
Synthesis

– Preparation by Fries rearrangement of 4-chloro-2-methylphenyl 4-isopropylbenzoate with aluminium chloride at 180° for 10 min [1074,1149].

yellow oil [1074,1149]; b.p. (NA); $n_D^{22} = 1.619$ [1074,1149];
Spectra (NA).

(5-Chloro-2-hydroxy-3-propylphenyl)(4-methylphenyl)methanone

[92739-94-1] $C_{17}H_{17}ClO_2$ mol.wt. 288.77



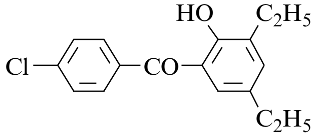
Synthesis

– Preparation by Fries rearrangement of 4-chloro-2-propyl-phenyl p-toluate with aluminium chloride at 160° for 15 min [1107].

m.p. 63–64° [1107]; Spectra (NA).

(4-Chlorophenyl)(3,5-diethyl-2-hydroxyphenyl)methanone

[108294-82-2] $C_{17}H_{17}ClO_2$ mol.wt. 288.77



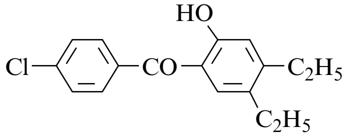
Synthesis

– Preparation by reaction of p-chlorobenzoyl chloride with 2,4-diethylphenol in the presence of aluminium chloride in tetrachloroethane at 105° for 22 h (53%) [92].

b.p.$_{1.4}$ 188° [92]; Spectra (NA).

(4-Chlorophenyl)(4,5-diethyl-2-hydroxyphenyl)methanone

[61750-26-3] $C_{17}H_{17}ClO_2$ mol.wt. 288.77



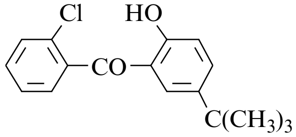
Synthesis

– Preparation by Fries rearrangement of 3,4-diethyl-phenyl p-chlorobenzoate with aluminium chloride in tetrachloroethane at 125° for 6 h [710].

b.p.$_{1.4}$ 188° [710]; Spectra (NA).

(2-Chlorophenyl)[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone

$C_{17}H_{17}ClO_2$ mol.wt. 288.77



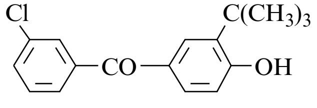
Synthesis

– Preparation by demethylation of 5-tert-butyl-2′-chloro-2-methoxybenzophenone with aluminium chloride in benzene at 50–60° for 5 h (70%) [9].

b.p.$_{12}$ 200–205° [9]; Spectra (NA).

(3-Chlorophenyl)[3-(1,1-dimethylethyl)-4-hydroxyphenyl]methanone

[124979-06-2] $C_{17}H_{17}ClO_2$ mol.wt. 288.77



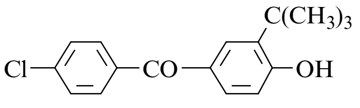
Synthesis

– Preparation by Friedel–Crafts acylation of o-tert-butyl-phenol with m-chlorobenzoyl chloride in ethylene dichloride in the presence of titanium tetrachloride, first at 0°, then at r.t. [816].

m.p. 173–174° [816]; Spectra (NA).

(4-Chlorophenyl)[3-(1,1-dimethylethyl)-4-hydroxyphenyl]methanone

[124979-05-1] $C_{17}H_{17}ClO_2$ mol.wt. 288.77



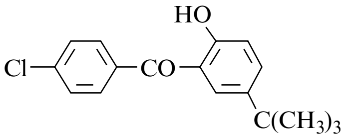
Synthesis

- Preparation by Friedel–Crafts acylation of o-tert-butyl-phenol with p-chlorobenzoyl chloride in ethylene dichloride in the presence of titanium tetrachloride, first at 0°, then at r.t. [816].

m.p. 205–206° [816]; Spectra (NA).

(4-Chlorophenyl)[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone

[72083-19-3] $C_{17}H_{17}ClO_2$ mol.wt. 288.77



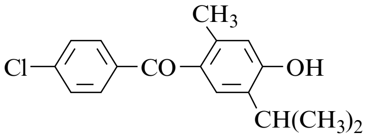
Syntheses

- Preparation by demethylation of 5-tert-butyl-4′-chloro-2-methoxybenzophenone with aluminium chloride in benzene at 50–60° for 5 h (68%) [9] or at 70° for 12 h [569].
- Obtained by photo-Fries rearrangement of p-tert-butylphenyl p-chlorobenzoate in benzene or in ethanol (55% and 48% yields, respectively) [154].
- Also refer to: [472,568].

m.p. 94–95° [9], 92–94° [154], 64–65° [569]. One of the reported melting points is obviously wrong. b.p.$_{12}$ 218–220° [9]; Spectra (NA).

(4-Chlorophenyl)[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]methanone

$C_{17}H_{17}ClO_2$ mol.wt. 288.77



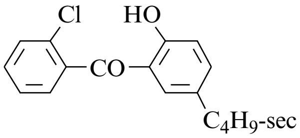
Synthesis

- Preparation by demethylation of 4′-chloro-4-methoxy-2-methyl-5-isopropylbenzophenone with boiling pyridinium chloride for 15 min (85%) [1166].

m.p. 156° [1166]; Spectra (NA).

(2-Chlorophenyl)[2-hydroxy-5-(1-methylpropyl)phenyl]methanone

[59746-95-1] $C_{17}H_{17}ClO_2$ mol.wt. 288.77



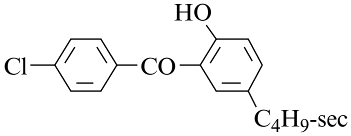
Synthesis

- Preparation by reaction of 2-chlorobenzotrichloride with 4-sec-butylphenol in hydrofluoric acid in the presence of water at −10°, then between 0° and −10° for 2 h, at r.t. for 7 h and at 80° for 30 min into an autoclave (72%) [213].
- Also refer to: [1165].

yellow oil [213]; b.p.$_{0.2}$ 152° [213]; Spectra (NA).

(4-Chlorophenyl)[2-hydroxy-5-(1-methylpropyl)phenyl]methanone

[59746-96-2]



$C_{17}H_{17}ClO_2$ mol.wt. 288.77

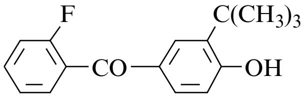
Synthesis

- Preparation by reaction of 4-chlorobenzotrichloride with 4-sec-butylphenol in hydrofluoric acid in the presence of water at −10°, then between 0° and −10° for 2 h, at r.t. for 7 h and at 80° for 30 min into an autoclave (51%) [213].
- Also refer to: [1165].

yellow oil [213]; b.p.$_1$ 175–177° [213]; Spectra (NA).

[3-(1,1-Dimethylethyl)-4-hydroxyphenyl](2-fluorophenyl)methanone

[124979-11-9]



$C_{17}H_{17}FO_2$ mol.wt. 272.32

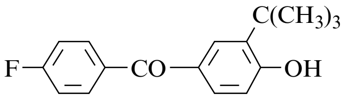
Synthesis

- Preparation by Friedel–Crafts acylation of o-tert-butylphenol with o-fluorobenzoyl chloride in ethylene dichloride in the presence of titanium tetrachloride, first at 0°, then at r.t. [816].

m.p. 152–153° [816]; Spectra (NA).

[3-(1,1-Dimethylethyl)-4-hydroxyphenyl](4-fluorophenyl)methanone

[124979-09-5]



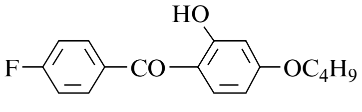
$C_{17}H_{17}FO_2$ mol.wt. 272.32

Synthesis

- Preparation by Friedel–Crafts acylation of o-tert-butylphenol with p-fluorobenzoyl chloride in ethylene dichloride in the presence of titanium tetrachloride, first at 0°, then at r.t. [816].

m.p. 203–204° [816]; Spectra (NA).

(4-Butoxy-2-hydroxyphenyl)(4-fluorophenyl)methanone



$C_{17}H_{17}FO_3$ mol.wt. 288.32

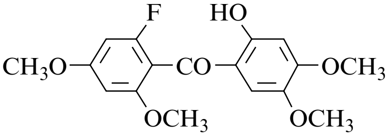
Synthesis

- Preparation by partial alkylation of 4′-fluoro-2,4-di-hydroxybenzophenone with a butyl halide in the presence of an alkali [1109].

m.p. and Spectra (NA).

(2-Fluoro-4,6-dimethoxyphenyl)(2-hydroxy-4,5-dimethoxyphenyl)methanone

[129103-95-3] $C_{17}H_{17}FO_6$ mol.wt. 336.32



Synthesis

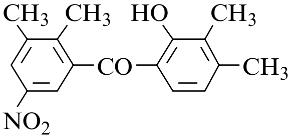
- Preparation from 2-iodo-4,5-dimethoxyphenyl 2-fluoro-4,6-dimethoxybenzoate by rearrangement on treatment with sec-butyllithium in a mixture of ethyl ether, hexane and tetrahydrofuran at −100° followed by heating to −70° for 2 h, then treatment with saturated aqueous ammonium chloride (89%) [58].

m.p. 133–134° [58];

^{1}H NMR [58], IR [58], MS [58].

(2,3-Dimethyl-5-nitrophenyl)(2-hydroxy-3,4-dimethylphenyl)methanone

[110969-70-5] $C_{17}H_{17}NO_4$ mol.wt. 299.33



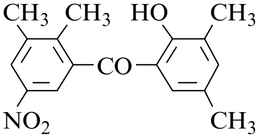
Synthesis

- Preparation by Fries rearrangement of 2,3-dimethylphenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (56%) [1003].

m.p. 165° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2,3-Dimethyl-5-nitrophenyl)(2-hydroxy-3,5-dimethylphenyl)methanone

[110969-73-8] $C_{17}H_{17}NO_4$ mol.wt. 299.33



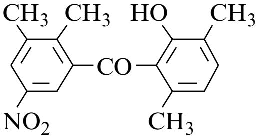
Synthesis

- Preparation by Fries rearrangement of 2,4-dimethylphenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (56%) [1003].

m.p. 133° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2,3-Dimethyl-5-nitrophenyl)(2-hydroxy-3,6-dimethylphenyl)methanone

[110969-75-0] $C_{17}H_{17}NO_4$ mol.wt. 299.33



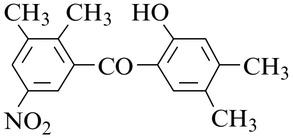
Synthesis

- Obtained by Fries rearrangement of 2,5-dimethylphenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (20%) [1003].

m.p. 120° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2,3-Dimethyl-5-nitrophenyl)(2-hydroxy-4,5-dimethylphenyl)methanone

[110969-80-7] $C_{17}H_{17}NO_4$ mol.wt. 299.33



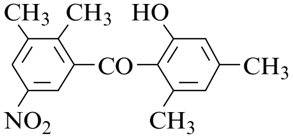
Synthesis

– Preparation by Fries rearrangement of 3,4-dimethylphenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (60%) [1003].

m.p. 169° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2,3-Dimethyl-5-nitrophenyl)(2-hydroxy-4,6-dimethylphenyl)methanone

[110993-12-9] $C_{17}H_{17}NO_4$ mol.wt. 299.33



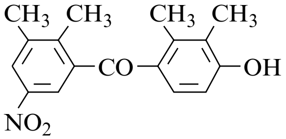
Synthesis

– Preparation by Fries rearrangement of 3,5-dimethylphenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (70%) [1003].

m.p. 121° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2,3-Dimethyl-5-nitrophenyl)(4-hydroxy-2,3-dimethylphenyl)methanone

[110969-71-6] $C_{17}H_{17}NO_4$ mol.wt. 299.33



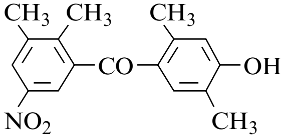
Synthesis

– Obtained by Fries rearrangement of 2,3-dimethylphenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (12%) [1003].

m.p. 212° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2,3-Dimethyl-5-nitrophenyl)(4-hydroxy-2,5-dimethylphenyl)methanone

[110969-76-1] $C_{17}H_{17}NO_4$ mol.wt. 299.33



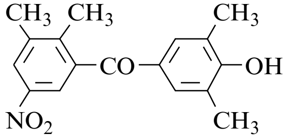
Synthesis

– Preparation by Fries rearrangement of 2,5-dimethylphenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (30%) [1003].

m.p. 232° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

(2,3-Dimethyl-5-nitrophenyl)(4-hydroxy-3,5-dimethylphenyl)methanone

[110969-78-3] $C_{17}H_{17}NO_4$ mol.wt. 299.33



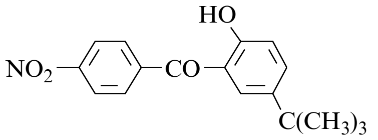
Synthesis

- Preparation by Fries rearrangement of 2,6-dimethylphenyl 2,3-dimethyl-5-nitrobenzoate with aluminium chloride at 160° for 2 h (55%) [1003].

m.p. 214° [1003]; ^{1}H NMR [1003], IR [1003], UV [1003].

[5-(1,1-Dimethylethyl)-2-hydroxyphenyl](4-nitrophenyl)methanone

$C_{17}H_{17}NO_4$ mol.wt. 299.33



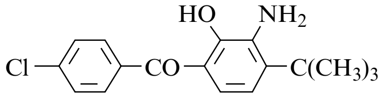
Synthesis

- Obtained (poor yield) by photo-Fries rearrangement of p-tert-butylphenyl p-nitrobenzoate in benzene (10%) [154].

m.p. 102–103° [154]; Spectra (NA).

[3-Amino-4-(1,1-dimethylethyl)-2-hydroxyphenyl](4-chlorophenyl)methanone

$C_{17}H_{18}ClNO_2$ mol.wt. 303.79



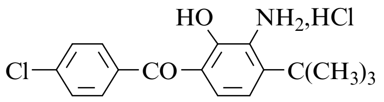
Synthesis

- Preparation by reduction of 4-tert-butyl-4′-chloro-2-hydroxy-3-nitrobenzophenone with titanium trichloride in a benzene/tetrahydrofuran mixture for 20 h at r.t. [472].

m.p. (NA); red oil [472]; ^{1}H NMR [472], IR [472], MS [472]; TLC [472].

[3-Amino-4-(1,1-dimethylethyl)-2-hydroxyphenyl](4-chlorophenyl)methanone *(Hydrochloride)*

$C_{17}H_{18}ClNO_2,HCl$ mol.wt. 340.25



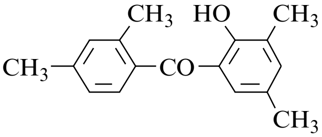
Synthesis

- Obtained by reaction of concentrated hydrochloric acid with 3-amino-4-tert-butyl-4′-chloro-2-hydroxy-benzophenone (52%) [472].

m.p. 153–158° [472]; Spectra: see the corresponding amino base.

(2,4-Dimethylphenyl)(2-hydroxy-3,5-dimethylphenyl)methanone

[86914-80-9] $C_{17}H_{18}O_2$ mol.wt. 254.33



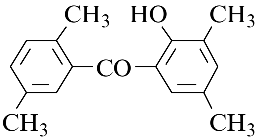
Synthesis

– Preparation by Fries rearrangement of 2,4-dimethylphenyl 2,4-dimethylbenzoate with aluminium chloride at 180° for 10 min [1074].

oil [1074]; b.p. and Spectra (NA).

(2,5-Dimethylphenyl)(2-hydroxy-3,5-dimethylphenyl)methanone

[86914-78-5] $C_{17}H_{18}O_2$ mol.wt. 254.33



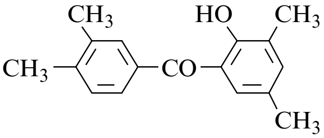
Synthesis

– Preparation by Fries rearrangement of 2,4-dimethylphenyl 2,5-dimethylbenzoate with aluminium chloride at 180° for 10 min [1074].

oil [1074]; b.p. and Spectra (NA).

(3,4-Dimethylphenyl)(2-hydroxy-3,5-dimethylphenyl)methanone

[86914-88-7] $C_{17}H_{18}O_2$ mol.wt. 254.33



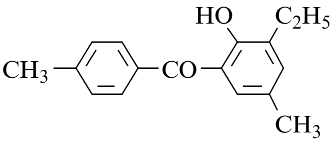
Synthesis

– Preparation by Fries rearrangement of 2,4-dimethyl-phenyl 3,4-dimethylbenzoate with aluminium chloride at 180° for 10 min [1074].

m.p. 68° [1074]; Spectra (NA).

(3-Ethyl-2-hydroxy-5-methylphenyl)(4-methylphenyl)methanone

[92739-95-2] $C_{17}H_{18}O_2$ mol.wt. 254.33



Synthesis

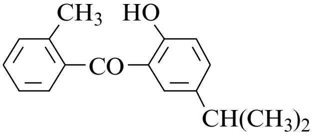
– Preparation by Fries rearrangement of 2-ethyl-4-methyl-phenyl p-toluate with aluminium chloride at 160° for 15 min [1107].

– Also refer to: [1076].

m.p. 38–39° [1107]; Spectra (NA).

[2-Hydroxy-5-(1-methylethyl)phenyl](2-methylphenyl)methanone

[170799-16-3] $C_{17}H_{18}O_2$ mol.wt. 254.33



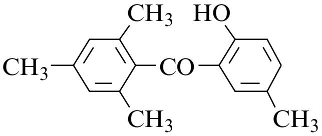
Synthesis

– Preparation by Fries rearrangement of p-isopropylphenyl o-toluate with aluminium chloride at 100–150° for 0.5–3 h (98%) [29].

oil [29]; b.p. and Spectra (NA).

(2-Hydroxy-5-methylphenyl)(2,4,6-trimethylphenyl)methanone

$C_{17}H_{18}O_2$ mol.wt. 254.33



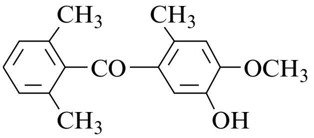
Synthesis

– Preparation by Fries rearrangement of p-cresyl 2,4,6-tri-methylbenzoate with aluminium chloride at 150° for 2 h (63%) [1151].

m.p. 86° [1151]; Spectra (NA).

(2,6-Dimethylphenyl)(5-hydroxy-4-methoxy-2-methylphenyl)methanone

$C_{17}H_{18}O_3$ mol.wt. 270.33



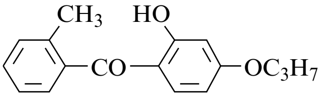
Synthesis

– Obtained by partial demethylation of 4,5-dimethoxy-2,2′,6′-trimethylbenzophenone [1072].

m.p. and Spectra (NA).

(2-Hydroxy-4-propoxyphenyl)(2-methylphenyl)methanone

[172479-21-9] $C_{17}H_{18}O_3$ mol.wt. 270.33



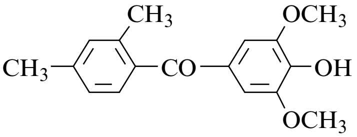
Synthesis

– Prepared by standard techniques [830].

m.p. (NA); UV [830].

(2,4-Dimethylphenyl)(4-hydroxy-3,5-dimethoxyphenyl)methanone

$C_{17}H_{18}O_4$ mol.wt. 286.33



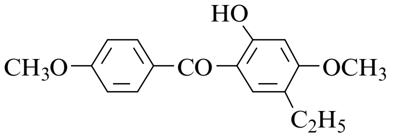
Synthesis

– Preparation by Friedel–Crafts acylation of m-xylene with 3,4,5-trimethoxybenzoyl chloride [429].

m.p. and Spectra (NA).

(5-Ethyl-2-hydroxy-4-methoxyphenyl)(4-methoxyphenyl)methanone

[66666-17-9] $C_{17}H_{18}O_4$ mol.wt. 286.33



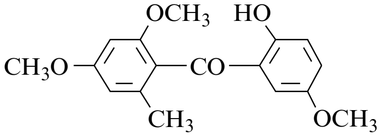
Synthesis

– Preparation by saponification of 2-(p-anisoyloxy)-4,4′-dimethoxy-5-ethylbenzophenone (SM) with potassium hydroxide in refluxing ethanol for 1 h (81%). SM was obtained by oxidation of 5-ethyl-6-methoxy-2,3-bis(p-methoxyphenyl)benzofuran with chromium trioxide in refluxing acetic acid for 45 min [1116].

oil [1116]; b.p. and Spectra (NA).

(2,4-Dimethoxy-6-methylphenyl)(2-hydroxy-5-methoxyphenyl)methanone

[78044-92-5] $C_{17}H_{18}O_5$ mol.wt. 302.33



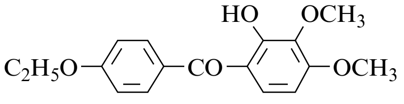
Synthesis

– Obtained by photo-Fries rearrangement of p-methoxy-phenyl 2,4-dimethoxy-6-methylbenzoate in benzene for 4 h under nitrogen (37%) [1167].

m.p. 120–123° [1167]; ^{1}H NMR [1167], IR [1167], UV [1167], MS [1167].

(4-Ethoxyphenyl)(2-hydroxy-3,4-dimethoxyphenyl)methanone

[69471-31-4] $C_{17}H_{18}O_5$ mol.wt. 302.33



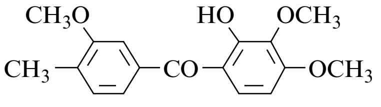
Synthesis

– Preparation by selective demethylation of 4′-ethoxy-2,3,4-trimethoxybenzophenone with aluminium chloride in nitrobenzene at 100° for 1 h (70%) [1168].

m.p. 113–114° [1168]; ^{1}H NMR [1168], IR [1168].

(2-Hydroxy-3,4-dimethoxyphenyl)(3-methoxy-4-methylphenyl)methanone

$C_{17}H_{18}O_5$ mol.wt. 302.33



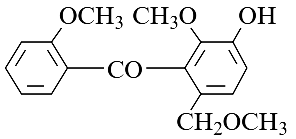
Synthesis

– Obtained by Friedel–Crafts acylation of pyrogallol trimethyl ether with 3-methoxy-4-methylbenzoyl chloride in the presence of aluminium chloride in boiling carbon disulfide for 4 h (22%) [1169,1170].

m.p. 109° [1169,1170]; Spectra (NA).

[3-Hydroxy-2-methoxy-6-(methoxymethyl)phenyl](2-methoxyphenyl) methanone

[133386-99-9] $C_{17}H_{18}O_5$ mol.wt. 302.33



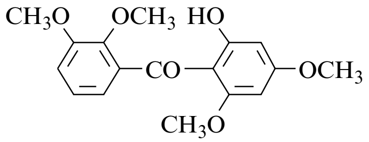
Synthesis

– Preparation from 4-hydroxy-3-methoxybenzyl methyl ether (SM) in two steps: first, direct lithiation of SM with n-butyl lithium in tetrahydrofuran at r.t., followed by quenching with o-anisaldehyde. Then, oxidation of the 8-(2-methoxyphenyl)-2,7-dimethoxybicyclo[4.2.0]octa-1,3,5-triene-3,8-diol so formed (good yield) [1171].

m.p. and Spectra (NA).

(2,3-Dimethoxyphenyl)(2-hydroxy-4,6-dimethoxyphenyl)methanone

[151417-67-3] $C_{17}H_{18}O_6$ mol.wt. 318.33



Synthesis

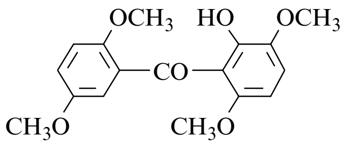
– Obtained, in mixture with 2′-hydroxy-2,3′,4,6-tetra-methoxybenzophenone, by reaction of 2,3-dimethoxy-benzoyl chloride with 1,3,5-trimethoxybenzene in ethyl ether in the presence of aluminium chloride for 15 h at r.t. (total yield: 92%) [1011].

– Also refer to: [1027].

m.p. (NA); ^{1}H NMR [1011].

(2,5-Dimethoxyphenyl)(2-hydroxy-3,6-dimethoxyphenyl)methanone

[109092-84-4] $C_{17}H_{18}O_6$ mol.wt. 318.33



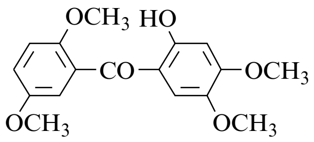
Synthesis

– Obtained by reaction of 2,3,6-trimethoxybenzoyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride in ethyl ether at r.t. for 2 days (17%) [1172] or for 1 h at 45–50°, then 20 h at r.t. (69%) [1160].

m.p. 115–116° [1172], 115° [1160]; Spectra (NA).

(2,5-Dimethoxyphenyl)(2-hydroxy-4,5-dimethoxyphenyl)methanone

[88133-95-3] $C_{17}H_{18}O_6$ mol.wt. 318.33



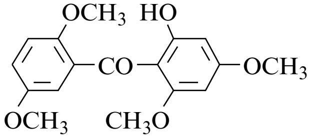
Synthesis

– Obtained by reaction of 2,5-dimethoxybenzoyl chloride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride in ethyl ether for 30 h [1173], or for 1 h at 45–50°, then 20 h at r.t. (65–70%) [1160].

m.p. 97–98° [1173], 82°5–83° [1160]; ^{1}H NMR [1173].

(2,5-Dimethoxyphenyl)(2-hydroxy-4,6-dimethoxyphenyl)methanone

[42833-59-0] $C_{17}H_{18}O_6$ mol.wt. 318.33



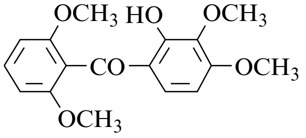
Synthesis

- Preparation by reaction of 2,5-dimethoxybenzoyl chloride with 1,3,5-trimethoxybenzene in the presence of aluminium chloride in ethyl ether for 28 h (minor product) [416], (52%, estimated, not isolated) [1160].

m.p. (NA); ^{1}H NMR [416], MS [416].

(2,6-Dimethoxyphenyl)(2-hydroxy-3,4-dimethoxyphenyl)methanone

[42833-55-6] $C_{17}H_{18}O_6$ mol.wt. 318.33



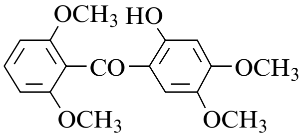
Synthesis

- Obtained by reaction of 2,6-dimethoxybenzoyl chloride with 1,2,3-trimethoxybenzene in the presence of aluminium chloride in ethyl ether for 20 h [416].

m.p. (NA); ^{1}H NMR [416], MS [416].

(2,6-Dimethoxyphenyl)(2-hydroxy-4,5-dimethoxyphenyl)methanone

[42833-53-4] $C_{17}H_{18}O_6$ mol.wt. 318.33



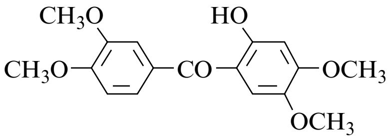
Synthesis

- Obtained by reaction of 2,6-dimethoxybenzoyl chloride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride in ethyl ether at r.t. for 40 h [416].

m.p. (NA); ^{1}H NMR [416], UV [416], MS [416].

(3,4-Dimethoxyphenyl)(2-hydroxy-4,5-dimethoxyphenyl)methanone

$C_{17}H_{18}O_6$ mol.wt. 318.33



Synthesis

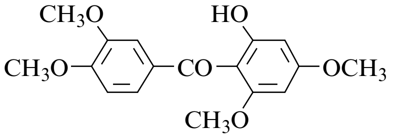
- Preparation by reaction of veratroyl chloride (3,4-dimethoxybenzoyl chloride) with hydroxy-hydroquinone trimethyl ether (1,2,4-trimethoxybenzene) in the presence of aluminium chloride,

- in ethyl ether at r.t. for 48 h [1136], (47%) [1174];
- in refluxing carbon disulfide for 8 h, then at r.t. for 12 h (37%) [1175].

m.p. 148–149° [1175], 145–146° [1174]; UV [1175].

(3,4-Dimethoxyphenyl)(2-hydroxy-4,6-dimethoxyphenyl)methanone

[62495-41-4]



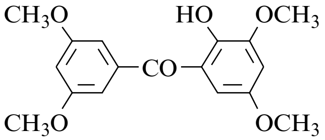
$C_{17}H_{18}O_6$ mol.wt. 318.33

Syntheses

- Preparation by reaction of 3,4-dimethoxybenzoyl chloride with 1,3,5-trimethoxybenzene in the presence of aluminium chloride in ethyl ether for 4 h (50%) [653].
- Preparation by reaction of 3,4-dimethoxybenzoyl chloride with 3,5-dimethoxyphenol in the presence of aluminium chloride in ethyl ether at r.t. for 48 h (49%) [1136].

m.p. 135–136° [653], 134° [1136]; ^{1}H NMR [653,1136], IR [1136], UV [1136].

(3,5-Dimethoxyphenyl)(2-hydroxy-3,5-dimethoxyphenyl)methanone



$C_{17}H_{18}O_6$ mol.wt. 318.33

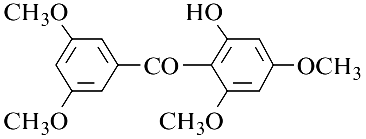
Synthesis

- Preparation by reaction of 3,5-dimethoxybenzoyl chloride with pyrogallol trimethyl ether in the presence of aluminium chloride in boiling carbon disulfide for 2 h (40%) [1176].

m.p. 123–124° [1176]; Spectra (NA).

(3,5-Dimethoxyphenyl)(2-hydroxy-4,6-dimethoxyphenyl)methanone

[53250-54-7]



$C_{17}H_{18}O_6$ mol.wt. 318.33

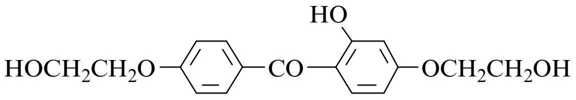
Synthesis

- Obtained by reaction of 3,5-dimethoxybenzoyl chloride with phloroglucinol trimethyl ether in ethyl ether in the presence of aluminium chloride at 25° for 48 h (16%) [167].

m.p. 118° [167]; Spectra (NA).

[2-Hydroxy-4-(2-hydroxyethoxy)phenyl][4-(2-hydroxyethoxy)phenyl] methanone

$C_{17}H_{18}O_6$ mol.wt. 318.33



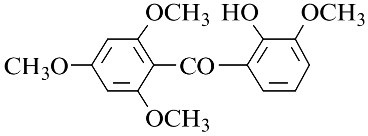
Synthesis

- Refer to: [93,116].

m.p. 128° [93]; UV [93]; pK_a [93]; TLC [116].

(2-Hydroxy-3-methoxyphenyl)(2,4,6-trimethoxyphenyl)methanone

[6343-00-6]



$C_{17}H_{18}O_6$ mol.wt. 318.33

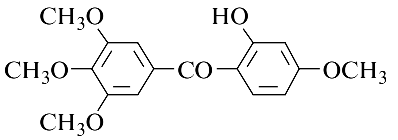
Synthesis

- Obtained, in mixture with 2-hydroxy-2′,3′,4,6-tetra-methoxybenzophenone, by reaction of 2,3-dimethoxy- benzoyl chloride with 1,3,5-trimethoxybenzene in ethyl ether in the presence of aluminium chloride for 15 h at r.t. (total yield: 92%) [1011].
- Also refer to: [1027].

m.p. (NA); ^{1}H NMR [1011].

(2-Hydroxy-4-methoxyphenyl)(3,4,5-trimethoxyphenyl)methanone

[62495-39-0]



$C_{17}H_{18}O_6$ mol.wt. 318.33

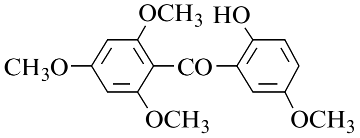
Synthesis

- Preparation by reaction of dimethyl sulfate with 2,4′-dihydroxy-3′,4,5′-trimethoxybenzophenone in the presence of potassium carbonate in refluxing acetone for 30 min (57%) [1136].

m.p. 106–107° [1136]; ^{1}H NMR [1136], IR [1136], UV [1136].

(2-Hydroxy-5-methoxyphenyl)(2,4,6-trimethoxyphenyl)methanone

[42832-64-4]



$C_{17}H_{18}O_6$ mol.wt. 318.33

Synthesis

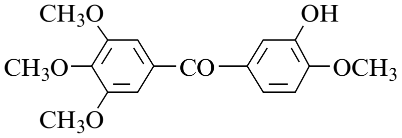
- Obtained by reaction of 2,5-dimethoxybenzoyl chloride with 1,3,5-trimethoxybenzene in the presence of aluminium chloride in ethyl ether for 28 h (major product) [416], (30%, estimated, not isolated) [1160].
- Also refer to: [1072].

m.p. (NA); ^{1}H NMR [416], MS [416].

(3-Hydroxy-4-methoxyphenyl)(3,4,5-trimethoxyphenyl)methanone

(Phenstatin)

[203448-32-2]



$C_{17}H_{18}O_6$ mol.wt. 318.33

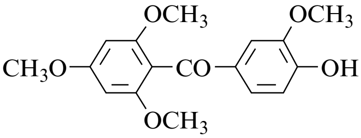
Synthesis

- Obtained by deprotection of 3-[(tert-butyldimethyl-silyl)oxy]-3′,4,4′,5′-tetramethoxybenzophenone (SM) in tetrahydrofuran with 1 M tetrabutyl-ammonium fluoride for 15 min under argon (83%), but only 30% overall yield [1177].

- SM was prepared in two steps: first, formation of N-[3-[(tert-butyldimethylsilyl)oxy]-4-methoxybenzoyl]morpholine (SM1) by action of 3-[(tert-butyldimethylsilyl)oxy]-4-methoxy-benzoyl chloride with morpholine in toluene for 4 h at r.t. under argon. Then, the amide SM1 was allowed to react with the lithium derivative prepared from 3,4,5-trimethoxybromobenzene [1178] and tert-butyllithium in tetrahydrofuran at −78° to give SM [1177].

m.p. 149–150° [1177]; ^{1}H NMR [1177], ^{13}C NMR [1177], IR [1177], MS [1177]; crystal data [1177]; TLC [1177].

(4-Hydroxy-3-methoxyphenyl)(2,4,6-trimethoxyphenyl)methanone



$C_{17}H_{18}O_6$ mol.wt. 318.33

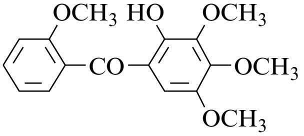
Synthesis

- Obtained by reaction of veratronitrile with phloroglucinol trimethyl ether (Hoesch reaction) (15%) [439].

m.p. 242° [439]; Spectra (NA).

(2-Hydroxy-3,4,5-trimethoxyphenyl)(2-methoxyphenyl)methanone

[42833-60-3]



$C_{17}H_{18}O_6$ mol.wt. 318.33

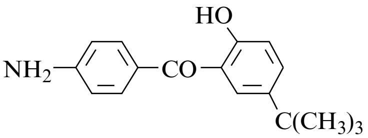
Synthesis

- Obtained by Friedel–Crafts acylation of 1,2,3,4-tetra-methoxybenzene with o-anisoyl chloride in the presence of aluminium chloride in ethyl ether for 44 h [416].

m.p. (NA); ^{1}H NMR [416], UV [416].

(4-Aminophenyl)[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone

[98031-50-6]



$C_{17}H_{19}NO_2$ mol.wt. 269.34

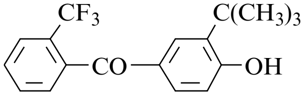
Synthesis

- Obtained by photo-Fries rearrangement of p-tert-butylphenyl p-aminobenzoate in benzene (12%) [154].

m.p. 98–100° [154]; Spectra (NA).

[3-(1,1-Dimethylethyl)-4-hydroxyphenyl][2-(trifluoromethyl)phenyl] methanone

[124979-17-5] $C_{18}H_{17}F_3O_2$ mol.wt. 322.33



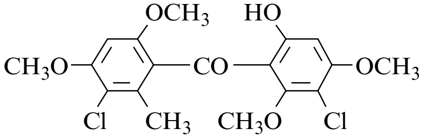
Synthesis

– Preparation by Friedel–Crafts acylation of o-tert-butylphenol with o-(trifluoromethyl) benzoyl chloride in ethylene dichloride in the presence of titanium tetrachloride, first at 0°, then at r.t. [816].

m.p. 160–161° [816]; Spectra (NA).

(3-Chloro-4,6-dimethoxy-2-methylphenyl) (3-chloro-6-hydroxy-2,4-dimethoxyphenyl) methanone

[68048-21-5] $C_{18}H_{18}Cl_2O_6$ mol.wt. 401.24



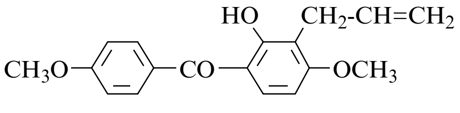
Synthesis

– Preparation by hydrogenolysis of 6-(benzyloxy)-3,3′-dichloro-2,4,4′, 6′-tetramethoxy-2′-methyl-benzophenone (SM) under hydrogen in the presence of 10% Pd/C at 25°. SM was obtained by condensation of 3-chloro-4,6-dimethoxy-2-methyl-benzoic acid with 4-chloro-3, 5-dimethoxyphenol benzyl ether in the presence of trifluoroacetic anhydride in methylene under nitrogen for 80 min (50%) [1179].

m.p. 196–197° [1179]; ^{1}H NMR [1179], IR [1179], MS [1179].

[2-Hydroxy-4-methoxy-3-(2-propenyl)phenyl](4-methoxyphenyl)methanone

[74079-07-5] $C_{18}H_{18}O_4$ mol.wt. 298.34



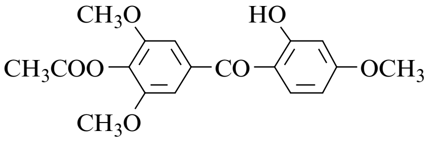
Synthesis

– Obtained by heating 2-(allyloxy)-4,4′-di-methoxybenzophenone (SM) to 240° that which initiates exothermic heating to 290° (60%) (Claisen rearrangement). SM was obtained by reaction of allyl bromide with resbenzophenone (90%) [1140].

m.p. and Spectra (NA).

[4-(Acetyloxy)-3,5-dimethoxyphenyl](2-hydroxy-4-methoxyphenyl)methanone

[62495-40-3] $C_{18}H_{18}O_7$ mol.wt. 346.34



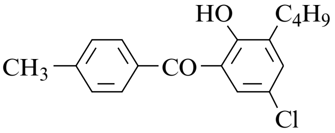
Synthesis

– Preparation by reaction of acetic anhydride with 2,4′-dihydroxy-3′,4,5′-trimethoxybenzophenone in the presence of pyridine at r.t. for 24 h (80%) [1136].

m.p. 124–125° [1136]; ^{1}H NMR [1136], IR [1136], UV [1136].

(3-Butyl-5-chloro-2-hydroxyphenyl)(4-methylphenyl)methanone

[92739-93-0] $C_{18}H_{19}ClO_2$ mol.wt. 302.80



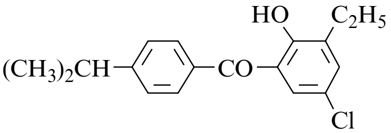
Synthesis

– Preparation by Fries rearrangement of 2-n-butyl-4-chlorophenyl p-toluate with aluminium chloride at 160° for 15 min [1107].

oil [1107]; b.p.$_{0.108}$ 148–150° [1107]; Spectra (NA).

(5-Chloro-3-ethyl-2-hydroxyphenyl)[4-(1-methylethyl)phenyl]methanone

[93575-43-0] $C_{18}H_{19}ClO_2$ mol.wt. 302.80



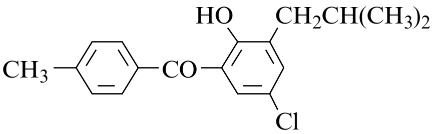
Synthesis

– Preparation by Fries rearrangement of 4-chloro-2-ethylphenyl 4-isopropylbenzoate with aluminium chloride at 160° for 15 min [1107].

m.p. 42–43° [1107]; Spectra (NA).

[5-Chloro-2-hydroxy-3-(2-methylpropyl)phenyl](4-methylphenyl)methanone

[93575-76-9] $C_{18}H_{19}ClO_2$ mol.wt. 302.80



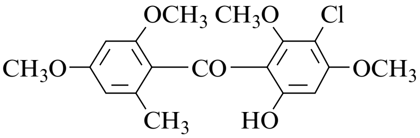
Synthesis

– Preparation by Fries rearrangement of 2-iso-butyl-4-chlorophenyl p-toluate with aluminium chloride at 160° for 15 min [1107].

m.p. 71–72° [1107]; Spectra (NA).

(3-Chloro-6-hydroxy-2,4-dimethoxyphenyl)(2,4-dimethoxy-6-methylphenyl) methanone

[68048-15-7] $C_{18}H_{19}ClO_6$ mol.wt. 366.80



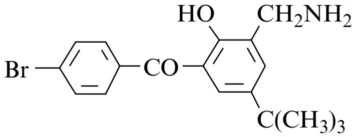
Synthesis

– Preparation by hydrogenolysis of 6-(benzyloxy)-3-chloro-2,2′,4,4′-tetramethoxy-6′-methyl-benzophenone (SM) with hydrogen in ethyl acetate/tetrahydrofuran in the presence of 10% Pd/C at 25°. SM was obtained by condensation of 2,4-dimethoxy-6-methylbenzoic anhydride with 4-chloro-3,5-dimethoxyphenol benzyl ether in the presence of trifluoroacetic anhydride in methylene chloride under nitrogen for 10 min (47%) [1179].

m.p. 140°5–142° [1179]; ^{1}H NMR [1179], IR [1179], MS [1179].

[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl](4-bromophenyl) methanone

[75061-00-6]



$C_{18}H_{20}BrNO_2$ mol.wt. 362.27

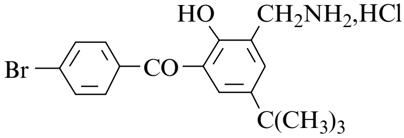
Synthesis

- Refer to: [817]; see the hydrochloride below.

m.p. and Spectra (NA).

[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl](4-bromophenyl) methanone *(Hydrochloride)*

[75060-65-0]



$C_{18}H_{20}BrNO_2$,HCl mol.wt. 398.73

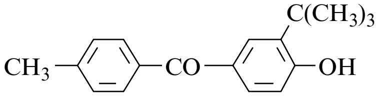
Synthesis

- Preparation by reaction of concentrated hydrochloric acid with 2-(4-bromo benzoyl)-4-tert-butyl-6-(N-chloro-acetylaminomethyl)phenol in refluxing ethanol for 20 h (89%) [817].

m.p. 240–245° [817]; Spectra (NA).

[3-(1,1-Dimethylethyl)-4-hydroxyphenyl](4-methylphenyl)methanone

[124979-04-4]



$C_{18}H_{20}O_2$ mol.wt. 268.36

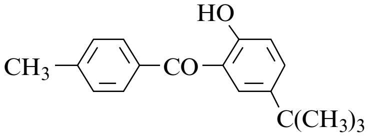
Synthesis

- Preparation by Friedel–Crafts acylation of o-tert-butylphenol with p-toluoyl chloride in ethylene dichloride in the presence of titanium tetrachloride, first at 0°, then at r.t. [816].

m.p. 209–210° [816]; Spectra (NA).

[5-(1,1-Dimethylethyl)-2-hydroxyphenyl](4-methylphenyl)methanone

[75919-94-7]



$C_{18}H_{20}O_2$ mol.wt. 268.36

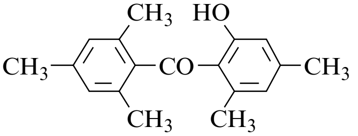
Synthesis

- Preparation by demethylation of 5-tert-butyl-2-methoxy-4′-methylbenzophenone with aluminium chloride in benzene between 50° and 60° for 5 h (85%) [9].

b.p.$_{12}$ 210–212° [9]; m.p. 94–95° [9]; Spectra (NA).

(2-Hydroxy-4,6-dimethylphenyl)(2,4,6-trimethylphenyl)methanone

[100923-74-8] $C_{18}H_{20}O_2$ mol.wt. 268.36



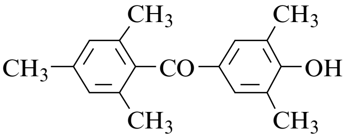
Synthesis

– Preparation by reaction of mesitoyl chloride with 3,5-dimethylphenol in the presence of aluminium chloride in refluxing nitromethane for 90 min (39%) [102].

m.p. 116–117° [102], 116° [94,95]; ^{1}H NMR [94,95,101,102], ^{13}C NMR [101], IR [94,95,102]; thermal behaviour [94,95].

(4-Hydroxy-3,5-dimethylphenyl)(2,4,6-trimethylphenyl)methanone

[69795-00-2] $C_{18}H_{20}O_2$ mol.wt. 268.36



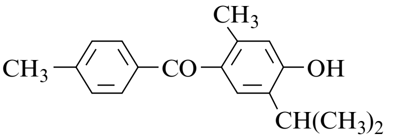
Synthesis

– Obtained by photo-Fries rearrangement of 2,6-di-methylphenyl mesitoate in pentane (5%), absorbed on a silica gel-pentane (18%) or on dry silica gel (37%) [64].

m.p. and Spectra (NA).

[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl](4-methylphenyl)methanone

[109250-36-4] $C_{18}H_{20}O_2$ mol.wt. 268.36



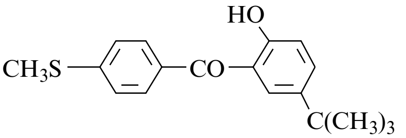
Synthesis

– Obtained by demethylation of 4-methoxy-2,4′-di-methyl-5-isopropylbenzophenone with refluxing pyridinium chloride [825].

m.p. 176° [825]; Spectra (NA).

[5-(1,1-Dimethylethyl)-2-hydroxyphenyl][4-(methylthio)phenyl]methanone

[75060-57-0] $C_{18}H_{20}O_2S$ mol.wt. 300.42

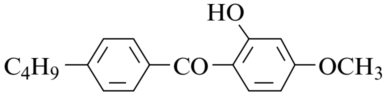


Synthesis

– Preparation by reaction of 30% methyl mercaptan with 2-(p-bromo benzoyl)-4-tert-butylphenol in the presence of sodium methoxide in refluxing methanol for 4 days (34%) [817].

m.p. 130–104° [817]. A typing error probably occurred in the published data. Spectra (NA).

(4-Butylphenyl)(2-hydroxy-4-methoxyphenyl)methanone



$C_{18}H_{20}O_3$ mol.wt. 284.36

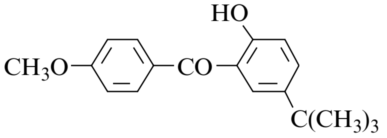
Synthesis

- Preparation by partial demethylation of 4′-butyl-2,4-dimethoxybenzophenone with aluminium chloride or aluminium bromide in chlorobenzene at 90–95° (good yield) [655].

m.p. and Spectra (NA).

[5-(1,1-Dimethylethyl)-2-hydroxyphenyl](4-methoxyphenyl)methanone

[116496-22-1]



$C_{18}H_{20}O_3$ mol.wt. 284.36

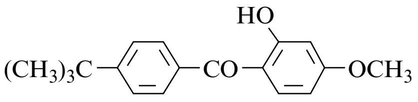
Syntheses

- Preparation by partial demethylation of 5-tert-butyl-2,4′-dimethoxybenzophenone with aluminium chloride in benzene at 50–60° for 5 h (76%) [9].
- Preparation by treatment of 2-hydroxy-4′-methoxy-benzophenone at 120° with a mixture of isobutylene/nitrogen (1:1) in the presence of a macroreticular acid ion exchanger (Wofatit OK 80) as catalyst, for 3 h (60%) [819].

b.p.$_{12}$ 235° [9], b.p.$_{0.15}$ 180–190° [819];
m.p. 93–94° [9]; Spectra (NA).

[4-(1,1-Dimethylethyl)phenyl](2-hydroxy-4-methoxyphenyl)methanone

[50739-53-2]



$C_{18}H_{20}O_3$ mol.wt. 284.36

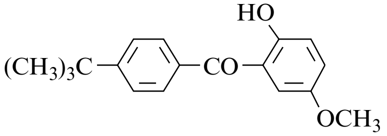
Synthesis

- Preparation by reaction of p-tert-butylbenzoyl chloride with resorcinol dimethyl ether,
  - in tetrachloroethane in the presence of aluminium chloride at 90° [222];
  - in chlorobenzene in the presence of titanium tetrachloride for 1 h at 120° (75%) [662].
- Also refer to: [78,684].

m.p. 75–77° [222], 73–75° [662]; UV [235].

[4-(1,1-Dimethylethyl)phenyl](2-hydroxy-5-methoxyphenyl)methanone

[162657-94-5]



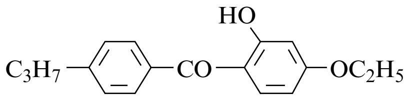
$C_{18}H_{20}O_3$ mol.wt. 284.36

Synthesis

- Preparation by partial demethylation of 4′-tert-butyl-2,5-dimethoxybenzophenone with aluminium chloride in benzene under nitrogen at 80° for 12 h (83%) [258,680].

viscous oil [680]; b.p. (NA); ^{1}H NMR [680], ^{13}C NMR [680], MS [680].

(4-Ethoxy-2-hydroxyphenyl)(4-propylphenyl)methanone



$C_{18}H_{20}O_3$ mol.wt. 284.36

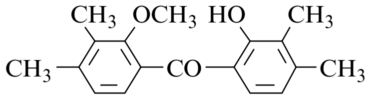
Synthesis

- Preparation by partial deethylation of 2,4-diethoxy-4′-propylbenzophenone with aluminium chloride in chlorobenzene at 80–100° [655].
- Also refer to: [78,222].

m.p. (NA); UV [235].

(2-Hydroxy-3,4-dimethylphenyl)(2-methoxy-3,4-dimethylphenyl)methanone

[54468-80-3]



$C_{18}H_{20}O_3$ mol.wt. 284.36

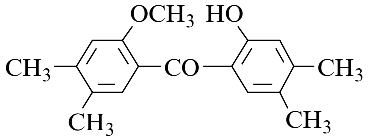
Synthesis

- Obtained by oxidation of 6,7-dimethyl-2-phenyl-3-(2-methoxy-3,4-dimethylphenyl)benzofuran with chromium trioxide, then saponification of the keto ester so formed, the 2-(benzoyloxy)-2′-methoxy-3,3′,4,4′-tetramethylbenzophenone [1152].

oil [1152]; b.p. (NA); IR [1152].

(2-Hydroxy-4,5-dimethylphenyl)(2-methoxy-4,5-dimethylphenyl)methanone

[54468-82-5]



$C_{18}H_{20}O_3$ mol.wt. 284.36

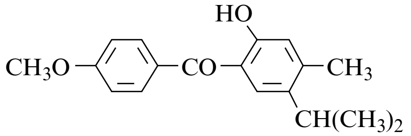
Synthesis

- Obtained by oxidation of 5,6-dimethyl-2-phenyl-3-(2-methoxy-4,5-dimethylphenyl)benzofuran with chromium trioxide, then saponification of the obtained keto ester, the 2-(benzoyloxy)-2′-methoxy-4,4′,5,5′-tetramethylbenzophenone [1152].

oil [1152]; b.p. (NA); IR [1152].

[2-Hydroxy-4-methyl-5-(1-methylethyl)phenyl](4-methoxyphenyl)methanone

[129375-12-8]



$C_{18}H_{20}O_3$ mol.wt. 284.36

Synthesis

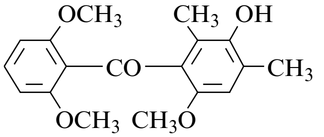
- Preparation by Fries rearrangement of 3-methyl-4-isopropylphenyl p-methoxybenzoate with titanium tetrachloride in nitromethane at 20° for 170 h (66%) [1180].

m.p. 100° [1180];

^{1}H NMR (Sadtler: standard n° 52709 M) [1180],
IR (Sadtler: standard n° 79766 K) [1180], UV [1180], MS [1180].

(2,6-Dimethoxyphenyl)(3-hydroxy-6-methoxy-2,4-dimethylphenyl)methanone

[42594-58-1]



$C_{18}H_{20}O_5$ mol.wt. 316.35

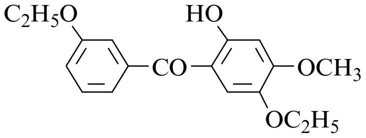
Synthesis

- Preparation by hydrogenolysis of 3-(benzyloxy)-2′,6,6′-tri-methoxy-2,4-dimethylbenzophenone with hydrogen in the presence of Pd/C in an ethyl acetate/methanol solution containing 70% perchloric acid (four drops) (96%) [1181].

m.p. 185–186° [1181];
^{1}H NMR [1181], IR [1181], UV [1181], MS [1181].

(5-Ethoxy-2-hydroxy-4-methoxyphenyl)(3-ethoxyphenyl)methanone

[51106-93-5]



$C_{18}H_{20}O_5$ mol.wt. 316.35

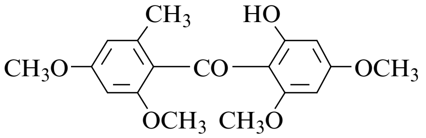
Synthesis

- Obtained (by-product) by acylation of 2,5-diethoxy-anisole with m-ethoxybenzoyl chloride in ethyl ether in the presence of aluminium chloride at r.t. for 48 h (<2%) [1163].

m.p. 79–80° [1163]; ^{1}H NMR [1163].

(2,4-Dimethoxy-6-methylphenyl)(2-hydroxy-4,6-dimethoxyphenyl)methanone

[93904-08-6]



$C_{18}H_{20}O_6$ mol.wt. 332.35

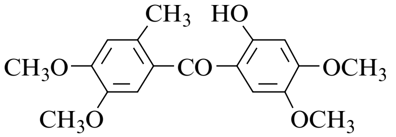
Synthesis

- Obtained by partial methylation of griseophenone C,
  - with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 24 h (45%) [1182];
  - with diazomethane in an ethyl ether/tetrahydrofuran mixture at 0° during 40 h (quantitative yield) [1182].

m.p. 124–125° [1182]; IR [1182], UV [1182].

(4,5-Dimethoxy-2-methylphenyl)(2-hydroxy-4,5-dimethoxyphenyl)methanone

[101744-11-0] $C_{18}H_{20}O_6$ mol.wt. 332.35



Syntheses

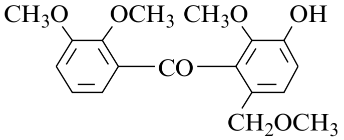
– Preparation by reaction of 2,4,5-trimethoxybenzoyl chloride with 4-methylveratrole in the presence of aluminium chloride in refluxing carbon disulfide for 8 h, then at r.t. for 12 h [1175].

– Preparation by reaction of 4,5-dimethoxy-2-methyl-benzoyl chloride with hydroxyhydroquinone trimethyl ether in the same conditions that previously [1175].

m.p. 138–139° [1175]; UV [1175].

(2,3-Dimethoxyphenyl)[3-hydroxy-2-methoxy-6-(methoxymethyl)phenyl] methanone

[133387-00-5] $C_{18}H_{20}O_6$ mol.wt. 332.35



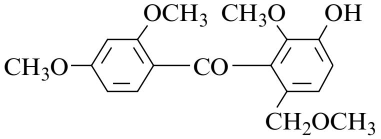
Synthesis

– Preparation from 4-hydroxy-3-methoxybenzyl methyl ether (SM) in two steps: first, direct lithiation of SM with n-butyl lithium in tetrahydrofuran at r.t., followed by quenching with 2,3-dimethoxybenzaldehyde. Then, oxidation of the 8-(2,3-dimetho xyphenyl)-2,7-di-methoxybicyclo[4.2.0] octa-1,3,5-triene-3,8-diol formed (good yield) [1171].

m.p. and Spectra (NA).

(2,4-Dimethoxyphenyl)[3-hydroxy-2-methoxy-6-(methoxymethyl)phenyl] methanone

[133386-98-8] $C_{18}H_{20}O_6$ mol.wt. 332.35



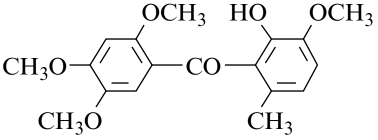
Synthesis

– Preparation from 4-hydroxy-3-methoxybenzyl methyl ether (SM1) in two steps: first, direct lithiation of SM1 with n-butyl lithium in tetrahydrofuran at r.t., followed by quenching with 2,4-dimethoxybenzaldehyde. Then, oxidation of the 8-(2,4-dimethoxyphenyl)-2,7-dimethoxybicyclo[4.2.0]octa-1,3,5-triene-3,8-diol formed (SM2) (good yield) [1171]. Actually, SM2 was quantitatively converted back into the expected ketone by heating a toluene solution to reflux for 14 h [1171].

m.p. and Spectra (NA).

(2-Hydroxy-3-methoxy-6-methylphenyl)(2,4,5-trimethoxyphenyl)methanone

[129103-92-0]



$C_{18}H_{20}O_6$ mol.wt. 332.35

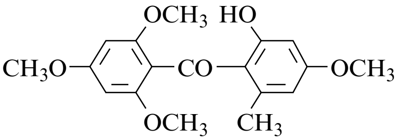
Synthesis

- Preparation from 2-iodo-6-methoxy-3-methylphenyl 2,4,5-trimethoxybenzoate on treatment with n-butyl-lithium in a mixture of ethyl ether, hexane and tetrahydrofuran at −70° for 2 h, followed by treatment with saturated aqueous ammonium chloride (40%) [58].

m.p. 160–162° [58]; ^{1}H NMR [58], IR [58], MS [58].

(2-Hydroxy-4-methoxy-6-methylphenyl)(2,4,6-trimethoxyphenyl)methanone

[74628-37-8]



$C_{18}H_{20}O_6$ mol.wt. 332.35

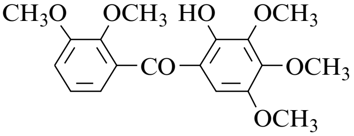
Synthesis

- Preparation by selective methylation of 2,4′-di-hydroxy-2′,4,6′-trimethoxy-6-methylbenzo-phenone [1183], with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 23 h (85%) [1184].

m.p. 161–162° [1184]; ^{1}H NMR [1184], MS [1184].

(2,3-Dimethoxyphenyl)(2-hydroxy-3,4,5-trimethoxyphenyl)methanone

[42833-83-0]



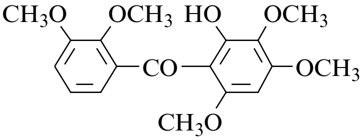
$C_{18}H_{20}O_7$ mol.wt. 348.35

Synthesis

- Obtained by reaction of 2,3-dimethoxybenzoyl chloride with 1,2,3,4-tetramethoxybenzene in the presence of aluminium chloride in ethyl ether for 42 h [416].

m.p. (NA); ^{1}H NMR [416], UV [416], MS [416].

(2,3-Dimethoxyphenyl)(2-hydroxy-3,4,6-trimethoxyphenyl)methanone



$C_{18}H_{20}O_7$ mol.wt. 348.35

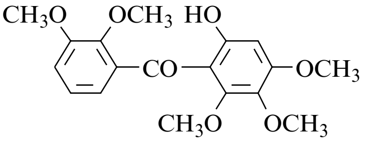
Synthesis

- Obtained by reaction of 2,3-dimethoxybenzoic acid with 1,2,3,5-tetramethoxybenzene in trifluoroacetic anhydride for 23 h at r.t. [1185].

m.p. and Spectra (NA); TLC [1185].

(2,3-Dimethoxyphenyl)(6-hydroxy-2,3,4-trimethoxyphenyl)methanone

[22804-59-7] $C_{18}H_{20}O_7$ mol.wt. 348.35



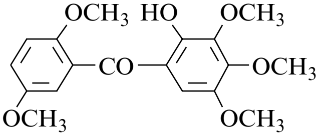
Synthesis

- Obtained by condensation of 2,3-dimethoxybenzoic acid with 1-acetoxy-3,4,5-trimethoxybenzene in trifluoroacetic anhydride, followed by hydrolysis of the reaction mixture (26%) [1186].

m.p. 110°8–111°2 [1186]; ^{1}H NMR [1186], UV [1186].

(2,5-Dimethoxyphenyl)(2-hydroxy-3,4,5-trimethoxyphenyl)methanone

[129168-53-2] $C_{18}H_{20}O_7$ mol.wt. 348.35



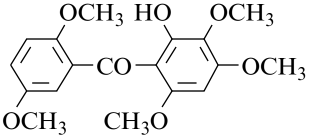
Syntheses

- Preparation by reaction of 2,5-dimethoxybenzoyl chloride with 1,2,3,4-tetramethoxybenzene in benzene in the presence of aluminium chloride at 50° (60%). The same result was obtained in ethyl ether at r.t. [1160].
- Also obtained (poor yield) by reaction of 2,3,4,5-tetra-methoxybenzoyl chloride with hydroquinone dimethyl ether in benzene in the presence of aluminium chloride (11%) [1160].

m.p. 74–74°5 [1160]; Spectra (NA).

(2,5-Dimethoxyphenyl)(2-hydroxy-3,4,6-trimethoxyphenyl)methanone

[23251-65-2] $C_{18}H_{20}O_7$ mol.wt. 348.35



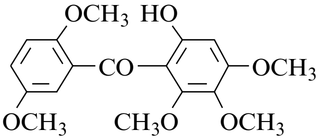
Syntheses

- Obtained by reaction of 2,5-dimethoxybenzoyl chloride with 1,2,3,5-tetramethoxybenzene in the presence of aluminium chloride in ethyl ether at r.t. for 18 h [416], (75%) [1160].
- Also obtained by selective demethylation of 2,2′,3,4,5′,6-hexamethoxybenzophenone with aluminium chloride in ethyl ether (30%) [1185].

m.p. 128–130° [1160,1185]; ^{1}H NMR [416,1185], UV [1185], MS [416].

(2,5-Dimethoxyphenyl)(6-hydroxy-2,3,4-trimethoxyphenyl)methanone

[22804-57-5] $C_{18}H_{20}O_7$ mol.wt. 348.35



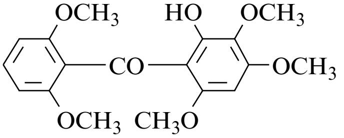
Synthesis

- Preparation by reaction of 2,5-dimethoxybenzoic acid with 1-acetoxy-3,4,5-trimethoxybenzene in the presence of trifluoroacetic anhydride for two weeks at r.t., followed by hydrolysis of the reaction mixture (52%) [1186], (45%) [1160].

yellow oil [1186]; m.p. 65–66° [1160]; ^{1}H NMR [1186], UV [1186].

(2,6-Dimethoxyphenyl)(2-hydroxy-3,4,6-trimethoxyphenyl)methanone

[22804-60-0] $C_{18}H_{20}O_7$ mol.wt. 348.35



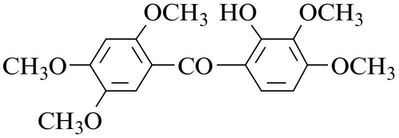
Synthesis

– Preparation by selective demethylation of 2,2′,3,4,6,6′-hexamethoxybenzophenone (SM) with aluminium chloride in refluxing ethyl ether (60%). SM was obtained by condensation of 2,6-dimethoxybenzoyl chloride with 1,2,3,5-tetramethoxybenzene in nitrobenzene with aluminium chloride for two days at r.t. (27%, m.p. 136–136°5) [1186].

m.p. 167–168° [1186]; ^{1}H NMR [1186], UV [1186].

(2-Hydroxy-3,4-dimethoxyphenyl)(2,4,5-trimethoxyphenyl)methanone

$C_{18}H_{20}O_7$ mol.wt. 348.35



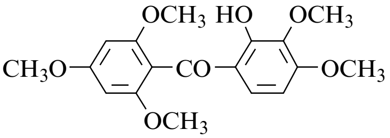
Synthesis

– Refer to: [1011] (compound **42**).

m.p. and Spectra (NA).

(2-Hydroxy-3,4-dimethoxyphenyl)(2,4,6-trimethoxyphenyl)methanone

[42833-67-0] $C_{18}H_{20}O_7$ mol.wt. 348.35



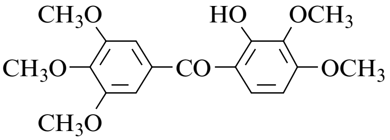
Synthesis

– Obtained by reaction of 2,3,4-tri methoxybenzoyl chloride with 1,3,5-trimethoxybenzene in the presence of aluminium chloride in ethyl ether for 15 h [416].

m.p. (NA); ^{1}H NMR [416].

(2-Hydroxy-3,4-dimethoxyphenyl)(3,4,5-trimethoxyphenyl)methanone

$C_{18}H_{20}O_7$ mol.wt. 348.35

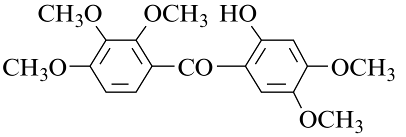


Synthesis

– Preparation by Friedel–Crafts acylation of pyrogallol trimethyl ether with 3,4,5-trimethoxybenzoyl chloride in carbon disulfide in the presence of aluminium chloride in a water bath [1169,1170].

m.p. 133–134° [1169,1170]; Spectra (NA).

(2-Hydroxy-4,5-dimethoxyphenyl)(2,3,4-trimethoxyphenyl)methanone



$C_{18}H_{20}O_7$ mol.wt. 348.35

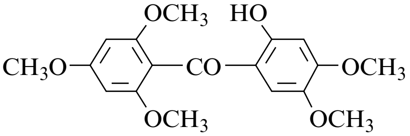
Synthesis

- Refer to: [1011] (compound **43**).

m.p. and Spectra (NA).

(2-Hydroxy-4,5-dimethoxyphenyl)(2,4,6-trimethoxyphenyl)methanone

[42833-68-1]



$C_{18}H_{20}O_7$ mol.wt. 348.35

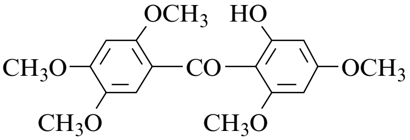
Syntheses

- Obtained (by-product) by partial demethylation of 2,2′,4,4′,5′,6-hexamethoxybenzophenone with boron trichloride in methylene chloride at 18° for 20 min (26%) [1187].
- Also obtained by Friedel–Crafts acylation of 1,3,5-trimethoxybenzene with 2,4,5-trimethoxybenzoyl chloride in the presence of aluminium chloride in ethyl ether for 21 h [416] or in boiling ethyl ether for 48 h (12%) [1187].

m.p. 171–173° [1188], 165–166° [1187]; ^{1}H NMR [416,1187], IR [1187], MS [1187].

(2-Hydroxy-4,6-dimethoxyphenyl)(2,4,5-trimethoxyphenyl)methanone

[76013-33-7]



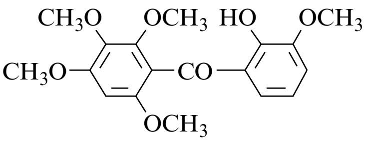
$C_{18}H_{20}O_7$ mol.wt. 348.35

Synthesis

- Preparation by partial demethylation of 2,2′,4,4′,5′,6-hexamethoxybenzophenone with boron trichloride in methylene chloride at 18° for 20 min (57%) [1187].

m.p. 129–130° [1187]; ^{1}H NMR [1187], IR [1187], MS [1187].

(2-Hydroxy-3-methoxyphenyl)(2,3,4,6-tetramethoxyphenyl)methanone



$C_{18}H_{20}O_7$ mol.wt. 348.35

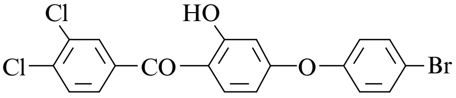
Synthesis

- Obtained by reaction of 2,3-dimethoxybenzoic acid with 1,2,3,5-tetramethoxybenzene in trifluoroacetic acid for 23 days at r.t. [1185].

m.p. and Spectra (NA); TLC [1185].

[4-(4-Bromophenoxy)-2-hydroxyphenyl](3,4-dichlorophenyl)methanone

[35698-03-4] $C_{19}H_{11}BrCl_2O_3$ mol.wt. 438.10



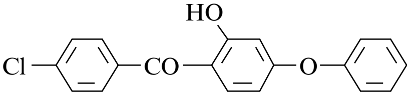
Synthesis

– Refer to: [839] (compound **33**).

m.p. (NA); UV [839].

(4-Chlorophenyl)(2-hydroxy-4-phenoxyphenyl)methanone

[35698-40-9] $C_{19}H_{13}ClO_3$ mol.wt. 324.76



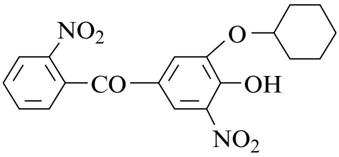
Synthesis

– Preparation by reaction of p-chlorobenzoyl chloride with 3-methoxydiphenyl ether in chloro-benzene in the presence of aluminium chloride: first at r.t., then for 4 h at 90–95° [839].

m.p. 125–127° [839]; UV [839].

[3-(Cyclohexyloxy)-4-hydroxy-5-nitrophenyl](2-nitrophenyl)methanone

[190585-64-9] $C_{19}H_{18}N_2O_7$ mol.wt. 386.36



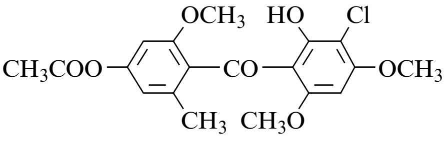
Synthesis

– Preparation by reaction of cyclohexene with 3,4-di-hydroxy-2′,5-dinitrobenzophenone in the presence of boron trifluoride ethyl ether complex for 12 h at reflux (24%). -Refer to: Chem. Abstr., **127**, 17465u (1997).

m.p. and Spectra (NA).

[4-(Acetyloxy)-2-methoxy-6-methylphenyl] (3-chloro-2-hydroxy-4,6-dimethoxyphenyl)-methanone

[95276-66-7] $C_{19}H_{19}ClO_7$ mol.wt. 394.81



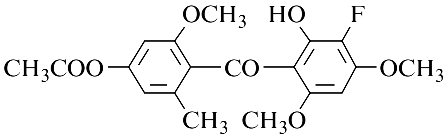
Synthesis

– Obtained by reaction of 4-acetoxy-2-methoxy-6-methylbenzoic acid with 2-chloro-3,5-di-methoxyphenol in the presence of trifluoroacetic anhydride at 20° for 18 h (17%) [1189] or at 25° (50%) [1190,1191].

– Also refer to: [1192].

m.p. 199–203° [1189]; Spectra (NA).

[4-(Acetyloxy)-2-methoxy-6-methylphenyl](3-fluoro-2-hydroxy-4,6-dimethoxyphenyl)-methanone



$C_{19}H_{19}FO_7$ mol.wt. 378.35

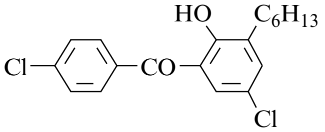
Synthesis

- Preparation by addition of 4-acetoxy-2-methoxy-6-methylbenzoic acid to 2-fluoro-3,5-dimethoxyphenol in the presence of trifluoroacetic anhydride (60%) [1190], first at 0°, then at 20–25° for 20 h (40%) [1191].

m.p. 195–200° [1190,1191]; IR [1191], UV [1190,1191].

(5-Chloro-3-hexyl-2-hydroxyphenyl)(4-chlorophenyl)methanone

[92739-91-8]



$C_{19}H_{20}Cl_2O_2$ mol.wt. 351.27

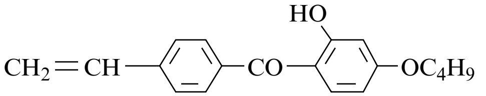
Synthesis

- Preparation by Fries rearrangement of 4-chloro-2-hexyl-phenyl p-chlorobenzoate with aluminium chloride at 160° for 15 min [1107].

oil [1107]; b.p.$_{0.133}$ 204° [1107]; Spectra (NA).

(4-Butoxy-2-hydroxyphenyl)(4-ethenylphenyl)methanone

[80167-01-7]



$C_{19}H_{20}O_3$ mol.wt. 296.37

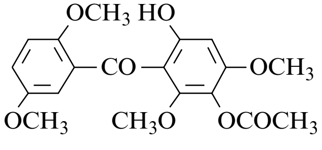
Synthesis

- Obtained by reaction of aqueous potassium hydroxide with 2-hydroxy-4-butoxy-4′-(2-bromoethyl)benzophenone in the presence of hydroquinone in refluxing methanol for 1.5 h with nitrogen bubbling (24%) [1147].

m.p. 78–80° [1147]; ^{1}H NMR [1147], ^{13}C NMR [1147], IR [1147], UV [1147], MS [1147].

[3-(Acetyloxy)-6-hydroxy-2,4-dimethoxyphenyl](2,5-dimethoxyphenyl) methanone

[129168-54-3]



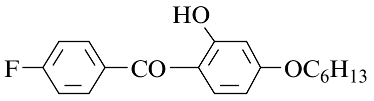
$C_{19}H_{20}O_8$ mol.wt. 376.36

Synthesis

- Obtained (poor yield) by reaction of 2,5-dimethoxybenzoic acid with 2,6-dimethoxy-1,4-hydroquinone diacetate in the presence of trifluoroacetic anhydride for two weeks at r.t. (<5%) [1160].

m.p. 135–135°5 [1160]; Spectra (NA).

(4-Fluorophenyl)[4-(hexyloxy)-2-hydroxyphenyl]methanone



$C_{19}H_{21}FO_3$ mol.wt. 316.37

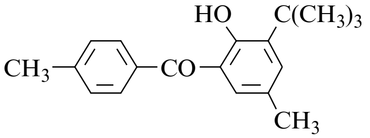
Synthesis

- Preparation by partial alkylation of 4′-fluoro-2,4-di-hydroxybenzophenone with an hexyl halide in the presence of an alkali [1109].

m.p. and Spectra (NA).

[3-(1,1-Dimethylethyl)-2-hydroxy-5-methylphenyl](4-methylphenyl) methanone

[93575-42-9]



$C_{19}H_{22}O_2$ mol.wt. 282.38

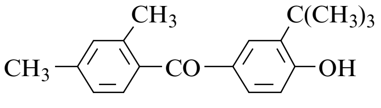
Synthesis

- Preparation by photo-Fries rearrangement of 2-tert-butyl-4-methylphenyl p-toluate in benzene under nitrogen for 32 h [1107].

m.p. 110–111° [1107]; Spectra (NA).

[3-(1,1-Dimethylethyl)-4-hydroxyphenyl](2,4-dimethylphenyl)methanone

[203786-32-7]



$C_{19}H_{22}O_2$ mol.wt. 282.38

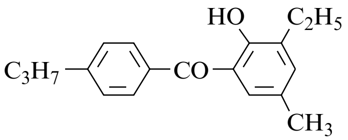
Synthesis

- Refer to: Chem. Abstr., **128**, 210826m (1998) (Japanese patent).

m.p. and Spectra (NA).

(3-Ethyl-2-hydroxy-5-methylphenyl)(4-propylphenyl)methanone

[93575-38-3]



$C_{19}H_{22}O_2$ mol.wt. 282.38

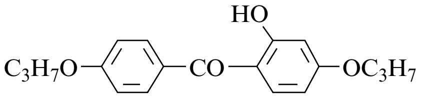
Synthesis

- Preparation by Fries rearrangement of 2-ethyl-4-methylphenyl 4-n-propylbenzoate with aluminium chloride at 160° for 15 min [1107].

oil [1107]; b.p. (NA); $n_D^{21} = 1.5861$ [1107]; Spectra (NA).

(2-Hydroxy-4-propoxyphenyl)(4-propoxyphenyl)methanone

[6131-39-1]



$C_{19}H_{22}O_4$ mol.wt. 314.38

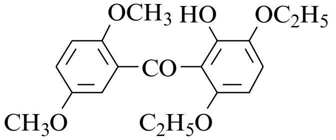
Synthesis

- Preparation by reaction of propyl bromide with 2,4,4′-trihydroxybenzophenone in the presence of potassium carbonate (50%) [380].

m.p. 60–60°5 [380]; IR [394], UV [380,394].

(3,6-Diethoxy-2-hydroxyphenyl)(2,5-dimethoxyphenyl)methanone

[110047-51-3]



$C_{19}H_{22}O_6$ mol.wt. 346.38

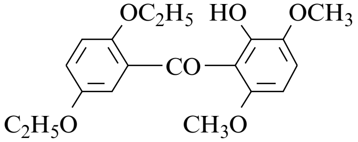
Synthesis

- Obtained (poor yield) by reaction of 3,6-diethoxy-2-methoxybenzoyl chloride with hydroquinone dimethyl ether in ethyl ether in the presence of aluminium chloride at r.t. for 2 days (4%) [1172].

m.p. 102–103° [1172]; Spectra (NA).

(2,5-Diethoxyphenyl)(2-hydroxy-3,6-dimethoxyphenyl)methanone

[110049-41-7]



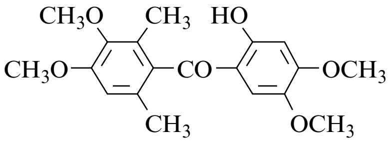
$C_{19}H_{22}O_6$ mol.wt. 346.38

Synthesis

- Obtained (poor yield) by reaction of 2,3,6-trimethoxy-benzoyl chloride with hydroquinone diethyl ether in ethyl ether in the presence of aluminium chloride at r.t. for 2 days (7%) [1172].

m.p. 100–101° [1172]; Spectra (NA).

(3,4-Dimethoxy-2,6-dimethylphenyl)(2-hydroxy-4,5-dimethoxyphenyl) methanone



$C_{19}H_{22}O_6$ mol.wt. 346.38

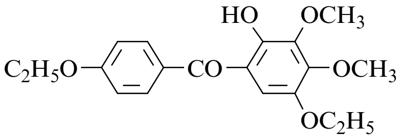
Synthesis

- Preparation by reaction of 2,4,5-trimethoxybenzoyl chloride with 3,5-dimethylveratrole in the presence of aluminium chloride in refluxing carbon disulfide for 8 h, then at r.t. for 12 h [1175].

m.p. 138–139° [1175]; UV [1175].

(5-Ethoxy-2-hydroxy-3,4-dimethoxyphenyl)(4-ethoxyphenyl)methanone

[69471-33-6] $C_{19}H_{22}O_6$ mol.wt. 346.38



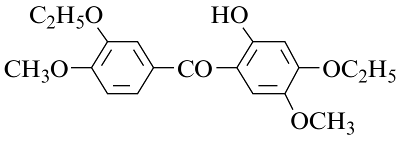
Synthesis

– Preparation by reaction of ethyl iodide with 4′-ethoxy-2,5-dihydroxy-3,4-dimethoxybenzo-phenone in the presence of potassium carbonate in refluxing acetone during 16 h (79%) [1168].

m.p. 60–61°5 [1168]; ^{1}H NMR [1168], IR [1168].

(4-Ethoxy-2-hydroxy-5-methoxyphenyl)(3-ethoxy-4-methoxyphenyl)methanone

[18008-38-3] $C_{19}H_{22}O_6$ mol.wt. 346.38



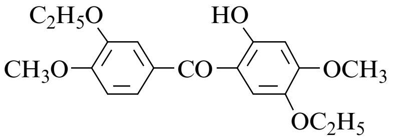
Synthesis

– Preparation by reaction of 3-ethoxy-4-methoxy-benzoyl chloride with 2,5-dimethoxyphenetole in the presence of aluminium chloride in ethyl ether at r.t. for 48 h (23%) [1174].

m.p. 132–133° [1174]; ^{1}H NMR [1174], IR [1174], UV[1174].

(5-Ethoxy-2-hydroxy-4-methoxyphenyl)(3-ethoxy-4-methoxyphenyl)methanone

[17892-44-3] $C_{19}H_{22}O_6$ mol.wt. 346.38



Syntheses

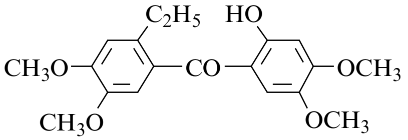
– Obtained by action of diethyl sulfate with 2,3′,5-tri-hydroxy-4,4′-dimethoxybenzophenone in the presence of potassium carbonate in refluxing acetone for 6 h (46%) [428].

– Preparation by reaction of 3-ethoxy-4-methoxy-benzoyl chloride with 2,4-dimethoxyphenetole in the presence of aluminium chloride in ethyl ether at r.t. for 48 h (26%) [1174].

m.p. 121–122° [1174], 121° [428]; ^{1}H NMR [1174], IR [1174], UV [1174].

(2-Ethyl-4,5-dimethoxyphenyl)(2-hydroxy-4,5-dimethoxyphenyl)methanone

$C_{19}H_{22}O_6$ mol.wt. 346.38



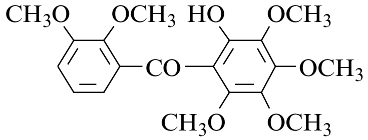
Synthesis

– Preparation by reaction of 2,4, 5-trimethoxybenzoyl chloride with 4-ethylveratrole in the presence of aluminium chloride in refluxing carbon disulfide for 8 h, then at r.t. for 12 h [1175].

m.p. 113–114° [1175]; UV [1175].

(2,3-Dimethoxyphenyl)(2-hydroxy-3,4,5,6-tetramethoxyphenyl)methanone

[22804-56-4] $C_{19}H_{22}O_8$ mol.wt. 378.38



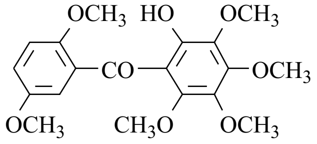
Synthesis

– Obtained by condensation of 2,3-dimethoxybenzoyl chloride with pentamethoxybenzene in the presence of aluminium chloride in refluxing ethyl ether for 1 h, then overnight at r.t. (19%) [1186].

m.p. 116°5–117°5 [1186]; ^{1}H NMR [1186], UV [1186].

(2,5-Dimethoxyphenyl)(2-hydroxy-3,4,5,6-tetramethoxyphenyl)methanone

[22961-80-4] $C_{19}H_{22}O_8$ mol.wt. 378.38



Syntheses

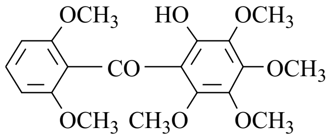
– Preparation by reaction of 2,5-dimethoxybenzoyl chloride with pentamethoxybenzene in nitrobenzene in the presence of aluminium chloride for 6 h at r.t. (38%) [1160], (13%) [1186].

– Preparation by selective demethylation of 2,2′,3,4,5,5′,6-heptamethoxybenzophenone with aluminium chloride in ethyl ether for 1.5 h at r.t. (42%) [1186].

yellow oil [1186]; m.p. 59° [1160]; ^{1}H NMR [1186], UV [1186].

(2,6-Dimethoxyphenyl)(2-hydroxy-3,4,5,6-tetramethoxyphenyl)methanone

[22804-62-2] $C_{19}H_{22}O_8$ mol.wt. 378.38



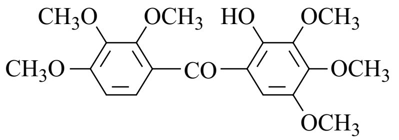
Synthesis

– Obtained (trace) by condensation of 2,6-dimethoxy-benzoyl chloride with pentamethoxybenzene in nitro-benzene with aluminium chloride for 4 h at r.t. (<1%) [1186].

oil [1186]; b.p. (NA); ^{1}H NMR [1186], UV [1186].

(2-Hydroxy-3,4,5-trimethoxyphenyl)(2,3,4-trimethoxyphenyl)methanone

[197355-26-3] $C_{19}H_{22}O_8$ mol.wt. 378.38



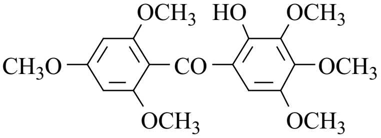
Synthesis

– Refer to: Chem. Abstr., **127**, 303322p (1997).

m.p. and Spectra (NA).

(2-Hydroxy-3,4,5-trimethoxyphenyl)(2,4,6-trimethoxyphenyl)methanone

[42833-85-2] $C_{19}H_{22}O_8$ mol.wt. 378.38



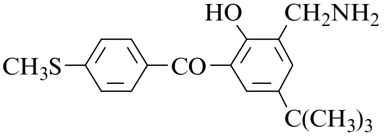
Synthesis

- Obtained by reaction of 2,4,6-trimethoxybenzoyl chloride with 1,2,3,4-tetramethoxybenzene in the presence of aluminium chloride in ethyl ether for18 h [416].

m.p. (NA); ^{1}H NMR [416].

[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl][4-(methylthio) phenyl]-methanone

[75061-01-7] $C_{19}H_{23}NO_2S$ mol.wt. 329.46



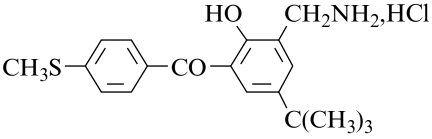
Synthesis

- Refer to: [817]; see the hydrochloride below.

m.p. and Spectra (NA).

[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl][4-(methylthio) phenyl]-methanone *(Hydrochloride)*

[75060-69-4] $C_{19}H_{23}NO_2S,HCl$ mol.wt. 365.92



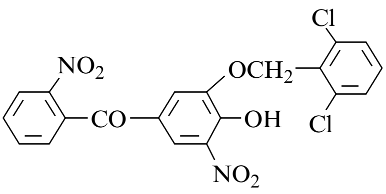
Synthesis

- Preparation by reaction of concentrated hydrochloric acid with 4-tert-butyl-6-(N-chloroacetylaminomethyl)-2-(4-methyl-thio benzoyl)phenol in refluxing ethanol for 20 h (86%) [817].

m.p. 205–208° [817]; Spectra (NA).

[3-[(2,6-Dichlorophenyl)methoxy]-4-hydroxy-5-nitrophenyl](2-nitrophenyl) methanone

[190585-65-0] $C_{20}H_{12}Cl_2N_2O_7$ mol.wt. 463.03



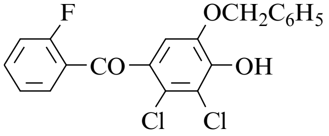
Synthesis

- Obtained by adding 2,6-dichlorobenzyl bromide to a solution of 3,4-dihydroxy-2′,5-dinitrobenzo-phenone in N,N-dimethylformamide, first treated with sodium hydride, then stirring for 2 h at 35° (66%). -Refer to: Chem. Abstr., **127**, 17465u (1997).

m.p. and Spectra (NA).

[2,3-Dichloro-4-hydroxy-5-(phenylmethoxy)phenyl](2-fluorophenyl) methanone

[103843-59-0]



$C_{20}H_{13}Cl_2FO_3$ mol.wt. 391.23

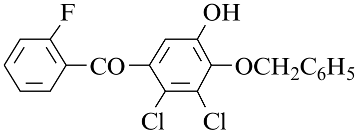
Synthesis

- Preparation by reaction of benzyl bromide with 2,3-dichloro-4,5-dihydroxy-2′-fluorobenzophenone in the presence of sodium hydride in N,N-dimethyl-formamide at r.t. for 15 min (71%) [841,842].

m.p. 156–157° [841,842]; ^{1}H NMR [841,842], IR [841,842]; X-ray analysis [841].

[2,3-Dichloro-5-hydroxy-4-(phenylmethoxy)phenyl](2-fluorophenyl) methanone

[103843-64-7]



$C_{20}H_{13}Cl_2FO_3$ mol.wt. 391.23

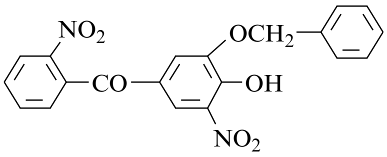
Synthesis

- Preparation by reaction of benzyl bromide with 2,3-dichloro-3,4-dihydroxy-2′-fluorobenzophenone in the presence of sodium hydride in N,N-dimethyl-formamide at 100° for 2 h (52%) [841].

m.p. 109–110° [841]; ^{1}H NMR [841], IR [841].

[4-Hydroxy-3-nitro-5-(phenylmethoxy)phenyl](2-nitrophenyl)methanone

[190585-63-8]



$C_{20}H_{14}N_2O_7$ mol.wt. 294.34

Synthesis

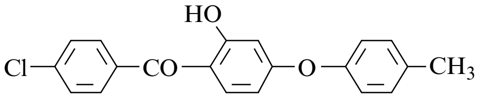
- Obtained by adding benzyl bromide to a solution of 3,4-dihydroxy-2′,5-dinitrobenzophenone in N,N-dimeth ylformamide, first treated with sodium hydride,

- then stirring for 2 h at 35° (66%);
- or then refluxing for 1 h and stirring for 12 h at 25° (75%). -Refer to: Chem. Abstr., **127**, 17465u (1997)^T.

m.p. (NA); ^{1}H NMR^T.

(4-Chlorophenyl)[2-hydroxy-4-(4-methylphenoxy)phenyl]methanone

[35698-48-7] $C_{20}H_{15}ClO_3$ mol.wt. 338.79



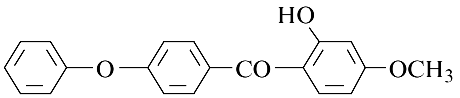
Synthesis

– Preparation by Fries rearrangement of 3-(p-chlorobenzoyloxy)-4′-methyldiphenyl ether in 1,2,4-trichlorobenzene in the presence of aluminium chloride at first for 30 min at 140°, then for 2 h at 200° [839].

m.p. 166–169° [839]; UV [839].

(2-Hydroxy-4-methoxyphenyl)(4-phenoxyphenyl)methanone

[127724-93-0] $C_{20}H_{16}O_4$ mol.wt. 320.34



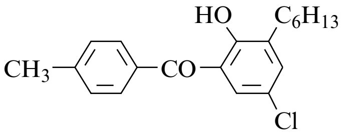
Synthesis

– Refer to: [668].

m.p. and Spectra (NA).

(5-Chloro-3-hexyl-2-hydroxyphenyl)(4-methylphenyl)methanone

[93739-92-9] $C_{20}H_{23}ClO_2$ mol.wt. 330.86



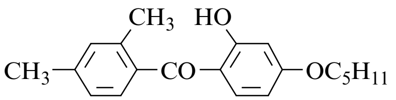
Synthesis

– Preparation by Fries rearrangement of 4-chloro-2-hexylphenyl p-toluate with aluminium chloride at 160° for 15 min [1107].

oil [1107]; b.p.$_{0.133}$ 192° [1107]; Spectra (NA).

(2,4-Dimethylphenyl)[2-hydroxy-4-(pentyloxy)phenyl]methanone

[36130-60-6] $C_{20}H_{24}O_3$ mol.wt. 312.41



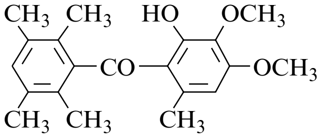
Synthesis

– Preparation by reaction of n-amyl chloride (1-chloropentane) with 2,4-dihydroxy-2′,4′-di-methylbenzophenone in the presence of potassium hydroxide and antimony triiodide in diethylene glycol at 140° for 1.5 h (85%) [801].

b.p.$_{0.4}$ 218–222° [801]; Spectra (NA).

(2-Hydroxy-3,4-dimethoxy-6-methyl)(2,3,5,6-tetramethylphenyl)methanone

[183725-95-3] $C_{20}H_{24}O_4$ mol.wt. 328.41



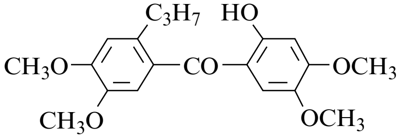
Syntheses

- Preparation by Friedel–Crafts acylation of 3,4,5-trimethoxy-toluene with 2,3,5,6-tetramethylbenzoyl chloride [1072].
- Also obtained by partial demethylation of 2,3,5,6,6′-penta-methyl-2′,3′,4′-trimethoxy-benzophenone [1072].

m.p. 137° [1072]; Spectra (NA).

(4,5-Dimethoxy-2-propylphenyl)(2-hydroxy-4,5-dimethoxyphenyl)methanone

$C_{20}H_{24}O_6$ mol.wt. 360.41



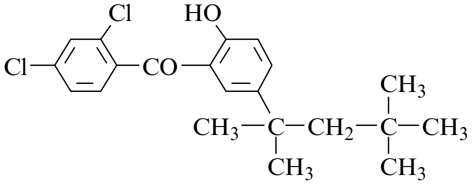
Synthesis

- Preparation by reaction of 2,4,5-trimethoxybenzoyl chloride with 4-propylveratrole in the presence of aluminium chloride in refluxing carbon disulfide for 8 h, then at r.t. for 12 h [1175].

m.p. 108–109° [1175]; UV [1175].

(2,4-Dichlorophenyl)[2-hydroxy-5-(1,1,3,3-tetramethylbutyl)phenyl]methanone

[93885-04-2] $C_{21}H_{24}Cl_2O_2$ mol.wt. 379.36



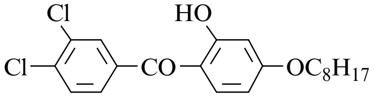
Synthesis

- Preparation by demethylation of 2-(2,4-di-chlorobenzoyl)-4-(1,1,3,3-tetramethyl-butyl) anisole (SM) with aluminium chloride in methylene chloride for 1 h at 10–12° (55%). SM was obtained by Friedel–Crafts acylation of 4-(1,1,3,3-tetramethylbutyl)-anisole with 2,4-dichlorobenzoyl chloride in methylene chloride in the presence of aluminium chloride for 30 min at 10° (74%) [1164].

m.p. 89–90° [1164]; Spectra (NA); GC [1164].

(3,4-Dichlorophenyl)[2-hydroxy-4-(octyloxy)phenyl]methanone

[36414-88-7] $C_{21}H_{24}Cl_2O_3$ mol.wt. 395.32



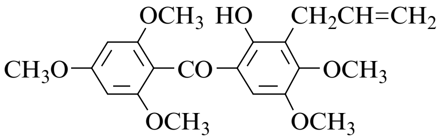
Synthesis

- Refer to: [235].

m.p. 51° [235]; UV [235].

[2-Hydroxy-4,5-dimethoxy-3-(2-propenyl)phenyl](2,4,6-trimethoxyphenyl) methanone

[42833-95-5] $C_{21}H_{24}O_7$ mol.wt. 388.42



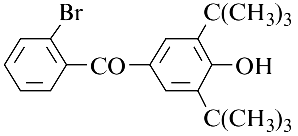
Synthesis

- Obtained by Claisen rearrangement of 2-(allyloxy)-2′,4,4′,5,6′-pentamethoxy-benzophenone in refluxing N,N-dimethyl-aniline for 4 h (62%) [416].

pale yellow oil [416]; b.p. (NA); ^{1}H NMR [416].

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](2-bromophenyl)methanone

[84700-54-9] $C_{21}H_{25}BrO_2$ mol.wt. 389.33



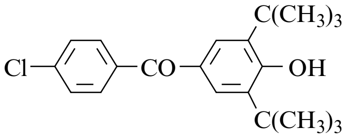
Synthesis

- Preparation by acylation of 2,6-di-tert-butylphenol with o-bromobenzoyl chloride in the presence of aluminium chloride (31%) [860], according to [857].

m.p. 74–75° [860]; IR [860].

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](4-chlorophenyl)methanone

[84700-53-8] $C_{21}H_{25}ClO_2$ mol.wt. 344.88



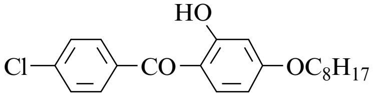
Synthesis

- Preparation by acylation of 2,6-di-tert-butylphenol with p-chlorobenzoyl chloride in the presence of aluminium chloride (41%) [860], according to [857].

m.p. 162–163° [860]; IR [860].

(4-Chlorophenyl)[2-hydroxy-4-(octyloxy)phenyl]methanone

[18190-30-2] $C_{21}H_{25}ClO_3$ mol.wt. 360.88



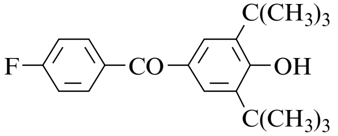
Synthesis

- Refer to: [235].

m.p. 60° [235]; UV [235].

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](4-fluorophenyl)methanone

[69451-08-7]



$C_{21}H_{25}FO_2$ mol.wt. 328.43

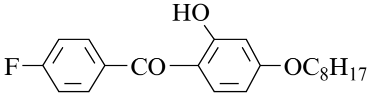
Synthesis

- Preparation by acylation of 2,6-di-tert-butylphenol with p-fluorobenzoyl chloride in the presence of aluminium chloride (35%) [860], according to [857].

m.p. 133–134° [860]; IR [860].

(4-Fluorophenyl)[2-hydroxy-4-(octyloxy)phenyl]methanone

[84794-99-0]



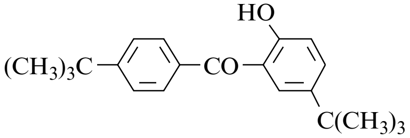
$C_{21}H_{25}FO_3$ mol.wt. 344.43

Synthesis

- Preparation by reaction of 1-bromooctane with 4′-fluoro-2,4-dihydroxybenzophenone in the presence of sodium bicarbonate in cyclohexanone for 5 h at 150° (72%) [1109].

m.p. 48–49° [1109]; Spectra (NA).

[5-(1,1-Dimethylethyl)-2-hydroxyphenyl][4-(1,1-dimethylethyl)phenyl] methanone



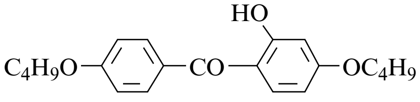
$C_{21}H_{26}O_2$ mol.wt. 310.44

Synthesis

- Obtained by UV light irradiation of p-tert-butyl-phenyl p-tert-butylbenzoate in benzene (48%) [154].

yellow oil [154]; b.p.$_{0.02}$ 150° [154]; Spectra (NA).

(4-Butoxy-2-hydroxyphenyl)(4-butoxyphenyl)methanone



$C_{21}H_{26}O_4$ mol.wt. 342.44

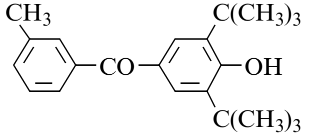
Synthesis

- Preparation by reaction of butyl bromide with 2,4,4′-trihydroxybenzophenone in the presence of potassium carbonate (50%) [380].

m.p. 95–96° [380]; UV [380].

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](3-methylphenyl)methanone

[84700-50-5] $C_{22}H_{28}O_2$ mol.wt. 324.46



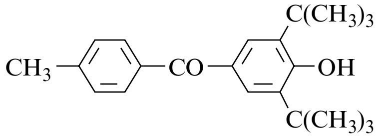
Synthesis

- Preparation by acylation of 2,6-di-tert-butylphenol with m-methylbenzoyl chloride in the presence of aluminium chloride (38%) [860], according to [857].

m.p. 99–100° [860]; IR [860].

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](4-methylphenyl)methanone

[84700-49-2] $C_{22}H_{28}O_2$ mol.wt. 324.46



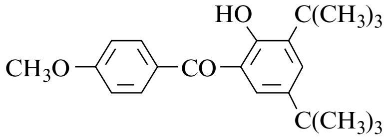
Synthesis

- Preparation by acylation of 2,6-di-tert-butylphenol with p-methylbenzoyl chloride in the presence of aluminium chloride (40%) [860], according to [857].

m.p. 131–132° [860]; IR [860].

[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl](4-methoxyphenyl)methanone

[80078-54-2] $C_{22}H_{28}O_3$ mol.wt. 340.46



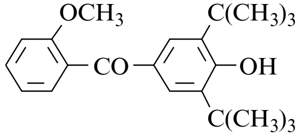
Syntheses

- Preparation by oxidation of 2,4-di-tert-butyl-6-(p-methoxybenzyl)phenol with silver oxide in boiling acetone for 40 min (24%) [1193].
- Preparation by treatment of 2-hydroxy-4′-methoxy-benzophenone at 120° with a mixture of isobutylene/nitrogen (1:1) in the presence of a macroreticular acid ion exchanger (Wofatit OK 80) as catalyst for 10 h (85%) [819].

b.p.$_{0.15}$ 200–205° [819]; m.p. 116–117° [1193]; ^{1}H NMR [1193], IR [1193], UV [1193], MS [1193].

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](2-methoxyphenyl)methanone

[28441-13-6] $C_{22}H_{28}O_3$ mol.wt. 340.46



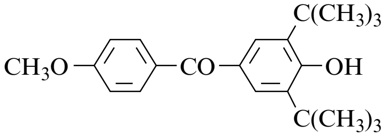
Synthesis

- Preparation by acylation of 2,6-di-tert-butylphenol with o-methoxybenzoyl chloride in the presence of aluminium chloride (36%) [860], according to [857].

m.p. 87–88° [860]; IR [860].

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](4-methoxyphenyl)methanone

[28440-99-5] $C_{22}H_{28}O_3$ mol.wt. 340.46



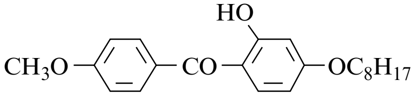
Synthesis

– Preparation by acylation of 2,6-di-tert-butylphenol with p-methoxybenzoyl chloride in the presence of aluminium chloride (61%) [860], according to [857].

m.p. 143–144° [860]; IR [860].

[2-Hydroxy-4-(octyloxy)phenyl](4-methoxyphenyl)methanone

[36469-90-6] $C_{22}H_{28}O_4$ mol.wt. 356.46



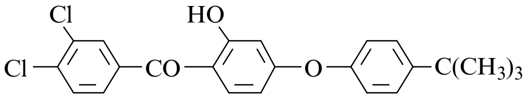
Synthesis

– Refer to: [235].

m.p. 55° [235]; UV [235].

(3,4-Dichlorophenyl)[4-[4-(1,1-dimethylethyl)phenoxy]-2-hydroxyphenyl] methanone

[35698-02-3] $C_{23}H_{20}Cl_2O_3$ mol.wt. 415.32



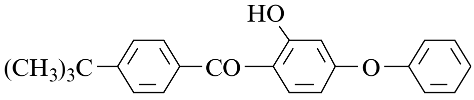
Synthesis

– Refer to: [839] (compound **32**).

m.p. (NA); UV [839].

[4-(1,1-Dimethylethyl)phenyl](2-hydroxy-4-phenoxyphenyl)methanone

[35698-42-1] $C_{23}H_{22}O_3$ mol.wt. 346.94



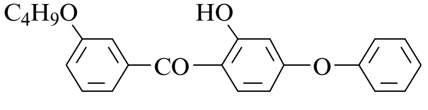
Synthesis

– Preparation by reaction of p-tert-butyl-benzoyl chloride with 3-methoxydiphenyl ether in chlorobenzene in the presence of aluminium chloride first at r.t., then for 4 h at 90–95° [839].

m.p. 88–90° [839]; UV [839].

(3-Butoxyphenyl)(2-hydroxy-4-phenoxyphenyl)methanone

[35698-55-6] $C_{23}H_{22}O_4$ mol.wt. 362.43



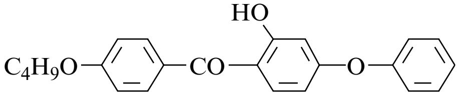
Synthesis

– Refer to: [839] (compound **13**).

m.p. (NA); UV [839].

(4-Butoxyphenyl)(2-hydroxy-4-phenoxyphenyl)methanone

[35698-52-3] $C_{23}H_{22}O_4$ mol.wt. 362.43



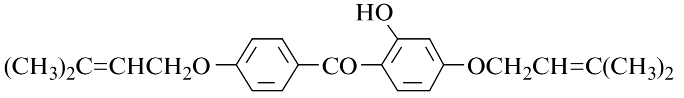
Synthesis

– Refer to: [839] (compound **10**).

m.p. (NA); UV [839].

[2-Hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl][4-[(3-methyl-2-butenyl)oxy] phenyl]-methanone

[63565-01-5] $C_{23}H_{26}O_4$ mol.wt. 366.46



Synthesis

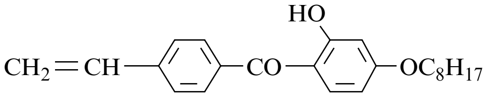
– Obtained (poor yield) by reaction of prenyl bromide with 2,4,4′-tri-hydroxy-benzophenone,

- in the presence of potassium carbonate in refluxing acetone for 3 h (6%) [831];
- in the presence of boron trifluoride-etherate in dioxane at 50–60° for 3 h (<1%) [832].

m.p. 74° [831], 73–74° [832]; ^{1}H NMR [831], IR [831], UV [831].

(4-Ethenylphenyl)[2-hydroxy-4-(octyloxy)phenyl]methanone

[80167-02-3] $C_{23}H_{28}O_3$ mol.wt. 352.47



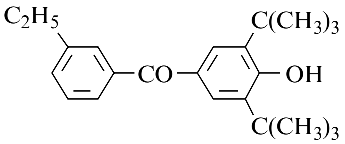
Synthesis

– Preparation by reaction of aqueous potassium hydroxide with 2-hydroxy-4-(octyloxy)-4′-(2-bromoethyl)-benzophenone in the presence of hydroquinone in refluxing methanol for 1.5 h with nitrogen bubbling (41%) [1147].

m.p. 57–59° [1147]; ^{1}H NMR [1147], IR [1147], UV [1147], MS [1147].

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](3-ethylphenyl)methanone

[84700-52-7] $C_{23}H_{30}O_2$ mol.wt. 338.49



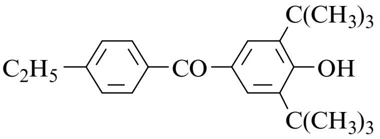
Synthesis

– Preparation by Friedel–Crafts acylation of 2,6-di-tert-butylphenol with 3-ethylbenzoyl chloride in the presence of aluminium chloride at –10° (50%) [860], according to [857].

m.p. 69–70° [860]; Spectra (NA).

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](4-ethylphenyl)methanone

[84700-51-6] $C_{23}H_{30}O_2$ mol.wt. 338.49



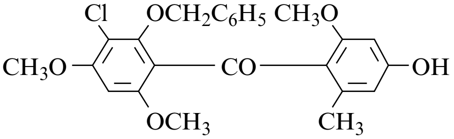
Synthesis

– Preparation by Friedel–Crafts acylation of 2,6-di-tert-butylphenol with p-ethylbenzoyl chloride in the presence of aluminium chloride at −10° (35%) [860] according to [857].

m.p. 136–137° [860]; Spectra (NA).

[3-Chloro-4,6-dimethoxy-2-(phenylmethoxy)phenyl] (4-hydroxy-2-methoxy-6-methyl-phenyl)methanone

$C_{24}H_{23}ClO_6$ mol.wt. 442.90



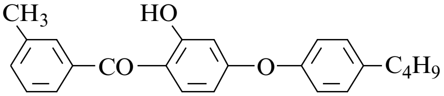
Synthesis

– Preparation by reaction of 2-(benzyloxy)-3-chloro-4,6-dimethoxybenzoyl chloride with mono(trimethylsilyl)derivative of 3-methoxy-5-methylphenol in the presence of stannic chloride (or titanium tetrachloride or aluminium chloride) in refluxing methylene chloride for 2 h [921].

m.p. and Spectra (NA).

[4-(4-Butylphenoxy)-2-hydroxyphenyl](3-methylphenyl)methanone

[35698-58-9] $C_{24}H_{24}O_3$ mol.wt. 360.45



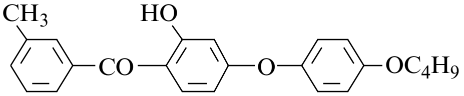
Synthesis

– Refer to: [839] (compound **16**).

m.p. (NA); UV [839].

[4-(4-Butoxyphenoxy)-2-hydroxyphenyl](3-methylphenyl)methanone

[35697-96-2] $C_{24}H_{24}O_4$ mol.wt. 376.45



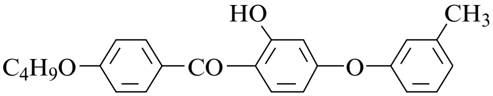
Synthesis

– Refer to: [839] (compound **25**).

m.p. (NA); UV [839].

(4-Butoxyphenyl)[2-hydroxy-4-(3-methylphenoxy)phenyl]methanone

[35698-62-5] $C_{24}H_{24}O_4$ mol.wt. 376.45



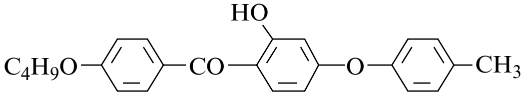
Synthesis

– Refer to: [839] (compound **20**).

m.p. (NA); UV [839].

(4-Butoxyphenyl)[2-hydroxy-4-(4-methylphenoxy)phenyl]methanone

[35698-59-0] $C_{24}H_{24}O_4$ mol.wt. 376.45



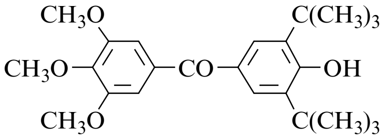
Synthesis

– Refer to: [839] (compound **17**).

m.p. (NA); UV [839].

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](3,4,5-trimethoxyphenyl) methanone

[54808-42-3] $C_{24}H_{32}O_5$ mol.wt. 400.52



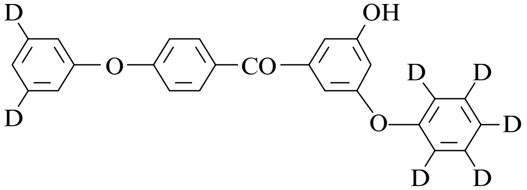
Synthesis

– Preparation by Friedel–Crafts acylation of 2,6-di-tert-butylphenol with 3,4,5-trimethoxybenzoyl chloride in the presence of stannic chloride in refluxing methylene chloride for 19 h (58%) [334].

m.p. 138–138°5 [334]; ^{1}H NMR [334], IR [334].

[3-Hydroxy-5-(phenoxy-*d5*)phenyl][4-(phenoxy-3,5-*d2*)phenyl]methanone

[176738-22-0] $C_{25}H_{11}D_7O_4$ mol.wt. 389.46



Synthesis

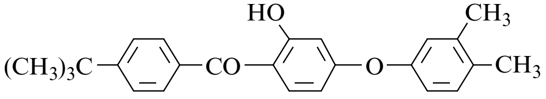
– Preparation by adding 3,5-dihydroxy-4′-(phenoxy-*d2*)benzophenone, potassium carbonate and copper powder to a solution of N-methyl-pyrrolidone/toluene. The obtained mixture was refluxed for 1 h, with removal of water.

– Then, adding bromobenzene-*d5* and heating at reflux again for 2 h (22%) (compound **22**) [906].

m.p. (NA); ^{1}H NMR [906], ^{13}C NMR [906].

[4-(1,1-Dimethylethyl)phenyl][4-(3,4-dimethylphenoxy)-2-hydroxyphenyl] methanone

[35697-99-5] $C_{25}H_{26}O_3$ mol.wt. 374.48



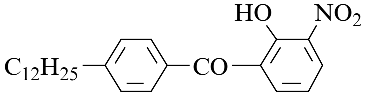
Synthesis

– Refer to: [839] (compound **28**).

m.p. (NA); UV [839].

(4-Dodecylphenyl)(2-hydroxy-3-nitrophenyl)methanone

[35698-24-9] $C_{25}H_{33}NO_4$ mol.wt. 411.54



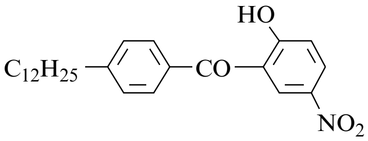
Synthesis

– Obtained by reaction of fuming nitric acid with 2-hydroxy-4′-dodecylbenzophenone in an acetic acid/acetic anhydride mixture (4:3) at 5–7° for 1 h (27%) [889,891].

m.p. and Spectra (NA).

(4-Dodecylphenyl)(2-hydroxy-5-nitrophenyl)methanone

[35698-23-8] $C_{25}H_{33}NO_4$ mol.wt. 411.54



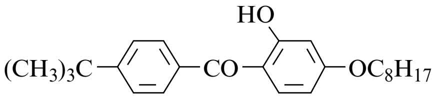
Synthesis

– Obtained by reaction of fuming nitric acid with 2-hydroxy-4′-dodecylbenzophenone in an acetic acid/acetic anhydride mixture (4:3) at 5–7° for 1 h (54%) [889,891].

m.p. and Spectra (NA).

[4-(1,1-Dimethylethyl)phenyl][2-hydroxy-4-(octyloxy)phenyl]methanone

[36419-37-1] $C_{25}H_{34}O_3$ mol.wt. 382.54



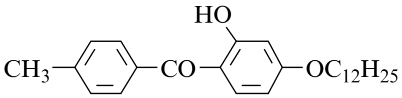
Synthesis

– Refer to: [235].

oil [235]; b.p. (NA); UV [235].

[4-(Dodecyloxy)-2-hydroxyphenyl](4-methylphenyl)methanone

[36130-67-3] $C_{26}H_{26}O_3$ mol.wt. 386.49



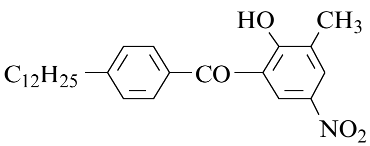
Synthesis

– Preparation by reaction of dodecyl chloride with 2,4-dihydroxy-4′-methylbenzophenone (92%), according to [801].

m.p. 50–51° [801]; Spectra (NA).

(4-Dodecylphenyl)(2-hydroxy-3-methyl-5-nitrophenyl)methanone

[35698-29-4] $C_{26}H_{35}NO_4$ mol.wt. 425.57



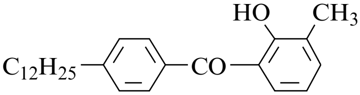
Synthesis

– Preparation by reaction of fuming nitric acid with 4′-dodecyl-2-hydroxy-3-methylbenzophenone in acetic acid/acetic anhydride (95%) [889,891].

m.p. and Spectra (NA).

(4-Dodecylphenyl)(2-hydroxy-3-methylphenyl)methanone

[35698-28-3] $C_{26}H_{36}O_2$ mol.wt. 380.57



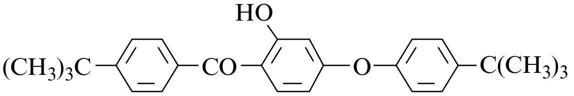
Synthesis

– Obtained (poor yield) by reaction of 2-hydroxy-3-methylbenzoyl chloride with dodecylbenzene in nitrobenzene in the presence of aluminium chloride for 6 h at 40° and at r.t. overnight (8%) [889,891].

b.p.$_{0.45–0.7}$ 210–217° [889,891]; Spectra (NA).

[[4-(1,1-Dimethylethyl)phenoxy]-2-hydroxyphenyl][4-(1,1-dimethylethyl) phenyl]-methanone

[35698-44-3] $C_{27}H_{30}O_3$ mol.wt. 402.53



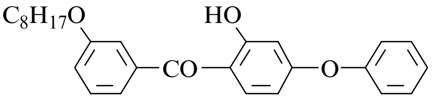
Synthesis

– Preparation by reaction of p-tert-butylbenzoyl chloride with 4-tert-butyl-3′-methoxydiphenyl ether in chlorobenzene in the presence of aluminium chloride first at r.t., then at 90–95° for 4 h [839].

m.p. 134–136° [839]; UV [839].

(2-Hydroxy-4-phenoxyphenyl)[3-(octyloxy)phenyl]methanone

[35698-56-7] $C_{27}H_{30}O_4$ mol.wt. 418.53



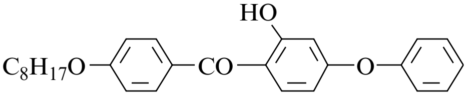
Synthesis

– Refer to: [839] (compound **14**).

m.p. (NA); UV [839].

(2-Hydroxy-4-phenoxyphenyl)[4-(octyloxy)phenyl]methanone

[35698-53-4] $C_{27}H_{30}O_4$ mol.wt. 418.53



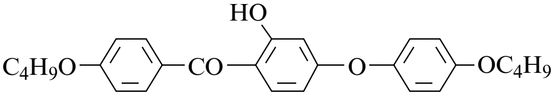
Synthesis

– Refer to: [839] (compound **11**).

m.p. (NA); UV [839].

[4-(4-Butoxyphenoxy)-2-hydroxyphenyl](4-butoxyphenyl)methanone

[35697-93-9] $C_{27}H_{30}O_5$ mol.wt. 434.68



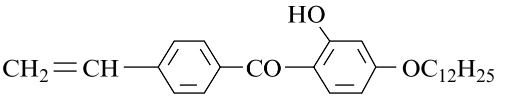
Synthesis

– Refer to: [839] (compound **22**).

m.p. (NA); UV [839].

[4-(Dodecyloxy)-2-hydroxyphenyl](4-ethenylphenyl)methanone

[80167-03-9] $C_{27}H_{36}O_3$ mol.wt. 408.58



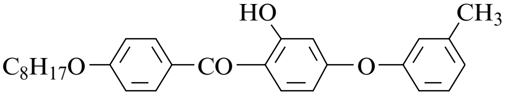
Synthesis

– Obtained by reaction of aqueous potassium hydroxide with 2-hydroxy-4-(dodecyloxy)- 4′-(2-bromoethyl)benzophenone in the presence of hydroquinone in refluxing methanol for 1.5 h with nitrogen bubbling (18%) [1147].

m.p. 49–50° [1147]; ^{1}H NMR [1147], IR [1147], UV [1147], MS [1147].

[2-Hydroxy-4-(3-methylphenoxy)phenyl][4-(octyloxy)phenyl]methanone

[35697-92-8] $C_{28}H_{32}O_4$ mol.wt. 432.56



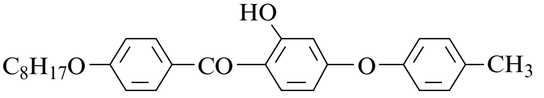
Synthesis

– Refer to: [839] (compound **21**).

m.p. (NA); UV [839].

[2-Hydroxy-4-(4-methylphenoxy)phenyl][4-(octyloxy)phenyl]methanone

[35698-60-3] $C_{28}H_{32}O_4$ mol.wt. 432.56



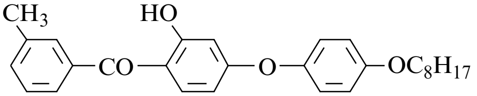
Synthesis

– Refer to: [839] (compound **18**).

m.p. (NA); UV [839].

[2-Hydroxy-4-[4-(octyloxy)phenoxy]phenyl](3-methylphenyl)methanone

[35697-97-3] $C_{28}H_{32}O_4$ mol.wt. 432.56



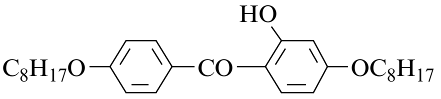
Synthesis

– Refer to: [839] (compound **26**).

m.p. (NA); UV [839].

[2-Hydroxy-4-(octyloxy)phenyl][4-(octyloxy)phenyl]methanone

$C_{29}H_{42}O_4$ mol.wt. 454.65



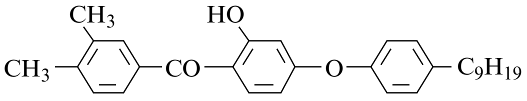
Synthesis

– Preparation by reaction of octyl bromide with 2,4,4′-trihydroxybenzophenone in the presence of potassium carbonate (60%) [380].

m.p. 60°5–61° [380]; UV [380].

(3,4-Dimethylphenyl)[2-hydroxy-4-(4-nonylphenoxy)phenyl]methanone

[35698-01-2] $C_{30}H_{36}O_3$ mol.wt. 444.61



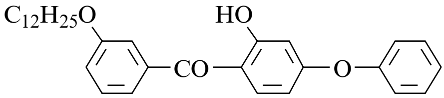
Synthesis

- Refer to: [839] (compound **31**).

m.p. (NA); UV [839].

[3-(Dodecyloxy)phenyl](2-hydroxy-4-phenoxyphenyl)methanone

[35698-57-8] $C_{31}H_{38}O_4$ mol.wt. 474.64



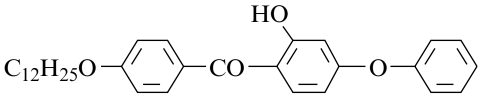
Synthesis

- Refer to: [839] (compound **15**).

m.p. (NA); UV [839].

[4-(Dodecyloxy)phenyl](2-hydroxy-4-phenoxyphenyl)methanone

[35698-54-5] $C_{31}H_{38}O_4$ mol.wt. 474.64



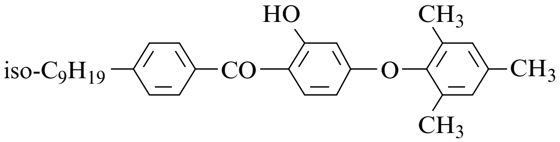
Synthesis

- Refer to: [839] (compound **12**).

m.p. (NA); UV [839].

[2-Hydroxy-4-(2,4,6-trimethylphenoxy)phenyl][4-(isononyloxy)phenyl]methanone

[36118-66-8] $C_{31}H_{38}O_4$ mol.wt. 474.64



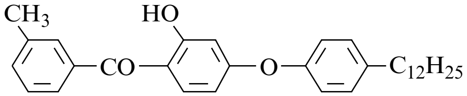
Synthesis

- Refer to: [839] (compound **29**).

m.p. (NA); UV [839].

[4-(4-Dodecylphenoxy)-2-hydroxyphenyl](3-methylphenyl)methanone

[35697-98-4] $C_{32}H_{40}O_3$ mol.wt. 472.67



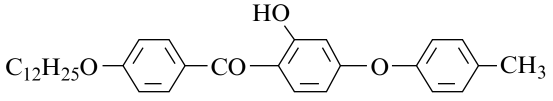
Synthesis

- Refer to: [839] (compound **27**).

m.p. (NA); UV [839].

[4-(Dodecyloxy)phenyl][2-hydroxy-4-(4-methylphenoxy)phenyl]methanone

[35698-61-4] $C_{32}H_{40}O_4$ mol.wt. 488.67



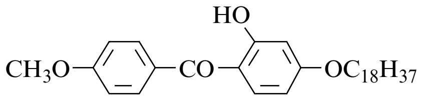
Synthesis

- Refer to: [839] (compound **19**).

m.p. (NA); UV [839].

[2-Hydroxy-4-(octadecyloxy)phenyl](4-methoxyphenyl)methanone

[36130-68-4] $C_{32}H_{48}O_4$ mol.wt. 496.73



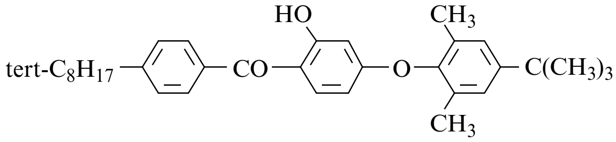
Synthesis

- Preparation by reaction of octadecyl chloride (1-chlorooctadecane) with 2,4-dihydroxy-4′-methoxybenzophenone (81%), according to [801].

m.p. 73–75° [801]; Spectra (NA).

[4-[4-(1,1-Dimethylethyl)-2,6-dimethylphenoxy]-2-hydroxyphenyl] [4-(1,1,3,3-tetra-methylbutyl)phenyl]methanone

[35698-00-1] $C_{33}H_{42}O_3$ mol.wt. 486.69



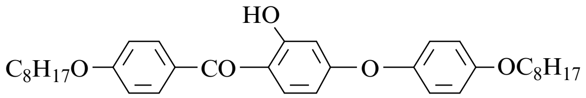
Synthesis

- Refer to: [839] (compound **30**).

m.p. (NA); UV [839].

[2-Hydroxy-4-[4-(octyloxy)phenoxy]phenyl][4-(octyloxy)phenyl]methanone

[35697-94-0] $C_{35}H_{46}O_5$ mol.wt. 546.75



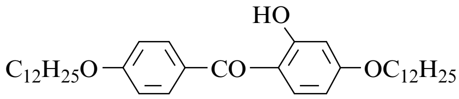
Synthesis

- Refer to: [839] (compound **23**).

m.p. (NA); UV [839].

[4-(Dodecyloxy)-2-hydroxyphenyl][4-(dodecyloxy)phenyl]methanone

$C_{37}H_{58}O_4$ mol.wt. 566.87



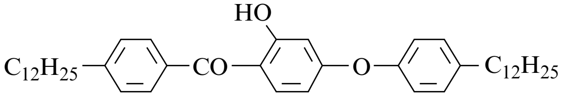
Synthesis

- Refer to: [98].

m.p. (NA); EPR [98].

[4-(4-Dodecylphenoxy)-2-hydroxyphenyl](4-dodecylphenyl)methanone

[35697-95-1] $C_{43}H_{62}O_3$ mol.wt. 626.96



Synthesis

- Refer to: [839] (compound **24**).

m.p. (NA); UV [839].

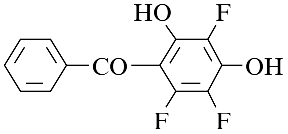
2.2 Dihydroxybenzophenones

2.2.1 Hydroxy Groups Located on the Same Ring

2.2.1.1 Substituents Located on the Hydroxylated Ring

Phenyl(2,3,5-trifluoro-4,6-dihydroxyphenyl)methanone

[32541-14-3] $C_{13}H_7F_3O_3$ mol.wt. 268.19



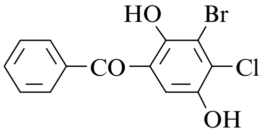
Synthesis

- Preparation by demethylation of 2,4-dimethoxy-3,5,6-trifluorobenzophenone (SM1) or of 2-hydroxy-4-methoxy-3,5,6-trifluorobenzophenone (SM2) with aluminium chloride in methylene chloride at 20° for 6 h (97% and 60% yields, respectively) [570]. SM1 was obtained in two steps: first, preparation of 4-methoxy-2,3,5,6-tetrafluorobenzophenone by treatment of 2,3,4,5,6-pentafluorobenzophenone with sodium methoxide in methanol at 20° for 3 days (91%). Then, this new ketone, by reaction with sodium methoxide in boiling methanol for 15 h gave SM1 (82%) [570]. The preparation of SM2 was also described in this book (94%) [570].

m.p. 173–176° [570]; IR [570], UV [570].

(3-Bromo-4-chloro-2,5-dihydroxyphenyl)phenylmethanone

[154700-58-0] $C_{13}H_8BrClO_3$ mol.wt. 327.56

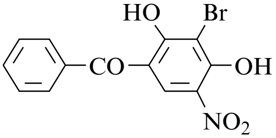


Synthesis

- Obtained by 10% Pd/C-catalyzed hydrogenolysis of *gem*-diphenylcyclopropane fused with 4-bromo-3,6-dichloro-benzoquinone in 1,4-dioxane in the presence of water (1%) for 6 h under atmospheric pressure at r.t. (27%) [1194].

m.p. (NA); ^{1}H NMR [1194].

(3-Bromo-2,4-dihydroxy-5-nitrophenyl)phenylmethanone



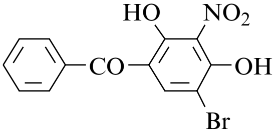
$C_{13}H_8BrNO_5$ mol.wt. 338.11

Syntheses

- Preparation by reaction of bromine with 2,4-dihydroxy-5-nitrobenzophenone in acetic acid at r.t. for 2 h [1195] or in hot acetic acid for 1 h [1196].
- Also obtained by reaction of nitric acid (d = 1.4) with 3,3′-dibenzoyl-5,5′-dibromo-2,2′,6,6′-tetrahydroxy-diphenyl thioether in acetic acid at r.t. for 1 h. The same result was obtained from 3,3′-dibenzoyl-5,5′-dibromo-4,4′,6,6′-tetrahydroxydiphenyl thioether [1195].
- Also obtained by action of nitric acid (d = 1.4) with 5-bromo-2,4-dihydroxybenzophenone in acetic acid, first at low temperature, then at r.t. According to the authors, there is a migration of the bromine atom during nitration [1196].

m.p. 208–209° [1195,1196]; Spectra (NA).

(5-Bromo-2,4-dihydroxy-3-nitrophenyl)phenylmethanone



$C_{13}H_8BrNO_5$ mol.wt. 338.11

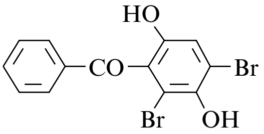
Synthesis

- Obtained by reaction of bromine with 2,4-dihydroxy-3-nitrobenzophenone in hot acetic acid solution then at r.t. for overnight [1196].

m.p. 110–111° [1196]; Spectra (NA).

(2,4-Dibromo-3,6-dihydroxyphenyl)phenylmethanone

[27065-46-9] $C_{13}H_8Br_2O_3$ mol.wt. 372.01



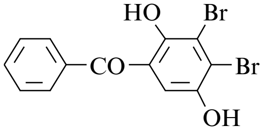
Syntheses

- Preparation by demethylation of 4,6-dibromo-2-hydroxy-5-methoxybenzophenone with aluminium chloride in boiling benzene for 10 min (72%) [571].
- Also obtained by saponification of 5-acetoxy-2-benzoyloxy- 4,6-dibromobenzophenone (SM) with potassium hydroxide in boiling ethanol for 1 h (72%) [1197]. SM was prepared in three steps: first, bromination of 5-hydroxy-2,3-diphenylbenzofuran (bromine/carbon tetrachloride), followed by acetylation of the 4,6-dibromo-5-hydroxy-2,3-diphenylbenzofuran so obtained (acetic anhydride/sodium acetate) and oxidation of the resulting 5-acetoxy-4,6-dibromo-2,3-diphenylbenzofuran (chromium trioxide/acetic acid).

m.p. 170–171° [1197], 170° [571]; Spectra (NA).

(3,4-Dibromo-2,5-dihydroxyphenyl)phenylmethanone

[154700-61-5] $C_{13}H_8Br_2O_3$ mol.wt. 372.01



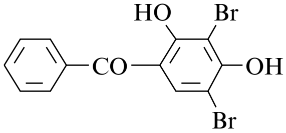
Synthesis

- Obtained by 10% Pd/C-catalyzed hydrogenolysis of *gem*-diphenylcyclopropane fused with 3,4,6-tribromo-benzoquinone in 1,4-dioxane in the presence of water (1%) for 6 h under atmospheric pressure at r.t. (33%) [1194].

m.p. (NA); ^{1}H NMR [1194].

(3,5-Dibromo-2,4-dihydroxyphenyl)phenylmethanone

[3286-96-2] $C_{13}H_8Br_2O_3$ mol.wt. 372.01



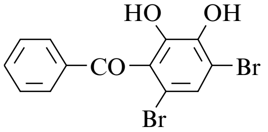
Syntheses

- Preparation by reaction of bromine with resbenzophenone in acetic acid [1195,1196], (44%) [1198].
- Also obtained by reaction of bromine with 3,3′-dibenzoyl-2,2′,6,6′-tetrahydroxydiphenyl thioether in acetic acid in a boiling water bath for 4 h. The same result was obtained from 3,3′-dibenzoyl-4,4′,6,6′-tetrahydroxydiphenyl thioether [1195].
- Also obtained by saponification of 4-acetoxy-2-benzoyloxy-3,5-dibromobenzophenone (SM) with potassium hydroxide in boiling ethanol for 1 h (73%). SM was prepared in three steps: first, bromination of 6-hydroxy-2,3-diphenylbenzofuran (bromine/carbon tetrachloride), followed by acetylation of the 5,7-dibromo-6-hydroxybenzofuran so obtained (acetic anhydride/sodium acetate) and oxidation of the resulting 6-acetoxy-5,7-dibromo-2,3-diphenylbenzofuran (chromium trioxide/acetic acid) [1197].

m.p. 151–152° [1198], 150–151° [1195,1196], 148–149° [1197];
Spectra (NA).

(4,6-Dibromo-2,3-dihydroxyphenyl)phenylmethanone

[65202-42-8] $C_{13}H_8Br_2O_3$ mol.wt. 372.01



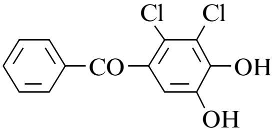
Synthesis

- Preparation by saponification of 3-acetoxy-2-(benzoyloxy)-4,6-dibromobenzophenone (SM) with potassium hydroxide in boiling ethanol (72%). SM was obtained by oxidation of 7-acetoxy-4,6-dibromo-2,3-diphenylbenzofuran with chromium trioxide in boiling acetic acid (70%) [579].

m.p. 205° [579]; Spectra (NA).

(2,3-Dichloro-4,5-dihydroxyphenyl)phenylmethanone

[103843-56-7] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



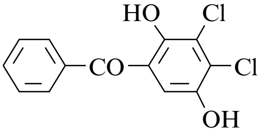
Synthesis

- Preparation by Friedel–Crafts acylation of 1,2-dichloro-3,4-dihydroxybenzene with benzoyl chloride in the presence of aluminium chloride in refluxing ethylene dichloride for 24 h (83%) [841,842].

m.p. 178–180° [841,842]; ^{1}H NMR [841], IR [841].

(3,4-Dichloro-2,5-dihydroxyphenyl)phenylmethanone

[21250-79-3] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



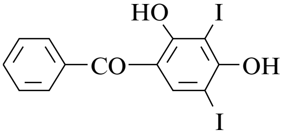
Syntheses

- Obtained by reaction of 2,3-dichloro-1,4-benzoquinone with benzaldehyde, either in the presence of benzoyl peroxide at 80° or in the absence of this one at 155° [840].
- Also obtained by 10% Pd/C-catalyzed hydrogenolysis of *gem*-diphenylcyclopropane fused with 3,4,6-trichloro-benzoquinone or with 6-bromo-3, 4-dichlorobenzoquinone in 1,4-dioxane in the presence of water (2%) for 2 h under atmospheric pressure at r.t. (59% and 75% yields, respectively) [1194].

m.p. (NA); ^{1}H NMR [1194].

(2,4-Dihydroxy-3,5-diiodophenyl)phenylmethanone

[33427-67-7] $C_{13}H_8I_2O_3$ mol.wt. 466.01



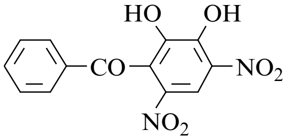
Syntheses

- Preparation by iodination of resbenzophenone,
  - with iodine and iodic acid in dilute ethanol for 1 h (62%) [460];
  - with iodine and potassium iodide in aqueous ammonia for 15 min [460];
  - with iodine monochloride in acetic acid for 2 h at r.t. [460].
- Also obtained from 2,4-dihydroxy-3,5-dinitrobenzophenone by methylation, reduction to 3,5-di-amino-2,4-dimethoxybenzophenone, subsequent diazotization and heating of the diazonium salt with potassium iodide, then demethylation of the 3,5-diiodo-2,4-dimethoxybenzophenone so formed [460].

m.p. 184–185° [460]; Spectra (NA).

(2,3-Dihydroxy-4,6-dinitrophenyl)phenylmethanone

[65202-37-1] $C_{13}H_8N_2O_7$ mol.wt. 304.22



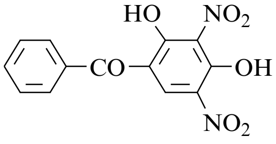
Synthesis

- Preparation by saponification of 3-acetoxy-2-(benzoyloxy)-4,6-dinitrobenzophenone (SM) with potassium hydroxide in refluxing ethanol (68%). SM was obtained by oxidation of 7-acetoxy-4,6-dinitro-2,3-diphenylbenzofuran with chromium trioxide in boiling acetic acid (81%) [579].

m.p. 264–265° [579]; Spectra (NA).

(2,4-Dihydroxy-3,5-dinitrophenyl)phenylmethanone

[27065-50-5] $C_{13}H_8N_2O_7$ mol.wt. 304.22



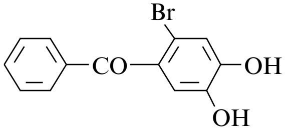
Syntheses

- Obtained by saponification of 4-acetoxy-2-benzoyloxy-3,5-dinitrobenzophenone (SM) with potassium hydroxide in boiling ethanol for 1 h (76%). SM was prepared in three steps: first, nitration of 6-hydroxy-2,3-diphenylbenzofuran (concentrated nitric acid/acetic acid), followed by acetylation of the 6-hydroxy-5,7-dinitro-2,3-diphenylbenzofuran so obtained (acetic anhydride/sodium acetate) and oxidation of the resulting 6-acetoxy-5,7-dinitro-2,3-diphenylbenzofuran (chromium trioxide/acetic acid) [1197].
- Preparation by nitration of resbenzophenone with 4 N nitric acid in the presence of sodium nitrite at r.t. for 8 days [460,1197], (60%) [227].

m.p. 187–188° [227], 185° [1197]; ^{1}H NMR [1197].

(2-Bromo-4,5-dihydroxyphenyl)phenylmethanone

[91197-10-3] $C_{13}H_9BrO_3$ mol.wt. 293.12



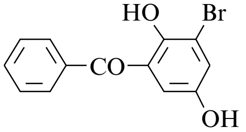
Synthesis

- Preparation by demethylation of 2-bromo-4,5-dimethoxy-benzophenone(SM)(m.p. 71–72°) with pyridinium chloride for 2 h at 180–200°. SM was obtained by reaction of benzoic acid with 1-bromo-3,4-dimethoxybenzene in the presence of phosphorous pentoxide in methanesulfonic acid for 30 min at 70° [1199].
- Also refer to: [1200].

brown oil [1199]; b.p. (NA); Spectra (NA).

(3-Bromo-2,5-dihydroxyphenyl)phenylmethanone

[112932-43-1] $C_{13}H_9BrO_3$ mol.wt. 293.12



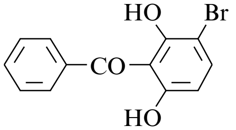
Synthesis

- Refer to: [1201,1202].

m.p. and Spectra (NA).

(3-Bromo-2,6-dihydroxyphenyl)phenylmethanone

$C_{13}H_9BrO_3$ mol.wt. 293.12



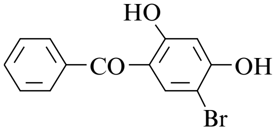
Syntheses

- Preparation by decarboxylation of 3-benzoyl-5-bromo-2,4-dihydroxybenzoic acid [1203], in the presence of concentrated hydrochloric acid in refluxing dilute acetic acid for 24 h (34%) [1204].
- Preparation by hydrolysis of 8-benzoyl-6-bromo-7-hydroxy-4-methylcoumarin [1204], in refluxing 10% sodium hydroxide for 2.5 h (49%) [1203].

m.p. 122–123° [1204], 122° [1203]; Spectra (NA).

(5-Bromo-2,4-dihydroxyphenyl)phenylmethanone

[3286-97-3] $C_{13}H_9BrO_3$ mol.wt. 293.12



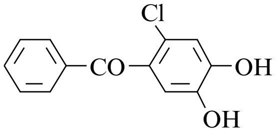
Syntheses

- Obtained by demethylation of 5-bromo-2-hydroxy-4-methoxybenzophenone with aluminium chloride in boiling benzene for 10 min (72%) [571].
- Preparation by reaction of bromine with 2,4-dihydroxy-benzophenone in acetic acid at r.t. overnight [1195,1196].
- Also refer to: [1198].

m.p. 148–149° [1195,1196], 148° [571]; Spectra (NA).

(2-Chloro-4,5-dihydroxyphenyl)phenylmethanone

[85525-22-0] $C_{13}H_9ClO_3$ mol.wt. 248.67



Syntheses

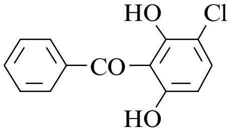
- Preparation by reaction of benzoyl chloride with 4-chloro-1,2-benzenediol in the presence of aluminium chloride at 140° for 4.5 h [1205].
- Preparation by total demethylation of 2-chloro-4,5-di-methoxybenzophenone (SM) with 48% hydrobromic acid in refluxing acetic acid for 17 h [1205]. SM was obtained by benzoylation of 4-chloro-1,2-dimethoxybenzene,

- with benzoyl chloride in the presence of iodine;
- with 2-pyridinyl benzoate in the presence of trifluoroacetic acid at 150° for 2 h in a sealed tube.

– Also refer to: [1199,1200].

m.p. 130–135° [1205]; Spectra (NA).

(3-Chloro-2,6-dihydroxyphenyl)phenylmethanone



$C_{13}H_9ClO_3$ mol.wt. 248.67

Syntheses

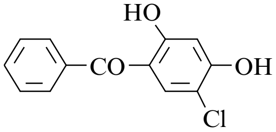
– Preparation by decarboxylation of 3-benzoyl-5-chloro-2,4-dihydroxybenzoic acid with concentrated hydrochloric acid in refluxing dilute acetic acid for 24 h [1203], (30%) [1204].

– Preparation by hydrolysis of 8-benzoyl-6-chloro-7-hydroxy-4-methylcoumarin with refluxing 10% aqueous sodium hydroxide or potassium hydroxide solution for 2.5 h [1204], (40%) [1203].

m.p. 119–120° [1203,1204]; Spectra (NA).

(5-Chloro-2,4-dihydroxyphenyl)phenylmethanone

[3286-95-1]



$C_{13}H_9ClO_3$ mol.wt. 248.67

Syntheses

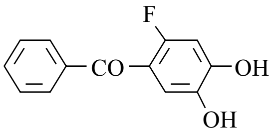
– Preparation by reaction of benzotrichloride with 4-chloro-resorcinol in 40% aqueous isopropanol solution at 70–80° (89%) [211].

– Preparation in two steps: first, chlorination of resorcinol dimethyl ether with sulfuryl chloride in chloroform, then Friedel–Crafts acylation of the 2,4-dimethoxychlorobenzene so obtained with benzoyl chloride in ethylene dichloride in the presence of aluminium chloride at r.t. for 3 h (81%) [478].

m.p. 142–143°5 [478], 142–143° [211]; ^{1}H NMR [478], IR [478], MS [478].

(2-Fluoro-4,5-dihydroxyphenyl)phenylmethanone

[85525-20-8]



$C_{13}H_9FO_3$ mol.wt. 232.21

Synthesis

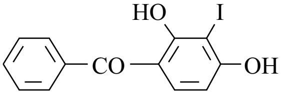
– Preparation by total demethylation of 2-fluoro-4,5-di-methoxybenzophenone (SM) with 48% hydrobromic acid in refluxing acetic acid for 17 h. SM was obtained on heating a mixture of 4-fluoro-1,2-dimethoxybenzene^T, 2-pyridinyl benzoate and trifluoroacetic acid at 100° for 4.5 h in a sealed tube [1205]. The starting dimethyl ether^T was prepared from 3,4-dimethoxyaniline by a Balz-Schiemann reaction [1206].

– Also refer to: [1200].

m.p. 169°5–171° [1205]; Spectra (NA).

(2,4-Dihydroxy-3-iodophenyl)phenylmethanone

[33427-62-2] $C_{13}H_9IO_3$ mol.wt. 340.12



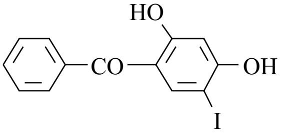
Synthesis

– Obtained by iodination of resbenzophenone with iodine and iodic acid in dilute ethanol for 30 min at r.t. (78%) [460].

m.p. 150–151° [460]; Spectra (NA).

(2,4-Dihydroxy-5-iodophenyl)phenylmethanone

[33427-72-4] $C_{13}H_9IO_3$ mol.wt. 340.12



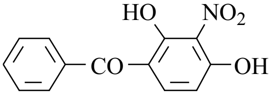
Synthesis

– Obtained from 2,4-dihydroxy-3,5-diiodobenzophenone in refluxing acetic acid for 8 h. There is an elimination of one iodine atom [460].

m.p. 151° [460]; Spectra (NA).

(2,4-Dihydroxy-3-nitrophenyl)phenylmethanone

[59746-91-7] $C_{13}H_9NO_5$ mol.wt. 259.22



Syntheses

– Preparation by reaction of benzotrichloride with 2-nitro-resorcinol in hydrofluoric acid in the presence of water at −10° for 4 h, then at r.t. overnight (98%) [213].

– Preparation by Fries rearrangement of 2-nitroresorcinol dibenzoate with aluminium chloride [1207],

  - in nitrobenzene at 100–110° for 3 h or at 25–28° for 70 h (42%);
  - without solvent, at 140° for 2 h (23%).

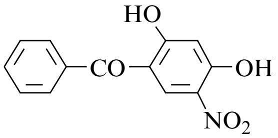
– Also obtained (poor yield) by reaction of concentrated nitric acid with resbenzophenone in acetic acid at r.t. for 1–2 h (6%) [1082].

– Also obtained (by-product) by reaction of benzoyl chloride with 2-nitroresorcinol in the presence of aluminium chloride in nitrobenzene in a boiling water bath for 1 h [1196].

m.p. 145° [1207], 144–145° [1196], 142° [213]; ^{1}H NMR [1082], IR [1082], MS [1082]; TLC [1082].

(2,4-Dihydroxy-5-nitrophenyl)phenylmethanone

[40990-79-2] $C_{13}H_9NO_5$ mol.wt. 259.22



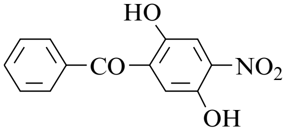
Syntheses

- Obtained by nitration of resbenzophenone,
  - with concentrated nitric acid in acetic acid at r.t. for 1–2 h (38%) [1082] or first at r.t., then at 60° [1195];
  - with 4 N nitric acid in the presence of sodium nitrite (trace) at r.t. for 8 days [1197];
  - also obtained by adding nitric acid (d = 1.4) to an ice cooled solution of resbenzophenone in acetic acid. Then, the ice bath was removed and the reaction stopped when the temperature reached 45° [1196].
- Preparation by refluxing hydrobromic acid with 2-hydroxy-4-methoxy-5-nitrobenzophenone for 3 h (61%) [571].
- Also obtained by reaction of nitric acid (d = 1.4) with 3,3′-dibenzoyl-4,4′,6,6′-tetrahydroxydiphenyl thioether in acetic acid at r.t. for 1 h [1195].

m.p. 144–145° [1195,1196], 143° [571]; ^{1}H NMR [1082], IR [1082], MS [1082]; TLC [1082].

(2,5-Dihydroxy-4-nitrophenyl)phenylmethanone

[40990-70-3] $C_{13}H_9NO_5$ mol.wt. 259.22



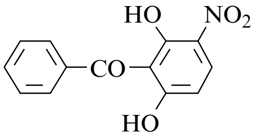
Syntheses

- Preparation by saponification of 5-(acetyloxy)-4-nitro-2-(p-nitrobenzoyloxy)benzophenone (SM) with potassium hydroxide in ethanol for 1 h (81%). SM was obtained by oxidation of 5-(acetyloxy)-6-nitro-2-(p-nitrophenyl)-3-phenylbenzofuran with chromium trioxide in refluxing acetic acid for 30 min [571].
- Preparation by demethylation of 2-hydroxy-5-methoxy-4-nitrobenzophenone with aluminium chloride in boiling benzene (62%) [571].

m.p. 167° [571]; Spectra (NA).

(2,6-Dihydroxy-3-nitrophenyl)phenylmethanone

$C_{13}H_9NO_5$ mol.wt. 259.22



Syntheses

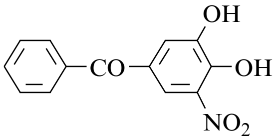
- Preparation by reaction of benzoic anhydride with 4-nitro-resorcinol in the presence of aluminium chloride in nitro-benzene on a steam bath for 3 h [1208].

- Preparation by nitration of 2,6-dihydroxybenzophenone with nitric acid (d = 1.42) at 0° for 10 min [1208].
- Preparation by Fries rearrangement,
  - of 4-nitroresorcinol 1-monobenzoate with aluminium chloride without solvent at 130–140° for 2 h (16%) or in nitrobenzene at 100° for 2 h (43%) [1209];
  - of 4-nitroresorcinol 3-monobenzoate that, under the above conditions, afforded the same mono-ketone [1209];
  - of 4-nitroresorcinol dibenzoate with aluminium chloride without solvent at 140° for 3 h or in nitrobenzene at 100° or 140° for 2 h [1209].

m.p. 159–160° [1209], 158° [1208]; Spectra (NA).

(3,4-Dihydroxy-5-nitrophenyl)phenylmethanone

[125628-96-8] $C_{13}H_9NO_5$ mol.wt. 259.22



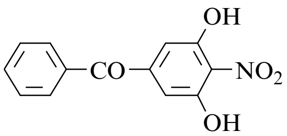
Synthesis

- Preparation by total demethylation of 3,4-dimethoxy-5-nitro-benzophenone with 48% hydrobromic acid in acetic acid at 110° for 30 h [1019].
- Also refer to: [1084,1210,1211].

m.p. 132° [1019]; Spectra (NA); pK_a [1084].

(3,5-Dihydroxy-4-nitrophenyl)phenylmethanone

[51787-06-5] $C_{13}H_9NO_5$ mol.wt. 259.22



Synthesis

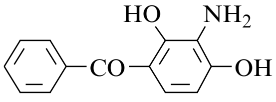
- Preparation from 3,5-dihydroxybenzophenone (and not from resorcinol 3-benzoate, as indicated in the paper) by nitration with aluminium nitrate in acetic acid for 4 h at r.t. (83%) [1212].

N.B.: This ketone was called 2-nitro-5-benzoyl resorcinol in the paper (table 1, compound **4**) [1212].

m.p. 121° [1212]; Spectra (NA).

(3-Amino-2,4-dihydroxyphenyl)phenylmethanone

[87119-03-7] $C_{13}H_{11}NO_3$ mol.wt. 229.24



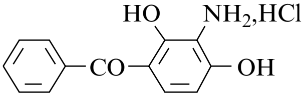
Synthesis

- Obtained from the corresponding hydrochloride described below [1082].

m.p. and Spectra (NA).

(3-Amino-2,4-dihydroxyphenyl)phenylmethanone *(Hydrochloride)*

[87119-04-8] $C_{13}H_{11}NO_3$,HCl mol.wt. 265.70



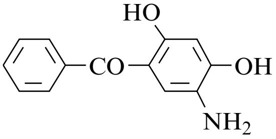
Synthesis

– Preparation by hydrogenation of 2,4-dihydroxy-3-nitro-benzophenone in chloroform/ethanol solution in the presence of 10% Pd/C, followed by treatment of the amino compound formed with concentrated hydrochloric acid in ethanol (61%) [1082].

m.p. 175–185° (d) [1082]; ^{1}H NMR [1082], IR [1082], MS [1082]; TLC [1082].

(5-Amino-2,4-dihydroxyphenyl)phenylmethanone

[87119-01-5] $C_{13}H_{11}NO_3$ mol.wt. 229.24



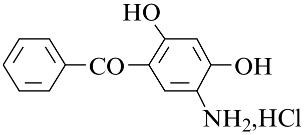
Synthesis

– Obtained from the corresponding hydrochloride described below [1082].

m.p. and Spectra (NA).

(5-Amino-2,4-dihydroxyphenyl)phenylmethanone *(Hydrochloride)*

[87119-02-6] $C_{13}H_{11}NO_3$,HCl mol.wt. 265.70



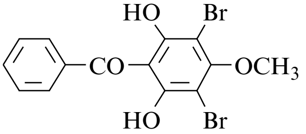
Synthesis

– Preparation by hydrogenation of 2,4-dihydroxy-5-nitro-benzophenone in chloroform/ethanol solution in the presence of 10% Pd/C, followed by treatment of the amino compound formed with concentrated hydrochloric acid in ethanol (85%) [1082].

m.p. 155–160° (d) [1082]; ^{1}H NMR [1082], IR [1082], MS [1082]; TLC [1082].

(3,5-Dibromo-2,6-dihydroxy-4-methoxyphenyl)phenylmethanone

$C_{14}H_{10}Br_2O_4$ mol.wt. 402.04



Synthesis

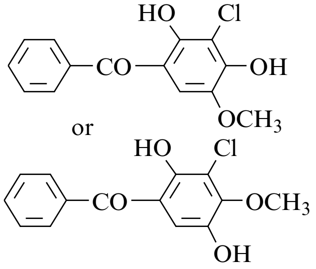
– Obtained by action of bromine with Cotoin in cooled chloroform [807].

N.B.: The formula proposed is the more likely.

m.p. 116° [807]; Spectra (NA).

[3-Chloro-2,4 (or 2,5)-dihydroxy-5 (or 4)-methoxyphenyl]phenylmethanone

[140708-51-6] $C_{14}H_{11}ClO_4$ mol.wt. 278.69



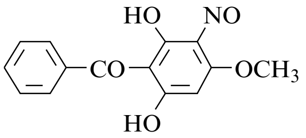
Synthesis

- Obtained by reaction of benzoyl chloride with 3-chloro-1,2,4-trimethoxybenzene in the presence of aluminium chloride in ethylene dichloride between 0° and 5°, then at r.t. for 8 h and at reflux for 1.5 h (58%) [704].

m.p. 181–182° [704]; ^{1}H NMR [704], IR [704], MS [704].

(2,6-Dihydroxy-4-methoxy-3-nitrosophenyl)phenylmethanone

$C_{14}H_{11}NO_5$ mol.wt. 273.25



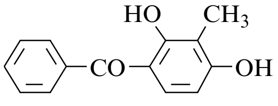
Synthesis

- Preparation by adding an aqueous solution of potassium nitrite to a solution of 2,6-dihydroxy-4-methoxy-benzophenone (so called Cotoin) in an acetic acid/ethanol mixture [808].

m.p. 153–154° [808]; Spectra (NA).

(2,4-Dihydroxy-3-methylphenyl)phenylmethanone

[52117-23-4] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

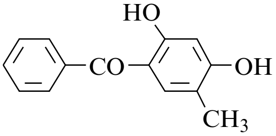
- Preparation by reaction of benzonitrile with 2,6-dihydroxy-toluene in the presence of zinc chloride and hydrochloric acid, followed by hydrolysis of the ketimine hydrochloride so formed with boiling water for 1 h (68%) (Hoesch reaction) [749].
- Preparation by Friedel–Crafts acylation of 3-methoxy-2-methylphenol with benzoyl chloride in the presence of aluminium chloride in boiling carbon disulfide for 1 h [749].
- Preparation by demethylation of 2-hydroxy-4-methoxy-3-methylbenzophenone with hydriodic acid in refluxing acetic anhydride (125–135°) for 2 h (63%) [750].
- Also obtained by methylation of resbenzophenone with methyl iodide in the presence of potassium hydroxide in refluxing methanol for 6 h [649] according to [751]. In this case, there is introduction of one methyl group on the benzene nucleus [649].
- Preparation by reaction of benzoic acid with 2-methylresorcinol in tetrachloroethane in the presence of boron trifluoride at 80° for 4 h (70%) [224].

- Also refer to: [221,597,805,867,1213–1217].

m.p. 177° [749,750], 176° [649], 173–174° [224]; ^{1}H NMR [224], UV [224].

(2,4-Dihydroxy-5-methylphenyl)phenylmethanone

[52220-71-0] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

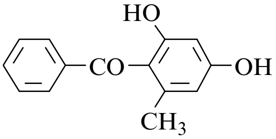
- Preparation by demethylation of 2-hydroxy-4-methoxy-5-methylbenzophenone with aluminium chloride in boiling benzene (62%) [752].
- Preparation by reaction of benzonitrile with 2,4-dihydroxy-toluene in the presence of zinc chloride and hydrochloric acid in ethyl ether, followed by hydrolysis of the ketimine hydrochloride so formed (65%) (Hoesch reaction) [805].
- Preparation by reaction of benzoyl chloride with 4-methylresorcinol in the presence of aluminium chloride [887].

N.B.: In the paper [234], the formula of the 2,4-dihydroxy-**6**-methylbenzophenone reported in the discussion (page 392, formula II) is correct since this product is prepared starting from orcinol. It must be pointed out that in the same communication, this compound has been erroneously named as the 2,4-dihydroxy-**5**-methylbenzophenone in the experimental part (page 394). The obtained compound is actually the 2,4-dihydroxy-**6**-methylbenzophenone already prepared by an other procedure [371].

m.p. 137°5–138° [805], 137° [752]; Spectra (NA).

(2,4-Dihydroxy-6-methylphenyl)phenylmethanone

[43221-40-5] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

- Preparation by Hoesch condensation of orcinol with benzonitrile (90%) [753], (50%) [371].
- Preparation by reaction of benzoyl chloride with orcinol in the presence of aluminium chloride in nitrobenzene at r.t. overnight, then heated in a water bath (temperature not quoted) for 1 h [234].

N.B.: In the paper [234], the formula of the 2,4-dihydroxy-**6**-methylbenzophenone reported in the discussion (page 392, formula II) is correct since this product is prepared starting from orcinol. It must be pointed out that in the same communication, this compound has been erroneously named as the 2, 4-di-hydroxy-**5**-methylbenzophenone in the experimental part (page 394). The obtained compound is actually the 2,4-dihydroxy-**6**-methylbenzophenone already prepared by an other procedure [371].

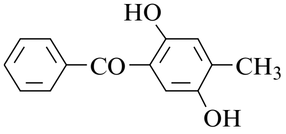
- Preparation from 3-methyl-3′-phenyl-5,5′-diisoxazolylmethane by performing hydrogenolysis and subsequent hydrolysis with hydrochloric acid (48%) [693].

- Orcinol by condensation with benzanilide imidochloride in the presence of aluminium chloride in ethyl ether gave a keto anil. This one was hydrolyzed by refluxing with ethanolic hydrochloric acid yielded the expected ketone (26%) [156].
- Also refer to: [1218].

m.p. 145–146° [753], 141° [156,371], 140° [693], 138° [234]; ^{1}H NMR [693,753], MS [693].

(2,5-Dihydroxy-4-methylphenyl)phenylmethanone

[59954-93-7] $C_{14}H_{12}O_3$ mol.wt. 228.25



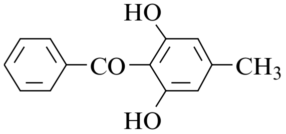
Syntheses

- Preparation by demethylation of,
  - 2,5-dimethoxy-4-methylbenzophenone on heating with pyridinium chloride for 20 min (95%) [726];
  - 2-hydroxy-5-methoxy-4-methylbenzophenone with aluminium chloride in boiling benzene for 10 min (65%) [752].

m.p. 152°5 [726], 152° [752]; Spectra (NA).

(2,6-Dihydroxy-4-methylphenyl)phenylmethanone

[68436-77-1] $C_{14}H_{12}O_3$ mol.wt. 228.25



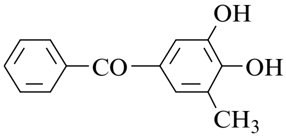
Syntheses

- Obtained by Fries rearrangement of orcinol dibenzoate with aluminium chloride at 160–170° for 90 min [253].
- Preparation by heating a mixture of benzoic acid, orcinol, zinc chloride and phosphorous oxychloride at 65° for 3 h [193].
- Also obtained by partial decarbonylation of 3-benzoyl-2,6-dihydroxy-4-methylbenzophenone by treatment with 85% sulfuric acid at r.t. for 4 h (quantitative yield) [253].
- Also obtained (poor yield) by Hoesch condensation of orcinol with benzonitrile (10%) [753].

m.p. 153–154° [753], 127° [253]; ^{1}H NMR [753].

(3,4-Dihydroxy-5-methylphenyl)phenylmethanone

[108055-13-6] $C_{14}H_{12}O_3$ mol.wt. 228.25



Synthesis

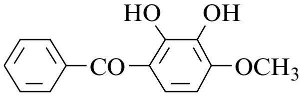
- Refer to: [1219].

m.p. and Spectra (NA).

(2,3-Dihydroxy-4-methoxyphenyl)phenylmethanone

(Alizarine Yellow A, monomethyl ether)

[35836-41-0] $C_{14}H_{12}O_4$ mol.wt. 244.25



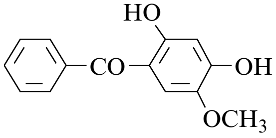
Syntheses

- Obtained by partial methylation of 2,3,4-trihydroxy-benzophenone,
  - with methyl iodide in the presence of potassium hydroxide in methanol at 100° for several hours [344] or in the presence of lithium carbonate in N,N-dimethylformamide at 30° for 15 h under nitrogen (30%) [369];
  - with dimethyl sulfate in the presence of alkali [379].
- Also obtained by reaction of methyl iodide with monosodium salt of 2,3,4-trihydroxy-benzophenone at 120° for several hours [344].
- Also obtained by adding a 5% sodium hydrogen carbonate solution to a 2,3-diacetoxy-4-methoxy-benzophenone solution in methanol and stirring at 30° for 2 h under nitrogen (25%) [369].
- Also refer to: [1220].

m.p. 172–174° [369], 165° [344], 164–165° [379]; ^{1}H NMR [369], ^{13}C NMR [369], UV [369], MS [369].

(2,4-Dihydroxy-5-methoxyphenyl)phenylmethanone

$C_{14}H_{12}O_4$ mol.wt. 244.25



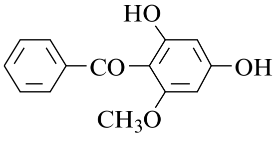
Synthesis

- Obtained by partial demethylation of 2,4,5-trimethoxy-benzophenone or 2-hydroxy-4,5-dimethoxybenzophenone with hydrobromic acid (d = 1.47) in acetic acid [761,762].

m.p. 183–185° [761,762]; Spectra (NA).

(2,4-Dihydroxy-6-methoxyphenyl)phenylmethanone *(Isocotoin)*

[81525-12-4] $C_{14}H_{12}O_4$ mol.wt. 244.25



Syntheses

- Preparation by reaction of benzonitrile with phloroglucinol monomethyl ether in the presence of zinc chloride and hydrochloric acid in ethyl ether, followed by hydrolysis of the ketimine hydrochloride so formed with boiling water for 15 min (good yield) (Hoesch reaction) [376,766].
- Preparation in one step involving the reaction of phlorobenzophenone with two mol of p-toluene-sulfonyl chloride in acetone in the presence of potassium carbonate followed by methylation with dimethyl sulfate and subsequent detosylation with ethanolic potassium hydroxide [373,685].
- Also refer to: [375].

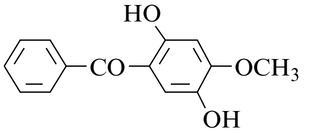
Isolation from natural sources

- From *Helichrysum triplinerve* (Asteraceae) [377].
- From *genus Leontonyx* [378].

m.p. 162° [376]; Spectra (NA).

(2,5-Dihydroxy-4-methoxyphenyl)phenylmethanone *(Cearoin)*

[52811-37-7] $C_{14}H_{12}O_4$ mol.wt. 244.25



Syntheses

- Obtained (poor yield) by reaction of benzoic acid with 2-methoxyhydroquinone in the presence of boron trifluoride-etherate, on heating at 100° for 30–45 min (5%) [650].
- Also obtained (poor yield) by oxidation of 2-hydroxy-4-methoxybenzophenone (Elbs reaction),
  - with lead tetraacetate in acetic acid at 100° for 5 h (3%) [650];
  - with potassium persulfate in aqueous potassium hydroxide (22%) [656].
- Preparation by oxidation of 2-hydroxy-4,5-dimethoxybenzophenone with nitric acid (d = 1.2) for 30 min at 15–20°, followed by reduction of the 2-benzoyl-5-methoxy-1,4-benzoquinone formed (93%) with sulfur dioxide in warm ethanol containing a drop of acetic acid for 1 h (79%) [763].
- Also refer to: [1221].

Isolation from natural sources

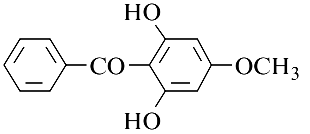
- From *Dalbergia cearensis* (Leguminosae) [760,1222–1224] and *Dalbergia miscolobium* [1222–1224].
- From *Dalbergia melanoxylon* Guill. et Perr. heartwood (Leguminosae-Lotoideae) [428].
- From the stem/bark of *Dalbergia volubilis* (Leguminosae) [1225].
- From the heartwood of *Dalbergia latifolia* Roxb. [1173].
- From the heartwood of *Dalbergia parviflora* Roxb. (Leguminosae) [1226].
- From *Dalbergia violacea* (Leguminosae) [1222].

m.p. 188–189° [763], 188° [656,1227], 187–188° [760], 187° [1225], 184°5–185°5 [1226], 183–185° [1228], 182–184° [428,1173], 182–183° [650];

^{1}H NMR [650,760,1225,1226], ^{13}C NMR [1226],
IR [650,760,1225,1226], UV [726 650], MS [760,1225,1226].

(2,6-Dihydroxy-4-methoxyphenyl)phenylmethanone *(Cotoin)*

[479-21-0] $C_{14}H_{12}O_4$ mol.wt. 244.25



Syntheses

- Obtained by reaction of benzonitrile with phloroglucinol monomethyl ether (Hoesch reaction) [376,685].

Also obtained by saponification of 2,6-diacetoxy-4-methoxybenzophenone with boiling aqueous potassium hydroxide solution for 15 min [807].

- Also refer to: [220,375,442,686,766,1229].

Isolation from natural sources

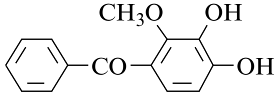
- From *Coto* bark (Lauraceae) [190,376,771,772,774,807,808,1230,1231];
- From the wood of *Aniba dukei* Kostermans (Lauraceae) [1232].

N.B.: In 1890 a careful quantitative analysis was kindly undertaken by Julius B. Cohen [771].

m.p. 131–132° [1232], 131° [376], 130–131° [774,807], 130° [771];
^{1}H NMR [675], IR [774,1232], UV [190].

(3,4-Dihydroxy-2-methoxyphenyl)phenylmethanone

[177703-29-6] $C_{14}H_{12}O_4$ mol.wt. 244.25



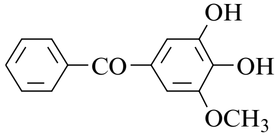
Synthesis

- Preparation by adding a 5% sodium bicarbonate solution to a 3,4-diacetoxy-2-methoxybenzophenone solution in methanol and stirring at 30° for 2 h under nitrogen (50%) [369].

m.p. 130–132° [369]; ^{1}H NMR [369], ^{13}C NMR [369], UV [369], MS [369]; pK_a [369].

(3,4-Dihydroxy-5-methoxyphenyl)phenylmethanone

$C_{14}H_{12}O_4$ mol.wt. 244.25



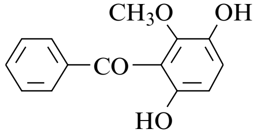
Synthesis

- Obtained by reaction of benzoyl chloride with 2,6-di-methoxyphenol in the presence of aluminium chloride in nitrobenzene, first at 2–3°, then at r.t. for 24 h (6–12% yield) [1233].

m.p. 168–169° [1233]; Spectra (NA).

(3,6-Dihydroxy-2-methoxyphenyl)phenylmethanone

[55137-06-9] $C_{14}H_{12}O_4$ mol.wt. 244.25



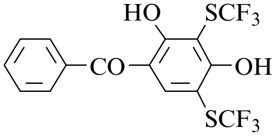
Synthesis

- Obtained from 2-benzoyl-1,4-benzoquinone [1234],
  - in refluxing methanol for 48 h without catalyst (26%);
  - in refluxing methanol for 12 h in the presence of zinc chloride (23%).

m.p. 141–143° [1234]; ^{1}H NMR [1234], IR [1234], UV [1234].

[2,4-Dihydroxy-3,5-bis[(trifluoromethyl)thio]phenyl]phenylmethanone

[66625-08-9] $C_{15}H_8F_6O_3S_2$ mol.wt. 414.35



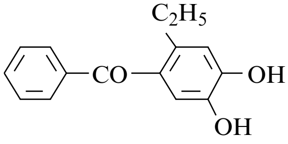
Synthesis

– Preparation by reaction of trifluoromethanesulfenyl chloride with 2,4-dihydroxybenzophenone in chloroform in the presence of a slight excess of pyridine, first at –40°, then 60° for 3 h (80%) [1235].

m.p. 91–95° [1235]; ^{1}H NMR [1235], IR [1235].

(2-Ethyl-4,5-dihydroxyphenyl)phenylmethanone

[91197-12-5] $C_{15}H_{14}O_3$ mol.wt. 242.27



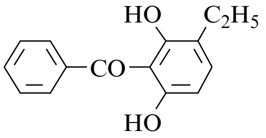
Synthesis

– Preparation by demethylation of 2-ethyl-4,5-dimethoxy-benzophenone (SM) with pyridinium chloride for 2 h at 180–200°. SM was obtained by reaction of benzoic acid with 1-ethyl-3,4-dimethoxybenzene in the presence of phosphorous pentoxide in methanesulfonic acid for 30 min at 70° [1199].

m.p. and Spectra (NA).

(3-Ethyl-2,6-dihydroxyphenyl)phenylmethanone

$C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

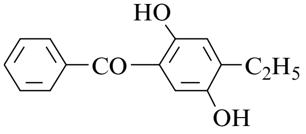
– Preparation from 8-benzoyl-6-ethyl-7-hydroxy-4-methyl-coumarin by action of,
  - a 20% aqueous sodium hydroxide solution at reflux for 2–2.5 h (51%) [1236];
  - a 10% aqueous sodium hydroxide solution at reflux for 4 h (40%) [1237].

– Also obtained by decarboxylation of 3-benzoyl-2,4-dihydroxy-5-ethylbenzoic acid with dilute hydrochloric acid (1:1) in a sealed tube at 160–170° [1238].

m.p. 128° [1237], 125–126° [1236], 125° [1238]; Spectra (NA).

(4-Ethyl-2,5-dihydroxyphenyl)phenylmethanone

[59623-16-4] $C_{15}H_{14}O_3$ mol.wt. 242.27



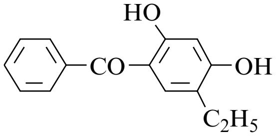
Synthesis

– Preparation by demethylation of 4-ethyl-2-hydroxy-5-methoxybenzophenone with aluminium chloride in boiling benzene for 10 min (55%) [370].

m.p. 86° [370]; Spectra (NA).

(5-Ethyl-2,4-dihydroxyphenyl)phenylmethanone

[50537-80-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

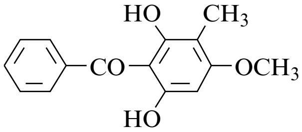
- Preparation by reaction of benzotrichloride with 4-ethyl-resorcinol in a 40% aqueous acetic acid solution at 70–80° (82%) [211].
- Preparation by Fries rearrangement of 4-ethylresorcinol dibenzoate with aluminium chloride in nitrobenzene at 50–60° for 3–4 h [1239].
- Preparation by reaction of benzoic acid with 4-ethylresorcinol,
  - in hydrofluoric acid at 100° in a stainless steel bomb (48%) [113];
  - in the presence of boron trifluoride in tetrachloroethane on a steam bath for 4 h [113].
- Preparation by demethylation of 5-ethyl-2-hydroxy-4-methoxybenzophenone with aluminium chloride in boiling benzene for 10 min (70%) [370].

m.p. 109° [370], 104° [113], 99°5–100°5 [211], 63–64° [1239]. One of the reported melting points is obviously wrong.

b.p.$_1$ 240–250° [113]; Spectra (NA).

(2,6-Dihydroxy-4-methoxy-3-methylphenyl)phenylmethanone

$C_{15}H_{14}O_4$ mol.wt. 258.27



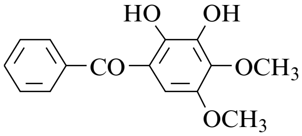
Synthesis

- Preparation by reaction of benzonitrile with 5-methoxy-4-methylresorcinol according to Hoesch method (50%) [805].

m.p. 143–144° [805]; Spectra (NA).

(2,3-Dihydroxy-4,5-dimethoxyphenyl)phenylmethanone

$C_{15}H_{14}O_5$ mol.wt. 274.27



Synthesis

- Refer to: [1221].

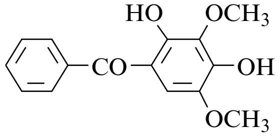
Isolation from natural source

- From *Machaerium scleroxylon* [1240].
- Also refer to: [416].

m.p. (NA) ^{1}H NMR [1240], IR [1240], UV [1240].

(2,4-Dihydroxy-3,5-dimethoxyphenyl)phenylmethanone

[42833-89-6] $C_{15}H_{14}O_5$ mol.wt. 274.27



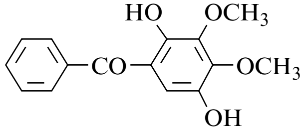
Synthesis

- Obtained by selective demethylation of 2-hydroxy-3,4,5-tri-methoxybenzophenone with refluxing aqueous piperidine for 45–65 h [416].

m.p. 182–184° [416]; UV [416], MS [416].

(2,5-Dihydroxy-3,4-dimethoxyphenyl)phenylmethanone *(Scleroin)*

[4646-78-0] $C_{15}H_{14}O_5$ mol.wt. 274.27



Synthesis

- Obtained (poor yield) by action of potassium persulfate with 2-hydroxy-3,4-dimethoxybenzophenone in the presence of ferrous sulfate in aqueous potassium hydroxide at r.t. for 3–4 h (10%) (Elbs reaction) [757].

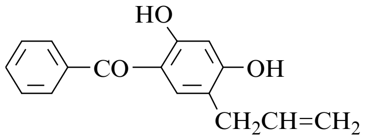
Isolation from natural source

- From *Machaerium scleroxylon* (Leguminosae) [675,1222,1224,1240].
- Also refer to: [416,1221,1241].

m.p. 143°5–144°5 [675], 140–142° [757]; ^{1}H NMR [675], IR [675], UV [675].

[2,4-Dihydroxy-5-(2-propenyl)phenyl]phenylmethanone

$C_{16}H_{14}O_3$ mol.wt. 254.29



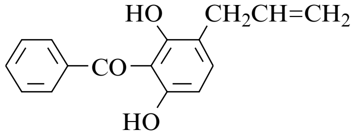
Synthesis

- Preparation by reaction of benzotrichloride with 4-allylresorcinol [211].

m.p. and Spectra (NA).

[2,6-Dihydroxy-3-(2-propenyl)phenyl]phenylmethanone

$C_{16}H_{14}O_3$ mol.wt. 254.29



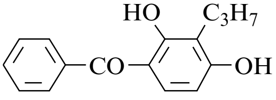
Synthesis

- Preparation from 6-allyl-8-benzoyl-4-methyl-umbelliferone (6-allyl-8-benzoyl-7-hydroxy-4-methyl-coumarin), by cleavage with boiling aqueous sodium hydroxide in the presence of sodium hydrosulfite under nitrogen for 3 h (90%) [269].

m.p. 80–81° [269]; Spectra (NA); TLC [269].

(2,4-Dihydroxy-3-propylphenyl)phenylmethanone

[79557-81-6] $C_{16}H_{16}O_3$ mol.wt. 256.30



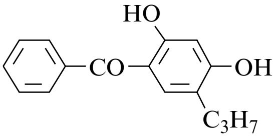
Synthesis

- Preparation by reaction of benzoic acid with 2-propyl-resorcinol in the presence of zinc chloride at 150° for 2.5 h (62%) [1242] (Nencki reaction).
- Also refer to: [168,597,1243].

m.p. 152–153° [1242]; Spectra (NA).

(2,4-Dihydroxy-5-propylphenyl)phenylmethanone

$C_{16}H_{16}O_3$ mol.wt. 256.30



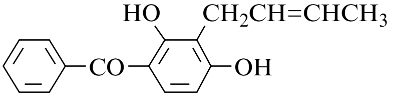
Synthesis

- Obtained by Fries rearrangement of 4-propylresorcinol dibenzoate with aluminium chloride in nitrobenzene at 50° for 4 h [1239].

m.p. 138–140° [1239]; b.p.$_9$ 240–245° [1239]; Spectra (NA).

[3-(2-Butenyl)-2,4-dihydroxyphenyl]phenylmethanone

[96836-08-7] $C_{17}H_{16}O_3$ mol.wt. 268.31



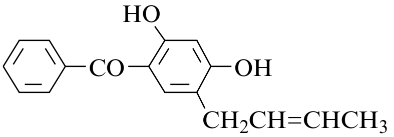
Synthesis

- Preparation by condensation of 2,4-dihydroxy-benzophenone with 1,3-butadiene in the presence of orthophosphoric acid in petroleum ether at 30–35° for 24 h (40%) [769].

m.p. 144–146° [769]; ^{1}H NMR [769].

[5-(2-Butenyl)-2,4-dihydroxyphenyl]phenylmethanone

[96859-90-4] $C_{17}H_{16}O_3$ mol.wt. 268.31



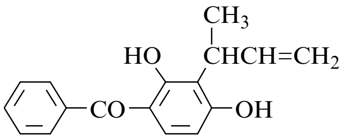
Synthesis

- Preparation by condensation of 2,4-dihydroxy-benzophenone with 1,3-butadiene in the presence of orthophosphoric acid in petroleum ether at 30–35° for 24 h (35%) [769].

m.p. 119–121° [769]; ^{1}H NMR [769].

[2,4-Dihydroxy-3-(1-methyl-2-propenyl)phenyl]phenylmethanone

[96836-07-6]



$C_{17}H_{16}O_3$ mol.wt. 268.31

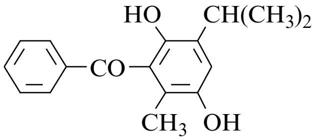
Synthesis

- Obtained (poor yield) by condensation of 2,4-di-hydroxybenzophenone with 1,3-butadiene in the presence of orthophosphoric acid in petroleum ether at 30–35° for 24 h (15%) [769].

m.p. 153–155° [769]; ^{1}H NMR [769].

[2,5-Dihydroxy-6-methyl-3-(1-methylethyl)phenyl]phenylmethanone

[101594-97-2]



$C_{17}H_{18}O_3$ mol.wt. 270.33

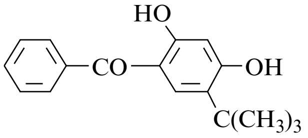
Synthesis

- Obtained by demethylation of 2-hydroxy-5-methoxy-6-methyl-3-isopropylbenzophenone or 3-hydroxy-6-methoxy-2-methyl-5-isopropylbenzophenone in refluxing pyridinium chloride for 20 min [726].

m.p. 147–147°5 [726]; Spectra (NA).

[5-(1,1-Dimethylethyl)-2,4-dihydroxyphenyl]phenylmethanone

[4211-67-0]



$C_{17}H_{18}O_3$ mol.wt. 270.33

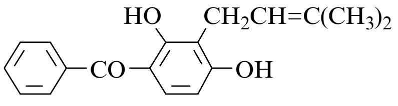
Synthesis

- Preparation by alkylation of resbenzophenone with isobutylene in benzene in the presence of p-toluenesulfonic acid [835].
- Also refer to: [1244–1246].

m.p. 141° [835,836];
Spectra (NA); gel chromatography [247].

[2,4-Dihydroxy-3-(3-methyl-2-butenyl)phenyl]phenylmethanone

[63565-04-8]



$C_{18}H_{18}O_3$ mol.wt. 282.34

Synthesis

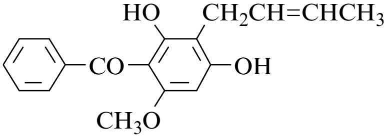
- Obtained (poor yield) by reaction of prenyl bromide with resbenzophenone,

- in the presence of sodium methoxide in refluxing methanol for 4 h (10%) [831];
- in the presence of boron trifluoride-etherate in dioxane at 60–70° for 2 h (<3%) [832].

m.p. 121° [831], 120° [832]; ^{1}H NMR [831], IR [831], UV [831].

[3-(2-Butenyl)-2,4-dihydroxy-6-methoxyphenyl]phenylmethanone

[96836-12-3]



$C_{18}H_{18}O_4$ mol.wt. 298.34

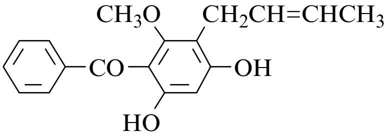
Synthesis

- Obtained by condensation of 2,4-dihydroxy-6-methoxybenzophenone with 1,3-butadiene in the presence of orthophosphoric acid in petroleum ether at 30–35° for 24 h (35%) [769].

m.p. 165–167° [769]; ^{1}H NMR [769].

[3-(2-Butenyl)-4,6-dihydroxy-2-methoxyphenyl]phenylmethanone

[96836-13-4]



$C_{18}H_{18}O_4$ mol.wt. 298.34

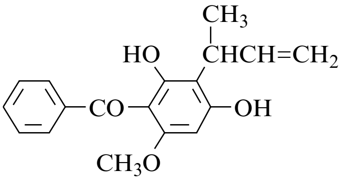
Synthesis

- Obtained by condensation of 2,4-dihydroxy-6-methoxybenzophenone with 1,3-butadiene in the presence of orthophosphoric acid in petroleum ether at 30–35° for 24 h (35%) [769].

m.p. 116–118° [769]; ^{1}H NMR [769].

[2,4-Dihydroxy-6-methoxy-3-(1-methyl-2-propenyl)phenyl]phenylmethanone

[96836-11-2]



$C_{18}H_{18}O_4$ mol.wt. 298.34

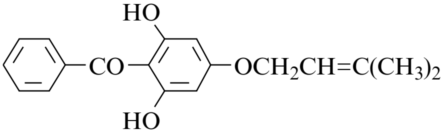
Synthesis

- Obtained (poor yield) by condensation of 2,4-di-hydroxy-6-methoxybenzophenone with 1,3-butadiene in the presence of orthophosphoric acid in petroleum ether at 30–35° for 24 h (15%) [769].

m.p. 117–119° [769]; ^{1}H NMR [769].

[2,6-Dihydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]phenylmethanone

[70219-83-9]



$C_{18}H_{18}O_4$ mol.wt. 298.34

Syntheses

- Obtained (trace) by reaction of 2-methyl-3-buten-2-ol with 2,4,6-trihydroxy-benzophenone in the presence of boron trifluoride-etherate in dioxane at 25–30° (<1%) [373].
- Also obtained (trace) by reaction of prenyl bromide with 2,4,6-trihydroxybenzophenone in the presence of sodium methoxide in refluxing methanol for 3 h (<1%) [374].

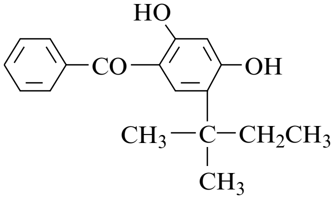
Isolation from natural source

- From *Leontonyx squarrosus* DC (Compositae) [378].

colourless oil [378]. This product is impure or in a metastable state.

m.p. 121° [373], 120–121° [374]; ^{1}H NMR [373,374,378], IR [373,374,378], UV [373,374], MS [378].

[2,4-Dihydroxy-5-(1,1-dimethylpropyl)phenyl]phenylmethanone



$C_{18}H_{20}O_3$ mol.wt. 284.36

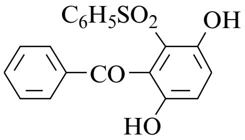
Synthesis

- Preparation by alkylation of resbenzophenone with 2-methylbutene in benzene in the presence of p-toluene-sulfonic acid [835].

m.p. 116° [835,836]; Spectra (NA).

[3,6-Dihydroxy-2-(phenylsulfonyl)phenyl]phenylmethanone

[145746-55-0]



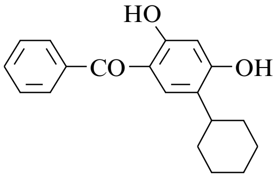
$C_{19}H_{14}O_5S$ mol.wt. 354.38

Synthesis

- Preparation by shaking an aqueous solution of sodium benzenesulfinate with a solution of benzoyl-1,4-benzo-quinone and trifluoroacetic acid in methylene chloride for 4 h at r.t. (85%) [1247].

m.p. 207–210° [1247]; ^{1}H NMR [1247], IR [1247], MS [1247].

(5-Cyclohexyl-2,4-dihydroxyphenyl)phenylmethanone



$C_{19}H_{20}O_3$ mol.wt. 296.37

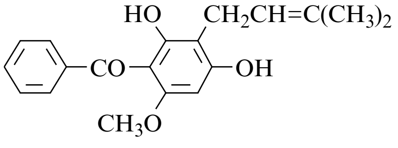
Synthesis

- Preparation by reaction of benzoic acid with 4-cyclohexyl-resorcinol in hydrofluoric acid at 100° in a stainless steel bomb [113].

m.p. 164° [113]; Spectra (NA).

[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]phenylmethanone

[81490-45-1]



$C_{19}H_{20}O_4$ mol.wt. 312.37

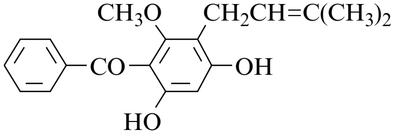
Synthesis

- Obtained by prenylation of 2, 4-dihydroxy-6-methoxybenzophenone (Isocotoin) with 2-methyl-3-buten-2-ol in the presence of boron trifluoride-etherate [685].

m.p. 160–161° [685]; ^{1}H NMR [685].

[4,6-Dihydroxy-2-methoxy-3-(3-methyl-2-butenyl)phenyl]phenylmethanone

[81490-46-2] $C_{19}H_{20}O_4$ mol.wt. 312.37



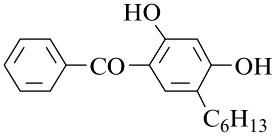
Synthesis

- Obtained by prenylation of 2,4-dihydroxy-6-methoxybenzophenone (Isocotoin) with 2-methyl-3-buten-2-ol in the presence of boron trifluoride-etherate [685].

m.p. 104–105° [685]; ^{1}H NMR [685].

(5-Hexyl-2,4-dihydroxyphenyl)phenylmethanone

[59746-92-8] $C_{19}H_{22}O_3$ mol.wt. 298.38



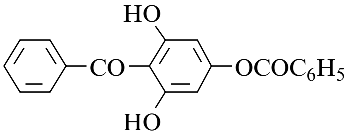
Syntheses

- Preparation by reaction of benzotrichloride with 4-hexyl-resorcinol in hydrofluoric acid in the presence of water at −10° for 4 h, then at r.t. overnight (80%) [213].
- Preparation by reaction of benzoic acid with 4-hexyl-resorcinol in the presence of boron trifluoride in tetrachloroethane on a steam bath for 4 h [113].
- Also refer to: [78].

m.p. 81–82° [113]; Spectra (NA).

[4-(Benzoyloxy)-2,6-dihydroxyphenyl]phenylmethanone

$C_{20}H_{14}O_5$ mol.wt. 334.33



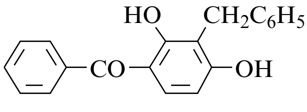
Synthesis

- Obtained by action of benzoyl chloride with phloro-benzophenone (2,4,6-trihydroxybenzophenone) in the presence of 1% potassium hydroxide aqueous solution at 0° (14%) [775].

m.p. 186° [775]; Spectra (NA).

[2,4-Dihydroxy-3-(phenylmethyl)phenyl]phenylmethanone

$C_{20}H_{16}O_3$ mol.wt. 304.35



Syntheses

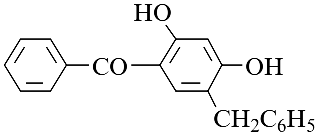
- Obtained (poor yield) by reaction of benzonitrile with 2-benzylresorcinol (8%) (Hoesch reaction) [846].
- Also obtained (poor yield) by hydrolysis of 3-benzyl-4-(benzyloxy)-2-hydroxybenzophenone (SM) with concentrated hydrochloric acid in boiling acetic acid for

2 h (2%). SM was obtained by reaction of benzyl chloride with resbenzophenone in the presence of potassium hydroxide in refluxing methanol for 5 h [846].

- Preparation by Friedel–Crafts acylation of 2-benzylresorcinol with benzoyl chloride in methylene chloride in the presence of aluminium chloride [168].

m.p. 159–160° [846]; Spectra (NA).

[2,4-Dihydroxy-5-(phenylmethyl)phenyl]phenylmethanone



$C_{20}H_{16}O_3$ mol.wt. 304.35

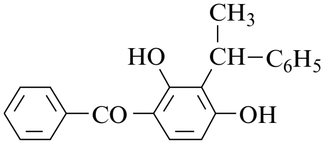
Synthesis

- Preparation by reaction of benzotrichloride with 4-benzyl-resorcinol [211].

m.p. and Spectra (NA).

[2,4-Dihydroxy-3-(1-phenylethyl)phenyl]phenylmethanone

[65221-07-0]



$C_{21}H_{18}O_3$ mol.wt. 318.37

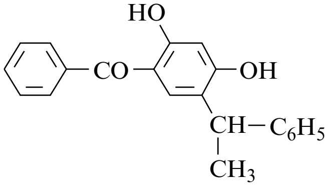
Synthesis

- Refer to: [233].

m.p. (NA); UV [233].

[2,4-Dihydroxy-5-(1-phenylethyl)phenyl]phenylmethanone

[43221-41-6]



$C_{21}H_{18}O_3$ mol.wt. 318.37

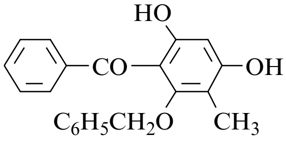
Synthesis

- Refer to: [233].

m.p. and Spectra (NA).

[4,6-Dihydroxy-3-methyl-2-(phenylmethoxy)phenyl]phenylmethanone

[74627-92-2]



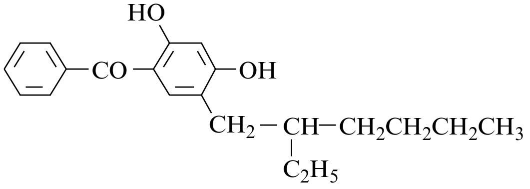
$C_{21}H_{18}O_4$ mol.wt. 334.37

Synthesis

- Preparation from 2-(benzyloxy)-4,6-dimethoxy-3-methyl-benzophenone by treatment with 10% hydrochloric acid in refluxing methanol for 40 min (89%) [838].

m.p. 155° [838]; ^{1}H NMR [838], MS [838].

[5-(2-Ethylhexyl)-2,4-dihydroxyphenyl]phenylmethanone



$C_{21}H_{26}O_3$ mol.wt. 326.44

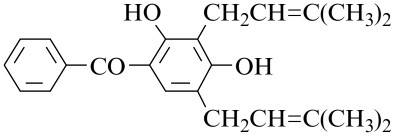
Synthesis

- Preparation by reaction of benzo-trichloride with 4-(2-ethylhexyl)-resorcinol [211].

m.p. and Spectra (NA).

[2,4-Dihydroxy-3,5-bis(3-methyl-2-butenyl)phenyl]phenylmethanone

[69443-76-1]



$C_{23}H_{26}O_3$ mol.wt. 350.46

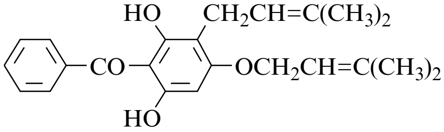
Synthesis

- Obtained (poor yield) by reaction of 2-methyl-3-buten-2-ol with resbenzophenone in the presence of boron trifluoride-etherate in dioxane at 60–70° for 2 h (3%) [832].

m.p. 74–75° [832]; ^{1}H NMR [832], IR [832], UV [832].

[2,6-Dihydroxy-3-(3-methyl-2-butenyl)-4-[(3-methyl-2-butenyl)oxy]phenyl]-phenylmethanone

[83611-01-2]



$C_{23}H_{26}O_4$ mol.wt. 366.46

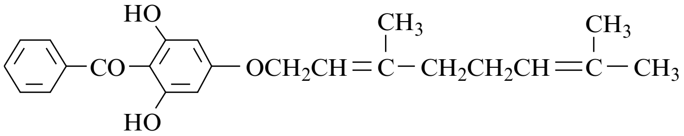
Synthesis

- Obtained (trace) by reaction of 2-methyl-3-buten-2-ol with 2,4,6-trihydroxy-benzophenone in the presence of boron trifluoride-etherate in dioxane at 25–30° (<1%) [373].

brown oil [373]; b.p. (NA); ^{1}H NMR [373], IR [373], UV [373].

[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2,6-dihydroxyphenyl]phenylmethanone *(E)*

[70219-85-1] $C_{23}H_{26}O_4$ mol.wt. 366.46



Synthesis

- Not yet described.

Isolation from natural source

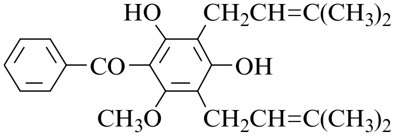
- From *Leontonyx spathulatus* Less. (Compositae) [377,378].

  colourless oil [378]; b.p. (NA); ^{1}H NMR [378], IR [378].

[2,4-Dihydroxy-6-methoxy-3,5-bis(3-methyl-2-butenyl)phenyl] phenylmethanone

(Vismiaphenone A)

[76444-61-6]



$C_{24}H_{28}O_4$ mol.wt. 380.48

Syntheses

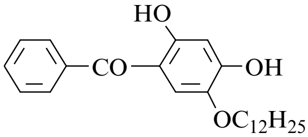
- Obtained by prenylation of 2,4-dihydroxy-6-methoxybenzophenone (Isocotoin) with 2-methyl-3-buten-2-ol in the presence of boron trifluoride-etherate [373,685].
- Also obtained first by methylation of 6-hydroxy-3,5-diprenyl-2,4-ditosyloxybenzophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 12 h. Then, the resulting compound was treated with 10% ethanolic potassium hydroxide for 2 h at 50–55° (17%) [373].

Isolation from natural source

- From the berries of *Vismia decipiens* Schlecht-Cham. (Guttiferae) [1248].

  oil [373,685,1248]; b.p. (NA); ^{1}H NMR [373,685,1248], IR [373,685,1248], UV [373,685,1248], MS [1248].

[2,4-Dihydroxy-5-(dodecyloxy)phenyl]phenylmethanone



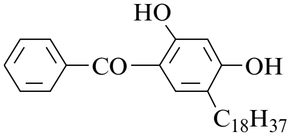
$C_{25}H_{34}O_4$ mol.wt. 398.54

Synthesis

- Refer to: [78].

m.p. and Spectra (NA).

(2,4-Dihydroxy-5-octadecylphenyl)phenylmethanone



$C_{31}H_{46}O_3$ mol.wt. 466.70

Synthesis

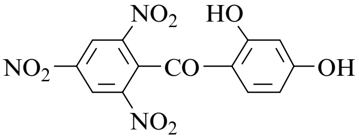
- Preparation by reaction of benzotrichloride with 4-stearyl-resorcinol [211].

m.p. and Spectra (NA).

2.2.1.2 Substituents Located on the Other Ring

(2,4-Dihydroxyphenyl)(2,4,6-trinitrophenyl)methanone

[188347-38-8]



$C_{13}H_7N_3O_9$ mol.wt. 349.21

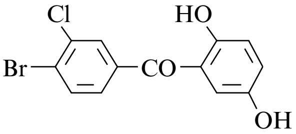
Synthesis

- Refer to: [1249] (compound **51**) NSC 338104 (no comments).

m.p. and Spectra (NA).

(4-Bromo-3-chlorophenyl)(2,5-dihydroxyphenyl)methanone

[161463-54-3]



$C_{13}H_8BrClO_3$ mol.wt. 327.56

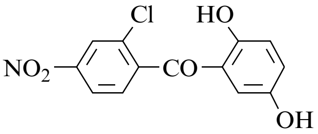
Synthesis

- Preparation by demethylation of 4-bromo-3-chloro-2′,5′-di-methoxybenzophenone (SM) with boron tribromide in methylene chloride at 0° for 15–17 h (70–95%). SM was prepared by reaction of 4-bromo-3-chlorobenzoic acid with 1,4-dimethoxybenzene in the presence of polyphosphoric acid at 60–70° for 6–7 h (40–83%) [1250].

m.p. (NA); ^{1}H NMR [1250], IR [1250].

(2-Chloro-4-nitrophenyl)(2,5-dihydroxyphenyl)methanone

[37884-01-8]



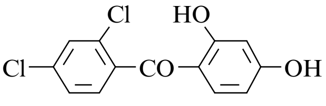
$C_{13}H_8ClNO_5$ mol.wt. 293.66

Synthesis

- Preparation by demethylation of 2-chloro-2′,5′-di-methoxy-4-nitrobenzophenone with an excess of boiling pyridinium chloride for 6 h (40%) [1073].

m.p. 265° [1073]; Spectra (NA).

(2,4-Dichlorophenyl)(2,4-dihydroxyphenyl)methanone



$C_{13}H_8Cl_2O_3$ mol.wt. 283.11

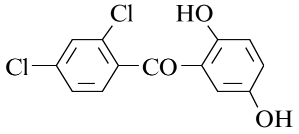
Synthesis

- Preparation by reaction of 2,4-dichlorobenzotrichloride with resorcinol [211], in hydrofluoric acid in the presence of water at −10°, then at 15° overnight and at 80° for 30 min [213].

m.p. and Spectra (NA).

(2,4-Dichlorophenyl)(2,5-dihydroxyphenyl)methanone

[37884-00-7] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



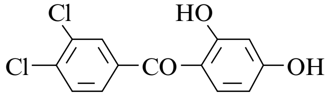
Syntheses

- Preparation by reaction of 2,4-dichlorobenzotrichloride with hydroquinone in hydrofluoric acid in the presence of water at −10°, then at 15° overnight and at 80° for 30 min [213].
- Also obtained by condensation reaction of hydroquinone dimethyl ether with excess of 2,4-dichlorobenzoic acid in the presence of polyphosphoric acid [1251].
- Preparation by demethylation of 2,4-dichloro-2′,5′-dimethoxybenzophenone,
  - with boiling excess pyridinium chloride for 1 h (95%) [1073];
  - with boron tribromide in methylene chloride at 0° (77%) [1251].

m.p. 145° [1073], 126° [1251]; ^{1}H NMR [1251], IR [1251].

(3,4-Dichlorophenyl)(2,4-dihydroxyphenyl)methanone

[36419-34-8] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



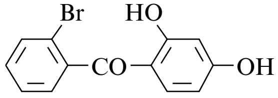
Synthesis

- Preparation by reaction of 3,4-dichlorobenzoic acid with resorcinol in the presence of boron trifluoride in tetrachloroethane on a steam bath for 4 h [113].
- Refer to: [235,1252].

m.p. 188° [113], 186° [235]; UV [113,235,1252].

(2-Bromophenyl)(2,4-dihydroxyphenyl)methanone

$C_{13}H_9BrO_3$ mol.wt. 293.12



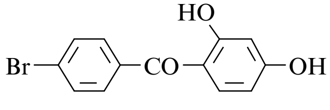
Synthesis

- Preparation by reaction of o-bromobenzotrichloride with resorcinol [211].

m.p. and Spectra (NA).

(4-Bromophenyl)(2,4-dihydroxyphenyl)methanone

[3286-88-2] $C_{13}H_9BrO_3$ mol.wt. 293.12

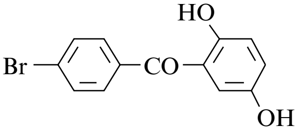


Synthesis

- Preparation by reaction of p-bromobenzonitrile with resorcinol in the presence of zinc chloride and hydrochloric acid in ethyl ether in an ice bath for 1 or 2 days, followed by hydrolysis of the ketimine hydrochloride so formed with boiling water (64%) [1253], (50%) [439].
- Also refer to: Chem. Abstr., **127**, 108921f (1997).

m.p. 169° [1253], 164° [439]; Spectra (NA).

(4-Bromophenyl)(2,5-dihydroxyphenyl)methanone



$C_{13}H_9BrO_3$ mol.wt. 293.12

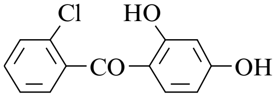
Synthesis

- Preparation by condensation of p-bromobenzoic acid with hydroquinone in the presence of aluminium chloride and sodium chloride at 180–200° for 2 min (32%) [129].

m.p. 153° [129]; Spectra (NA).

(2-Chlorophenyl)(2,4-dihydroxyphenyl)methanone

[50685-40-0]



$C_{13}H_9ClO_3$ mol.wt. 248.67

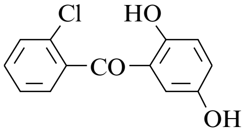
Syntheses

- Preparation by reaction of o-chlorobenzoyl chloride with O,O-bis(trimethylsilyl)resorcinol in the presence of stannic chloride in refluxing methylene chloride for 2 h (80%) [921].
- Preparation by Fries rearrangement of m-methoxyphenyl o-chlorobenzoate with aluminium chloride without solvent at 180° (40%) [155].
- Preparation by reaction of o-chlorobenzotrichloride with resorcinol in dilute isopropanol at 70–80° (85%) [211].
- Preparation by condensation of resorcinol and o-chlorobenzoic acid with boron trifluoride-etherate in carbon tetrachloride [1254], according to [360].

m.p. 138° [155], 135°5–136°5 [211], 131–132° [921]; Spectra (NA).

(2-Chlorophenyl)(2,5-dihydroxyphenyl)methanone

[37883-99-1]



$C_{13}H_9ClO_3$ mol.wt. 248.67

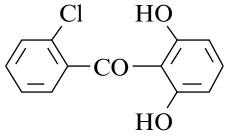
Syntheses

- Preparation by reaction of o-chlorobenzoyl chloride with O,O-bis(trimethylsilyl)hydroquinone in the presence of stannic chloride in refluxing methylene chloride for 2 h (84%) [921].
- Preparation by demethylation of 2′-chloro-2,5-dimethoxy-benzophenone with an excess of boiling pyridinium chloride for 1 h (63%) [1073].
- Also obtained by photochemical addition of o-chlorobenzaldehyde to 1,4-benzoquinone in benzene in the presence of benzophenone for 5 days (78%) [1255].

m.p. 160° [1073]; Spectra (NA).

(2-Chlorophenyl)(2,6-dihydroxyphenyl)methanone

[100334-93-8] $C_{13}H_9ClO_3$ mol.wt. 248.67



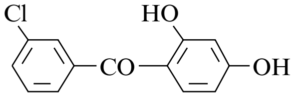
Synthesis

- Preparation by hydrolysis of 8-(o-chlorobenzoyl)-7-hydroxy-4-methylcoumarin (SM) with sodium hydroxide in refluxing dilute ethanol for 3 h (72%).SM was obtained by Fries rearrangement of 7-(o-chlorobenzoyloxy)-4-methylcoumarin with aluminium chloride, first at 180°, then at 185–190° for 1.5 h (65%, m.p. 165°). -Refer to: Chem. Abstr., **114**, 42490n (1991)^{T}.

m.p. 140$^{\circ T}$; IR^{T}.

(3-Chlorophenyl)(2,4-dihydroxyphenyl)methanone

$C_{13}H_9ClO_3$ mol.wt. 248.67



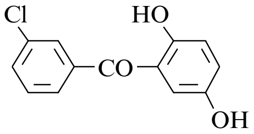
Synthesis

- Preparation by reaction of m-chlorobenzonitrile with resorcinol (52%) (Hoesch reaction) [1256].

m.p. 197-197°5 [1256]; Spectra (NA).

(3-Chlorophenyl)(2,5-dihydroxyphenyl)methanone

[161463-59-8] $C_{13}H_9ClO_3$ mol.wt. 248.67



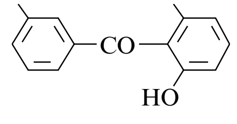
Synthesis

- Preparation by demethylation of 3′-chloro-2,5-dimethoxy-benzophenone (SM) with boron tribromide in methylene chloride at 0° for 15–17 h (70–95%). SM was prepared by reaction of m-chlorobenzoic acid with 1,4-dimethoxy-benzene in the presence of polyphosphoric acid at 60–70° for 6–7 h (40–83%) [1250].

m.p. (NA); ^{1}H NMR [1250], IR [1250].

(3-Chlorophenyl)(2,6-dihydroxyphenyl)methanone

[131425-89-3] $C_{13}H_9ClO_3$ mol.wt. 248.67



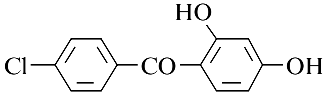
Synthesis

- Preparation by hydrolysis of 8-(m-chlorobenzoyl)-7-hydroxy-4-methylcoumarin (SM) with sodium hydroxide in refluxing dilute ethanol for 3 h (67%). SM was obtained by Fries rearrangement of 7-(m-chlorobenzoyloxy)-4-methylcoumarin with aluminium chloride, first at 185°,
- then at 190–195° for 1 h (64%, m.p. 245°).
- Refer to: Chem. Abstr., **114**, 42490n (1991)^{T}.

m.p. 150$^{\circ T}$; IR^{T}.

(4-Chlorophenyl)(2,4-dihydroxyphenyl)methanone

[18239-10-6] $C_{13}H_9ClO_3$ mol.wt. 248.67



Syntheses

– Obtained by reaction of p-chlorobenzonitrile with resorcinol in the presence of zinc chloride and hydrochloric acid in ethyl ether (Hoesch reaction) in ice during 24 h and hydrolysis of 4′-chloro-2,4-dihydroxybenzophenone imide hydrochloride so formed (72%) with boiling water for 30 min (46%) [1253], (39%) [1256].

– Also obtained by reaction of p-chlorobenzoic acid with resorcinol,

- in hydrofluoric acid at 100° in a stainless steel bomb [113];
- in the presence of Zeolite-H-beta in refluxing p-chlorotoluene (162°) for 7 h, with stirring and azeotropic removal of water (4%) [53].

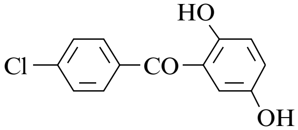
– Preparation by reaction of 4-chlorobenzotrichloride with resorcinol in 40% aqueous isopropanol solution at 70–80° (95%) [211].

– Also refer to: [235,778,1252,1257].

m.p. 155° [1253], 152° [235], 151–152° [1256], 151° [113], 150° [215]; 149°5–150°5 [211]; UV [113,215,235].

(4-Chlorophenyl)(2,5-dihydroxyphenyl)methanone

[91290-75-4] $C_{13}H_9ClO_3$ mol.wt. 248.67



Syntheses

– Preparation by demethylation of 4′-chloro-2,5-dimethoxy-benzophenone (SM) with boron tribromide in methylene chloride,

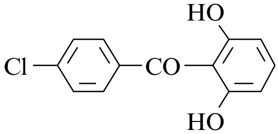
- at 0° (95%) [1251];
- first at −30° for 30 min, then at 22° for 5 h. SM was obtained by Friedel–Crafts acylation of hydroquinone dimethyl ether with p-chlorobenzoyl chloride in methylene chloride in the presence of aluminium chloride at 0° for 8 h (85%) [1258].

– Also obtained by photochemical addition of p-chlorobenzaldehyde to 1,4-benzoquinone in benzene in the presence of benzophenone for 5 days (73%) [1255].

m.p. 132° [1251]; ^1H NMR [1251], IR [1251].

(4-Chlorophenyl)(2,6-dihydroxyphenyl)methanone

[29627-01-8] $C_{13}H_9ClO_3$ mol.wt. 248.67



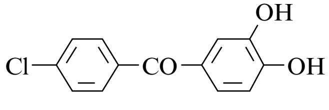
Synthesis

– Preparation by hydrolysis of 8-(p-chlorobenzoyl)-7-hydroxy-4-methylcoumarin (SM) with sodium hydroxide in refluxing dilute ethanol for 3 h (71%). SM was obtained by Fries rearrangement of 7-(p-chlorobenzoyloxy)-4-methylcoumarin with aluminium chloride, first at 180°, then at 185–190° for 1.5 h (60%, m.p. 214°). -Refer to: Chem. Abstr., **114**, 42490n (1991)^{T}.

m.p. 130°^{T}; IR^{T}.

(4-Chlorophenyl)(3,4-dihydroxyphenyl)methanone

[134612-84-3] $C_{13}H_9ClO_3$ mol.wt. 248.67



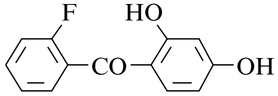
Syntheses

- Preparation by demethylation of 4′-chloro-4-hydroxy-3-methoxybenzophenone with hydrobromic acid in refluxing aqueous acetic acid [1019].
- Preparation by Friedel–Crafts reaction of p-chlorobenzoyl chloride with veratrole [900].

m.p. 190° [900], 174–176° [1019]; Spectra (NA).

(2,4-Dihydroxyphenyl)(2-fluorophenyl)methanone

[19390-38-6] $C_{13}H_9FO_3$ mol.wt. 232.21



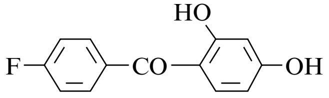
Synthesis

- Preparation by reaction of 2-fluorobenzoyl chloride with resorcinol dimethyl ether in the presence of aluminium chloride in ethylene dichloride at 60° for 90 min [476], (59%) [1031].

m.p. 112–114° [1031], 109–111° [476]; Spectra (NA).

(2,4-Dihydroxyphenyl)(4-fluorophenyl)methanone

[84794-97-8] $C_{13}H_9FO_3$ mol.wt. 232.21



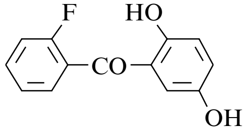
Synthesis

- Preparation by Friedel–Crafts acylation of resorcinol with p-fluorobenzoyl chloride in the presence of aluminium chloride in ethylene dichloride at 50° for 1.5 h (90%) [1109].
- Also refer to: [1259].

m.p. 171–172° [1109]; Spectra (NA).

(2,5-Dihydroxyphenyl)(2-fluorophenyl)methanone

[176547-98-1] $C_{13}H_9FO_3$ mol.wt. 232.21



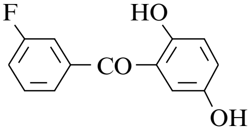
Synthesis

- Preparation by total demethylation of 2′-fluoro-2,5-di-methoxybenzophenone (SM) with boron tribromide in methylene chloride, first at −70°, then at 25° for 24 h (86%). SM was obtained by Friedel–Crafts acylation of hydroquinone dimethyl ether with o-fluorobenzoyl chloride in methylene chloride in the presence of aluminium chloride at 0° for 4 h (79%) [258].

m.p. 118–119° [258]; ^{1}H NMR [258].

(2,5-Dihydroxyphenyl)(3-fluorophenyl)methanone

[161463-61-2] $C_{13}H_9FO_3$ mol.wt. 232.21



Synthesis

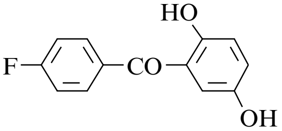
– Preparation by demethylation of 3′-fluoro-2,5-dimethoxy-benzophenone (SM) with boron tribromide in methylene chloride at 0° for 15–17 h (70–95%) [1250] or at 22° [258]. SM was prepared from hydroquinone dimethyl ether,

- by reaction with m-fluorobenzoic acid in the presence of polyphosphoric acid at 60–70° for 6–7 h (40–83%) [1250];
- by reaction with m-fluorobenzoyl chloride in methylene chloride in the presence of aluminium chloride at 0° (78%) [258].

m.p. (NA); ^{1}H NMR [1250], IR [1250].

(2,5-Dihydroxyphenyl)(4-fluorophenyl)methanone

[83235-21-6] $C_{13}H_9FO_3$ mol.wt. 232.21



Syntheses

– Preparation by total demethylation of 4′-fluoro-2,5-di-methoxybenzophenone (SM) with boron tribromide in methylene chloride, first at −30° for 30 min, then at 22° for 5 h (80%). SM was obtained by Friedel–Crafts acylation of hydroquinone dimethyl ether with p-fluorobenzoyl chloride in methylene chloride in the presence of aluminium chloride at 0° for 8 h (90%) [258,1258].

– Also obtained by photochemical addition of p-fluorobenzaldehyde to 1,4-benzoquinone in benzene in the presence of benzophenone for 5 days (61%) [1255].

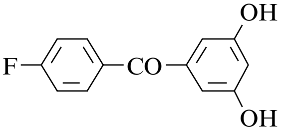
– Also obtained by UV light irradiation of α-hydroxy(p-fluorobenzyl)-1,4-benzoquinone in benzene for 72 h (45%) [1260].

– Also refer to: [261].

m.p. 140–141° [1258,1260]; ^{1}H NMR [1258,1260], IR [1260].

(3,5-Dihydroxyphenyl)(4-fluorophenyl)methanone

[148253-51-4] $C_{13}H_9FO_3$ mol.wt. 232.21



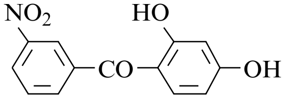
Synthesis

– Preparation by total demethylation of 3, 5-dimethoxy-4′-fluorobenzophenone (SM) with 48% hydrobromic acid in refluxing acetic acid under nitrogen for 20 h (91%) [905,906].

SM was obtained by oxidation of 3,5-dimethoxy-4′-fluorobenzhydrol with pyridinium chlorochromate in the presence of sodium acetate in methylene chloride at r.t. under nitrogen for 30 min (84%) [905].

m.p. 142–143° [905,906]; ^{1}H NMR [905,906], ^{13}C NMR [905,906], IR [905,906], MS [905,906].

(2,4-Dihydroxyphenyl)(3-nitrophenyl)methanone



$C_{13}H_9NO_5$ mol.wt. 259.22

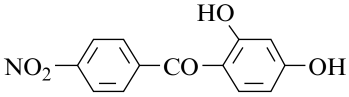
Syntheses

- Obtained by reaction of m-nitrobenzonitrile with resorcinol in the presence of zinc chloride and hydrochloric acid in ethyl ether, followed by hydrolysis of the resulting ketimine hydrochloride in boiling water (73%) [215], for 30 min [1261], (11%) [1262] (Hoesch reaction).
- Preparation by reaction of m-nitrobenzotrichloride with resorcinol [211].

m.p. 228° [215,1261,1262]; Spectra (NA).

(2,4-Dihydroxyphenyl)(4-nitrophenyl)methanone

[6994-40-7]



$C_{13}H_9NO_5$ mol.wt. 259.22

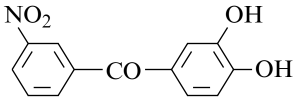
Synthesis

- Preparation by reaction of p-nitrobenzonitrile with resorcinol in the presence of zinc chloride and hydrochloric acid in ethyl ether, followed by hydrolysis of the ketimine hydrochloride so formed with boiling water for 30 min [439,1110,1261], (17%) [1262] (Hoesch reaction).

m.p. 203° [1261,1262], 200° [439]; Spectra (NA).

(3,4-Dihydroxyphenyl)(3-nitrophenyl)methanone

[203060-36-0]



$C_{13}H_9NO_5$ mol.wt. 259.22

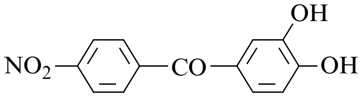
Synthesis

- Refer to: Chem. Abstr., **128**, 181082h (1998).

m.p. and Spectra (NA).

(3,4-Dihydroxyphenyl)(4-nitrophenyl)methanone

[203060-35-9]



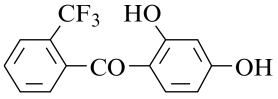
$C_{13}H_9NO_5$ mol.wt. 259.22

Synthesis

- Refer to: Chem. Abstr., **128**, 181082h (1998).

m.p. and Spectra (NA).

(2,4-Dihydroxyphenyl)[2-(trifluoromethyl)phenyl]methanone



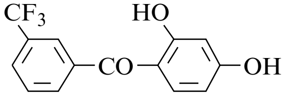
$C_{14}H_9F_3O_3$ mol.wt. 282.22

Synthesis

- Preparation by reaction of o-(trifluoromethyl) benzoyl chloride with resorcinol in the presence of aluminium chloride in refluxing carbon disulfide for 20 h [239].

m.p. 168–168°5 [239]; Spectra (NA).

(2,4-Dihydroxyphenyl)[3-(trifluoromethyl)phenyl]methanone



$C_{14}H_9F_3O_3$ mol.wt. 282.22

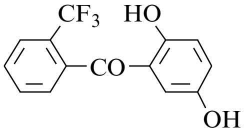
Synthesis

- Preparation by reaction of m-(trifluoromethyl) benzoyl chloride with resorcinol in the presence of aluminium chloride in refluxing carbon disulfide for 20 h [239].

m.p. 175°5–176° [239]; Spectra (NA).

(2,5-Dihydroxyphenyl)[2-(trifluoromethyl)phenyl]methanone

[161463-62-3]



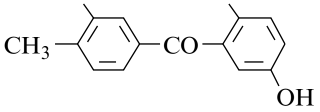
$C_{14}H_9F_3O_3$ mol.wt. 282.22

Synthesis

- Preparation by demethylation of 2,5-dimethoxy-2′-(tri-fluoromethyl)benzophenone (SM) with boron tribromide at 0° for 15–17 h (70–95%). SM was prepared by reaction of 2-(trifluoromethyl)benzoic acid with 1,4-dimethoxybenzene in the presence of polyphosphoric acid at 60–70° for 6–7 h (40–83%) [1250].

m.p. (NA); ^{1}H NMR [1250], IR [1250].

(3-Chloro-4-methylphenyl)(2,5-dihydroxyphenyl)methanone



$C_{14}H_{11}ClO_3$ mol.wt. 262.69

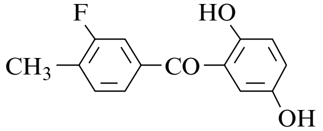
Synthesis

- Preparation by demethylation of 3′-chloro-2, 5-dimethoxy-4′-methylbenzophenone (SM) with boron tribromide in methylene chloride at 0° for 15–17 h (70–95%). SM was prepared by reaction of 3-chloro-4-methylbenzoic acid with 1,4-dimethoxybenzene in the presence of polyphosphoric acid at 60–70° for 6–7 h (40–83%) [1250].

m.p. (NA); ^{1}H NMR [1250], IR [1250].

(2,5-Dihydroxyphenyl)(3-fluoro-4-methylphenyl)methanone

[161463-63-4] $C_{14}H_{11}FO_3$ mol.wt. 246.24



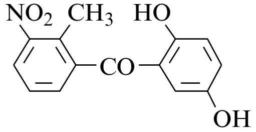
Synthesis

– Preparation by demethylation of 2,5-dimethoxy-3′-fluoro-4′-methylbenzophenone (SM) with boron tribromide in methylene chloride at 0° for 15–17 h (70–95%). SM was prepared by reaction of 3-fluoro-4-methylbenzoic acid with 1,4-dimethoxybenzene in the presence of polyphosphoric acid at 60–70° for 6–7 h (40–83%) [1250].

m.p. (NA); ^{1}H NMR [1250], IR [1250].

(2,5-Dihydroxyphenyl)(2-methyl-3-nitrophenyl)methanone

[161463-58-7] $C_{14}H_{11}NO_5$ mol.wt. 273.25



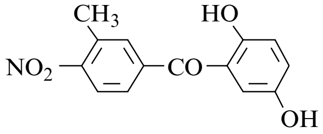
Synthesis

– Preparation by demethylation of 2,5-dimethoxy-2′-methyl-3′-nitrobenzophenone (SM) with boron tribromide in methylene chloride at 0° for 15–17 h (70–95%). SM was prepared by reaction of 2-methyl-3-nitrobenzoic acid with 1,4-dimethoxybenzene in the presence of polyphosphoric acid at 60–70° for 6–7 h (40–83%) [1250].

m.p. (NA); ^{1}H NMR [1250], IR [1250].

(2,5-Dihydroxyphenyl)(3-methyl-4-nitrophenyl)methanone

[153907-08-5] $C_{14}H_{11}NO_5$ mol.wt. 273.25



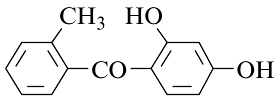
Synthesis

– Preparation by demethylation of 2,5-dimethoxy-3′-methyl-4′-nitrobenzophenone with boron tribromide in methylene chloride at 0° (81%) [1251].

m.p. 150° [1251]; ^{1}H NMR [1251], IR [1251].

(2,4-Dihydroxyphenyl)(2-methylphenyl)methanone

[14446-07-2] $C_{14}H_{12}O_3$ mol.wt. 228.25



Synthesis

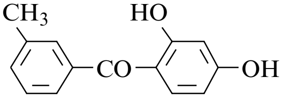
– Preparation by reaction of o-toluic acid (2-methylbenzoic acid) with resorcinol,

- in the presence of zinc chloride for 20 min at 160° (42%), (Nencki reaction) [1263];
- in the presence of Amberlyst-15 in refluxing 4-chlorotoluene (162°) for 2 h (91%) [53];
- in the presence of Zeolite-H-beta (previously activated at 400°) in refluxing mesitylene or 4-chlorotoluene for 3 h (with removal of water) (76%) [53,196].

– Also refer to: [1264].

m.p. 126–127° [1263], 121–123° [53,196]; ^{1}H NMR [53,196].

(2,4-Dihydroxyphenyl)(3-methylphenyl)methanone



$C_{14}H_{12}O_3$ mol.wt. 228.25

Synthesis

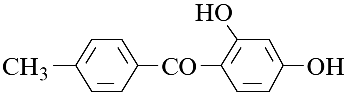
– Preparation by reaction of m-toluic acid with resorcinol,

- in the presence of zinc chloride and phosphorous oxychloride at 65° for 3 h [193];
- in the presence of zinc chloride at 140° for 20 min (22%) [1088] (Nencki reaction).

m.p. 168° [1088]; Spectra (NA).

(2,4-Dihydroxyphenyl)(4-methylphenyl)methanone

[40444-43-7]



$C_{14}H_{12}O_3$ mol.wt. 228.25

Synthesis

– Preparation by reaction of p-toluic acid (4-methyl-benzoic acid) with resorcinol,

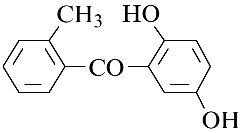
- in the presence of zinc chloride for 20 min at 165° (25%) (Nencki reaction) [1265];
- in the presence of Zeolite-H-beta in refluxing 4-chlorotoluene (162°) for 16–18 h (70%) [53];
- in the presence of boron trifluoride in tetrachloroethane on a steam bath for 4 h [113].

– Also refer to: [298,391,435,778,779,1266–1269].

m.p. 139° [113], 138° [1265]; UV [113].

(2,5-Dihydroxyphenyl)(2-methylphenyl)methanone

[83235-18-1]



$C_{14}H_{12}O_3$ mol.wt. 228.25

Syntheses

– Obtained by photochemical addition of o-tolualdehyde to 1,4-benzoquinone in benzene in the presence of benzophenone for 5 days (65%) [1255].

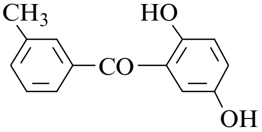
– Obtained by UV light irradiation of α-hydroxy (o-methyl-benzyl)-1,4-benzoquinone in benzene for 72 h (35%) [1260].

– Also refer to: [261].

m.p. 106–108° [1260]; ^{1}H NMR [1260], IR [1260].

(2,5-Dihydroxyphenyl)(3-methylphenyl)methanone

[83235-19-2] $C_{14}H_{12}O_3$ mol.wt. 228.25



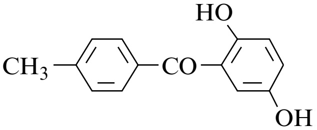
Synthesis

- Obtained by UV light irradiation of α-hydroxy(m-methyl-benzyl)-1,4-benzoquinone in benzene for 72 h (37%) [1260].
- Also refer to: [261].

m.p. 114–116° [1260]; ^{1}H NMR [1260], IR [1260].

(2,5-Dihydroxyphenyl)(4-methylphenyl)methanone

[83235-20-5] $C_{14}H_{12}O_3$ mol.wt. 228.25



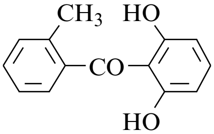
Syntheses

- Obtained by photochemical addition of p-tolualdehyde to 1,4-benzoquinone in benzene in the presence of benzophenone (79%) [1255].
- Obtained by UV light irradiation of α-hydroxy(p-methyl-benzyl)-1,4-benzoquinone in benzene for 72 h (43%) [1260].

m.p. 137–139° [1260]; ^{1}H NMR [1260], IR [1260].

(2,6-Dihydroxyphenyl)(2-methylphenyl)methanone

$C_{14}H_{12}O_3$ mol.wt. 228.25



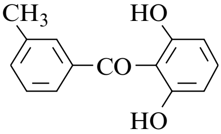
Synthesis

- Obtained from 8-(o-toluoyl)-4-methylumbelliferone by refluxing with N aqueous sodium hydroxide for 30 min (54%) [1263].

m.p. 140° [1263]; Spectra (NA).

(2,6-Dihydroxyphenyl)(3-methylphenyl)methanone

$C_{14}H_{12}O_3$ mol.wt. 228.25



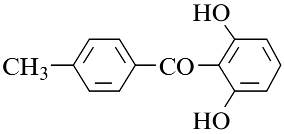
Synthesis

- Obtained from 8-(m-toluoyl)-4-methylumbelliferone by refluxing with N aqueous sodium hydroxide for 30 min (41%) [1088].

m.p. 145° [1088]; Spectra (NA).

(2,6-Dihydroxyphenyl)(4-methylphenyl)methanone

$C_{14}H_{12}O_3$ mol.wt. 228.25



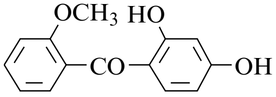
Synthesis

- Obtained from 8-(p-toluoyl)-4-methylumbelliferone by refluxing with N aqueous sodium hydroxide for 30 min (45%) [1265].

m.p. 125° [1265]; Spectra (NA).

(2,4-Dihydroxyphenyl)(2-methoxyphenyl)methanone

[79215-32-0] $C_{14}H_{12}O_4$ mol.wt. 244.25



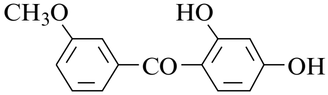
Syntheses

- Preparation by oxidation of 6-acetoxy-2,3-bis (2-methoxy-phenyl)benzofuran with chromium trioxide in refluxing acetic acid for 30 min, followed by saponification of the resulting keto ester (65%) with potassium hydroxide in refluxing ethanol for 1 h (66%) [1117].
- Preparation by condensation of resorcinol and o-anisic acid with boron trifluoride-etherate in carbon tetrachloride [1254], according to [360].
- Also obtained by hydrolysis of various substituted keto anils^T with potassium hydroxide in refluxing ethanol for 8 h [415],
  ^{T}4-(N-phenyl-o-anisimidoyl)resorcinol;
  ^{T}4-(N-o-tolyl-o-anisimidoyl)resorcinol;
  ^{T}4-[N-(p-methoxyphenyl)-o-anisimidoyl]resorcinol;
  ^{T}4-[N-(p-ethoxyphenyl)-o-anisimidoyl]resorcinol.
- Also refer to: [1259].

m.p. 247–248° [415], 175° [1117]. One of the reported melting points is obviously wrong. IR [1117].

(2,4-Dihydroxyphenyl)(3-methoxyphenyl)methanone

$C_{14}H_{12}O_4$ mol.wt. 244.25



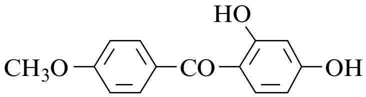
Syntheses

- Preparation by reaction of m-anisoyl chloride with resorcinol in the presence of aluminium chloride in nitrobenzene at r.t. for 48 h (86%) [1137].
- Obtained (poor yield) by condensation of m-anisic acid with resorcinol in the presence of zinc chloride at 160° for 10 min (Nencki reaction) (4%) [1137].

m.p. 176° [1137]; Spectra (NA).

(2,4-Dihydroxyphenyl)(4-methoxyphenyl)methanone

[5298-27-1] $C_{14}H_{12}O_4$ mol.wt. 244.25



Syntheses

- Preparation by reaction of p-methoxybenzoic acid (p-anisic acid) with resorcinol,
  - in the presence of Zeolite-H-beta in refluxing p-chlorotoluene (162°) for 3 h, with stirring and azeotropic removal of water (55%) [53];
  - in the presence of zinc chloride at 160° (Nencki reaction) [188];
  - in the presence of zinc chloride and phosphorous oxychloride during 3 h at 65° [193];

- in the presence of zinc chloride and a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 40°. Then, during 1.5 h, phosphorous trichloride was added and the mixture heated at 60° for 16 h (98%) [194];
- in the presence of boron trifluoride without solvent at 160° for 2 h in a sealed tube (67%) [185] or in tetrachloroethane on a steam bath for 4 h (81%) [113];
- in hydrofluoric acid at 100° in a stainless steel bomb [113].

– Preparation by condensation of p-anisoyl chloride with resorcinol in the presence of aluminium chloride in nitrobenzene during 48 h at r.t. (73%) [1137].

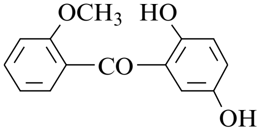
– Also obtained by hydrolysis of various substituted keto anils^T with potassium hydroxide in refluxing ethanol for 8 h [415],
^{T}4-(N-phenyl-p-anisimidoyl)resorcinol;
^{T}4-(N-o-tolyl-p-anisimidoyl)resorcinol;
^{T}4-[N-(p-methoxyphenyl)-p-anisimidoyl]resorcinol;
^{T}4-[N-(p-ethoxyphenyl)-p-anisimidoyl]resorcinol.

– Also refer to: [235,626].

m.p. 165° [113,188], 164° [185], 163°4–164°8 [194], 163° [235], 160° [1137], 158–159° [415]; UV [113,235]; paper chromatography [383].

(2,5-Dihydroxyphenyl)(2-methoxyphenyl)methanone

[140660-43-1] $C_{14}H_{12}O_4$ mol.wt. 244.25



Syntheses

– Obtained by photochemical addition of o-anisaldehyde to 1,4-benzoquinone in benzene in the presence of benzophenone for 5 days (72%) [1255].

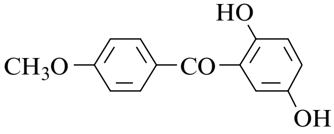
– Preparation by irradiation of a solution of 1,4-benzo-quinone and salicylaldehyde in benzene under nitrogen for 5 days (62%) [259].

– Also refer to: [1270].

m.p. 145–148° [259]; ^{1}H NMR [259], IR [259], MS [259]; TLC [259].

(2,5-Dihydroxyphenyl)(4-methoxyphenyl)methanone

[160720-40-1] $C_{14}H_{12}O_4$ mol.wt. 244.25



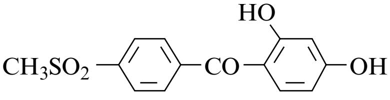
Synthesis

– Obtained by photochemical addition of p-anisaldehyde to 1,4-benzoquinone in benzene in the presence of benzophenone for 5 days (77%) [1255].

m.p. and Spectra (NA).

(2,4-Dihydroxyphenyl)[4-(methylsulfonyl)phenyl]methanone

[36419-33-7] $C_{14}H_{12}O_5S$ mol.wt. 292.31



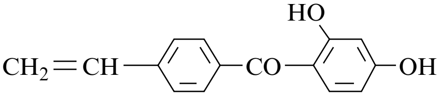
Synthesis

– Refer to: [235].

m.p. 202° [235]; UV [235].

(2,4-Dihydroxyphenyl)(4-ethenylphenyl)methanone

[66787-22-2] $C_{15}H_{12}O_3$ mol.wt. 240.26



Syntheses

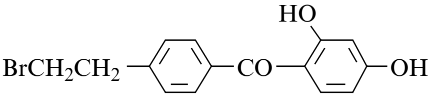
– Preparation by reaction of aqueous potassium hydroxide with 2,4-dihydroxy-4′-(2-bromo-ethyl)benzophenone in the presence of hydroquinone in refluxing methanol for 1.5 h with nitrogen bubbling (40%) [1147].

– Preparation, first by dehydrobromination of 2,4-diacetoxy-4′-(1-bromoethyl)benzophenone with tri-n-butylamine in the presence of picric acid as polymerization inhibitor in refluxing N,N-di-methylacetamide (140°) for 80 min, under nitrogen, then hydrolysis of the resulting new keto ester (2,4-diacetoxy-4′-vinylbenzophenone) (42%) with sodium bicarbonate in refluxing aqueous methanol for 1 h (33%) [1271].

m.p. 96° [1271,1272], 91–93° [1147]; ^{1}H NMR [1147,1271], IR [1147,1271], UV[1147,1271], MS [1147].

[4-(2-Bromoethyl)phenyl](2,4-dihydroxyphenyl)methanone

[80167-04-0] $C_{15}H_{13}BrO_3$ mol.wt. 321.17



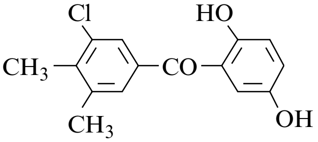
Synthesis

– Preparation by reaction of p-(2-bromoethyl)-benzonitrile with resorcinol (Hoesch reaction) (53%) [1147].

m.p. 113–115° [1147]; Spectra (NA).

(3-Chloro-4,5-dimethylphenyl)(2,5-dihydroxyphenyl)methanone

[161463-57-6] $C_{15}H_{13}ClO_3$ mol.wt. 276.72



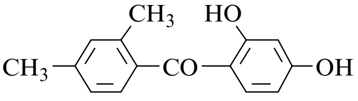
Synthesis

N.B.: This compound is mentioned in [Chem. Abstr., **122**, 187188v (1995)]. Nevertheless, the benzophenone in question is not described in the original paper [1250]. Actually, in this one, all substituted benzophenones without exception have the two ***ortho*** positions to the carbonyl group occupied.

m.p. and Spectra (NA).

(2,4-Dihydroxyphenyl)(2,4-dimethylphenyl)methanone

[36130-59-3] $C_{15}H_{14}O_3$ mol.wt. 242.27



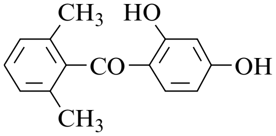
Synthesis

- Refer to: [801].

m.p. and Spectra (NA).

(2,4-Dihydroxyphenyl)(2,6-dimethylphenyl)methanone

[147809-19-6] $C_{15}H_{14}O_3$ mol.wt. 242.27



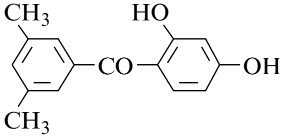
Synthesis

- Obtained by Friedel–Crafts acylation of resorcinol with 2,6-dimethylbenzoic acid in refluxing p-chlorotoluene (162°), with stirring and azeotropic removal of water,
  - in the presence of Amberlyst-15 for 2 h (98%) [53];
  - in the presence of Zeolite-H-beta for 5 h (32%) [53].
- Also refer to: [1264].

m.p. and Spectra (NA).

(2,4-Dihydroxyphenyl)(3,5-dimethylphenyl)methanone

[36419-35-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



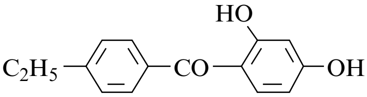
Synthesis

- Refer to: [1252] (Japanese patent) and [235].

m.p. 158° [235]; UV [235].

(2,4-Dihydroxyphenyl)(4-ethylphenyl)methanone

[66802-91-3] $C_{15}H_{14}O_3$ mol.wt. 242.27



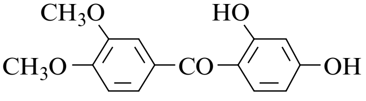
Synthesis

- Preparation by reaction of resorcinol with p-ethyl-benzoic acid in tetrachloroethane in the presence of boron trifluoride at 80° for 4 h (59%) [1271].
- Also refer to: [1273,1274].

m.p. 111–113° [1271]; b.p.$_{0.1}$ 195–200° [1271]; ^{1}H NMR [1271], IR [1271], UV [1271].

(2,4-Dihydroxyphenyl)(3,4-dimethoxyphenyl)methanone

[128996-02-1] $C_{15}H_{14}O_5$ mol.wt. 274.27



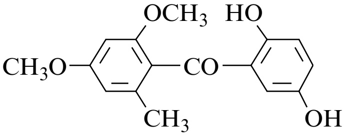
Syntheses

- Preparation by reaction of veratric acid with resorcinol in the presence of zinc chloride at 160° for 7 min (24%) (Nencki reaction) [1137].
- Preparation by reaction of veratroyl chloride with resorcinol in the presence of aluminium chloride in nitrobenzene during 3 days (60–64%) [1137].
- Also obtained from β-(3-hydroxy-4-veratroylphenoxy)propionic acid with aqueous sodium hydroxide solution [218].

m.p. 177° [1137], 149° [218]. One of the reported melting points is obviously wrong. Spectra (NA).

(2,5-Dihydroxyphenyl)(2,4-dimethoxy-6-methylphenyl)methanone

[78044-94-7] $C_{16}H_{16}O_5$ mol.wt. 288.30



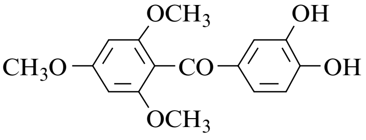
Synthesis

- Obtained by photo-Fries rearrangement of 4-hydroxy-phenyl 2,4-dimethoxy-6-methylbenzoate in benzene under nitrogen for 4 h (33%) [1167].

m.p. 228–229° [1167]; ^{1}H NMR [1167], IR [1167], UV [1167], MS [1167].

(3,4-Dihydroxyphenyl)(2,4,6-trimethoxyphenyl)methanone *(Cotogenin)*

$C_{16}H_{16}O_6$ mol.wt. 304.30



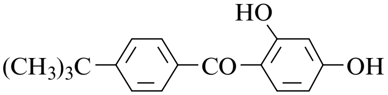
Synthesis

- Obtained by reaction of 3,4-(diacetoxy) benzonitrile with phloroglucinol trimethyl ether in the presence of zinc chloride and hydrochloric acid, followed by hydrolysis of the resulting ketimine hydrochloride (16%) (Hoesch reaction) [1275].
- Also refer to: [450,772].

m.p. 219–220° [1275], 217° [450], 210° [447]; Spectra (NA).

(2,4-Dihydroxyphenyl)[4-(1,1-dimethylethyl)phenyl]methanone

[21332-56-9] $C_{17}H_{18}O_3$ mol.wt. 270.33



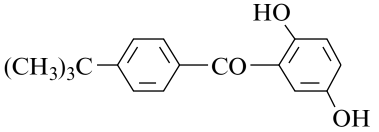
Synthesis

- Refer to: [235,1276].

m.p. 162° [235]; UV [235].

(2,5-Dihydroxyphenyl)[4-(1,1-dimethylethyl)phenyl]methanone

[169696-58-6] $C_{17}H_{18}O_3$ mol.wt. 270.33



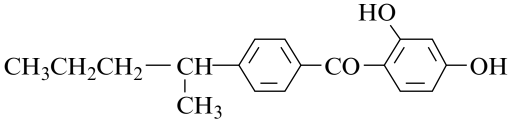
Synthesis

– Preparation by total demethylation of 4′-tert-butyl-2,5-dimethoxybenzophenone (SM) with boron tribromide in methylene chloride, first at −30° for 30 min, then at 22° for 5 h (86%). SM was obtained by Friedel–Crafts acylation of hydroquinone dimethyl ether with p-tert-butylbenzoyl chloride in methylene chloride in the presence of aluminium chloride at 0° for 8 h (84%) [1258].

m.p. 115–116° [1258]; ^{1}H NMR [1258].

(2,4-Dihydroxyphenyl)[4-(1-methylbutyl)phenyl]methanone

$C_{18}H_{20}O_3$ mol.wt. 284.36



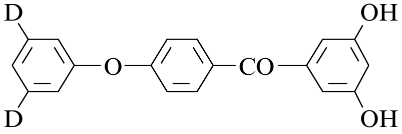
Synthesis

– Preparation by reaction of p-sec-amyl-benzoic acid with resorcinol in the presence of boron trifluoride in tetra-chloroethane on a steam bath for 4 h [113].

b.p.$_{0.75}$ 235–240° [113]; Spectra (NA).

(3,5-Dihydroxyphenyl)(4-phenoxy-3,5-d_2-phenyl)methanone

[176738-21-9] $C_{19}H_{12}D_2O_4$ mol.wt. 308.33



Synthesis

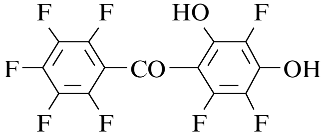
– Preparation by demethylation of 3,5-dimethoxy-4′-(phenoxy-d_5)benzophenone with 48% hydrobromic acid in refluxing acetic acid for 16 h (92%) (compound **21**). The acidic treatment exchanges exclusively the deuterium atoms in the 2”, 4” and 6” positions, because of the mesomeric inductive effect of the exocyclic oxygen [906].

m.p. (NA), white solid [906]; ^{1}H NMR [906], ^{13}C NMR [906].

2.2.1.3 Substituents Located on Both Rings

(2,3,4,5,6-Pentafluorophenyl)(2,3,5-trifluoro-4,6-dihydroxyphenyl)methanone

[32541-20-1] $C_{13}H_2F_8O_3$ mol.wt. 358.14



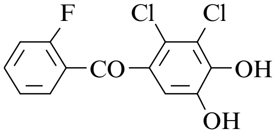
Synthesis

– Preparation by total demethylation of 2, 4-dimethoxy-2′,3,3′,4′,5,5′,6,6′-octafluorobenzophenone in methylene chloride with aluminium chloride (2 mol) at 20° (73%) [570].

m.p. 118–120° [570]; IR [570].

(2,3-Dichloro-4,5-dihydroxyphenyl)(2-fluorophenyl)methanone

[103843-57-8] $C_{13}H_7Cl_2FO_3$ mol.wt. 301.10



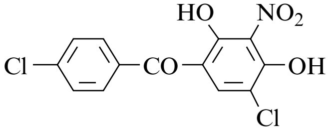
Synthesis

– Preparation by reaction of 2-fluorobenzoyl chloride with 3,4-dichloro-1,2-dihydroxybenzene in the presence of aluminium chloride in refluxing ethylene dichloride during 24 h (75%) [841,842].

m.p. 164–165° [841,842]; ^{1}H NMR [841,842], IR [841,842].

(5-Chloro-2,4-dihydroxy-3-nitrophenyl)(4-chlorophenyl)methanone

$C_{13}H_7Cl_2NO_5$ mol.wt. 328.11



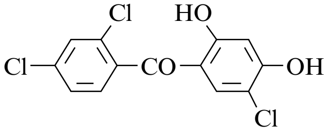
Synthesis

– Obtained by reaction of concentrated nitric acid with 4′,5-dichloro-2,4-dihydroxybenzophenone (50%) [1082].

m.p. 120° [1082]; TLC [1082]; ^{1}H NMR [1082], IR [1082], MS [1082].

(5-Chloro-2,4-dihydroxyphenyl)(2,4-dichlorophenyl)methanone

$C_{13}H_7Cl_3O_3$ mol.wt. 317.55



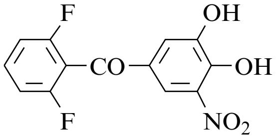
Synthesis

– Preparation by reaction of 2,4-dichlorobenzotrichloride with 4-chlororesorcinol [211].

m.p. and Spectra (NA).

(2,6-Difluorophenyl)(3,4-dihydroxy-5-nitrophenyl)methanone

[134612-45-6] $C_{13}H_7F_2NO_5$ mol.wt. 295.20



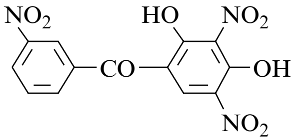
Synthesis

– Preparation by demethylation of 2′,6′-difluoro-4-hydroxy-3-methoxy-5-nitrobenzophenone with hydrobromic acid in refluxing aqueous acetic acid [1019].

m.p. 194–196° [1019]; Spectra (NA).

(2,4-Dihydroxy-3,5-dinitrophenyl)(3-nitrophenyl)methanone

$C_{13}H_7N_3O_9$ mol.wt. 349.21



Synthesis

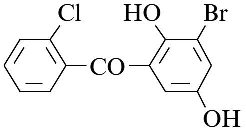
– Preparation by slowly adding (2 h) nitric acid (d = 1.42) to a solution of resbenzophenone in concentrated sulfuric acid (d = 1.84) at 5–10°. The mixture was allowed to stand overnight, then heated at 50° for 30 min (quantitative yield) [227].

N.B.: The first crop from 95% ethanol contained 12–14% of the 2,4-dihydroxy -3,5-dinitrobenzophenone.

m.p. 178–180° [227]; Spectra (NA).

(3-Bromo-2,5-dihydroxyphenyl)(2-chlorophenyl)methanone

[153907-03-0] $C_{13}H_8BrClO_3$ mol.wt. 327.56



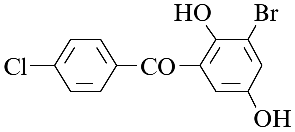
Synthesis

- Preparation by reaction of boron tribromide with 3-bromo-2′-chloro-2,5-dimethoxybenzophenone in methylene chloride at 0° (95%) [1251].

m.p. 150° [1251]; ^{1}H NMR [1251], IR [1251].

(3-Bromo-2,5-dihydroxyphenyl)(4-chlorophenyl)methanone

[153907-04-1] $C_{13}H_8BrClO_3$ mol.wt. 327.56



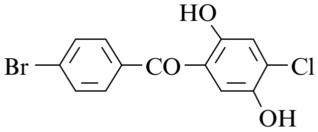
Synthesis

- Preparation by demethylation of 3-bromo-4′-chloro-2,5-di-methoxybenzophenone with boron tribromide in methylene chloride at 0° (86%) [1251].

m.p. 90° [1251]; ^{1}H NMR [1251], IR [1251].

(4-Bromophenyl)(4-chloro-2,5-dihydroxyphenyl)methanone

[161463-53-2] $C_{13}H_8BrClO_3$ mol.wt. 327.56



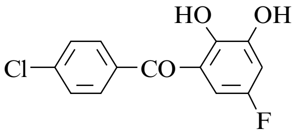
Synthesis

- Preparation by demethylation of 4′-bromo-4-chloro-2,5-di-methoxybenzophenone (SM) with boron tribromide in methylene chloride at 0° for 15–17 h (70–95%). SM was prepared by reaction of 4-bromobenzoic acid with 2-chloro-1,4-dimethoxybenzene in the presence of polyphosphoric acid at 60–70° for 6–7 h (40–83%) [1250].

m.p. (NA); ^{1}H NMR [1250], IR [1250].

(4-Chlorophenyl)(5-fluoro-2,3-dihydroxyphenyl)methanone

[92735-05-2] $C_{13}H_8ClFO_3$ mol.wt. 266.65



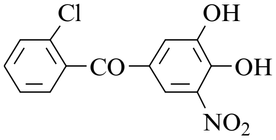
Synthesis

- Obtained by action of dilute hydrogen peroxide with 4′-chloro-5-fluoro-3-formyl-2-hydroxy-benzophenone in refluxing aqueous sodium hydroxide under argon for 30 min [1077].

m.p. 124–126° [1077]; ^{1}H NMR [1077], IR [1077].

(2-Chlorophenyl)(3,4-dihydroxy-5-nitrophenyl)methanone

[134612-46-7] $C_{13}H_8ClNO_5$ mol.wt. 293.66



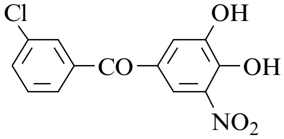
Synthesis

– Preparation by demethylation of 2′-chloro-4-hydroxy-3-methoxy-5-nitrobenzophenone with hydrobromic acid in refluxing aqueous acetic acid [1019].

m.p. 129–131° [1019]; Spectra (NA).

(3-Chlorophenyl)(3,4-dihydroxy-5-nitrophenyl)methanone

[134612-47-8] $C_{13}H_8ClNO_5$ mol.wt. 293.66



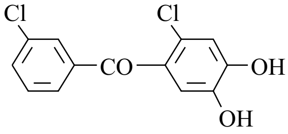
Synthesis

– Preparation by demethylation of 3′-chloro-4-hydroxy-3-methoxy-5-nitrobenzophenone with hydrobromic acid in refluxing aqueous acetic acid [1019].

m.p. 143–145° [1019]; Spectra (NA).

(2-Chloro-4,5-dihydroxyphenyl)(3-chlorophenyl)methanone

[91197-11-4] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



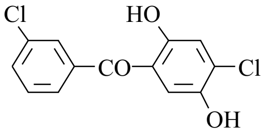
Synthesis

– Preparation by demethylation of 2,3′-dichloro-4,5-di-methoxybenzophenone (SM) with pyridinium chloride for 2 h at 180–200°. SM was obtained by reaction of m-chlorobenzoic acid with 1-chloro-3,4-dimethoxybenzene in the presence of phosphorous pentoxide in methanesulfonic acid for 30 min at 70° [1199].

m.p. and Spectra (NA).

(4-Chloro-2,5-dihydroxyphenyl)(3-chlorophenyl)methanone

[161463-60-1] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



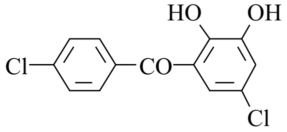
Synthesis

– Preparation by demethylation of 3′,4-dichloro-2,5-di-methoxybenzophenone (SM) with boron tribromide in methylene chloride at 0° for 15–17 h (70–95%). SM was prepared by reaction of m-chlorobenzoic acid with 2-chloro-1,4-dimethoxybenzene in the presence of polyphosphoric acid at 60–70° for 6–7 h (40–83%) [1250].

m.p. (NA); ^{1}H NMR [1250], IR [1250].

(5-Chloro-2,3-dihydroxyphenyl)(4-chlorophenyl)methanone

[92735-01-8] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



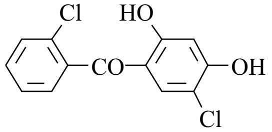
Synthesis

- Obtained by action of dilute hydrogen peroxide with 4′,5-di-chloro-3-formyl-2-hydroxybenzophenone in refluxing aqueous sodium hydroxide under argon for 30 min [1077].

m.p. 133–135° [1077]; ^{1}H NMR [1077], IR [1077].

(5-Chloro-2,4-dihydroxyphenyl)(2-chlorophenyl)methanone

[50685-42-2] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



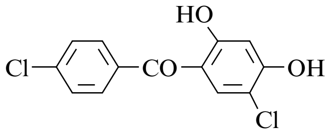
Synthesis

- Preparation by reaction of 2-chlorobenzotrichloride with 4-chlororesorcinol in isopropanol at 70–80° (81%) [211].

m.p. 187–188° [211]; Spectra (NA).

(5-Chloro-2,4-dihydroxyphenyl)(4-chlorophenyl)methanone

[50685-41-1] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



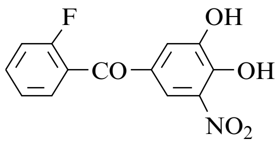
Syntheses

- Preparation by demethylation of 4′,5-dichloro-2-hydroxy-4-methoxybenzophenone with a 47% aqueous solution of hydrobromic acid in refluxing acetic acid for 18 h under nitrogen (22%) [1082].
- Preparation by reaction of 4-chlorobenzotrichloride with 4-chlororesorcinol in 40% aqueous isopropanol solution at 70–80° (85%) [211].

m.p. 188°5–189°5 [211], 170–190° [1082]. A typing error probably occurred in the published data. ^{1}H NMR [1082], IR [1082], MS [1082]; TLC [1082].

(3,4-Dihydroxy-5-nitrophenyl)(2-fluorophenyl)methanone

[125628-97-9] $C_{13}H_8FNO_5$ mol.wt. 277.21



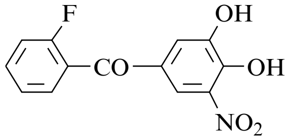
Synthesis

- Preparation by demethylation of 2′-fluoro-4-hydroxy-3-methoxy-5-nitrobenzophenone with 48% hydrobromic acid in refluxing acetic acid for 4 h (86%) [1019,1084].
- Also refer to: [1277–1281].

m.p. 169–171° [1019,1084]; ^{1}H NMR [1084], MS [1084]; LD_{50} [1282].

(3,4-Dihydroxy-5-nitrophenyl)[2-(fluoro-^{18}F)phenyl]methanone

[172546-74-6] $C_{13}H_8FNO_5$ mol.wt. 276.21



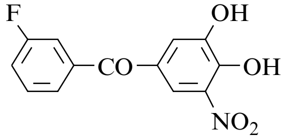
Synthesis

- Preparation by demethylation of 2′-[^{18}F] fluoro-3,4-di-methoxy-5-nitrobenzophenone (SM) in dimethyl sulfoxide with aqueous hydrobromic acid first at r.t. and at 140° for 30 min. SM was obtained by action of [^{18}F] cesium fluoride with 3,4-dimethoxy-2′,5-dinitro-benzophenone in dimethyl sulfoxide for 10 min at 150° in a silicone-coated tube (Vacutainer).
- Refer to: Chem. Abstr., **127**, 17465u (1997).

m.p. and Spectra (NA).

(3,4-Dihydroxy-5-nitrophenyl)(3-fluorophenyl)methanone

[134612-43-4] $C_{13}H_8FNO_5$ mol.wt. 277.21



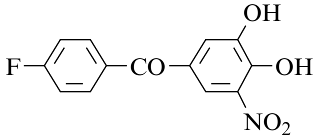
Synthesis

- Preparation by demethylation of 3′-fluoro-4-hydroxy-3-methoxy-5-nitrobenzophenone with hydrobromic acid in refluxing aqueous acetic acid [1019].

m.p. 124–126° [1019]; Spectra (NA).

(3,4-Dihydroxy-5-nitrophenyl)(4-fluorophenyl)methanone

[134612-44-5] $C_{13}H_8FNO_5$ mol.wt. 277.21



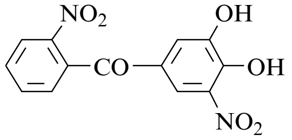
Synthesis

- Preparation by demethylation of 4′-fluoro-4-hydroxy-3-methoxy-5-nitrobenzophenone with hydrobromic acid in refluxing aqueous acetic acid [1019].

m.p. 171–173° [1019]; Spectra (NA).

(3,4-Dihydroxy-5-nitrophenyl)(2-nitrophenyl)methanone

[190523-00-3] $C_{13}H_8N_2O_7$ mol.wt. 304.22



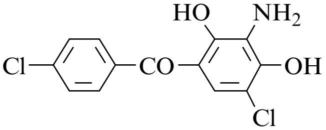
Synthesis

- Preparation by demethylation of 4-hydroxy-3-methoxy-2′,5-dinitrobenzophenone with 48% hydrobromic acid in refluxing acetic acid for 6 h (70%). -Refer to: Chem. Abstr., **127**, 17465u (1997)^{T}.

m.p. (NA); ^{1}H NMR^{T}, MS^{T}.

(3-Amino-5-chloro-2,4-dihydroxyphenyl)(4-chlorophenyl)methanone

[87119-05-9]



$C_{13}H_9Cl_2NO_3$ mol.wt. 298.12

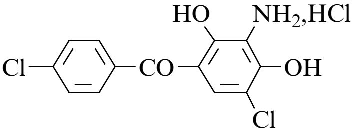
Synthesis

- Obtained from the corresponding hydrochloride described below [1082].

m.p. and Spectra (NA).

(3-Amino-5-chloro-2,4-dihydroxyphenyl)(4-chlorophenyl)methanone *(Hydrochloride)*

[87119-06-0]



$C_{13}H_9Cl_2NO_3$,HCl mol.wt. 334.59

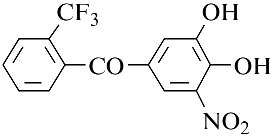
Synthesis

- Preparation by hydrogenation of 4′,5-dichloro-2,4-di-hydroxy-3-nitrobenzophenone in chloroform/ethanol solution in the presence of 10% Pd/C, followed by treatment of the resulting amino compound with concentrated hydrochloric acid in ethanol (51%) [1082].

m.p. 180° [1082]; ^{1}H NMR [1082], IR [1082], MS [1082]; TLC [1082].

(3,4-Dihydroxy-5-nitrophenyl)[2-(trifluoromethyl)phenyl]methanone

[134612-50-3]



$C_{14}H_8F_3NO_5$ mol.wt. 327.22

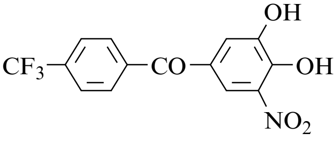
Synthesis

- Preparation by demethylation of 4-hydroxy-3-methoxy-5-nitro-2′-(trifluoromethyl)benzophenone with hydrobromic acid in refluxing aqueous acetic acid [1019].

m.p. 146–148° [1019]; Spectra (NA).

(3,4-Dihydroxy-5-nitrophenyl)[4-(trifluoromethyl)phenyl]methanone

[134611-76-0]



$C_{14}H_8F_3NO_5$ mol.wt. 327.22

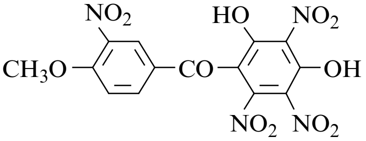
Synthesis

- Preparation by demethylation of 4-hydroxy-3-methoxy-5-nitro-4′-(trifluoromethyl)benzophenone first with 33% hydrobromic acid in acetic acid at 90° for 18 h, then with 48% hydrobromic acid in aqueous acetic acid at 110° for 18 h [1019].

m.p. 116–118° [1019]; Spectra (NA).

(2,4-Dihydroxy-3,5,6-trinitrophenyl)(4-methoxy-3-nitrophenyl)methanone

[67246-07-5] $C_{14}H_8N_4O_{12}$ mol.wt. 424.24



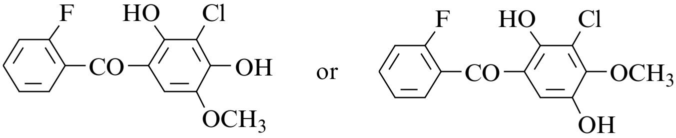
Synthesis

- Obtained by partial methylation of 2,4,4′-trihydroxy-3,5,3′,6-tetranitrobenzophenone with dimethyl sulfate in the presence of potassium carbonate in acetone [592].

m.p. 75° [592]; ^{1}H NMR [592].

[3-Chloro-2,4 (or 2,5)-dihydroxy-5 (or 4)-methoxyphenyl](2-fluorophenyl) methanone

[140708-53-8] $C_{14}H_{10}ClFO_4$ mol.wt. 296.68



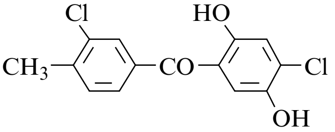
Synthesis

- Obtained by reaction of o-fluorobenzoyl chloride with 3-chloro-1,2,4-trimethoxybenzene in the presence of aluminium chloride in ethylene dichloride between 0° and 5°, then at r.t. for 8 h and at reflux for 1.5 h (71%) [704].

m.p. 136–137° [704]; Spectra (NA).

(4-Chloro-2,5-dihydroxyphenyl)(3-chloro-4-methylphenyl)methanone

[161463-56-5] $C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14



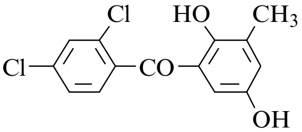
Synthesis

- Preparation by demethylation of 3′,4-dichloro-2,5-di-methoxy-4′-methylbenzophenone (SM) with boron tribromide in methylene chloride at 0° for 15–17 h (70–95%). SM was prepared by reaction of 3-chloro-4-methylbenzoic acid with 2-chloro-1,4-dimethoxy-benzene in the presence of polyphosphoric acid at 60–70° for 6–7 h (40–83%) [1250].

m.p. (NA); ^{1}H NMR [1250], IR [1250].

(2,4-Dichlorophenyl)(2,5-dihydroxy-3-methylphenyl)methanone

[153907-06-3] $C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14



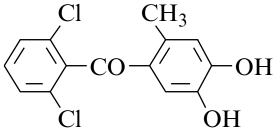
Synthesis

- Preparation by demethylation of 2′,4′-dichloro-2,5-di-methoxy-3-methylbenzophenone with boron tribromide in methylene chloride at 0° (77%) [1251].

m.p. 150° [1251]; ^{1}H NMR [1251], IR [1251].

(2,6-Dichlorophenyl)(4,5-dihydroxy-2-methylphenyl)methanone

[91197-05-6] $C_{14}H_{10}Cl_2O_3$ mol.wt. 297.14



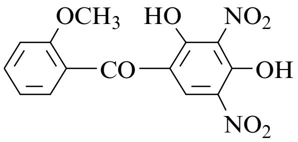
Syntheses

- Preparation by total demethylation of 2′,6′-dichloro-4,5-di-methoxy-2-methylbenzophenone (SM) [1072], with pyridinium chloride for 2 h at 180–200° [1199]. SM was obtained by reaction of 2,6-dichlorobenzoic acid with 1,2-dimethoxy-4-methylbenzene in the presence of phosphorous pentoxide in methanesulfonic acid for 30 min at 70° [1199].
- Preparation by Friedel–Crafts acylation of 4-methylpyrocatechol with 2,6-dichlorobenzoyl chloride [1072].
- Also refer to: [1200].

m.p. 206–207° [1199], 201–203° [1072]; Spectra (NA).

(2,4-Dihydroxy-3,5-dinitrophenyl)(2-methoxyphenyl)methanone

[79204-68-5] $C_{14}H_{10}N_2O_8$ mol.wt. 334.24



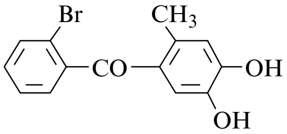
Synthesis

- Preparation by oxidation of 6-acetoxy-2,3-bis(o-methoxy-phenyl)-5,7-dinitrobenzofuran with chromium trioxide in acetic acid, followed by saponification of the keto ester so formed with potassium hydroxide in ethanol (55%) [1117].

m.p. 280° [1117]; IR [1117].

(2-Bromophenyl)(4,5-dihydroxy-2-methylphenyl)methanone

[91197-04-5] $C_{14}H_{11}BrO_3$ mol.wt. 307.14



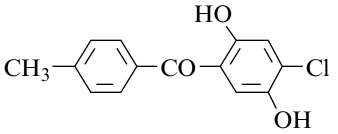
Synthesis

- Preparation by demethylation of 2′-bromo-4,5-dimethoxy-2-methylbenzophenone (SM) with pyridinium chloride for 2 h at 180–200°. SM was obtained by reaction of o-bromo-benzoic acid with 1,2-dimethoxy-4-methylbenzene in the presence of phosphorous pentoxide in methanesulfonic acid for 30 min at 70° [1199].

m.p. 177–178° [1199]; Spectra (NA).

(4-Chloro-2,5-dihydroxyphenyl)(4-methylphenyl)methanone

[161463-55-4] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



Synthesis

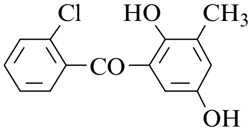
- Preparation by demethylation of 4-chloro-2,5-dimethoxy-4′-methylbenzophenone (SM) with boron tribromide in methylene chloride at 0° for 15–17 h (70–95%).

SM was prepared by reaction of p-toluic acid with 2-chloro-1,4-di-methoxybenzene in the presence of polyphosphoric acid at 60–70° for 6–7 h (40–83%) [1250].

m.p. (NA); ^{1}H NMR [1250], IR [1250].

(2-Chlorophenyl)(2,5-dihydroxy-3-methylphenyl)methanone

[153907-05-2] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



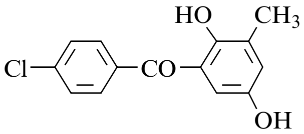
Synthesis

- Preparation by demethylation of 2′-chloro-2,5-dimethoxy-3-methylbenzophenone with boron tribromide in methylene chloride at 0° (76%) [1251].

m.p. 140° [1251]; ^{1}H NMR [1251], IR [1251].

(4-Chlorophenyl)(2,5-dihydroxy-3-methylphenyl)methanone

[153907-07-4] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



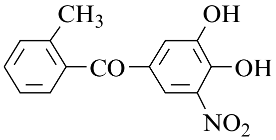
Synthesis

- Preparation by demethylation of 4′-chloro-2,5-dimethoxy-3-methylbenzophenone with boron tribromide in methylene chloride at 0° (70%) [1251].

m.p. 190° [1251]; ^{1}H NMR [1251], IR [1251].

(3,4-Dihydroxy-5-nitrophenyl)(2-methylphenyl)methanone

[134612-48-9] $C_{14}H_{11}NO_5$ mol.wt. 273.25



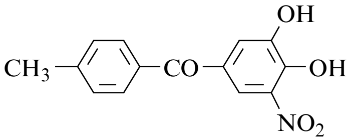
Synthesis

- Preparation by demethylation of 4-hydroxy-3-methoxy-2′-methyl-5-nitrobenzophenone with hydrobromic acid in refluxing aqueous acetic acid [1019].

m.p. 164–166° [1019]; Spectra (NA).

(3,4-Dihydroxy-5-nitrophenyl)(4-methylphenyl)methanone

[134308-13-7] $C_{14}H_{11}NO_5$ mol.wt. 273.25



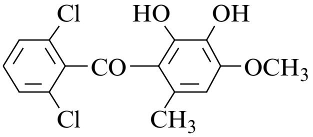
Synthesis

- Preparation by demethylation of 4-hydroxy-3-methoxy-4′-methyl-5-nitrobenzophenone with hydrobromic acid in refluxing aqueous acetic acid [1019].
- Also refer to: [1283–1285].

m.p. 146–148° [1019]; Spectra (NA).

(2,6-Dichlorophenyl)(2,3-dihydroxy-4-methoxy-6-methylphenyl)methanone

[183725-80-6] $C_{15}H_{12}Cl_2O_4$ mol.wt. 327.16



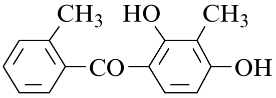
Synthesis

- Preparation by partial demethylation of 2′,6′-dichloro-2,3,4-trimethoxy-6-methylbenzophenone in acetic acid in the presence of 30% hydrobromic acid for 2 h at 75° (39%) [1072].

m.p. 182° [1072]; Spectra (NA).

(2,4-Dihydroxy-3-methylphenyl)(2-methylphenyl)methanone

[147809-15-2] $C_{15}H_{14}O_3$ mol.wt. 242.27



Synthesis

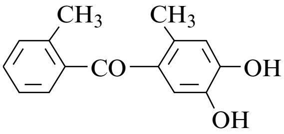
- Preparation by treating o-toluic acid with 2-methylresorcinol in the presence of Zeolite-H-beta (previously activated at 400°), in refluxing p-chlorotoluene or n-decane for 2–3 h (with water removal) (74%) [53,196].

- Also refer to: [1264].

m.p. 187–188°5 [53,196]; ^{1}H NMR [53,196].

(4,5-Dihydroxy-2-methylphenyl)(2-methylphenyl)methanone

[91197-07-8] $C_{15}H_{14}O_3$ mol.wt. 242.27



Synthesis

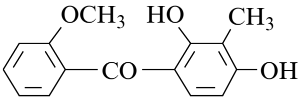
- Preparation by demethylation of 4,5-dimethoxy-2,2′-di-methylbenzophenone (SM) with pyridinium chloride for 2 h at 180–200°. SM was obtained by reaction of o-toluic acid with 1,2-dimethoxy-4-methylbenzene in the presence of phosphorous pentoxide in methanesulfonic acid for 20 min at 70° [1199].

- Also refer to: [1200].

m.p. 149–150° [1199]; Spectra (NA).

(2,4-Dihydroxy-3-methylphenyl)(2-methoxyphenyl)methanone

[85636-84-6] $C_{15}H_{14}O_4$ mol.wt. 258.27



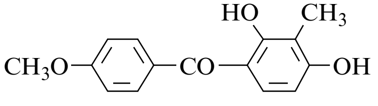
Synthesis

- Refer to: [193].

m.p. and Spectra (NA).

(2,4-Dihydroxy-3-methylphenyl)(4-methoxyphenyl)methanone

[79861-83-9]



$C_{15}H_{14}O_4$ mol.wt. 258.27

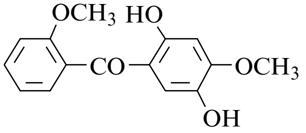
Synthesis

- Preparation by reaction of p-anisic acid with 2-methyl-resorcinol in tetrachloroethane in the presence of boron trifluoride at 80° for 4 h (66%) [224].

m.p. 194–195° [224]; ^{1}H NMR [224].

(2,5-Dihydroxy-4-methoxyphenyl)(2-methoxyphenyl)methanone

[42833-90-9]



$C_{15}H_{14}O_5$ mol.wt. 274.27

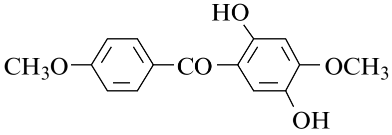
Synthesis

- Preparation by reaction of 2-hydroxy-2′,4,5-trimethoxy-benzophenone with DDQ (2,3-Dichloro-5,6-dicyano-1,4-benzoquinone) in refluxing benzene for 1.5 h, followed by complete evaporation of the solvent, then treatment of the resulting brown oil in boiling methanol (58%) [416].

m.p. 195–196° [416]; UV [416], MS [416].

(2,5-Dihydroxy-4-methoxyphenyl)(4-methoxyphenyl)methanone

[42045-63-6]



$C_{15}H_{14}O_5$ mol.wt. 274.27

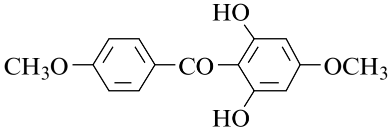
Syntheses

- Preparation by reaction of p-anisic acid with 2-methoxyhydroquinone in the presence of boron trifluoride-etherate, heating at 100° for 30–45 min (26%) [650].
- Also obtained (poor yield) by reaction of 2-hydroxy-4,4′-dimethoxybenzo phenone with lead tetraacetate in acetic acid at 100° for 5 h (1%) [650].

m.p. 164–165° [650]; ^{1}H NMR [650], IR [650], UV [650].

(2,6-Dihydroxy-4-methoxyphenyl)(4-methoxyphenyl)methanone

[55051-89-3]



$C_{15}H_{14}O_5$ mol.wt. 274.27

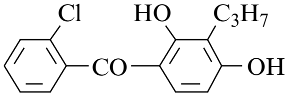
Synthesis

- Obtained by partial methylation of 2,4′,6-trihydroxy-4-methoxybenzphenone with diazomethane in ethyl ether [1286].

m.p. (NA); ^{1}H NMR [1286].

(2-Chlorophenyl)(2,4-dihydroxy-3-propylphenyl)methanone

[115296-10-1] $C_{16}H_{15}ClO_3$ mol.wt. 290.75



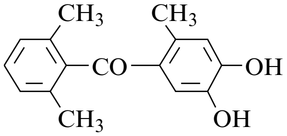
Synthesis

- Obtained (poor yield) by reaction of o-chlorobenzoyl chloride with 1,3-dimethoxy-2-propylbenzene in methylene chloride in the presence of aluminium chloride, first at 0° for 2 h and at r.t. for 1 h (10%) [1029].

oil [1029]; b.p. and Spectra (NA).

(4,5-Dihydroxy-2-methylphenyl)(2,6-dimethylphenyl)methanone

[91197-06-7] $C_{16}H_{16}O_3$ mol.wt. 256.30



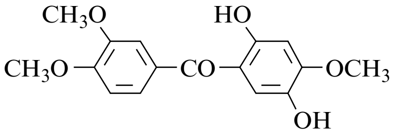
Synthesis

- Preparation by demethylation of 4,5-dimethoxy-2,2′,6′-tri-methylbenzophenone (SM) with pyridinium chloride for 2 h at 180–200°. SM was obtained by reaction of 2,6-di-methylbenzoic acid with 1,2-dimethoxy-4-methylbenzene in methanesulfonic acid in the presence of phosphorous pentoxide for 30 min at 70° [1199].
- Also refer to: [1200].

m.p. 193–195° [1199]; Spectra (NA).

(2,5-Dihydroxy-4-methoxyphenyl)(3,4-dimethoxyphenyl)methanone

[62495-45-8] $C_{16}H_{16}O_6$ mol.wt. 304.30



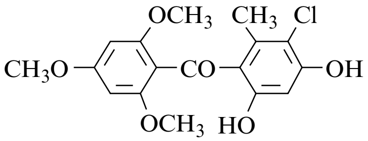
Synthesis

- Obtained (poor yield) by oxidation of 2-hydroxy-3′,4,4′,5-tetramethoxybenzophenone with manganese (III) acetate dihydrate in refluxing acetic acid for 2 h (9%) [1136].

m.p. 208–209° [1136]; ^{1}H NMR [1136], IR [1136], UV [1136].

(3-Chloro-4,6-dihydroxy-2-methylphenyl)(2,4,6-trimethoxyphenyl)methanone

[68048-19-1] $C_{17}H_{17}ClO_6$ mol.wt. 352.77



Synthesis

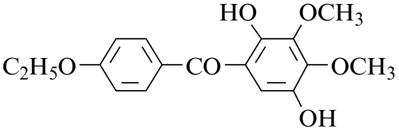
- Preparation by hydrogenolysis of 4,6-bis(benzyloxy)-3-chloro-2′,4′,6′-trimethoxy-2-methylbenzophenone (SM) with hydrogen in ethyl acetate/

tetrahydrofuran in the presence of 10% Pd/C at 25°. SM was obtained by condensation of 4,6-bis(benzyloxy)-3-chloro-2-methylbenzoic acid with phloroglucinol trimethyl ether in the presence of trifluoroacetic anhydride in methylene chloride under nitrogen for 3 min (87%) [1179].

m.p. 132–133° [1179]; ^{1}H NMR [1179], IR [1179], MS [1179].

(2,5-Dihydroxy-3,4-dimethoxyphenyl)(4-ethoxyphenyl)methanone

[69471-32-5]



$C_{17}H_{18}O_6$ mol.wt. 318.33

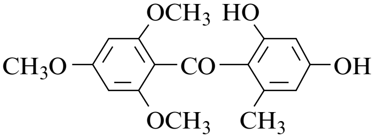
Synthesis

- Preparation by oxidation of 4′-ethoxy-2-hydroxy-3,4-dimethoxybenzophenone with potassium persulfate (24%) (Elbs reaction) [1168].

m.p. 140–142° [1168]; ^{1}H NMR [1168], IR [1168].

(2,4-Dihydroxy-6-methylphenyl)(2,4,6-trimethoxyphenyl)methanone

[76631-09-9]



$C_{17}H_{18}O_6$ mol.wt. 318.33

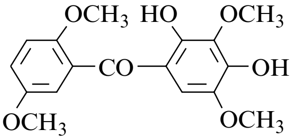
Synthesis

- Preparation by reaction of triethylamine with 1-(2,4,6-trimethoxyphenyl)-1,3,5,7-octanetetraone (SM) in refluxing tetrahydrofuran in a nitrogen atmosphere for 38 h (quantitative yield) [1287]. SM can be obtained according to two methods:

- acylation of the trilithium salt of 2,4,6-heptanetrione (prepared with LDA in tetrahydrofuran at 0°) with methyl 2,4,6-trimethoxybenzoate in tetrahydrofuran for 17 h at r.t. (55%);
- acylation of the dilithium salt of 2,4-pentanedione with methyl 3-(2,4,6-trimethoxyphenyl)-3-oxopropanoate (sodium salt) (42%).

m.p. 169–173° [1287]; ^{1}H NMR [1287], IR [1287], MS [1287]; TLC [1287].

(2,4-Dihydroxy-3,5-dimethoxyphenyl)(2,5-dimethoxyphenyl)methanone



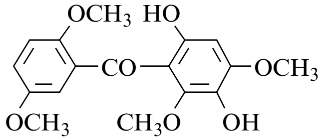
$C_{17}H_{18}O_7$ mol.wt. 334.33

Synthesis

- Obtained (poor yield) by reaction of 2,3,4,5-tetramethoxy-benzoyl chloride with hydroquinone dimethyl ether in benzene in the presence of aluminium chloride (16%) [1160].

m.p. and Spectra (NA).

(3,6-Dihydroxy-2,4-dimethoxyphenyl)(2,5-dimethoxyphenyl)methanone



$C_{17}H_{18}O_7$ mol.wt. 334.33

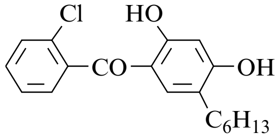
Synthesis

- Preparation by reaction of 2,5-dimethoxybenzoic acid with 2,6-dimethoxyhydroquinone in trifluoroacetic anhydride [1185].

m.p. and Spectra (NA).

(2-Chlorophenyl)(5-hexyl-2,4-dihydroxyphenyl)methanone

[50685-43-3]



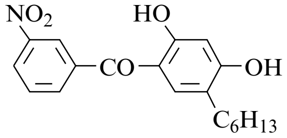
$C_{19}H_{21}ClO_3$ mol.wt. 332.83

Synthesis

- Preparation by reaction of 2-chlorobenzotrichloride with 4-hexylresorcinol in dilute isobutanol at 70–80° (77%) [211].

m.p. and Spectra (NA).

(5-Hexyl-2,4-dihydroxyphenyl)(3-nitrophenyl)methanone



$C_{19}H_{21}NO_5$ mol.wt. 343.38

Synthesis

- Refer to: [113].

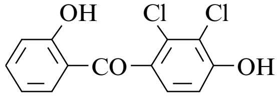
m.p. 85° [113]; Spectra (NA).

2.2.2 *Hydroxy Groups Located on Both Rings*

2.2.2.1 Substituents Located on One Ring

(2,3-Dichloro-4-hydroxyphenyl)(2-hydroxyphenyl)methanone

[72482-30-5]



$C_{13}H_8Cl_2O_3$ mol.wt. 283.11

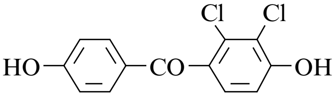
Synthesis

- Preparation by total demethylation of 2,3-dichloro-2′,4-dimethoxybenzophenone (SM) with pyridinium chloride at 200° for 1 h. SM was obtained by reaction of o-anisoyl chloride with 2,3-dichloroanisole in ethylene dichloride in the presence of aluminium chloride first at –10° for 2.5 h, then at +5° [476].
- Also refer to: [967].

m.p. 197–201° [476]; Spectra (NA).

(2,3-Dichloro-4-hydroxyphenyl)(4-hydroxyphenyl)methanone

[78697-41-3] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



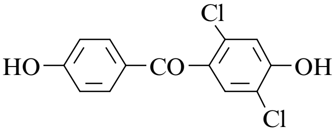
Synthesis

- Obtained by Friedel–Crafts acylation of 2,3-dichloro-anisole with p-methoxybenzoyl chloride in the presence of aluminium chloride at r.t. [979] or in benzene at 75–80° for 1 h (53%) [1288].

m.p. 208–210° [1288]; Spectra (NA).

(2,5-Dichloro-4-hydroxyphenyl)(4-hydroxyphenyl)methanone

[98155-78-3] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



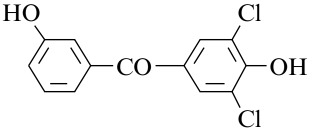
Synthesis

- Refer to: [901] (compound **20**).

m.p. and Spectra (NA).

(3,5-Dichloro-4-hydroxyphenyl)(3-hydroxyphenyl)methanone

[92005-28-2] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



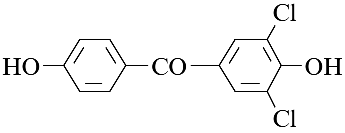
Synthesis

- Refer to: [1289].

m.p. and Spectra (NA).

(3,5-Dichloro-4-hydroxyphenyl)(4-hydroxyphenyl)methanone

[92005-19-1] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



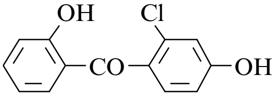
Synthesis

- Preparation by acylation of 2,6-dichlorophenol with p-(trichloromethyl)phenyl p-(trichloromethyl)benzoate in methylene chloride in the presence of aluminium chloride at 0–5° over 1 h, then at r.t. for 1 h, followed by alkaline hydrolysis of the resulting keto ester [325] (Japanese patent).

m.p. and Spectra (NA).

(2-Chloro-4-hydroxyphenyl)(2-hydroxyphenyl)methanone

[126165-47-7] $C_{13}H_{9}ClO_{3}$ mol.wt. 248.67



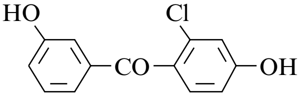
Synthesis

– Preparation by Fries rearrangement of m-chlorophenyl salicylate with aluminium chloride without solvent at 180–182° for 3 h (40%) [506].

m.p. 130° [506]; Spectra (NA).

(2-Chloro-4-hydroxyphenyl)(3-hydroxyphenyl)methanone

[126165-56-8] $C_{13}H_{9}ClO_{3}$ mol.wt. 248.67



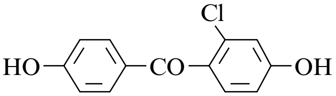
Synthesis

– Obtained (by-product) by Fries rearrangement of m-chloro-phenyl m-methoxybenzoate with aluminium chloride at 182–184° for 3 h (15%) [506].

m.p. 200° [506]; Spectra (NA).

(2-Chloro-4-hydroxyphenyl)(4-hydroxyphenyl)methanone

[98155-77-2] $C_{13}H_{9}ClO_{3}$ mol.wt. 248.67



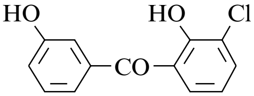
Synthesis

– Preparation by reaction of bis(4-trichloromethylphenoxy)-ketone with m-chlorophenol in methylene chloride in the presence of aluminium chloride, first at 0° for 1.5 h, then at 20° for 3 h, then hydrolysis of the intermediate product so obtained with 20% sodium hydroxide aqueous solution at 20° for 7 h (64%) [901].

m.p. 176–178° (anhydrous) and 100–108° (hemihydrate) [901]; Spectra (NA).

(3-Chloro-2-hydroxyphenyl)(3-hydroxyphenyl)methanone

[126165-53-5] $C_{13}H_{9}ClO_{3}$ mol.wt. 248.67



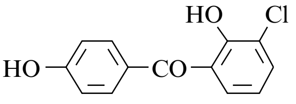
Synthesis

– Obtained (by-product) by Fries rearrangement of o-chloro-phenyl m-methoxybenzoate with aluminium chloride at 182–184° for 3 h (13%) [506].

m.p. 121° [506]; Spectra (NA).

(3-Chloro-2-hydroxyphenyl)(4-hydroxyphenyl)methanone

[126165-62-6] $C_{13}H_9ClO_3$ mol.wt. 248.67



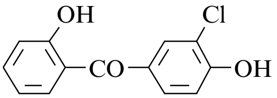
Synthesis

- Obtained (by-product) by Fries rearrangement of o-chloro-phenyl p-methoxybenzoate with aluminium chloride at 180° for 3 h (10%) [506].

m.p. 152° [506]; Spectra (NA).

(3-Chloro-4-hydroxyphenyl)(2-hydroxyphenyl)methanone

[123861-94-9] $C_{13}H_9ClO_3$ mol.wt. 248.67



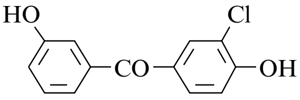
Syntheses

- Preparation by Fries rearrangement of o-chlorophenyl salicylate with aluminium chloride at 180–183° for 3 h (35%) [506].
- Preparation by hydrolysis of 2-chloro-4-salicyloylphenyl salicylate [346], a secondary product obtained by Fries rearrangement of o-chlorophenyl salicylate at 180–183° for 3 h (20%) [506].
- Also obtained (by-product) by Fries rearrangement of o-chlorophenyl 2-(nicotinoyloxy)benzoate with aluminium chloride at 150–152° for 2 h (4%) [346].

m.p. 113° [346,506]; Spectra (NA).

(3-Chloro-4-hydroxyphenyl)(3-hydroxyphenyl)methanone

[92005-08-8] $C_{13}H_9ClO_3$ mol.wt. 248.67



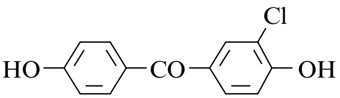
Synthesis

- Preparation by Fries rearrangement of o-chlorophenyl m-methoxybenzoate with aluminium chloride at 182–184° for 3 h (62%) [506].

m.p. 183° [506]; Spectra (NA).

(3-Chloro-4-hydroxyphenyl)(4-hydroxyphenyl)methanone

[92005-17-9] $C_{13}H_9ClO_3$ mol.wt. 248.67



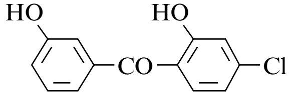
Synthesis

- Preparation by Fries rearrangement of o-chlorophenyl p-methoxybenzoate with aluminium chloride at 180° for 3 h (57%) [506].

m.p. 204° [506]; Spectra (NA).

(4-Chloro-2-hydroxyphenyl)(3-hydroxyphenyl)methanone

[126165-57-9] $C_{13}H_9ClO_3$ mol.wt. 248.67



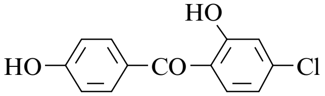
Synthesis

- Preparation by Fries rearrangement of m-chlorophenyl m-methoxybenzoate with aluminium chloride at 182–184° for 3 h (55%) [506].

m.p. 132° [506]; Spectra (NA).

(4-Chloro-2-hydroxyphenyl)(4-hydroxyphenyl)methanone

[126165-44-4] $C_{13}H_9ClO_3$ mol.wt. 248.67



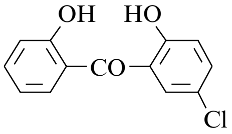
Synthesis

- Preparation by Fries rearrangement of m-chlorophenyl p-methoxybenzoate with aluminium chloride without solvent at 182–185° for 3 h (47%) [506].

m.p. 170° [506]; Spectra (NA).

(5-Chloro-2-hydroxyphenyl)(2-hydroxyphenyl)methanone

[76237-02-0] $C_{13}H_9ClO_3$ mol.wt. 248.67



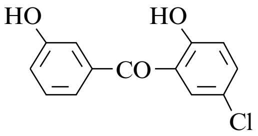
Syntheses

- Obtained by Fries rearrangement of p-chlorophenyl salicylate with aluminium chloride at 208–210° for 2.5 h (20%) [506].
- Also obtained by UV light irradiation of p-chlorophenyl salicylate in methanol for 8 h [1290].

m.p. 68–69° [1290], 59–60° [506]; ^{1}H NMR [1290], ^{13}C NMR [1290], IR [1290], UV [1290], MS [1290].

(5-Chloro-2-hydroxyphenyl)(3-hydroxyphenyl)methanone

[126165-59-1] $C_{13}H_9ClO_3$ mol.wt. 248.67



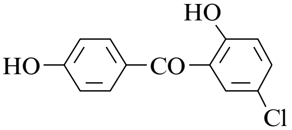
Synthesis

- Preparation by Fries rearrangement of p-chlorophenyl m-methoxybenzoate with aluminium chloride at 182–185° for 3 h (60%) [506].

m.p. 148° [506]; Spectra (NA).

(5-Chloro-2-hydroxyphenyl)(4-hydroxyphenyl)methanone

[126165-40-0] $C_{13}H_9ClO_3$ mol.wt. 248.67



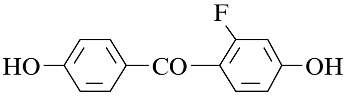
Synthesis

- Preparation by Fries rearrangement of p-chlorophenyl p-methoxybenzoate with aluminium chloride without solvent at 182–185° for 3 h (67%) [506].

m.p. 160° [506]; Spectra (NA).

(2-Fluoro-4-hydroxyphenyl)(4-hydroxyphenyl)methanone

[98155-81-8] $C_{13}H_9FO_3$ mol.wt. 232.21



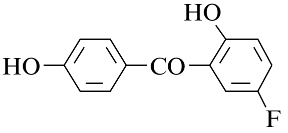
Synthesis

- Refer to: [901] (compound **30**).

m.p. and Spectra (NA).

(5-Fluoro-2-hydroxyphenyl)(4-hydroxyphenyl)methanone

[159300-38-6] $C_{13}H_9FO_3$ mol.wt. 232.21



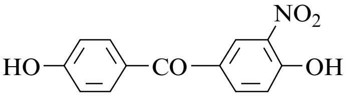
Synthesis

- Obtained by demethylation of 5-fluoro-2-hydroxy-4′-methoxybenzophenone with an excess of boron tribromide under nitrogen first at −70° for 2 min, then at 0° for 1 h (47%) [1106].

m.p. 104–105° [1106]; ^{1}H NMR [1106,1108], MS [1106,1108]; HPLC [1106,1108].

(4-Hydroxy-3-nitrophenyl)(4-hydroxyphenyl)methanone

[94737-85-6] $C_{13}H_9NO_5$ mol.wt. 259.22



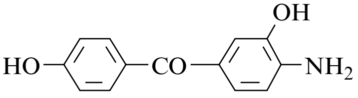
Synthesis

- Refer to: [325] (Japanese patent).

m.p. and Spectra (NA).

(4-Amino-3-hydroxyphenyl)(4-hydroxyphenyl)methanone

[136134-35-5] $C_{13}H_{11}NO_3$ mol.wt. 229.24

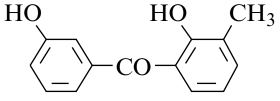


Synthesis

- Preparation by hydrolysis of 6-(4-hydroxybenzoyl)-benzoxazolinone in aqueous sodium hydroxide [564], according to [565].

m.p. (NA); MS [564].

(2-Hydroxy-3-methylphenyl)(3-hydroxyphenyl)methanone



$C_{14}H_{12}O_3$ mol.wt. 228.25

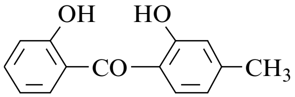
Synthesis

- Obtained by Fries rearrangement of o-cresyl m-anisate with aluminium chloride without solvent at 120° or 160° for 2 h [340].

m.p. 144° [340]; Spectra (NA).

(2-Hydroxy-4-methylphenyl)(2-hydroxyphenyl)methanone

[86415-67-0]



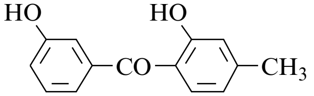
$C_{14}H_{12}O_3$ mol.wt. 228.25

Synthesis

- Refer to: [294].

m.p. and Spectra (NA).

(2-Hydroxy-4-methylphenyl)(3-hydroxyphenyl)methanone



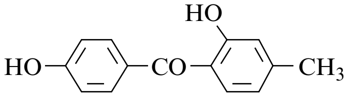
$C_{14}H_{12}O_3$ mol.wt. 228.25

Synthesis

- Obtained by Fries rearrangement of m-cresyl m-anisate with aluminium chloride without solvent at 120° or 160° for 2 h [340].

m.p. 105° [340]; Spectra (NA).

(2-Hydroxy-4-methylphenyl)(4-hydroxyphenyl)methanone



$C_{14}H_{12}O_3$ mol.wt. 228.25

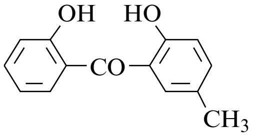
Synthesis

- Preparation by reaction of p-hydroxybenzoic acid with m-cresol in the presence of boron trifluoride at 150° for 3 h (68%) [150].

m.p. 148–149° [150]; Spectra (NA).

(2-Hydroxy-5-methylphenyl)(2-hydroxyphenyl)methanone

[93097-75-7]



$C_{14}H_{12}O_3$ mol.wt. 228.25

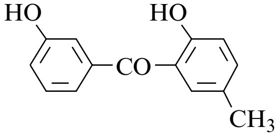
Syntheses

- Preparation by Fries rearrangement of p-tolyl salicylate with aluminium chloride at 140° for 3 h (70%) [345].
- Also obtained by reaction of o-methoxybenzoyl chloride with p-cresol in the presence of aluminium chloride at 100° for 24 h [683].
- Also refer to: [1135].

m.p. 143–144° [345]; Spectra (NA).

(2-Hydroxy-5-methylphenyl)(3-hydroxyphenyl)methanone

[57855-38-6]



$C_{14}H_{12}O_3$ mol.wt. 228.25

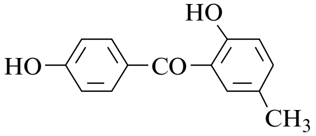
Syntheses

- Preparation by demethylation of 2-hydroxy-3′-methoxy-5-methylbenzophenone with hydrobromic acid in acetic acid [1132,1291].
- Also obtained by Fries rearrangement of p-tolyl m-anisate with aluminium chloride without solvent at 120° or 160° for 2 h (20–30%) [340].
- Also refer to: [1292].

m.p. 136° [340]; Spectra (NA).

(2-Hydroxy-5-methylphenyl)(4-hydroxyphenyl)methanone

[25148-21-4]



$C_{14}H_{12}O_3$ mol.wt. 228.25

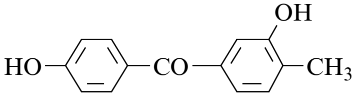
Syntheses

- Preparation by demethylation of 4′-hydroxy-2-methoxy-5-methylbenzophenone (m.p. 160°) with aluminium chloride at 150° [683].
- Preparation by diazotization of 4′-amino-2-hydroxy-5-methylbenzophenone (m.p. 137°), followed by hydrolysis of the resulting diazonium salt [683].

m.p. 150–151° [683]; Spectra (NA).

(3-Hydroxy-4-methylphenyl)(4-hydroxyphenyl)methanone

[75731-48-5]



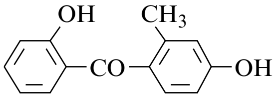
$C_{14}H_{12}O_3$ mol.wt. 228.25

Synthesis

- Refer to: [352,355].

m.p. and Spectra (NA).

(4-Hydroxy-2-methylphenyl)(2-hydroxyphenyl)methanone



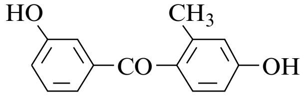
$C_{14}H_{12}O_3$ mol.wt. 228.25

Syntheses

- Obtained by Fries rearrangement,
  - of m-cresyl salicylate (1 mol) with aluminium chloride (4 mol) at 140° for 3 h (80%) [345];
  - of m-cresyl o-methoxybenzoate (1 mol) with aluminium chloride (2.6 mol) at 120° or 160° for 2 h (low yields) [340].

m.p. 146° [340,345]; Spectra (NA).

(4-Hydroxy-2-methylphenyl)(3-hydroxyphenyl)methanone



$C_{14}H_{12}O_3$ mol.wt. 228.25

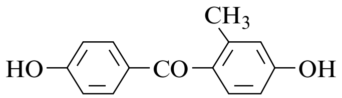
Synthesis

- Obtained by Fries rearrangement of m-cresyl m-anisate with aluminium chloride without solvent at 120° or 160° for 2 h [340].

m.p. 173° [340]; Spectra (NA).

(4-Hydroxy-2-methylphenyl)(4-hydroxyphenyl)methanone

[98155-72-7]



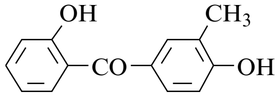
$C_{14}H_{12}O_3$ mol.wt. 228.25

Synthesis

- Preparation by adding m-cresol to a mixture of bis(4-tri-chloromethylphenoxy)ketone and aluminium chloride in methylene chloride at 0° for 1.5 h, then at 20° for 3 h. Then, hydrolysis of the intermediate product so obtained with 20% sodium hydroxide aqueous solution at 20° for 7 h (40%) [901].

m.p. 82–85° [901]; Spectra (NA).

(4-Hydroxy-3-methylphenyl)(2-hydroxyphenyl)methanone



$C_{14}H_{12}O_3$ mol.wt. 228.25

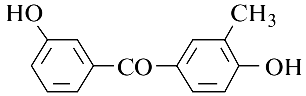
Synthesis

- Preparation by Fries rearrangement of o-cresyl salicylate with aluminium chloride without solvent at 140° for 3 h (80%) [345].

m.p. 112° [345]; Spectra (NA).

(4-Hydroxy-3-methylphenyl)(3-hydroxyphenyl)methanone

[92005-62-4]



$C_{14}H_{12}O_3$ mol.wt. 228.25

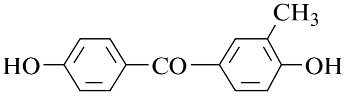
Syntheses

- Obtained by Fries rearrangement of o-cresyl m-methoxy-benzoate with aluminium chloride between 120° and 160° for 2 h (20–30%) [340].
- Preparation by demethylation of 3,4′-dimethoxy-3′-methylbenzophenone with 48% hydrobromic acid in an acetic anhydride/acetic acid mixture (1:1) for 15 h at reflux (78%) [352].

m.p. 174–175° [352], 172° [340]; Spectra (NA).

(4-Hydroxy-3-methylphenyl)(4-hydroxyphenyl)methanone

[92005-11-3] $C_{14}H_{12}O_3$ mol.wt. 228.25



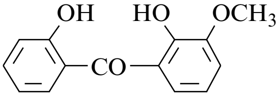
Synthesis

- Preparation by acylation of o-cresol with p-(trichloro-methyl)phenyl p-(trichloro-methyl)benzoate in methylene chloride in the presence of aluminium chloride at 0–5° over 1 h, then at r.t. for 1 h, followed by alkaline hydrolysis of the resulting keto ester [325] (Japanese patent).

m.p. and Spectra (NA).

(2-Hydroxy-3-methoxyphenyl)(2-hydroxyphenyl)methanone

[117574-12-6] $C_{14}H_{12}O_4$ mol.wt. 244.25



Synthesis

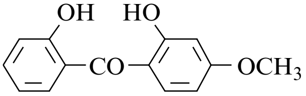
- Refer to: [1293].

m.p. and Spectra (NA).

(2-Hydroxy-4-methoxyphenyl)(2-hydroxyphenyl)methanone

(Dioxybenzone, Cyasorb UV 24)

[131-53-3] $C_{14}H_{12}O_4$ mol.wt. 244.25



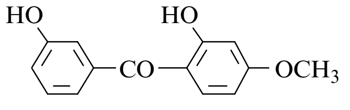
Syntheses

- Preparation by condensation of salicylic acid and m-methoxyphenol [1135].
- Preparation by reaction of o-anisoyl chloride with 1,3-di-methoxybenzene in the presence of aluminium chloride in chlorobenzene, first at 0°, then at 88° (21%) [684,1294].
- Preparation by partial demethylation of 2-hydroxy-2′,4′-dimethoxybenzophenone or 2,2′,4′-tri-methoxybenzophenone with aluminium chloride or aluminium bromide in chlorobenzene at 90–95° (good yield) [655].
- Preparation by partial methylation of 2,2′,4-trihydroxybenzophenone with dimethyl sulfate in alkaline solution (50%) [380].
- Also refer to: [225,235,666,1293,1295].

b.p.$_1$ 170–175° [684,1294];
m.p. 71–72° [1135], 70° [93], 69–70° [380], 69° [236]; ^{1}H NMR [303], IR [1135], UV [93,235,236,241,242,303,380,830], MS [1135];
pK_a [93,115]; TLC [116,244];
gel permeation chromatography [246,247]; vapour pressure [236];
paper chromatography [383].

(2-Hydroxy-4-methoxyphenyl)(3-hydroxyphenyl)methanone

[84394-12-7] $C_{14}H_{12}O_4$ mol.wt. 244.25



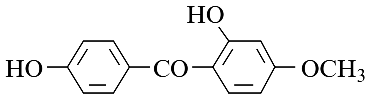
Synthesis

– Preparation by condensation of m-methoxyphenol with m-hydroxybenzoic acid in the presence of stannic chloride at reflux for 8 h (26%) [1296].

m.p. 113–114° [1296]; IR [1296].

(2-Hydroxy-4-methoxyphenyl)(4-hydroxyphenyl)methanone

[33257-86-2] $C_{14}H_{12}O_4$ mol.wt. 244.25



Syntheses

– Obtained by condensation of 2-hydroxy-4-methoxy-benzoic acid with phenol,
  - in the presence of stannic chloride at 115–120° for 3–4 h [190];
  - in the presence of zinc chloride at 115–120° for 3–4 h [237].

– Preparation by reaction of p-hydroxybenzoic acid with m-methoxyphenol in the presence of phosphorous oxychloride at 60–70° for 1.5 h (36%) [626,1297], 1213].

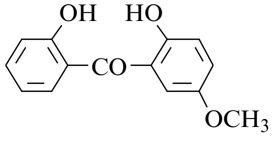
– Also refer to: [592,627,1016,1017,1298,1299].

m.p. 200° [626], 136–138° [190,237]. One of the reported melting points is obviously wrong.

IR [626], UV [190,237].

(2-Hydroxy-5-methoxyphenyl)(2-hydroxyphenyl)methanone

[83570-57-4] $C_{14}H_{12}O_4$ mol.wt. 244.25



Syntheses

– Preparation by saponification of 2′-(acetylamino)-2-hydroxy-5-methoxybenzophenone (SM) with 10% sodium hydroxide (37%). SM was obtained by photo-Fries rearrangement of p-methoxyphenyl 2-(acetylamino)-benzoate in benzene for 2.5 h (40%) [1115].

– Also obtained by photo-Fries rearrangement of p-methoxyphenyl salicylate in hexane for 4 h (8%) or in methanol for 11 h (23%) [1025].

– Also obtained by photo-Fries rearrangement of p-methoxyphenyl o-acetoxybenzoate in benzene, followed by saponification of the keto ester so formed [1148].

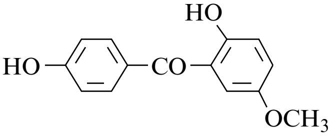
– Preparation by partial demethylation of 2,2′,5-trimethoxybenzophenone with aluminium chloride in benzene at 50° under nitrogen atmosphere for 12 h (40%) [395].

m.p. 93° [1025], 88–90° [395];

^{1}H NMR [98,395,1025], IR [395,1025], UV [395], MS [395]; TLC [395].

(2-Hydroxy-5-methoxyphenyl)(4-hydroxyphenyl)methanone

[80427-40-3] $C_{14}H_{12}O_4$ mol.wt. 244.25



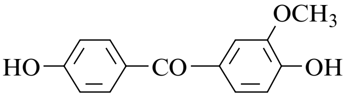
Synthesis

- Obtained by partial demethylation of 2,4′,5-trimethoxy-benzophenone (SM) with aluminium chloride in nitro-methane at 20° for 24 h. SM was obtained by Friedel–Crafts acylation of 1,4-dimethoxy-benzene with p-anisoyl chloride in the presence of stannic chloride in nitromethane at 20° for 1 h [679].
- Also refer to: [325] (Japanese patent).

m.p. 154° [679]; IR [679], UV [679].

(4-Hydroxy-3-methoxyphenyl)(4-hydroxyphenyl)methanone

[147904-63-0] $C_{14}H_{12}O_4$ mol.wt. 244.25



Synthesis

- Refer to: [1300].

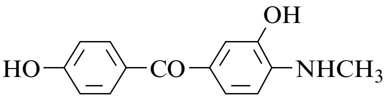
Isolation from natural source

- Identified from lignin dimers [1300].

m.p. (NA); GC-MS [1300].

[3-Hydroxy-4-(methylamino)phenyl](4-hydroxyphenyl)methanone

[136134-36-6] $C_{14}H_{13}NO_3$ mol.wt. 243.26



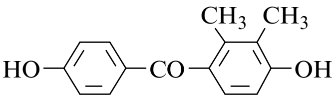
Synthesis

- Preparation from 6-(4-hydroxy-benzoyl)-3-methyl-benzoxazolinone by alkaline hydrolysis with boiling 10% aqueous sodium hydroxide solution [564].

m.p. and Spectra (NA).

(4-Hydroxy-2,3-dimethylphenyl)(4-hydroxyphenyl)methanone

[98155-73-8] $C_{15}H_{14}O_3$ mol.wt. 242.27



Synthesis

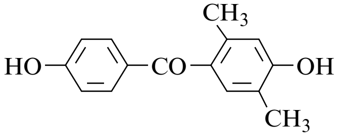
- Refer to: [901] (compound **2**).

m.p. and Spectra (NA).

(4-Hydroxy-2,5-dimethylphenyl)(4-hydroxyphenyl)methanone

[98155-75-0]

$C_{15}H_{14}O_3$ mol.wt. 242.27



Synthesis

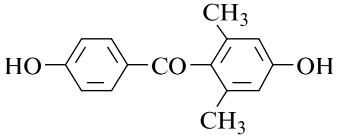
– Refer to: [901] (compound **5**).

m.p. and Spectra (NA).

(4-Hydroxy-2,6-dimethylphenyl)(4-hydroxyphenyl)methanone

[93899-05-9]

$C_{15}H_{14}O_3$ mol.wt. 242.27



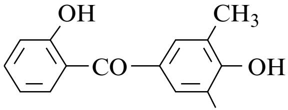
Synthesis

– Preparation by reaction of p-hydroxybenzoic acid with 3,5-xylenol in the presence of zinc chloride and a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 40°. Then, during 1.5 h, phosphorous trichloride was added and the mixture heated at 70° for 16 h (90%) [194].
– Also refer to: [901] (compound **3**).

m.p. and Spectra (NA).

(4-Hydroxy-3,5-dimethylphenyl)(2-hydroxyphenyl)methanone

$C_{15}H_{14}O_3$ mol.wt. 242.27



Synthesis

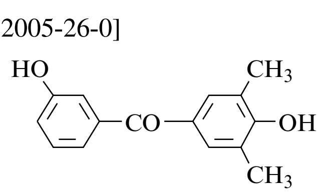
– Obtained by photo-Fries rearrangement of 2,6-dimethyl-phenyl salicylate (by-product) [219].

m.p. (NA); UV [219].

(4-Hydroxy-3,5-dimethylphenyl)(3-hydroxyphenyl)methanone

[92005-26-0]

$C_{15}H_{14}O_3$ mol.wt. 242.27



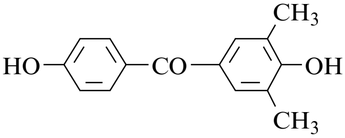
Synthesis

– Preparation by acylation of 2,6-dimethylphenol with m-(tri-chloromethyl)phenyl m-(trichloromethyl)benzoate in methylene chloride in the presence of aluminium chloride at 0–5° over 1 h, then at r.t. for 1 h, followed by alkaline hydrolysis of the resulting keto ester [325] (Japanese patent).

m.p. and Spectra (NA).

(4-Hydroxy-3,5-dimethylphenyl)(4-hydroxyphenyl)methanone

[92005-13-5] $C_{15}H_{14}O_3$ mol.wt. 242.27



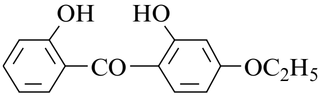
Synthesis

- Preparation by acylation of 2,6-dimethylphenol with p-(trichloromethyl)phenyl p-(trichloromethyl)benzoate in methylene chloride in the presence of aluminium chloride at 0–5° over 1 h, then at r.t. for 1 h, followed by alkaline hydrolysis of the resulting keto ester [325] (Japanese patent).

m.p. and Spectra (NA).

(4-Ethoxy-2-hydroxyphenyl)(2-hydroxyphenyl)methanone

$C_{15}H_{14}O_4$ mol.wt. 258.27



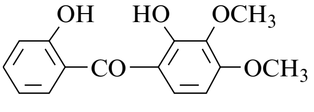
Synthesis

- Preparation by partial deethylation of 2,4-diethoxy-2′-hydroxybenzophenone with aluminium chloride in chlorobenzene at 80–100° [655].

m.p. and Spectra (NA).

(2-Hydroxy-3,4-dimethoxyphenyl)(2-hydroxyphenyl)methanone

$C_{15}H_{14}O_5$ mol.wt. 274.27



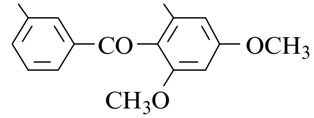
Synthesis

- Preparation by action of o-anisoyl chloride with pyrogallol trimethyl ether in the presence of aluminium chloride.
- Refer to: Chem. Abstr., **9**, 609^{5-6} $(1915)^T$.

m.p. $127°^T$; Spectra (NA).

(2-Hydroxy-4,6-dimethoxyphenyl)(3-hydroxyphenyl)methanone

[34425-65-5] $C_{15}H_{14}O_5$ mol.wt. 274.27



Synthesis

- Not yet described.

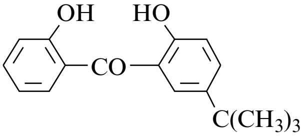
Isolation from natural source

- From the heartwood of *Allanblackia floribunda* Oliver (Guttiferae) [171].

m.p. 105–107° [171]; ^{1}H NMR [171], IR [171], UV [171], MS [171].

[5-(1,1-Dimethylethyl)-2-hydroxyphenyl](2-hydroxyphenyl)methanone

[125182-25-4]



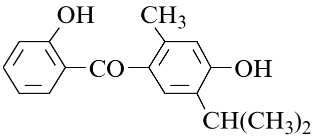
$C_{17}H_{18}O_3$ mol.wt. 270.33

Synthesis

- Obtained by treatment of 2,2′-dihydroxybenzophenone at 120° with a mixture of isobutylene/nitrogen (1:1) in the presence of a macroreticular acid ion exchanger (Wofatit OK 80) as catalyst for 1 h (40%) [819].

b.p.$_{0.15}$ 180–185° [819]; Spectra (NA).

[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl](2-hydroxyphenyl)methanone



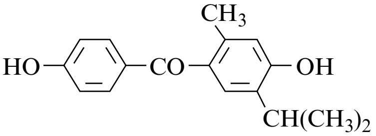
$C_{17}H_{18}O_3$ mol.wt. 270.33

Synthesis

- Preparation by demethylation of 2′,4-dimethoxy-2-methyl-5-isopropylbenzophenone with refluxing pyridinium chloride at 205–215° for 2 h (61%) [824].

b.p.$_{0.6}$ 195–200° [824]; m.p. 117–118° [824]; Spectra (NA).

[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl](4-hydroxyphenyl)methanone



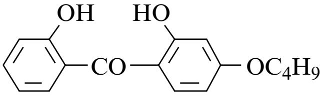
$C_{17}H_{18}O_3$ mol.wt. 270.33

Synthesis

- Obtained by demethylation of 4,4′-dimethoxy-2-methyl-5-isopropylbenzophenone in refluxing pyridinium chloride (205–215°) for 2 h (20%) [824].

b.p.$_{0.6}$ 200–210° [824]; Spectra (NA).

(4-Butoxy-2-hydroxyphenyl)(2-hydroxyphenyl)methanone



$C_{17}H_{18}O_4$ mol.wt. 286.33

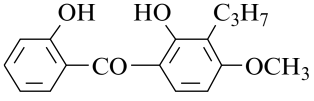
Synthesis

- Refer to: [383].

m.p. and Spectra (NA); paper chromatography [383].

(2-Hydroxy-4-methoxy-3-propylphenyl)(2-hydroxyphenyl)methanone

[115296-05-4] $C_{17}H_{18}O_4$ mol.wt. 286.33



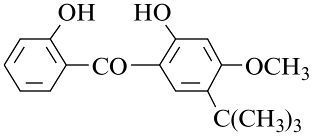
Syntheses

- Preparation by partial methylation of 2,2′,4-trihydroxy-3-propylbenzophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing 2-butanone for overnight (93%) [1029].
- Also obtained (trace) by reaction of o-anisoyl chloride with 1,3-dimethoxy-2-propylbenzene in methylene chloride in the presence of aluminium chloride, first at 0° for 2 h and at r.t. for 1 h [1029].

oil [1029]; b.p. and Spectra (NA).

[5-(1,1-Dimethylethyl)-2-hydroxy-4-methoxyphenyl](2-hydroxyphenyl) methanone

$C_{18}H_{20}O_4$ mol.wt. 300.35



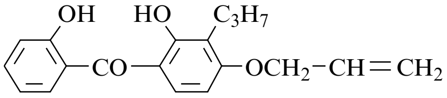
Synthesis

- Preparation by selective methylation of 5-tert-butyl-2,2′,4-trihydroxybenzophenone with dimethyl sulfate in refluxing methyl ethyl ketone in the presence of potassium carbonate [835].

m.p. 116°5 [835,836]; Spectra (NA).

(2-Hydroxyphenyl)[2-hydroxy-4-(2-propenyloxy)-3-propylphenyl]methanone

[115308-88-8] $C_{19}H_{20}O_4$ mol.wt. 312.37



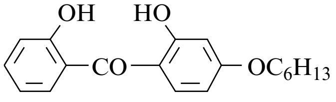
Synthesis

- Obtained by reaction of allyl bromide with 2,2′,4-trihydroxy-3-propylbenzophenone in the presence of potassium carbonate and potassium iodide in refluxing methyl ethyl ketone for 3 h (30%) [1029].

oil [1029]; b.p. and Spectra (NA).

[4-(Hexyloxy)-2-hydroxyphenyl](2-hydroxyphenyl)methanone

[65221-06-9] $C_{19}H_{22}O_4$ mol.wt. 314.38

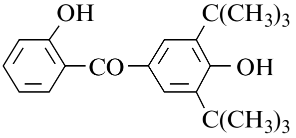


Synthesis

- Refer to: [233].

m.p. (NA); UV [233].

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](2-hydroxyphenyl)methanone



$C_{21}H_{26}O_3$ mol.wt. 326.44

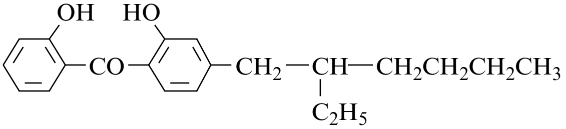
Synthesis

- Obtained by UV light irradiation of phenyl 3,5-di-tert-butyl-4-hydroxybenzoate in isooctane [1301].

m.p. 119° [1301]; Spectra (NA).

[4-(2-Ethylhexyl)-2-hydroxyphenyl](2-hydroxyphenyl)methanone

[84875-84-3] $C_{21}H_{26}O_3$ mol.wt. 326.44

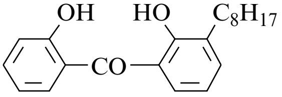


Synthesis

- Refer to: [1302].

m.p. and Spectra (NA).

(2-Hydroxy-3-octylphenyl)(2-hydroxyphenyl)methanone



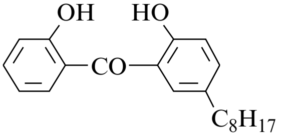
$C_{21}H_{26}O_3$ mol.wt. 326.44

Synthesis

- Obtained by photo-Fries rearrangement of o-octylphenyl salicylate (major product) [219].

m.p. (NA); UV [219].

(2-Hydroxy-5-octylphenyl)(2-hydroxyphenyl)methanone



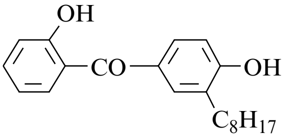
$C_{21}H_{26}O_3$ mol.wt. 326.44

Synthesis

- Obtained by photo-Fries rearrangement of p-octylphenyl salicylate (major product) [219].

m.p. (NA); UV [219].

(4-Hydroxy-3-octylphenyl)(2-hydroxyphenyl)methanone



$C_{21}H_{26}O_3$ mol.wt. 326.44

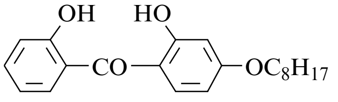
Synthesis

- Obtained by photo-Fries rearrangement of o-octylphenyl salicylate (by-product) [219].

m.p. (NA); UV [219].

[2-Hydroxy-4-(octyloxy)phenyl](2-hydroxyphenyl)methanone

[85-24-5] $C_{21}H_{26}O_4$ mol.wt. 342.44



Synthesis

– Preparation by reaction of octyl bromide with 2,2′,4-tri-hydroxybenzophenone,

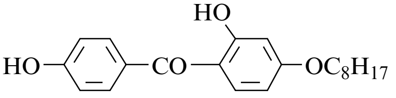
- in the presence of potassium carbonate (50%) [380];
- in the presence of sodium carbonate in refluxing dilute ethanol [1303].

– Also refer to: [888,892,1104,1215,1295,1304–1313].

m.p. 90°5–91° [380]; UV [380]; paper chromatography [383].

[2-Hydroxy-4-(octyloxy)phenyl](4-hydroxyphenyl)methanone

$C_{21}H_{26}O_4$ mol.wt. 342.44



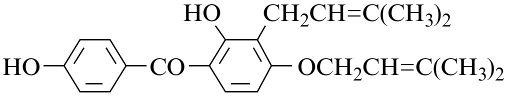
Synthesis

– Refer to: [1310] (Japanese patent).

m.p. and Spectra (NA).

[2-Hydroxy-3-(3-methyl-2-butenyl)-4-[(3-methyl-2-butenyl)oxy]phenyl]-(4-hydroxyphenyl)methanone

[63565-06-0] $C_{23}H_{26}O_4$ mol.wt. 366.46



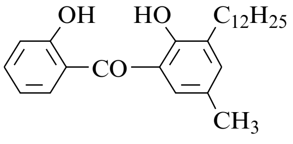
Synthesis

– Obtained (poor yield) by reaction of prenyl bromide with 2,4,4′-trihydroxy-benzophenone in the presence of potassium carbonate in refluxing acetone for 3 h (7%) [831].

m.p. 80° [831]; ^{1}H NMR [831], IR [831], UV [831].

(3-Dodecyl-2-hydroxy-5-methylphenyl)(2-hydroxyphenyl)methanone

$C_{26}H_{36}O_3$ mol.wt. 396.57

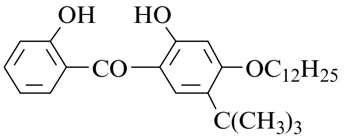


Synthesis

– Obtained by photo-Fries rearrangement of 2-dodecyl-4-methylphenyl salicylate (major product) [219].

m.p. (NA); UV [219].

[5-(1,1-Dimethylethyl)-4-(dodecyloxy)-2-hydroxyphenyl](2-hydroxyphenyl) methanone



$C_{29}H_{42}O_4$ mol.wt. 454.65

Synthesis

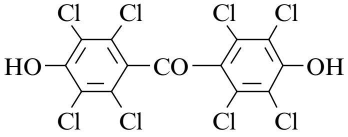
- Preparation by selective alkylation of 5-tert-butyl-2,2′,4-trihydroxybenzophenone with lauryl bromide in refluxing methyl ethyl ketone in the presence of potassium carbonate [835].

m.p. 81° [835,836]; Spectra (NA).

2.2.2.2 Substituents Located on Both Rings

Symmetrical ketones

Bis(2,3,5,6-tetrachloro-4-hydroxyphenyl)methanone



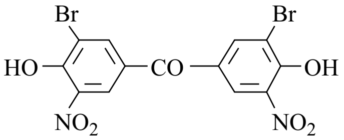
$C_{13}H_2Cl_8O_3$ mol.wt. 489.78

Synthesis

- Refer to: [901] (compound **12**).

m.p. and Spectra (NA).

Bis(3-bromo-4-hydroxy-5-nitrophenyl)methanone



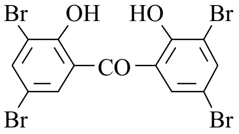
$C_{13}H_6Br_2N_2O_7$ mol.wt. 462.01

Synthesis

- Preparation by adding sodium nitrite to an hot solution of 3,3′,5,5′-tetrabromo-4,4′-dihydroxybenzophenone in acetic acid [326].

m.p. 246° [326]; Spectra (NA).

Bis(3,5-dibromo-2-hydroxyphenyl)methanone



$C_{13}H_6Br_4O_3$ mol.wt. 529.80

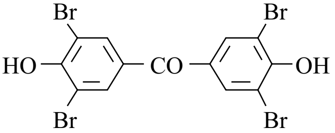
Synthesis

- Obtained by treatment of 2,2′-dihydroxybenzophenone in hot acetic acid with bromine (60%) [1314].
- Also refer to: [297].

m.p. 178°5–180°5 [1314]; Spectra (NA).

Bis(3,5-dibromo-4-hydroxyphenyl)methanone

[28818-29-3]



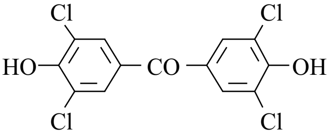
$C_{13}H_6Br_4O_3$ mol.wt. 529.80

Syntheses

- Obtained by action of bromine with 4,4′-dihydroxy-benzophenone [327,329], (86%) [1314].
- Also obtained by oxidation of α,α-bis(3,5-dibromo-4-hydroxyphenyl)dichloroethylene by heating with chromium trioxide (81%) [1315].

m.p. 230–232° [1314], 225–226° [326,1315], 213–214° [327,329]; Spectra (NA).

Bis(3,5-dichloro-4-hydroxyphenyl)methanone



$C_{13}H_6Cl_4O_3$ mol.wt. 352.00

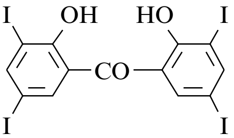
Synthesis

- Preparation by reaction of chlorine with 4,4′-dihydroxy-benzophenone in acetic acid [326].
- Also refer to: [1316,1317] (Japanese papers).

m.p. 231–232° [326]; Spectra (NA).

Bis(2-hydroxy-3,5-diiodophenyl)methanone

[33417-57-1]



$C_{13}H_6I_4O_3$ mol.wt. 717.80

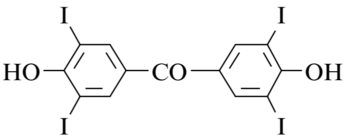
Synthesis

- Preparation by iodination of 2,2′-dihydroxybenzophenone,
  - with iodine and iodic acid in dilute ethanol for 20 min (77%) [460];
  - with iodine and potassium iodide in aqueous ammonia [460].

m.p. 225° [460]; Spectra (NA).

Bis(4-hydroxy-3,5-diiodophenyl)methanone

[15198-16-0]



$C_{13}H_6I_4O_3$ mol.wt. 717.80

Syntheses

- Preparation by iodination of 4,4′-dihydroxy-benzophenone,
  - with iodine and potassium iodide in ammonia for 1 h (65%) [460];

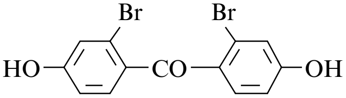
- with iodine and iodic acid in dilute ethanol for 15 min [460];
- with iodine monochloride (Wijs'chloride) at r.t. [460].

– Also obtained (by-product) by reaction of 4-hydroxy-3,5-diiodophenylpyruvic acid with 4-hydroxy-3,5-diiodobenzoic acid (6%) [333], according to the methods [1318,1319].

m.p. 255° (d) [333], 254° (d) [460]; IR [333], UV [333].

Bis(2-bromo-4-hydroxyphenyl)methanone

[98155-80-7] $C_{13}H_8Br_2O_3$ mol.wt. 372.01



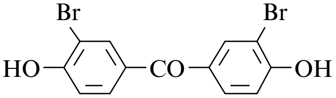
Synthesis

– Refer to: [901] (compound **29**).

m.p. and Spectra (NA).

Bis(3-bromo-4-hydroxyphenyl)methanone

[5423-21-2] $C_{13}H_8Br_2O_3$ mol.wt. 372.01



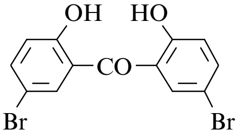
Synthesis

– Preparation by demethylation of 3,3′-dibromo-4,4′-di-methoxybenzophenone (SM) with pyridinium chloride at 210° for 20 min (85%) [313]. SM was obtained by action of bromine with 4,4′-dimethoxybenzophenone in acetic acid for 1 h in daylight (77%).

m.p. 214–215° [313]; ^{1}H NMR [313], IR [313].

Bis(5-bromo-2-hydroxyphenyl)methanone

$C_{13}H_8Br_2O_3$ mol.wt. 372.01



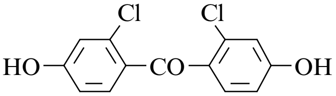
Synthesis

– Refer to: [297].

m.p. and Spectra (NA).

Bis(2-chloro-4-hydroxyphenyl)methanone

[94323-04-3] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



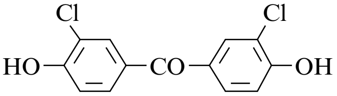
Synthesis

– Refer to: [901] (compound **13**).

m.p. and Spectra (NA); hemihydrate [901].

Bis(3-chloro-4-hydroxyphenyl)methanone

[79616-16-3] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



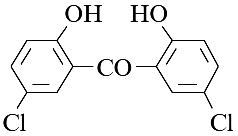
Synthesis

– Preparation by demethylation of 3,3′-dichloro-4,4′-di-methoxybenzophenone (SM) with pyridinium chloride at 210° for 20 min (84%) [313]. SM was obtained by chlorination of 4,4′-dimethoxybenzophenone with sulfuryl chloride in methylene chloride at 45° for 5 h (74%).

m.p. 206–207° [313]; ^{1}H NMR [313], IR [313].

Bis(5-chloro-2-hydroxyphenyl)methanone

[6178-89-8] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



Synthesis

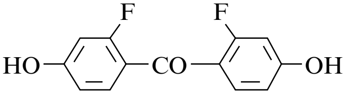
– Preparation by oxidation of 2,2′-methylenebis(4-chloro-anisole) with chromium trioxide in refluxing acetic acid, followed by demethylation of the 5,5′-dichloro-2,2′-di-methoxybenzophenone so obtained with aluminium chloride in chlorobenzene at 60° for 5.5 h [295], (92%) [1320].

– Also refer to: [297,1321–1323].

m.p. 151–152° [1320]; Spectra (NA).

Bis(2-fluoro-4-hydroxyphenyl)methanone

[98155-79-4] $C_{13}H_8F_2O_3$ mol.wt. 250.20



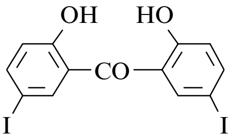
Synthesis

– Refer to: [901] (compound **28**).

m.p. and Spectra (NA).

Bis(2-hydroxy-5-iodophenyl)methanone

[33417-58-2] $C_{13}H_8I_2O_3$ mol.wt. 466.01

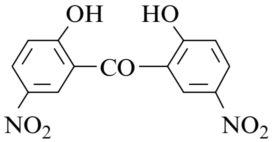


Synthesis

– Obtained by iodination of 2,2′-dihydroxybenzophenone with iodine monochloride in acetic acid for 24 h at r.t. (23%) [460].

m.p. 194–195° [460]; Spectra (NA).

Bis(2-hydroxy-5-nitrophenyl)methanone



$C_{13}H_8N_2O_7$ mol.wt. 304.22

Synthesis

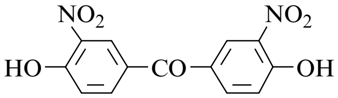
N.B.: The 2,7-dinitroxanthone was obtained on heating 2,2′-dimethoxy-5,5′-dinitrobenzophenone (m.p. 188°) with 75% sulfuric acid at 150° for 1 h via 2,2′-dihydroxy-5,5′-dinitrobenzophenone [288].

- Also refer to: [187,1324].

m.p. and Spectra (NA).

Bis(4-hydroxy-3-nitrophenyl)methanone

[37567-35-4]



$C_{13}H_8N_2O_7$ mol.wt. 304.22

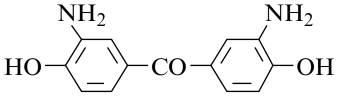
Synthesis

- Preparation by hydrolysis of 4,4′-dichloro-3,3′-dinitro-benzophenone with 5–15% sodium hydroxide at 155–160° for 1–1.5 h (97–98%) [1071].

m.p. 195°2–195°5 [1071]; Spectra (NA).

Bis(3-amino-4-hydroxyphenyl)methanone

[22445-98-3]



$C_{13}H_{12}N_2O_3$ mol.wt. 244.25

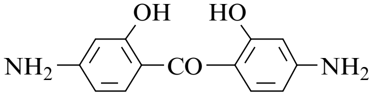
Synthesis

- Preparation by hydrogenation of 4,4′-dihydroxy-3,3′-di-nitrobenzophenone in the presence of Raney nickel in water at 85–90° for 1 h under 100 atmospheres (69%) [1071].

m.p. >220° (d) [1071]; Spectra (NA).

Bis(4-amino-2-hydroxyphenyl)methanone

[107516-91-6]



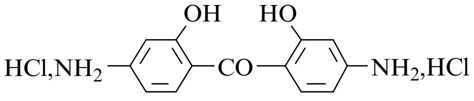
$C_{13}H_{12}N_2O_3$ mol.wt. 244.25

Synthesis

- Preparation from 4-amino-2′-hydroxy-2-methoxy-4′-nitrobenzophenone by refluxing with freshly distilled constant-boiling hydriodic acid at 140° for 7 h (81%) [286].

m.p. 247–248° [286]; Spectra (NA).

Bis(4-amino-2-hydroxyphenyl)methanone *(Dihydrochloride)*



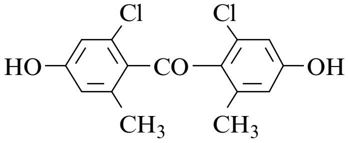
$C_{13}H_{12}N_2O_3$,2HCl mol.wt. 317.17

Synthesis

- Preparation from 4,4′-diamino-2,2′-di-hydroxybenzophenone with hydrochloric acid in refluxing dilute ethanol (74%) [286].

m.p. 208° (d) [286]; Spectra (NA).

Bis(2-chloro-4-hydroxy-6-methylphenyl)methanone



$C_{15}H_{12}Cl_2O_3$ mol.wt. 311.16

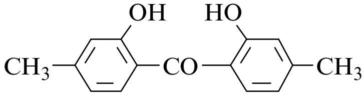
Synthesis

- Refer to: [901] (compound **35**).

m.p. and Spectra (NA).

Bis(2-hydroxy-4-methylphenyl)methanone

[24018-76-6]



$C_{15}H_{14}O_3$ mol.wt. 242.27

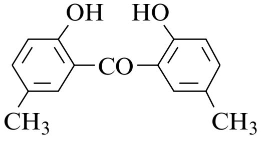
Synthesis

- Refer to: [668] and also [1325–1327] (Japanese papers).

m.p. and Spectra (NA).

Bis(2-hydroxy-5-methylphenyl)methanone

[27404-62-2]



$C_{15}H_{14}O_3$ mol.wt. 242.27

Syntheses

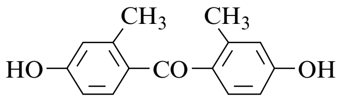
- Preparation by Friedel–Crafts acylation of p-methoxytoluene with 2-methoxy-5-methylbenzoyl chloride in carbon disulfide in the presence of aluminium chloride, first at 0° for 5 h, then at r.t. overnight and at reflux for 2 h (74%) [1153].
- Also obtained by total demethylation of 2,2′-dimethoxy-5,5′-dimethylbenzophenone (m.p. 84°) with hydriodic acid in refluxing acetic acid for 7 h (83%) [1153].
- Also obtained (poor yield) by Fries rearrangement of p-cresyl 2-methoxy-5-methylbenzoate in nitrobenzene with aluminium chloride for 20 min at 130° (9%) [1153].

- Also obtained (poor yield) by alkaline melting of p-cresolphthalein at 200° with potassium hydroxide [1153,1328].
- Also obtained by fusion of 2,7-dimethylfluoran with potassium hydroxide at 220–240° [617].
- Also refer to: [1154,1329,1330].

m.p. 107° [1154], 106–107° [1153,1329], 104–106° [617], 104–105° [1328]; IR [1154], UV [1154], MS [1329].

Bis(4-hydroxy-2-methylphenyl)methanone

[98155-74-9]



$C_{15}H_{14}O_3$ mol.wt. 242.27

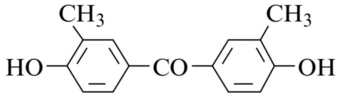
Synthesis

- Refer to: [901] (compound **4**).

m.p. and Spectra (NA).

Bis(4-hydroxy-3-methylphenyl)methanone

[94323-02-1]



$C_{15}H_{14}O_3$ mol.wt. 242.27

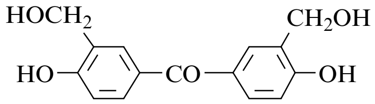
Syntheses

- Obtained by condensation of o-cresol with carbon tetrachloride in the presence of metallic halides (zinc chloride, aluminium chloride or stannic chloride) at 100–130° in an autoclave [642].
- Obtained by oxidation of o-cresaurin in 5% sodium hydroxide solution by passing a very slow stream of air during several days (41%) [642].

m.p. 240° [642]; Spectra (NA).

Bis[4-hydroxy-3-(hydroxymethyl)phenyl]methanone

[74697-55-5]



$C_{15}H_{14}O_5$ mol.wt. 274.27

Synthesis

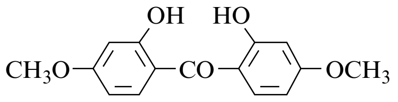
- Refer to: [309].

m.p. and Spectra (NA).

Bis(2-hydroxy-4-methoxyphenyl)methanone

(UV 12, Uvinul D-49, Uvinul 490, Uvinul 3049)

[131-54-4]



$C_{15}H_{14}O_5$ mol.wt. 274.27

Syntheses

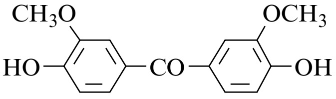
- Preparation by reaction of 2-hydroxy-4-methoxy-benzoic acid with resorcinol monomethyl ether in the presence of zinc chloride and phosphorous oxychloride at 70–75° for 2 h (27%) [402].

- Preparation by reaction of 2,4-dimethoxybenzoyl chloride with 1,3-dimethoxy benzene in the presence of aluminium chloride,
  - in a chlorobenzene/N,N-dimethylformamide mixture (22:1) at 115° (72%) [235,657];
  - in chlorobenzene at 90° [222].
- Preparation by partial demethylation of 2-hydroxy-2′,4,4′-trimethoxybenzophenone or 2,2′,4,4′-tetramethoxybenzophenone with aluminium chloride or aluminium bromide in chlorobenzene at 90–95° (good yield). The same result is obtained using ethylene dichloride or nitrobenzene as the solvent [655].
- Preparation by reaction of phosgene with resorcinol dimethyl ether in the presence of aluminium chloride [215].
- Also refer to: [78,84,222,225,227,228,241,298,666,668,684].

m.p. 139–140° [402], 137° [215], 136–137° [650], 135° [236],
133–136° [93], 133–135° [222];
EPR [98], IR [650], UV [93,215,235,236,240,241,650,684,830];
TLC [116]; vapour pressure [236]; pK_a [93];
gel permeation chromatography [247].

Bis(4-hydroxy-3-methoxyphenyl)methanone

[5623-44-9] $C_{15}H_{14}O_5$ mol.wt. 274.27



Syntheses

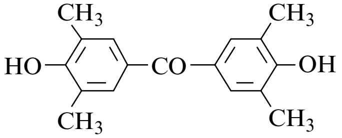
- Obtained from 4-hydroxy-3,3′,4′-trimethoxy-1,2-di-phenyl-1,2-ethanediol (*Z* configuration) after 3 h radiolysis of aqueous sodium hydroxide solution [1331].
- Also obtained by heating a mixture of the 4,4′-dihydroxy-3,3′-dimethoxydiphenylmethane, 2 N sodium hydroxide and nitrobenzene for 2 h at 170° (21%) [1332,1333].
- Also obtained by mild oxidation of various materials^T with cupric hydroxide in aqueous sodium hydroxide at 170° for 5 h in a stainless steal bomb,
  ^{T}three polymers prepared from isoeugenol by oxidative coupling with cuprous chloride in pyridine at 100° under oxygen [1334];
  ^{T}dehydrodiisoeugenol [1334];
  ^{T}milled wood lignin [1334];
  ^{T}lignin and related products [1335].
- Also refer to: [1336–1338].

N. B.: Na salt [215].

m.p. (NA); IR [215];

TLC [1334]; GLC [1334,1335]; GC and GC-MS [1332,1333].

Bis(4-hydroxy-3,5-dimethylphenyl)methanone



$C_{17}H_{18}O_3$ mol.wt. 270.33

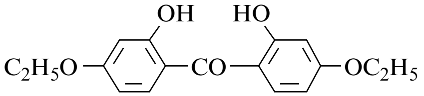
Synthesis

- Refer to: Chem. Abstr., **127**, 109367s (1997).

m.p. and Spectra (NA).

Bis(4-ethoxy-2-hydroxyphenyl)methanone

[15889-67-5]



$C_{17}H_{18}O_5$ mol.wt. 302.33

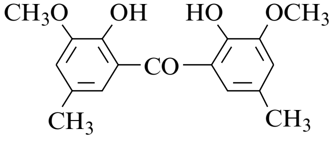
Synthesis

- Preparation by partial dealkylation of 2-hydroxy-2′-methoxy-4,4′-diethoxybenzophenone, 2-hydroxy-2′,4,4′-triethoxybenzophenone or 2,2′,4,4′-tetraethoxybenzophenone with aluminium chloride in chlorobenzene at 80–100° [655].

m.p. and Spectra (NA).

Bis(2-hydroxy-3-methoxy-5-methylphenyl)methanone

[17772-33-7]



$C_{17}H_{18}O_5$ mol.wt. 302.33

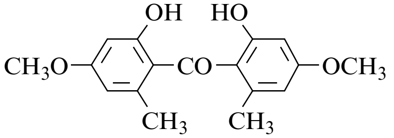
Synthesis

- Obtained by heating a mixture of the 2,2′-dihydroxy-3,3′-dimethoxy-5,5′-dimethyldiphenylmethane, 2 N sodium hydroxide and nitrobenzene for 2 h at 170° (24%) [1332,1333].

m.p. and Spectra (NA); GC and GC-MS [1332,1333].

Bis(2-hydroxy-4-methoxy-6-methylphenyl)methanone

[78135-60-1]



$C_{17}H_{18}O_5$ mol.wt. 302.33

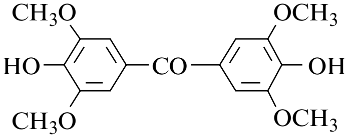
Synthesis

- Preparation by reduction of 4′,6-dimethoxy-4,6′-di-methylspiro[78135-60-1] -2′,3 (*2H*) dione with zinc dust in acetic acid at r.t. for 1 h (94%) [1339].

m.p. 159–160° [1339]; ^{1}H NMR [1339], MS [1339].

Bis(4-hydroxy-3,5-dimethoxyphenyl)methanone

[34007-64-2] $C_{17}H_{18}O_7$ mol.wt. 334.33



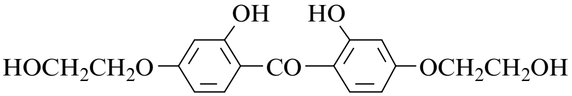
Synthesis

- Obtained by heating a mixture of the 4,4′-dihydroxy-3,3′,5,5′-tetramethoxy-diphenylmethane (disyringyl-methane), 2 N sodium hydroxide and nitrobenzene for 2 h at 170° (18%) [1332,1333].

m.p. and Spectra (NA); GC and GC-MS [1332,1333].

Bis[2-hydroxy-4-(2-hydroxyethoxy)phenyl]methanone

[15577-13-6] $C_{17}H_{18}O_7$ mol.wt. 334.33



Synthesis

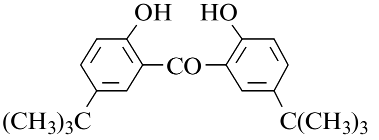
- Preparation by bubbling ethylene oxide into a hot mixture of 2,2′,4,4′-tetrahydroxy-benzophenone and aqueous sodium hydroxide during 1 h at 55° (98%) [1340].

- Also refer to: [93,116].

m.p. 152° [93]; UV [93]; pK_a [93]; TLC [116].

Bis[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone

[25446-98-4] $C_{21}H_{26}O_3$ mol.wt. 326.44

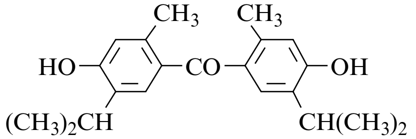


Syntheses

- Obtained by photo-Fries rearrangement of p-tert-butylphenyl carbonate,
  - in ethanol for 24 h (26%) [68];
  - in benzene for 24 h (36%) [68];
  - in ethylene dichloride [1341], where the rearrangement proceeds via the formation of an intermediate ester, the p-tert-butylphenyl 5-tert-butylsalicylate [1341].
- Also obtained by treatment of 2,2′-dihydroxybenzophenone at 120° with a mixture of isobutylene/nitrogen (1:1) in the presence of a macroreticular acid ion exchanger (Wofatit OK 80) as catalyst for 1 h (30%) [819].

b.p.$_{0.15}$ 200–205° [819]; m.p. 104–106° [68]; IR [68].

Bis[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]methanone



$C_{21}H_{26}O_3$ mol.wt. 326.44

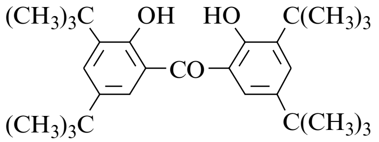
Synthesis

- Obtained by total demethylation of 5,5′-di-isopropyl-4,4′-dimethoxy-2,2′-dimethyl-benzophenone with refluxing pyridinium chloride (205–215°) for 3 h (26%) [824].

b.p.$_{0.9}$ 215–225° [824]; Spectra (NA).

Bis[3,5-bis(1,1-dimethylethyl)-2-hydroxyphenyl]methanone

[30381-72-7]



$C_{29}H_{42}O_3$ mol.wt. 438.65

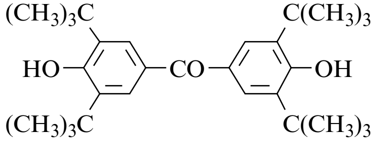
Syntheses

- Preparation by oxidation of bis (2-hydroxy-3,5-di-tert-butylphenyl) methane (SM) with chromium trioxide in acetic anhydride. SM was obtained by condensation of 2,4-di-tert-butylphenol with formaldehyde under acidic condition [1342].
- Preparation by treatment of 2,2′-dihydroxybenzophenone at 120° with a mixture of isobutylene/nitrogen (1:1) in the presence of a macroreticular acid ion exchanger (Wofatit OK 80) as catalyst for 10 h (75%) [819].

b.p.$_{0.19}$ 230–240° [819]; m.p. 202–204° [1342]; MS [1342].

Bis[3,5-bis(1,1-dimethylethyl)-4-hydroxyphenyl]methanone

[29372-72-3]



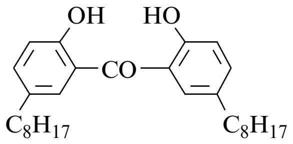
$C_{29}H_{42}O_3$ mol.wt. 438.65

Synthesis

- Preparation by demethylation of 3,3′, 5,5′-tetra-tert-butyl-4,4′-dimethoxy-benzophenone (SM) by means of sodium thioethoxide, under nitrogen, in refluxing N,N-dimethylformamide for 15 h (95%). SM was obtained by a two-step synthesis: at first, total methylation of 3,3′,5,5′-tetra-tert-butyl-4,4′-dihydroxydiphenylmethane with methyl iodide in the presence of sodium hydride, under nitrogen, in refluxing tetrahydrofuran for 2 h. Then, by adding a solution of chromium trioxide in dilute sulfuric acid to an acetonic solution of the dimethyl ether previously formed (82%) and stirring at r.t. for 70 h, one obtains the expected ketone SM (86%) [334].

m.p. 226°5–227° [334]; ^{1}H NMR [334], IR [334].

Bis(2-hydroxy-5-octylphenyl)methanone



$C_{29}H_{42}O_3$ mol.wt. 438.65

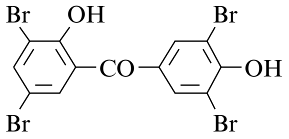
Synthesis

- Obtained by photo-Fries rearrangement of p-octylphenyl 5-octylsalicylate (major product) [219].

m.p. (NA); UV [219].

Asymmetric ketones

(3,5-Dibromo-2-hydroxyphenyl)(3,5-dibromo-4-hydroxyphenyl)methanone



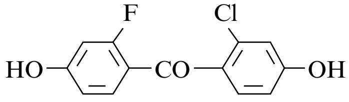
$C_{13}H_6Br_4O_3$ mol.wt. 529.80

Synthesis

- Preparation by reaction of bromine with 2,4′-dihydroxy-benzophenone in acetic acid (84%) [1314].

m.p. 193–195°5 [1314]; Spectra (NA).

(2-Chloro-4-hydroxyphenyl)(2-fluoro-4-hydroxyphenyl)methanone



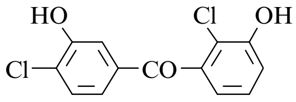
$C_{13}H_8ClFO_3$ mol.wt. 266.65

Synthesis

- Refer to: [901] (compound **32**).

m.p. and Spectra (NA).

(2-Chloro-3-hydroxyphenyl)(4-chloro-3-hydroxyphenyl)methanone



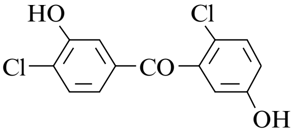
$C_{13}H_8Cl_2O_3$ mol.wt. 283.11

Synthesis

- Preparation by demethylation of 2,4′-dichloro-3,3′-di-methoxybenzophenone with aluminium chloride in chlorobenzene at 60° for 5.5 h (88%) [1320].

m.p. 157–158°5 [1320]; Spectra (NA).

(2-Chloro-5-hydroxyphenyl)(4-chloro-3-hydroxyphenyl)methanone



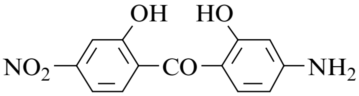
$C_{13}H_8Cl_2O_3$ mol.wt. 283.11

Synthesis

- Preparation by diazotization of 3′,5-diamino-2,4′-dichloro-benzophenone, followed by treatment of the diazonium salt obtained with boiling 70% sulfuric acid (160°) for 10 min (32%) [1320].

m.p. 161° [1320]; Spectra (NA).

(4-Amino-2-hydroxyphenyl)(2-hydroxy-4-nitrophenyl)methanone



$C_{13}H_{10}N_2O_5$ mol.wt. 274.23

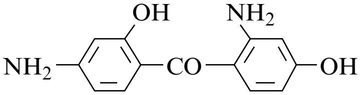
Syntheses

- Preparation by hydrolysis of 4-acetamido-2,2′-di-hydroxy-4′-nitrobenzophenone with refluxing 20% hydrochloric acid for 3 h [286].
- Preparation by demethylation of 4-amino-2′-hydroxy-2-methoxy-4′-nitrobenzophenone with 50% hydrobromic acid in refluxing acetic acid for 6 h (78%) [286].

m.p. 224–226° [286]; Spectra (NA).

(2-Amino-4-hydroxyphenyl)(4-amino-2-hydroxyphenyl)methanone

[107518-30-9]



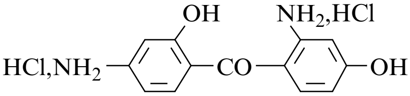
$C_{13}H_{12}N_2O_3$ mol.wt. 244.25

Synthesis

- Preparation from 2-amino-2′-hydroxy-4-methoxy-4′-nitrobenzophenone by refluxing with freshly distilled constant-boiling hydriodic acid at 140° for 7 h (63%) [286].

m.p. 190–191° [286]; Spectra (NA).

(2-Amino-4-hydroxyphenyl)(4-amino-2-hydroxyphenyl)methanone
(Dihydrochloride)



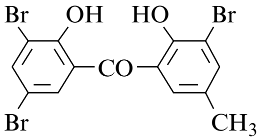
$C_{13}H_{12}N_2O_3$,2HCl mol.wt. 317.17

Synthesis

- Preparation from 2,4′-diamino-2′,4-dihydroxy-benzophenone with hydrochloric acid in refluxing dilute ethanol (72%) [286].

m.p. 218° (d) [286]; Spectra (NA).

(3-Bromo-2-hydroxy-5-methylphenyl)(3,5-dibromo-2-hydroxyphenyl) methanone



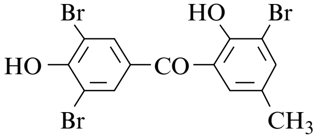
$C_{14}H_9Br_3O_3$ mol.wt. 464.94

Synthesis

- Obtained by reaction of excess bromine with 2,2′-di-hydroxy-5-methylbenzophenone in acetic acid [683].

m.p. 190° [683]; Spectra (NA).

(3-Bromo-2-hydroxy-5-methylphenyl)(3,5-dibromo-4-hydroxyphenyl) methanone



$C_{14}H_9Br_3O_3$ mol.wt. 464.94

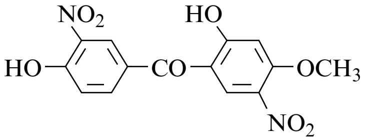
Synthesis

- Obtained by reaction of bromine with 2,4′-dihydroxy-5-methylbenzophenone in acetic acid [683].

m.p. 211°5–212°5 [683]; Spectra (NA).

(2-Hydroxy-4-methoxy-5-nitrophenyl)(4-hydroxy-3-nitrophenyl)methanone

[67246-05-3]



$C_{14}H_{10}N_2O_8$ mol.wt. 334.24

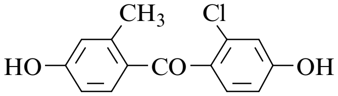
Synthesis

- Obtained by reaction of nitric acid (d = 1.42) with 2,4′-dihydroxy-4-methoxybenzophenone in acetic acid at 32° [592].

m.p. 185° [592]; ^{1}H NMR [592].

(2-Chloro-4-hydroxyphenyl)(4-hydroxy-2-methylphenyl)methanone

[98155-83-0]



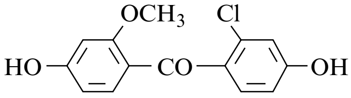
$C_{14}H_{11}ClO_3$ mol.wt. 262.69

Synthesis

- Refer to: [901] (compound **34**).

m.p. and Spectra (NA).

(2-Chloro-4-hydroxyphenyl)(4-hydroxy-2-methoxyphenyl)methanone



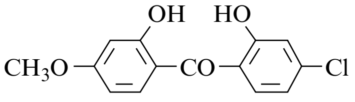
$C_{14}H_{11}ClO_4$ mol.wt. 278.69

Synthesis

- Refer to: [901] (compound **36**).

m.p. and Spectra (NA).

(4-Chloro-2-hydroxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone



$C_{14}H_{11}ClO_4$ mol.wt. 278.69

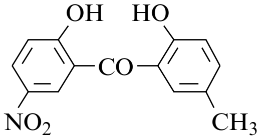
Synthesis

- Preparation by reaction of 2,4-dimethoxybenzoyl chloride with m-chloroanisole in chlorobenzene in the presence of aluminium chloride at 90° [222].

m.p. and Spectra (NA).

(2-Hydroxy-5-methylphenyl)(2-hydroxy-5-nitrophenyl)methanone

[145804-70-2]



$C_{14}H_{11}NO_5$ mol.wt. 273.25

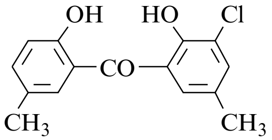
Synthesis

- Obtained by cleavage of 2-methyl-7-nitroxanthone with 10% potassium hydroxide in refluxing methanol for 12 h [719].

m.p. 146–148° [719]; Spectra (NA).

(3-Chloro-2-hydroxy-5-methylphenyl)(2-hydroxy-5-methylphenyl)methanone

[27404-63-3]



$C_{15}H_{13}ClO_3$ mol.wt. 276.72

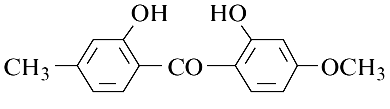
Synthesis

- Refer to: [1329] (compound **IV**).

m.p. 146–147° [1329]; MS [1329].

(2-Hydroxy-4-methoxyphenyl)(2-hydroxy-4-methylphenyl)methanone

[105515-30-8]



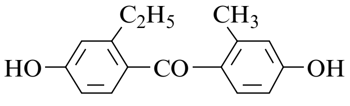
$C_{15}H_{14}O_4$ mol.wt. 258.27

Synthesis

- Refer to: [1343] (Japanese patent).

m.p. and Spectra (NA).

(2-Ethyl-4-hydroxyphenyl)(4-hydroxy-2-methylphenyl)methanone



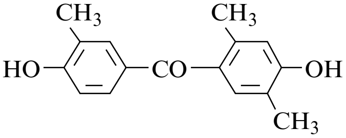
$C_{16}H_{16}O_3$ mol.wt. 256.30

Synthesis

- Refer to: [901] (compound **6**).

m.p. and Spectra (NA).

(4-Hydroxy-2,5-dimethylphenyl)(4-hydroxy-3-methylphenyl)methanone



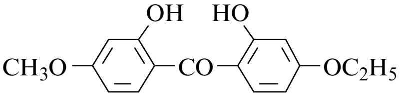
$C_{16}H_{16}O_3$ mol.wt. 256.30

Synthesis

– Refer to: [901] (compound **8**).

m.p. and Spectra (NA).

(4-Ethoxy-2-hydroxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone



$C_{16}H_{16}O_5$ mol.wt. 288.30

Syntheses

– Preparation by reaction of 2,4-dimethoxybenzoyl chloride with 1,3-diethoxybenzene in the presence of aluminium chloride,

- in a chlorobenzene/N,N-dimethylformamide mixture (22:1) at 115° [657];
- in chlorobenzene at 90° [222].

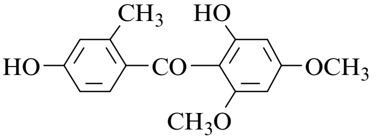
– Preparation by partial demethylation of 4-ethoxy-2-hydroxy-2′,4′-dimethoxybenzophenone or 4-ethoxy-2,2′,4′-trimethoxybenzophenone with aluminium chloride or aluminium bromide in chlorobenzene at 90–95° (good yield) [655].

– Also refer to: [235].

m.p. (NA); UV [235].

(2-Hydroxy-4,6-dimethoxyphenyl)(4-hydroxy-2-methylphenyl)methanone

[4650-75-3]



$C_{16}H_{16}O_5$ mol.wt. 288.30

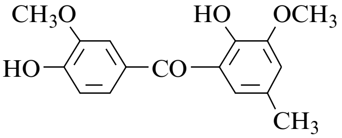
Synthesis

– Refer to: [114].

m.p. (NA); MS [114].

(2-Hydroxy-3-methoxy-5-methylphenyl)(4-hydroxy-3-methoxyphenyl) methanone

[199735-29-0]



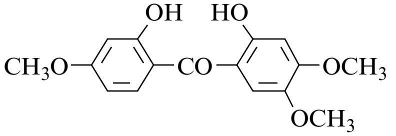
$C_{16}H_{16}O_5$ mol.wt. 288.30

Synthesis

– Obtained by heating a mixture of the 2,4′-dihydroxy-3,3′-dimethoxy-5-methyldiphenylmethane, 2 N sodium hydroxide and nitrobenzene for 2 h at 170° (10%) [1332,1333].

m.p. and Spectra (NA); GC and GC-MS [1332,1333].

(2-Hydroxy-4,5-dimethoxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone



$C_{16}H_{16}O_6$ mol.wt. 304.30

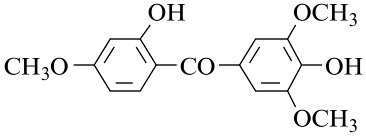
Synthesis

- Preparation by reaction of 2-hydroxy-4-methoxy-benzoic acid with 3,4-dimethoxyphenol in the presence of zinc chloride and phosphorous oxychloride on heating at 65–70° for 1 h [650].
- Also refer to: [649].

m.p. 147–148° [650]; IR [650], UV [650].

(4-Hydroxy-3,5-dimethoxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone

[62495-38-9]



$C_{16}H_{16}O_6$ mol.wt. 304.30

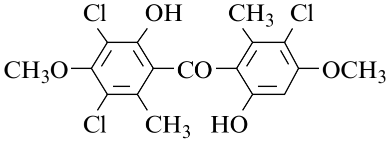
Synthesis

- Preparation by reaction of 3,4,5-trimethoxybenzoic acid with 3-methoxyphenol in the presence of phosphorous oxychloride and zinc chloride at 65–70° for 2 h (32%) [1136].

m.p. 142–143° [1136]; ^{1}H NMR [1136], IR [1136], UV [1136].

(3-Chloro-6-hydroxy-4-methoxy-2-methylphenyl) (3,5-dichloro-2-hydroxy-4-methoxy-6-methylphenyl)methanone

[69709-92-8]



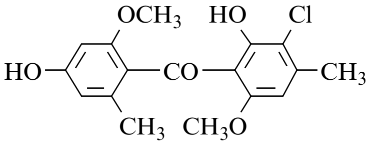
$C_{17}H_{15}Cl_3O_5$ mol.wt. 405.66

Synthesis

- Preparation by hydrogenolysis of 2, 2′-bis (benzyloxy)-3,5,5′-trichloro-4, 4′-dimethoxy-6,6′-dimethylbenzophenone (SM) with hydrogen in the presence of 10% Pd/C in ethyl acetate containing concentrated hydrochloric acid (5 drops) (>90%) [1339]. SM was obtained by Friedel–Crafts acylation of 5-(benzyloxy)-2-chloro-3-methoxytoluene with 2-(benzyloxy)-3,5-dichloro-4-methoxy-6-methylbenzoic acid in the presence of trifluoroacetic anhydride in refluxing ethylene dichloride for 5 h (28%).
- Also refer to: [1344].

m.p. 137–138° [1339]; ^{1}H NMR [1339], MS [1339].

(3-Chloro-2-hydroxy-6-methoxy-4-methylphenyl)(4-hydroxy-2-methoxy-6-methyl-phenyl)methanone



$C_{17}H_{17}ClO_5$ mol.wt. 336.77

Synthesis

- Not yet described.

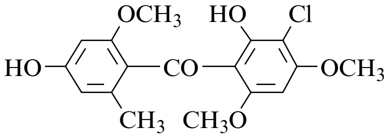
Isolation from natural source

- From cultures of *Penicillium patulum* [1345].

m.p. 181–182° [1345]; IR [1345], UV [1345], MS [1345].

(3-Chloro-2-hydroxy-4,6-dimethoxyphenyl)(4-hydroxy-2-methoxy-6-methylphenyl)-methanone
(Griseophenone A)

[2151-17-9]



$C_{17}H_{17}ClO_6$ mol.wt. 352.77

Syntheses

- Obtained by acylation of 2-chloro-3-,5-dimethoxy-phenol,
  - with 4-acetoxy-2-methoxy-6-methylbenzoyl chloride in the presence of aluminium chloride in nitrobenzene at r.t. for 20 h (35%) [1189] (Friedel–Crafts reaction);
  - with 4-acetoxy-2-methoxy-6-methylbenzoic acid in the presence of trifluoroacetic anhydride at 20° for 18 h (50%) [1189];
  - with 2-methoxy-4-methoxycarbonyl-6-methylbenzoyl chloride in the presence of aluminium chloride in nitrobenzene at r.t. [1346].
- Also obtained by hydrogenation of dehydrogriseofulvin,
  - with hydrogen in the presence of Pd/C in ethanol [1347];
  - over Rh/C containing 3% of selenium in ethanol (70%) [1346].
- Also obtained by saponification of two intermediate esters^T (formed in the above reactions) with 2.5% sodium hydroxide in dilute methanol at 25° (quantitative yields) [1189],

  ^{T}of 4′-acetoxy-3-chloro-2-hydroxy-2′,4,6-trimethoxy-6′-methylbenzophenone;
  ^{T}of 4′-acetoxy-3-chloro-2-(trifluoroacetoxy)-2′,4,6-trimethoxybenzophenone.
- Also obtained from dehydrogriseofulvin by reductive scission with chromous chloride or with zinc in acetic acid [1192].
- Also obtained from 2-chloro-3,5-dimethoxyphenyl 4-hydroxy-2-methoxy-6-methylbenzoate,
  - by Fries rearrangement in the presence of titanium tetrachloride in nitrobenzene at 20° for 18 h (65%) [1189];
  - by light-catalyzed Fries rearrangement in ethanol (10–15%) [1192], at 40° for 66 h (8%) [1189].

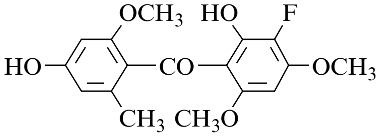
- Also refer to: [1348,1349].

Isolation from natural source

- From cultures of *Penicillium patulum* [1182,1345,1346,1350].

  m.p. 213–214° [1182,1350], 212°5–215° [1189], 212–214° [1345], 210–212° [1192]; IR [1182,1345,1346], UV [1192,1345,1346], MS [1345]; paper chromatography [1182].

(3-Fluoro-2-hydroxy-4,6-dimethoxyphenyl)(4-hydroxy-2-methoxy-6-methylphenyl)-methanone



$C_{17}H_{17}FO_6$ mol.wt. 336.32

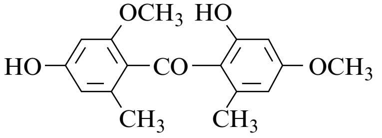
Syntheses

- Obtained by addition of 4-acetoxy-2-methoxy-6-methylbenzoic acid to 2-fluoro-3,5-dimethoxy-phenol in the presence of trifluoroacetic anhydride, first at 0°, then at 20–25° for 20 h (21%) [1191].
- Preparation by saponification of the corresponding acetate [1190], with 5% aqueous sodium hydroxide at 20° under nitrogen for 2 h (quantitative yield) [1191].

  m.p. 200–203° [1190], double melting point: 186–190°, then 200–203° [1191]; IR [1191], UV [1190,1191].

(2-Hydroxy-4-methoxy-6-methylphenyl)(4-hydroxy-2-methoxy-6-methylphenyl)-methanone

[81574-67-6]



$C_{17}H_{18}O_5$ mol.wt. 302.33

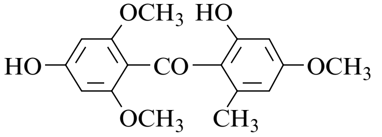
Synthesis

- Preparation by reduction of the 2′, 6-dimethoxy-4,6′-dimethylspiro[81574-67-6]-3(2*H*),4′-dione (SM) with zinc dust in acetic acid for 1 h (83%). SM was obtained from methyl 4-methoxy-2-(2,4-dimethoxy-6-methyl-phenoxy)-6-methylbenzoate by treatment with titanium tetrachloride and hydrogen chloride for 40 h (65%, m.p. 190–192°) [1184].

  m.p. 176–177° [1184]; ^{1}H NMR [1184], MS [1184].

(4-Hydroxy-2,6-dimethoxyphenyl)(2-hydroxy-4-methoxy-6-methylphenyl) methanone

[74628-36-7]



$C_{17}H_{18}O_6$ mol.wt. 318.33

Synthesis

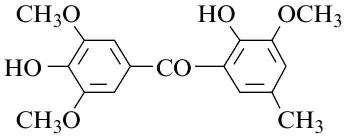
- Preparation by reduction of the 2′,6, 6′-trimethoxy-4-methylspiro[74628-36-7]-3(2*H*),4′-dione (SM) with zinc dust in acetic acid [1183], for 1 h (82%).

SM was obtained from methyl 4-methoxy-2-(2,4,6-trimethoxy-phenoxy) -6-methylbenzoate by treatment with titanium tetrachloride and hydrogen chloride for 12 h (90%, m.p. 273–274°) [1184].

m.p. 192–193° [1184]; ^{1}H NMR [1184], MS [1184].

(4-Hydroxy-3,5-dimethoxyphenyl)(2-hydroxy-3-methoxy-5-methylphenyl) methanone

[25138-53-8] $C_{17}H_{18}O_6$ mol.wt. 318.33



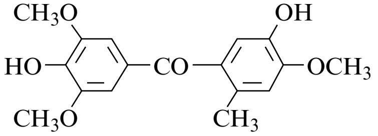
Synthesis

- Obtained (poor yield) by heating a mixture of 2,4′-di-hydroxy-3,3′,5′-trimethoxy-5-methyldiphenylmethane, 2 N sodium hydroxide and nitrobenzene for 2 h at 170° (5%) [1332,1333].

m.p. and Spectra (NA); GC and GC-MS [1332,1333].

(4-Hydroxy-3,5-dimethoxyphenyl)(5-hydroxy-4-methoxy-2-methylphenyl) methanone

[199735-38-1] $C_{17}H_{18}O_6$ mol.wt. 318.33



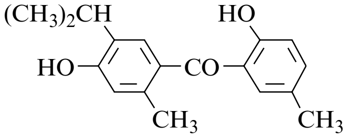
Synthesis

- Obtained (poor yield) by heating a mixture of 4,5′-dihydroxy-3,4′,5-trimethoxy-2-methyldiphenyl-methane, 2 N sodium hydroxide and nitrobenzene for 2 h at 170° (4%) [1332,1333].

m.p. and Spectra (NA); GC and GC-MS [1332,1333].

[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl](2-hydroxy-5-methylphenyl) methanone

$C_{18}H_{20}O_3$ mol.wt. 284.36

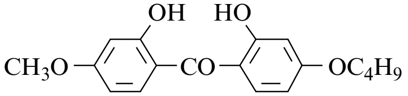


Synthesis

- Obtained by demethylation of 2′,4-dimethoxy-2,5′-di-methyl-5-isopropylbenzophenone with pyridinium chloride at reflux (205–215°) for 1.5 h (30%) [824].

b.p.$_{0.7}$ 200–210° [824]; Spectra (NA).

(4-Butoxy-2-hydroxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone



$C_{18}H_{20}O_5$ mol.wt. 316.35

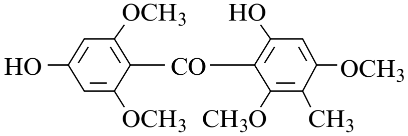
Syntheses

- Preparation by partial demethylation of 4-butoxy-2-hydroxy-2′,4′-dimethoxybenzophenone with aluminium chloride or aluminium bromide in chlorobenzene at 90–95° (good yield) [655].
- Preparation by reaction of 2,4-dimethoxybenzoyl chloride with 1,3-dibutoxybenzene in the presence of aluminium chloride,
    - in a chlorobenzene/N,N-dimethylformamide mixture (22:1) at 115° [657];
    - in chlorobenzene at 90° [222].
- Also refer to: [235].

m.p. (NA); UV [235].

(6-Hydroxy-2,4-dimethoxy-3-methylphenyl)(4-hydroxy-2,6-dimethoxyphenyl) methanone

[81574-66-5]



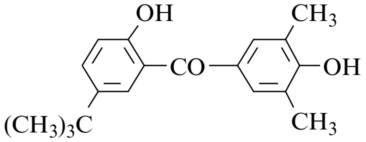
$C_{18}H_{20}O_7$ mol.wt. 348.35

Synthesis

- Preparation by reduction of 2′,4,6,6′-tetramethoxy-5-methyl-spiro[81574-66-5]-3(*2H*),4′-dione (SM) with zinc dust in acetic acid for 1 h (85%). SM was prepared from methyl 2,4-dimethoxy-6-(2,4,6-trimethoxyphenoxy)-3-methylbenzoate by treatment with titanium tetrachloride and hydrogen chloride for 65 h (78%, m.p. 265–267°) [1184].

m.p. 239–241° [1184]; ^{1}H NMR [1184], MS [1184].

[5-(1,1-Dimethylethyl)-2-hydroxyphenyl](4-hydroxy-3,5-dimethylphenyl) methanone



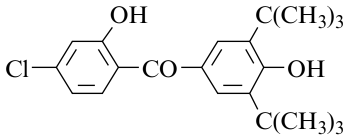
$C_{19}H_{22}O_3$ mol.wt. 298.38

Synthesis

- Obtained by photo-Fries rearrangement of 2,6-di-methylphenyl 5-tert-butylsalicylate (by-product) [219].

m.p. and Spectra (NA).

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](4-chloro-2-hydroxyphenyl) methanone



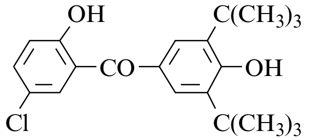
$C_{21}H_{25}ClO_3$ mol.wt. 360.88

Synthesis

- Obtained by UV light irradiation of m-chlorophenyl 3,5-di-tert-butyl-4-hydroxybenzoate in isooctane [1301].

m.p. 162°3 [1301]; Spectra (NA).

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](5-chloro-2-hydroxyphenyl) methanone



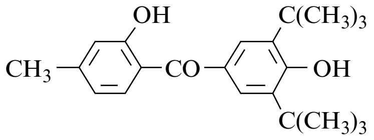
$C_{21}H_{25}ClO_3$ mol.wt. 360.88

Synthesis

- Obtained by UV light irradiation of p-chlorophenyl 3,5-di-tert-butyl-4-hydroxybenzoate in isooctane [1301].

m.p. 164–165° [1301]; Spectra (NA).

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](2-hydroxy-4-methylphenyl) methanone



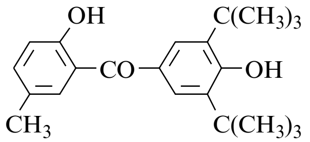
$C_{22}H_{28}O_3$ mol.wt. 340.46

Synthesis

- Obtained by UV light irradiation of m-cresyl 3,5-di-tert-butyl-4-hydroxybenzoate in isooctane [1301].

m.p. 126° [1301]; Spectra (NA).

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](2-hydroxy-5-methylphenyl) methanone



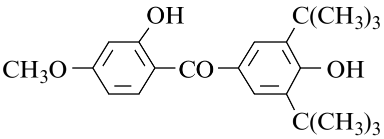
$C_{22}H_{28}O_3$ mol.wt. 340.46

Synthesis

- Obtained by UV light irradiation of p-cresyl 3,5-di-tert-butyl-4-hydroxybenzoate in isooctane [1301].

m.p. 148–149° [1301]; Spectra (NA).

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](2-hydroxy-4-methoxyphenyl) methanone



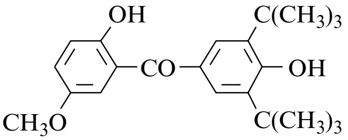
$C_{22}H_{28}O_4$ mol.wt. 356.46

Synthesis

– Obtained by UV light irradiation of m-methoxy-phenyl 3,5-di-tert-butyl-4-hydroxybenzoate in isooctane [1301].

m.p. 137–138° [1301]; Spectra (NA).

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](2-hydroxy-5-methoxyphenyl) methanone



$C_{22}H_{28}O_4$ mol.wt. 356.46

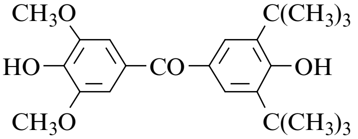
Synthesis

– Obtained by UV light irradiation of p-methoxyphenyl 3,5-di-tert-butyl-4-hydroxybenzoate in isooctane (60%) [1301].

m.p. 142–143° [1301]; Spectra (NA).

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](4-hydroxy-3,5-dimethoxyphenyl)-methanone

[54808-43-4]



$C_{23}H_{30}O_5$ mol.wt. 386.49

Synthesis

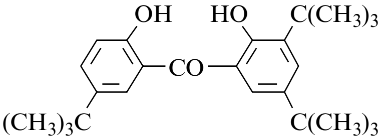
– Preparation by selective demethylation of 3,5-di-tert-butyl-4-hydroxy-3′,4′,5′-trimethoxybenzophenone (SM),

- with 45% anhydrous hydrobromic acid in acetic acid at r.t. for 48 h (49%) [334];
- by adding a freshly prepared ethereal magnesium iodide solution to a solution of SM in toluene, elimination of ethyl ether by distillation, toluene being added to maintain the original volume. Then, heating at reflux for 10 h (68%) [334].

m.p. 181°5–182°5 [334]; ^{1}H NMR [334], IR [334].

[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl][5-(1,1-dimethylethyl)-2-hydroxy-phenyl]methanone

[125182-26-5]



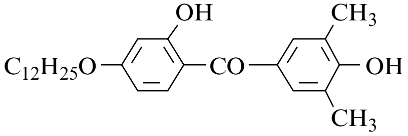
$C_{25}H_{34}O_3$ mol.wt. 382.54

Synthesis

– Obtained by treatment of 2,2′-dihydroxy-benzophenone at 120° with a mixture of isobutylene/nitrogen (1:1) in the presence of a macroreticular acid ion exchanger (Wofatit OK 80) as catalyst for 5 h (40%) [819].

b.p.$_{0.15}$ 215–220° [819]; Spectra (NA).

[4-(Dodecyloxy)-2-hydroxyphenyl](4-hydroxy-3,5-dimethylphenyl)methanone



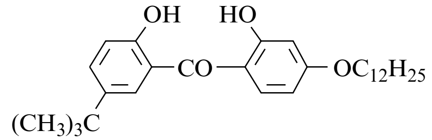
$C_{27}H_{38}O_4$ mol.wt. 426.60

Synthesis

- Obtained by photo-Fries rearrangement of 2,6-di-methylphenyl 4-dodecyloxysalicylate (by-product) [219].

m.p. and Spectra (NA).

[4-(Dodecyloxy)-2-hydroxyphenyl][5-(1,1-dimethylethyl)-2-hydroxyphenyl] methanone



$C_{29}H_{42}O_4$ mol.wt. 454.65

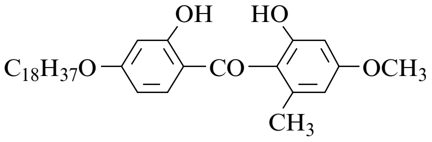
Synthesis

- Obtained by photo-Fries rearrangement of p-tert-butylphenyl 4-dodecyloxysalicylate (major product) [219].

m.p. (NA); UV [219].

(2-Hydroxy-4-methoxy-6-methylphenyl)[2-hydroxy-4-(octadecyloxy)phenyl] methanone

[128464-15-3]



$C_{33}H_{50}O_5$ mol.wt. 526.76

Synthesis

- Refer to: [1351] and [1352] (Japanese patent).

m.p. and Spectra (NA).

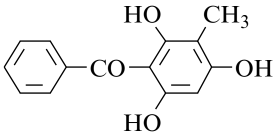
2.3 Trihydroxybenzophenones

2.3.1 Hydroxy Groups Located on the Same Ring

2.3.1.1 Substituents Located on the Hydroxylated Ring

Phenyl(2,4,6-trihydroxy-3-methylphenyl)methanone

[68223-56-3]



$C_{14}H_{12}O_4$ mol.wt. 244.25

Synthesis

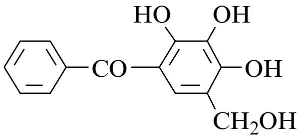
- Preparation by reaction of benzonitrile with 2-methyl-phloroglucinol in the presence of zinc chloride and hydrochloric acid in ethyl ether, followed by hydrolysis of the ketimine hydrochloride so formed (51%) [805,806].

- Also refer to: [1353,1354] (Chinese papers).

m.p. 146–147° [806], 139–140° [805]; Spectra (NA).

Phenyl[2,3,4-trihydroxy-5-(hydroxymethyl)phenyl]methanone

[138250-28-9] $C_{14}H_{12}O_5$ mol.wt. 260.25



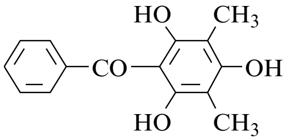
Synthesis

- Refer to: [1355–1357].

m.p. and Spectra (NA).

Phenyl(2,4,6-trihydroxy-3,5-dimethylphenyl)methanone

[22744-25-8] $C_{15}H_{14}O_4$ mol.wt. 258.27



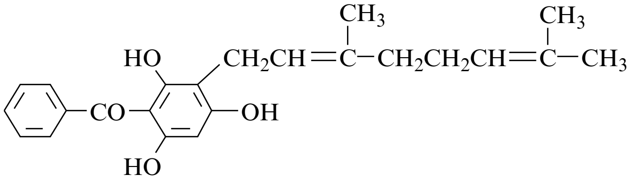
Synthesis

- Preparation by reaction of benzonitrile with 2,4-dimethyl-phloroglucinol (42%) (Hoesch reaction) [1358].

m.p. 134° [1358]; Spectra (NA).

[3-(3,7-Dimethyl-2,6-octadienyl)-2,4,6-trihydroxyphenyl]phenylmethanone

[70219-87-3] *(E)*
[76015-48-0] *(Z)* $C_{23}H_{26}O_4$ mol.wt. 366.46



Synthesis

- Not yet described.

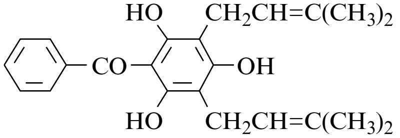
Isolation from natural sources

- From *Leontonyx spathulatus* Less. (Compositae) [378];
- From *Leontonyx Squarrosus* DC (Compositae) [378];
- From *Helichrysum crispum* Less. (Compositae) [1359];
- From *Helichrysum monticola* Hilliard (Compositae) [1360].

colourless oil [378,1360]; b.p. (NA);
^{1}H NMR [378,1360], IR [378,1360], MS [378,1360].

Phenyl[2,4,6-trihydroxy-3,5-bis(3-methyl-2-butenyl)phenyl]methanone

[70219-84-0] $C_{23}H_{26}O_4$ mol.wt. 366.46



Syntheses

- Obtained (poor yield) by reaction of 2-methyl-3-buten-2-ol with 2,4,6-trihydroxybenzophenone in the presence of boron trifluoride-etherate in dioxane at 25–30° (6%) [373].
- Also obtained (poor yield) by reaction of prenyl bromide with 2,4,6 trihydroxybenzophenone in the presence of sodium methoxide in refluxing methanol for 3 h [373], (<1%) [374].

Isolation from natural sources

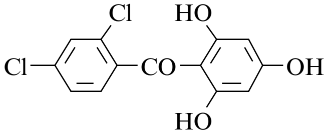
- From *Leontonyx spathulatus Less* and from *Leontonyx sqarrosus* DC (Compositae) [378];
- From *Helichrysum crispum* Less (Compositae) [1359].

Colourless oil [378]. This product is impure or in a metastable state.
m.p. 94–95° [373,374]; ^{1}H NMR [373,374,378], IR [373,374,378], UV [373,374], MS [378].

2.3.1.2 Substituents Located on the Other Ring

(2,4-Dichlorophenyl)(2,4,6-trihydroxyphenyl)methanone

[61101-87-9] $C_{13}H_8Cl_2O_4$ mol.wt. 299.11



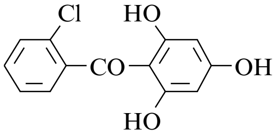
Synthesis

- Preparation by reaction of 2,4-dichlorobenzoyl chloride with O,O,O-tris(trimethylsilyl) phloroglucinol in the presence of stannic chloride in refluxing methylene chloride for 2 h (76%) [921].

oily product, not distillable [921]; b.p. and Spectra (NA).

(2-Chlorophenyl)(2,4,6-trihydroxyphenyl)methanone

[61101-84-6] $C_{13}H_9ClO_4$ mol.wt. 264.66

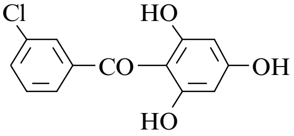


Synthesis

- Preparation by adding O,O,O-tris(trimethylsilyl) phloro-glucinol to a solution of o-chlorobenzoyl chloride and stannic chloride in methylene chloride and the resulting solution stirred overnight at r.t. (80%) [921].

m.p. 141–143° [921]; Spectra (NA).

(3-Chlorophenyl)(2,4,6-trihydroxyphenyl)methanone



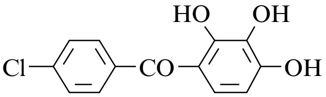
$C_{13}H_9ClO_4$ mol.wt. 264.66

Synthesis

- Preparation by reaction of m-chlorobenzonitrile with phloroglucinol (67%) (Hoesch reaction) [1256].

m.p. 169°5–170° [1256]; Spectra (NA).

(4-Chlorophenyl)(2,3,4-trihydroxyphenyl)methanone



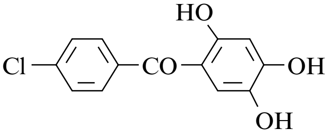
$C_{13}H_9ClO_4$ mol.wt. 264.66

Synthesis

- Preparation by reaction of p-chlorobenzonitrile with pyrogallol in the presence of zinc chloride and hydrochloric acid in ethyl ether, followed by hydrolysis of the resulting ketimine hydrochloride with boiling water for 10 min under carbon dioxide (25%) [439].

m.p. 157–158° [439]; Spectra (NA).

(4-Chlorophenyl)(2,4,5-trihydroxyphenyl)methanone



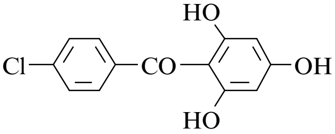
$C_{13}H_9ClO_4$ mol.wt. 264.66

Synthesis

- Preparation by reaction of p-chlorobenzonitrile with hydroxyhydroquinone in the presence of zinc chloride and hydrochloric acid in ethyl ether, followed by hydrolysis of the resulting ketimine hydrochloride with boiling water for 30 min under carbon dioxide (Hoesch reaction) (55%) [439].

m.p. 260° [439]; Spectra (NA).

(4-Chlorophenyl)(2,4,6-trihydroxyphenyl)methanone



$C_{13}H_9ClO_4$ mol.wt. 264.66

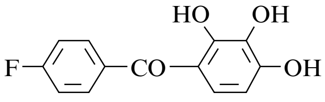
Synthesis

- Preparation by reaction of p-chlorobenzonitrile with phloroglucinol (43%) (Hoesch reaction) [1256].

m.p. 169–169°5 [1256]; Spectra (NA).

(4-Fluorophenyl)(2,3,4-trihydroxyphenyl)methanone

[84795-00-6] $C_{13}H_9FO_4$ mol.wt. 248.21



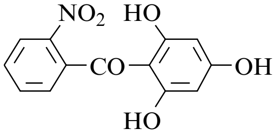
Synthesis

- Preparation by reaction of p-fluorobenzotrichloride with pyrogallol in ethanol at 65° for 30 min (80%) [1109].

m.p. 149–150° [1109]; Spectra (NA).

(2-Nitrophenyl)(2,4,6-trihydroxyphenyl)methanone

[61736-69-4] $C_{13}H_9NO_6$ mol.wt. 275.22



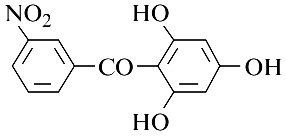
Synthesis

- Obtained by reaction of o-nitrobenzoyl chloride with phloroglucinol in the presence of aluminium chloride in ethyl ether at 0° for 3 h (20%) [1122].
- Also refer to: [974].

m.p. 182–184° [1122]; ^{1}H NMR [1122], IR [1122], UV [1122], MS [1122].

(3-Nitrophenyl)(2,4,6-trihydroxyphenyl)methanone

$C_{13}H_9NO_6$ mol.wt. 275.22



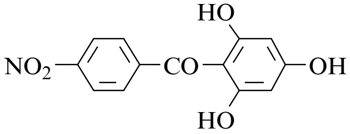
Synthesis

- Preparation by reaction of m-nitrobenzonitrile with phloroglucinol in the presence of zinc chloride and hydrochloric acid in ethyl ether, followed by hydrolysis of the resulting ketimine hydrochloride with boiling water for 30 min (Hoesch reaction) [1261,1262].

m.p. 194° [1261,1262]; Spectra (NA).

(4-Nitrophenyl)(2,4,6-trihydroxyphenyl)methanone

$C_{13}H_9NO_6$ mol.wt. 275.22

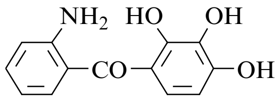


Synthesis

- Preparation by reaction of p-nitrobenzonitrile with phloroglucinol in the presence of zinc chloride and hydrochloric acid, followed by hydrolysis of the resulting ketimine hydrochloride with boiling water for 10–30 min [1261,1262], (46%) [439] (Hoesch reaction).

m.p. 246–247° [1261,1262], 244–245° [439];
monohydrate [439,1262]; Spectra (NA).

(2-Aminophenyl)(2,3,4-trihydroxyphenyl)methanone



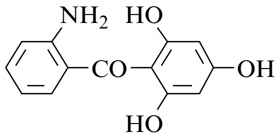
$C_{13}H_{11}NO_4$ mol.wt. 245.23

Synthesis

- Refer to: [974].

m.p. and Spectra (NA).

(2-Aminophenyl)(2,4,6-trihydroxyphenyl)methanone



$C_{13}H_{11}NO_4$ mol.wt. 245.23

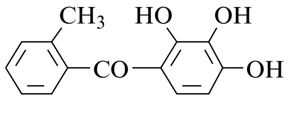
Synthesis

- Presumably obtained by treatment of 2-nitro-2′,4′,6′-tri-hydroxybenzophenone in the presence of tin and hydrochloric acid at r.t., before cyclodehydration and quantitative conversion into 1,3-dihydroxyacridan-9-one [974].
- Also refer to: [975,1122].

m.p. and Spectra (NA).

(2-Methylphenyl)(2,3,4-trihydroxyphenyl)methanone

[120506-54-9]



$C_{14}H_{12}O_4$ mol.wt. 244.25

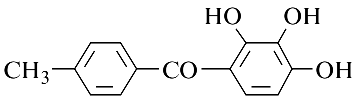
Synthesis

- Preparation by acylation of pyrogallol with o-toluic acid in the presence of Amberlyst-15 in refluxing toluene with azeotropical water removal [419].
- Also refer to: [1361–1363] (Japanese patents).

m.p. and Spectra (NA).

(4-Methylphenyl)(2,3,4-trihydroxyphenyl)methanone

[120506-55-0]



$C_{14}H_{12}O_4$ mol.wt. 244.25

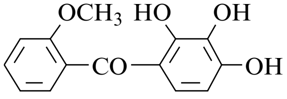
Synthesis

- Preparation by acylation of pyrogallol with p-toluic acid in the presence of Amberlyst-15 in refluxing toluene with azeotropical water removal [419].

m.p. and Spectra (NA).

(2-Methoxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[156333-16-3] $C_{14}H_{12}O_5$ mol.wt. 260.25



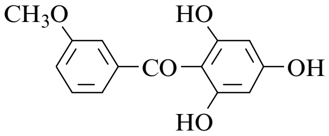
Synthesis

– Preparation by condensation of pyrogallol and o-anisic acid with boron trifluoride-etherate in carbon tetrachloride [1254], according to [360].

m.p. and Spectra (NA).

(3-Methoxyphenyl)(2,4,6-trihydroxyphenyl)methanone

[21554-79-0] $C_{14}H_{12}O_5$ mol.wt. 260.25



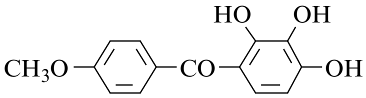
Synthesis

– Obtained (poor yield) by reaction of m-anisoyl chloride with phloroglucinol in the presence of aluminium chloride in ethyl ether at r.t. for 60 h [343], (11%) [341,422].

m.p. 168–171° [341]; ^{1}H NMR [341], IR [341], UV [341].

(4-Methoxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[105443-50-3] $C_{14}H_{12}O_5$ mol.wt. 260.25



Synthesis

– Preparation by reaction of p-anisic acid with pyrogallol,

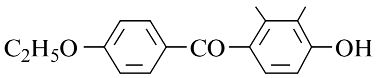
- in tetrachloroethane bubbling boron trifluoride at 110° for 1 h (84%) [420];
- in the presence of Amberlyst-15 in refluxing toluene with azeotropical water removal [419].

– Also refer to: [1364].

m.p. and Spectra (NA).

(4-Ethoxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[69471-29-0] $C_{15}H_{14}O_5$ mol.wt. 274.27



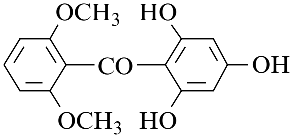
Synthesis

– Preparation by reaction of p-ethoxybenzoic acid with pyrogallol in the presence of boron trifluoride-ethyl ether complex at 100° for 2 h (73%) [1168].

m.p. 104–105° [1168]; ^{1}H NMR [1168], IR [1168], MS [1168].

(2,6-Dimethoxyphenyl)(2,4,6-trihydroxyphenyl)methanone

[61101-86-8] $C_{15}H_{14}O_6$ mol.wt. 290.27



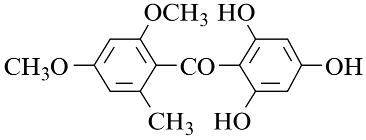
Syntheses

- Preparation by reaction of 2,6-dimethoxybenzoyl chloride with O,O,O-tris(trimethylsilyl) phloroglucinol in the presence of stannic chloride in refluxing methylene chloride for 2 h (64%) [921].
- Also obtained (poor yield) by reaction of 2,6-dimethoxy-benzonitrile with phloroglucinol in the presence of zinc chloride and hydrochloric acid in ethyl ether at 0° for 5 days, followed by hydrolysis of the resulting ketimine with boiling water for 2 h (7%) (Hoesch reaction) [439].

m.p. 216–218° [439], 195–202° (d) [921]; Spectra (NA).

(2,4-Dimethoxy-6-methylphenyl)(2,4,6-trihydroxyphenyl)methanone

[38071-50-0] $C_{16}H_{16}O_6$ mol.wt. 304.30



Synthesis

- Obtained by treatment of methyl 7-(4-orcinyl)-3,5,7-trioxoheptanoate dimethyl ether with aqueous potassium hydroxide (25%) [1365].

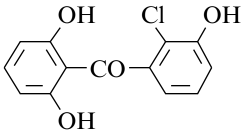
m.p. and Spectra (NA).

2.3.2 Hydroxy Groups Located on Both Rings

2.3.2.1 Substituents Located on One Ring

(2-Chloro-3-hydroxyphenyl)(2,6-dihydroxyphenyl)methanone

$C_{13}H_9ClO_4$ mol.wt. 264.66



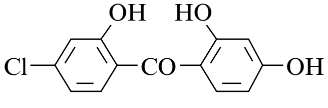
Synthesis

- Preparation by reaction of 2-chloro-3-hydroxybenzoyl chloride with bis(trimethylsilyl) derivative of resorcinol in the presence of stannic chloride (or titanium tetrachloride or aluminium chloride) in refluxing methylene chloride for 2 h [921].

m.p. and Spectra (NA).

(4-Chloro-2-hydroxyphenyl)(2,4-dihydroxyphenyl)methanone

[95481-60-0] $C_{13}H_9ClO_4$ mol.wt. 264.66



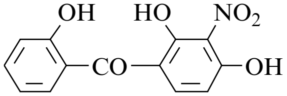
Synthesis

- Preparation by reaction of 4-chlorosalicylic acid with resorcinol in the presence of zinc chloride and a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 27°. Then, during 2 h, phosphorous trichloride was added between 27° and 37° and the mixture heated at 60° for 16 h [194].
- Also refer to: [1366].

pale yellow crystals [194]; m.p. and Spectra (NA).

(2,4-Dihydroxy-3-nitrophenyl)(2-hydroxyphenyl)methanone

[69169-87-5] $C_{13}H_9NO_6$ mol.wt. 275.22



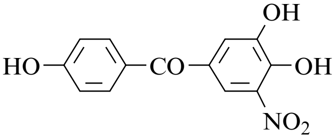
Synthesis

- Obtained (poor yield) by action of salicylic acid with 2-nitroresorcinol in the presence of zinc chloride and phosphorous oxychloride at 60–65° for 3 h (10%) [406].

m.p. 118–120° [406]; Spectra (NA).

(3,4-Dihydroxy-5-nitrophenyl)(4-hydroxyphenyl)methanone

[134612-51-4] $C_{13}H_9NO_6$ mol.wt. 275.22



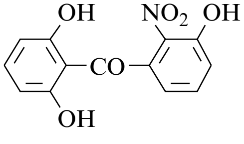
Synthesis

- Preparation by demethylation of 3,4′-dimethoxy-4-hydroxy-5-nitrobenzophenone with hydrobromic acid in refluxing acetic acid [1019].

m.p. 212–214° [1019]; Spectra (NA).

(2,6-Dihydroxyphenyl)(3-hydroxy-2-nitrophenyl)methanone

$C_{13}H_9NO_6$ mol.wt. 275.22



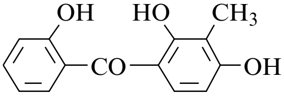
Synthesis

- Preparation by reaction of 3-hydroxy-2-nitrobenzoyl chloride with bis(trimethylsilyl) derivative of resorcinol in the presence of stannic chloride (or titanium tetrachloride or aluminium chloride) in refluxing methylene chloride for 2 h [921].

m.p. and Spectra (NA).

(2,4-Dihydroxy-3-methylphenyl)(2-hydroxyphenyl)methanone

[57654-18-9] $C_{14}H_{12}O_4$ mol.wt. 244.25



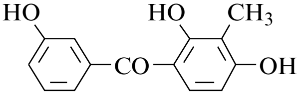
Synthesis

- Preparation by reaction of salicylic acid with 2-methyl-resorcinol in the presence of zinc chloride and phosphorous oxychloride for 4 h at 60–65° (55%) [303].

m.p. 145°5–147° [303]; ^{1}H NMR [303], IR [303], UV [303], MS [303].

(2,4-Dihydroxy-3-methylphenyl)(3-hydroxyphenyl)methanone

[61227-13-2] $C_{14}H_{12}O_4$ mol.wt. 244.25



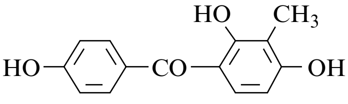
Syntheses

- -Preparation by condensation of m-acetoxybenzonitrile with 2-methylresorcinol in the presence of zinc chloride in ethyl ether (Hoesch reaction) (42%) [413].
- -Also obtained by [414] according to the method [415].

m.p. 181° [413]; Spectra (NA).

(2,4-Dihydroxy-3-methylphenyl)(4-hydroxyphenyl)methanone

[79861-84-0] $C_{14}H_{12}O_4$ mol.wt. 244.25



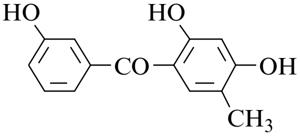
Synthesis

- Preparation by reaction of p-hydroxybenzoic acid with 2-methylresorcinol in tetrachloroethane in the presence of boron trifluoride at 80° for 4 h (79%) [224].

m.p. 227–228° [224]; ^{1}H NMR [224].

(2,4-Dihydroxy-5-methylphenyl)(3-hydroxyphenyl)methanone

[61227-14-3] $C_{14}H_{12}O_4$ mol.wt. 244.25



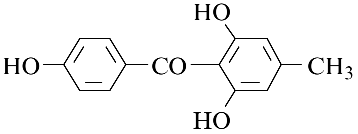
Syntheses

- Preparation by condensation of m-acetoxybenzonitrile with 4-methylresorcinol (Hoesch reaction) (70%) [413].
- Also obtained by [414] according to the method [415].

m.p. 180° [413]; Spectra (NA).

(2,6-Dihydroxy-4-methylphenyl)(4-hydroxyphenyl)methanone

[190728-23-5]



$C_{14}H_{12}O_4$ mol.wt. 244.25

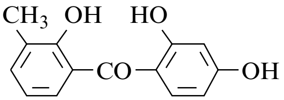
Synthesis

- Refer to: Chem. Abstr., **127**, 34137f (1997).

m.p. and Spectra (NA).

(2,4-Dihydroxyphenyl)(2-hydroxy-3-methylphenyl)methanone

[107412-87-3]



$C_{14}H_{12}O_4$ mol.wt. 244.25

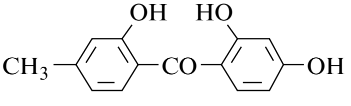
Synthesis

- Preparation by heating o-cresotic acid (3-methylsalicylic acid or 2-hydroxy-3-methylbenzoic acid) and resorcinol with zinc chloride and phosphorous oxychloride [1367].

m.p. 116–117° [1367]; Spectra (NA).

(2,4-Dihydroxyphenyl)(2-hydroxy-4-methylphenyl)methanone

[92254-59-6]



$C_{14}H_{12}O_4$ mol.wt. 244.25

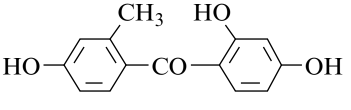
Synthesis

- Preparation by reaction of 4-methylsalicylic acid (m-cresotic acid or 2-hydroxy-4-methylbenzoic acid) with resorcinol,
  - in the presence of zinc chloride and a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 27°. Then, during 2 h, phosphorous trichloride was added between 27° and 37° and the mixture heated at 60° for 16 h [194];
  - in the presence of zinc chloride and phosphorous oxychloride [1367].
- Also refer to: [1368].

pale yellow crystals [194]; m.p. 153–154° [1367]; Spectra (NA).

(2,4-Dihydroxyphenyl)(4-hydroxy-2-methylphenyl)methanone

[4520-99-4]



$C_{14}H_{12}O_4$ mol.wt. 244.25

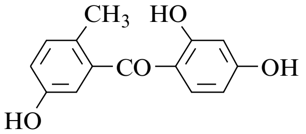
Synthesis

- Refer to: [114].

m.p. (NA); MS [114].

(2,4-Dihydroxyphenyl)(5-hydroxy-2-methylphenyl)methanone

[42470-88-2] $C_{14}H_{12}O_4$ mol.wt. 244.25



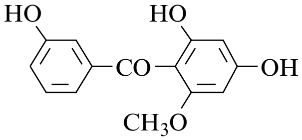
Synthesis

- Preparation by condensation of 5-hydroxy-2-methylbenzoic acid with resorcinol in the presence of zinc chloride and phosphorous oxychloride at 75° for 1 h (78%) [1369].

m.p. 162° [1369]; ^{1}H NMR [1369], IR [1369], UV [1369], MS [1369].

(2,4-Dihydroxy-6-methoxyphenyl)(3-hydroxyphenyl)methanone

[61227-12-1] $C_{14}H_{12}O_5$ mol.wt. 260.25



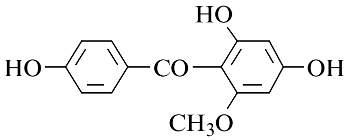
Syntheses

- Preparation by condensation of m-acetoxybenzonitrile with phloroglucinol monomethyl ether (Hoesch reaction) (33%) [413].
- Also obtained by [414] according to the method [415].

m.p. 178° [413]; Spectra (NA).

(2,4-Dihydroxy-6-methoxyphenyl)(4-hydroxyphenyl)methanone

[56308-11-3] $C_{14}H_{12}O_5$ mol.wt. 260.25



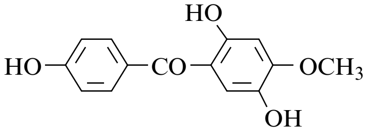
Synthesis

- Refer to: [1370].

m.p. and Spectra (NA).

(2,5-Dihydroxy-4-methoxyphenyl)(4-hydroxyphenyl)methanone

[58115-06-3] $C_{14}H_{12}O_5$ mol.wt. 260.25



Synthesis

- Obtained by partial demethylation of 2-hydroxy-4,4′,5-trimethoxybenzoenone with hydriodic acid in acetic anhydride [428].

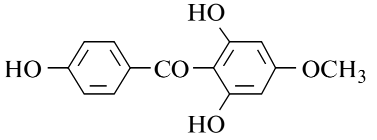
Isolation from natural source

- From *Dalbergia melanoxylon* Guill. et Perr. heartwood (Leguminosae-Lotoideae) [428].

m.p. 228–229°5 [428]; ^{1}H NMR [428].

(2,6-Dihydroxy-4-methoxyphenyl)(4-hydroxyphenyl)methanone

[55051-85-9] $C_{14}H_{12}O_5$ mol.wt. 260.25



Synthesis

- Refer to: [1371].

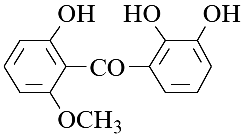
Isolation from natural sources

- From *Aniba duckei* Kostern (Lauraceae) [1286];
- From rhizome of *Anemarrhena asphodeloides* [1372], according to [1371] or from *Anemarrhena asphodeloides Bge* [1373].

m.p. 179–181° [1286], 146–150° [1373]. There is discrepancy between the two melting points.
^{1}H NMR [1286,1373], ^{13}C NMR [1373], IR [1286],
UV [1286], MS [1286,1373]; TLC [1372].

(2,3-Dihydroxyphenyl)(2-hydroxy-6-methoxyphenyl)methanone

[25577-00-8] $C_{14}H_{12}O_5$ mol.wt. 260.25



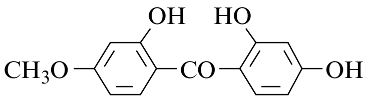
Synthesis

- Preparation by partial demethylation of 2,2′,3′, 6-tetra-methoxybenzophenone with boron tribromide in benzene at r.t. for 5 h (76%) [412].

m.p. 147–150° [412]; ^{1}H NMR [412], IR [412], UV [412], MS [412].

(2,4-Dihydroxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone

[7392-62-3] $C_{14}H_{12}O_5$ mol.wt. 260.25



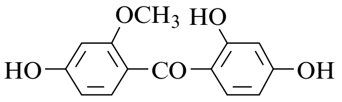
Synthesis

- Obtained by action of 2-hydroxy-4-methoxybenzoic acid with resorcinol in the presence of zinc chloride and phosphorous oxychloride at 60–65° for 3 h (30%) [406].
- Also refer to: [405,1135,1374].

m.p. 101–102° [406]; Spectra (NA).

(2,4-Dihydroxyphenyl)(4-hydroxy-2-methoxyphenyl)methanone

[71655-03-3] $C_{14}H_{12}O_5$ mol.wt. 260.25



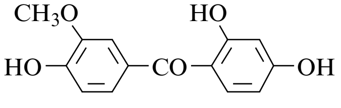
Synthesis

- Preparation by reaction of β-resorcylic acid with resorcinol monomethyl ether in the presence of zinc chloride and a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 27°. Then, during 2 h, phosphorous trichloride was added between 27° and 37° and the mixture heated at 60° for 16 h [194].

- Also refer to: [1375] (Japanese patent).

yellow crystals [194]; m.p. and Spectra (NA).

(2,4-Dihydroxyphenyl)(4-hydroxy-3-methoxyphenyl)methanone



$C_{14}H_{12}O_5$ mol.wt. 260.25

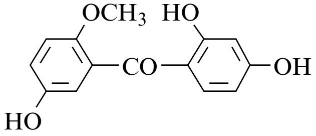
Synthesis

- Preparation by reaction of vanillonitrile (4-hydroxy-3-methoxybenzonitrile) with resorcinol (Hoesch reaction) (20%) [440].

m.p. 210° [440]; Spectra (NA).

(2,4-Dihydroxyphenyl)(5-hydroxy-2-methoxyphenyl)methanone

[61227-15-4]



$C_{14}H_{12}O_5$ mol.wt. 260.25

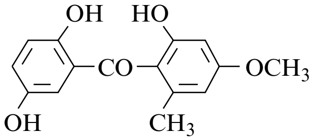
Synthesis

- Preparation by treatment of N-[5-acetoxy-α-(2,4-di-hydroxyphenyl)-2-methoxybenzylidene]aniline with 20% sulfuric acid at reflux for 5 h under nitrogen (92%) [413].

m.p. 194° [413]; IR [413].

(2,5-Dihydroxyphenyl)(2-hydroxy-4-methoxy-6-methylphenyl)methanone

[78044-96-9]



$C_{15}H_{14}O_5$ mol.wt. 274.27

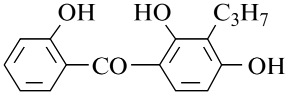
Synthesis

- Preparation by partial demethylation of 2′,5′-dihydroxy-2,4-dimethoxy-6-methylbenzophenone with boron trichloride in methylene chloride at r.t. for 5 days (82%) [1167].

m.p. 176–179° [1167]; ^{1}H NMR [1167], IR [1167], UV [1167].

(2,4-Dihydroxy-3-propylphenyl)(2-hydroxyphenyl)methanone

[115296-04-3]



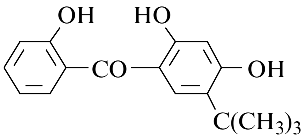
$C_{16}H_{16}O_4$ mol.wt. 272.30

Synthesis

- Obtained by total demethylation of 2,2′,4-trimethoxy-3-propylbenzophenone or of 2,4-dimethoxy-2′-hydroxy-3-propylbenzophenone with pyridinium chloride at 180° for 3 h [1029].
- Also refer to: [168].

m.p. 110–113° [1029]; Spectra (NA).

[2,4-Dihydroxy-5-(1,1-dimethylethyl)phenyl](2-hydroxyphenyl)methanone



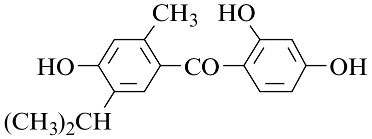
$C_{17}H_{18}O_4$ mol.wt. 286.33

Synthesis

- Preparation by alkylation of 2,2′,4-trihydroxybenzophenone with isobutylene in benzene in the presence of p-toluenesulfonic acid for 2 h at 65–75° (70%) [835,836].

m.p. 194–196° [835,836]; UV [836].

(2,4-Dihydroxyphenyl)[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl] methanone



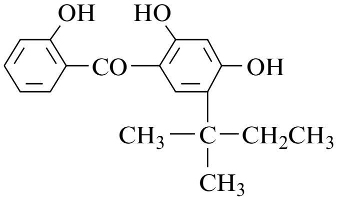
$C_{17}H_{18}O_4$ mol.wt. 286.33

Synthesis

- Obtained by demethylation of 2′,4,4′-trimethoxy-2-methyl-5-isopropylbenzophenone in refluxing pyridinium chloride (205–215°) for 1.3 h (34%) [824].

$b.p._{0.9}$ 220–230° [824]; Spectra (NA).

[2,4-Dihydroxy-5-(1,1-dimethylpropyl)phenyl](2-hydroxyphenyl)methanone



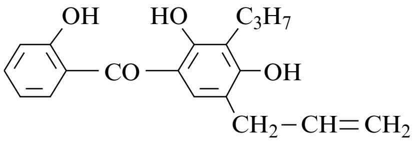
$C_{18}H_{20}O_4$ mol.wt. 300.35

Synthesis

- Preparation by alkylation of 2,2′,4-trihydroxy-benzophenone with 2-methylbutene in benzene in the presence of concentrated sulfuric acid (82%) [835].

m.p. 142–144° [835,836]; Spectra (NA).

[2,4-Dihydroxy-5-(2-propenyl)-3-propylphenyl](2-hydroxyphenyl)methanone



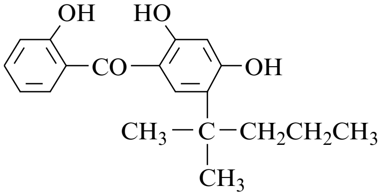
$C_{19}H_{20}O_4$ mol.wt. 312.37

Synthesis

- Obtained (poor yield) by heating 4-allyloxy-2,2′-dihydroxy-3-propylbenzophenone at 180° for 6 h (19%) [1029] (Claisen rearrangement).

m.p. and Spectra (NA).

[5-(1,1-Dimethylbutyl)-2,4-dihydroxyphenyl](2-hydroxyphenyl)methanone



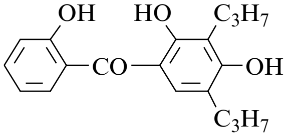
$C_{19}H_{22}O_4$ mol.wt. 314.38

Synthesis

- Preparation by alkylation of 2,2′,4-trihydroxy-benzophenone with 2-methylpentene in benzene in the presence of concentrated sulfuric acid [835].

m.p. 125° [835,836]; Spectra (NA).

(2,4-Dihydroxy-3,5-dipropylphenyl)(2-hydroxyphenyl)methanone



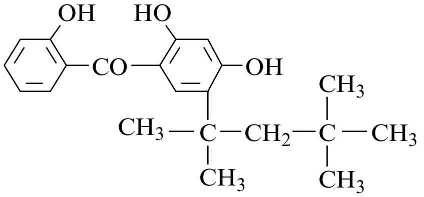
$C_{19}H_{22}O_4$ mol.wt. 314.38

Synthesis

- Preparation by catalytic hydrogenation of 5-allyl-3-propyl-2,2′,4-trihydroxybenzophenone in ethyl acetate in the presence of 5% Pd/C (94%) [1029].

m.p. 110–112° [1029]; Spectra (NA).

[2,4-Dihydroxy-5-(1,1,3,3-tetramethylbutyl)phenyl](2-hydroxyphenyl) methanone



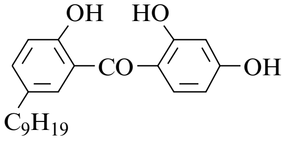
$C_{21}H_{26}O_4$ mol.wt. 342.44

Synthesis

- Preparation by alkylation of 2,2′, 4-trihydroxy-benzophenone with 2,2,4-trimethylpentene in benzene in the presence of concentrated sulfuric acid [835].

m.p. 165–168° [835,836]; Spectra (NA).

(2,4-Dihydroxyphenyl)(2-hydroxy-5-nonylphenyl)methanone



$C_{22}H_{28}O_4$ mol.wt. 356.46

Synthesis

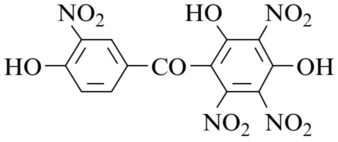
- Preparation by reaction of β-resorcylic acid with p-nonyl-phenol in the presence of a mixture of polyphosphoric acid/85% phosphoric acid (60:40) at 40–45°. Then, during 2 h, phosphorous trichloride was added and the mixture heated at 60° for 16 h (73%) [194].

m.p. and Spectra (NA).

2.3.2.2 Substituents Located on Both Rings

(2,4-Dihydroxy-3,5,6-trinitrophenyl)(4-hydroxy-3-nitrophenyl)methanone

[67246-03-1]



$C_{13}H_6N_4O_{12}$ mol.wt. 410.21

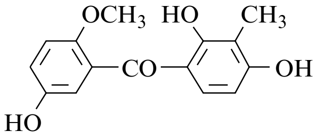
Synthesis

- Obtained by reaction of nitric acid (d = 1.42) with 4-hydroxy-2′,4′-dimethoxybenzophenone in acetic acid at 32° [592].

m.p. 110° [592]; ^{1}H NMR [592].

(2,4-Dihydroxy-3-methylphenyl)(5-hydroxy-2-methoxyphenyl)methanone

[61227-17-6]



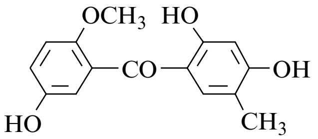
$C_{15}H_{14}O_5$ mol.wt. 274.27

Synthesis

- Preparation by hydrolysis of N-[5-acetoxy-α-(2,4-di-hydroxyphenyl)-2-methoxy-3-methylbenzylidene]aniline with refluxing 20% sulfuric acid under nitrogen during 2 h (90%) [413].

m.p. 210° [413]; Spectra (NA).

(2,4-Dihydroxy-5-methylphenyl)(5-hydroxy-2-methoxyphenyl)methanone



$C_{15}H_{14}O_5$ mol.wt. 274.27

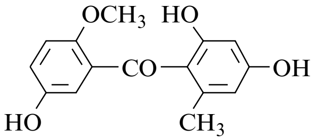
Syntheses

- Preparation by hydrolysis of N-[5-acetoxy-α-(2,4-di-hydroxyphenyl)-2-methoxy-5-methylbenzylidene]-aniline with refluxing 20% sulfuric acid during 5 h under nitrogen (90%) [413].
- Also obtained by [414] according to the method [415].

m.p. 199° [413]; Spectra (NA).

(2,4-Dihydroxy-6-methylphenyl)(5-hydroxy-2-methoxyphenyl)methanone

[61227-16-5]



$C_{15}H_{14}O_5$ mol.wt. 274.27

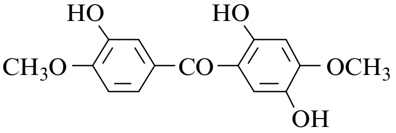
Synthesis

N.B.: This compound, mentioned in [Chem. Abstr., **86**, 16504d (1977)], is not described in the original paper [413], where the sole corresponding ketone indicated is the2,4,5′-trihydroxy-2′-methoxy-5-methylbenzophenone or (2,4-dihydroxy-**5**-methylphenyl) (5-hydroxy-2-methoxy-phenyl)methanone.

m.p. and Spectra (NA).

(2,5-Dihydroxy-4-methoxyphenyl)(3-hydroxy-4-methoxyphenyl)methanone
(Melanoxoin)

[58115-05-2]



$C_{15}H_{14}O_6$ mol.wt. 290.27

Synthesis

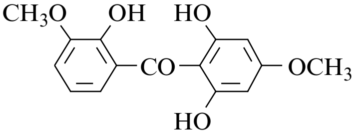
- Not yet described.

Isolation from natural source

- From *Dalbergia melanoxylon* Guill. et Perr. heartwood (Leguminosae-Lotoideae) [428].

m.p. 232–234° [428]; IR [428], UV [428].

(2,6-Dihydroxy-4-methoxyphenyl)(2-hydroxy-3-methoxyphenyl)methanone



$C_{15}H_{14}O_6$ mol.wt. 290.27

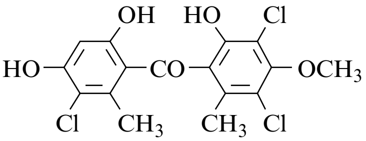
Synthesis

- Obtained by condensation of 2,3-dimethoxybenzoic acid and 1,3,5-trimethoxybenzene in the presence of aluminium chloride, zinc chloride and phosphorous oxychloride, prior to cyclisation into 1-hydroxy-3,5-di-methoxyxanthone [1241].

m.p. and Spectra (NA).

(3-Chloro-4,6-dihydroxy-2-methylphenyl)(3,5-dichloro-2-hydroxy-4-methoxy-6-methylphenyl)methanone

[69709-89-3]



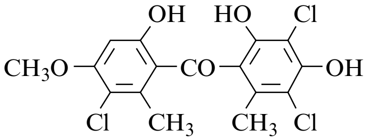
$C_{16}H_{13}Cl_3O_5$ mol.wt. 391.63

Synthesis

- Preparation by hydrogenolysis of 2,2′,4′-tris-(benzyloxy)-3,5,5′-trichloro-4-methoxy-6,6′-di-methylbenzophenone (SM) in the presence of 10% Pd/C in ethyl acetate containing concentrated hydrochloric acid (5 drops) (>90%) [1339]. SM was obtained by Friedel–Crafts acylation of 3,5-bis(benzyloxy)-2-chlorotoluene with 2-(benzyloxy)-3,5-dichloro-4-methoxy-6-methylbenzoic acid in the presence of trifluoroacetic anhydride in refluxing ethylene dichloride for 5 h (42%).
- Also refer to: [1344].

m.p. 168°5–169°5 [1339]; ^{1}H NMR [1339], MS [1339].

(3-Chloro-6-hydroxy-4-methoxy-2-methylphenyl)(3,5-dichloro-2,4-dihydroxy-6-methylphenyl)methanone



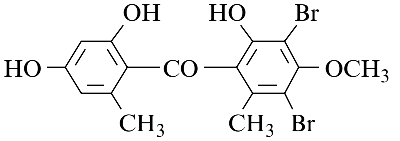
$C_{16}H_{13}Cl_3O_5$ mol.wt. 391.63

Synthesis

– Preparation by hydrogenolysis of 2,2′,4-tris-(benzyloxy)-3,5,5′-trichloro-4′-methoxy-6,6′-di-methylbenzophenone (SM) in the presence of 10% Pd/C in ethyl acetate containing concentrated hydrochloric acid (5 drops) (>90%) [1339]. SM was obtained by Friedel–Crafts acylation of 5-(benzyloxy)-2-chloro-3-methoxytoluene with 2,4-bis-(benzyloxy)-3,5-dichloro-6-methylbenzoic acid in the presence of trifluoroacetic anhydride in refluxing ethylene dichloride for 5 h (42%).

m.p. 174–175°5 [1339]; ^{1}H NMR [1339], MS [1339].

(3,5-Dibromo-2-hydroxy-4-methoxy-6-methylphenyl)(2,4-dihydroxy-6-methylphenyl)-methanone



$C_{16}H_{14}Br_2O_5$ mol.wt. 446.09

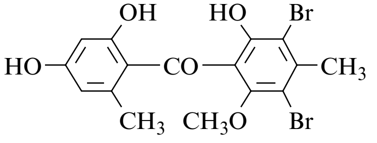
Synthesis

– Preparation by hydrogenolysis of 2,2′,4′-tris-(benzyloxy)-3,5-dibromo-4-methoxy-6,6′-dimethyl-benzophenone (SM) in the presence of 10% Pd/C in ethanol/ethyl acetate mixture at r.t. and atmospheric pressure. SM was obtained by condensation of orcinol dibenzyl ether with 3,5-dibromoeverninic acid benzyl ether in the presence of a trifluoroacetic anhydride/trifluoroacetic acid mixture under nitrogen in refluxing chloroform for 5 h [1376].

m.p. and Spectra (NA).

(3,5-Dibromo-2-hydroxy-6-methoxy-4-methylphenyl)(2,4-dihydroxy-6-methylphenyl)-methanone

[39803-81-1]



$C_{16}H_{14}Br_2O_5$ mol.wt. 446.09

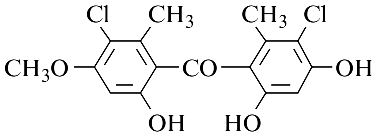
Synthesis

– Preparation by hydrogenolysis of 2,2′,4′-tris-(benzyloxy)-3,5-dibromo-6-methoxy-4,6′-dimethyl-benzophenone (SM) in ethanol/trifluoroacetic acid mixture in the presence of 10% Pd/C at atmospheric pressure (quantitative yield). SM was obtained by condensation of O-benzyl-O-methyl-3,5-dibromo-γ-orsellinic acid—2-(benzyloxy)-3,5-dibromo-6-methoxy-4-methylbenzoic acid—with orcinol dibenzyl ether in the presence of trifluoroacetic anhydride/trifluoroacetic acid mixture under nitrogen in refluxing chloroform for 6.25 h [1376].

m.p. and Spectra (NA).

(3-Chloro-4,6-dihydroxy-2-methylphenyl)(3-chloro-6-hydroxy-4-methoxy-2-methylphenyl)methanone

[78135-54-3]



$C_{16}H_{14}Cl_2O_5$ mol.wt. 357.19

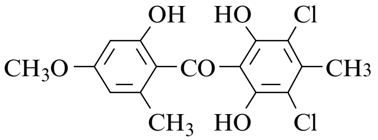
Synthesis

- Preparation by hydrogenolysis (4 bars) of 4,6,6′-tris(benzyloxy)-3,3′-dichloro-4′-methoxy-2,2′-dimethylbenzophenone (m.p. 168–169°) in ethyl acetate in the presence of 10% Pd/C and 5 drops of concentrated hydrochloric acid (75%) [1377].

m.p. 180–182° [1377]; ^{1}H NMR [1377], IR [1377].

(3,5-Dichloro-2,6-dihydroxy-4-methylphenyl)(2-hydroxy-4-methoxy-6-methylphenyl)-methanone

[23565-77-7]



$C_{16}H_{14}Cl_2O_5$ mol.wt. 357.19

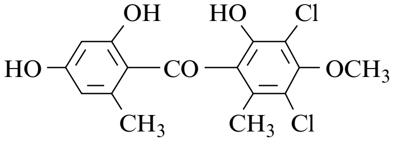
Synthesis

- Obtained (poor yield) by photo-Fries rearrangement of 2,4-dichloro-5-hydroxy-3-methylphenyl 2-hydroxy-4-methoxy-6-methylbenzoate in ethanol at 20° for 75 h (3%) [754].

m.p. 142–144° [754]; ^{1}H NMR [754], IR [754], UV [754].

(3,5-Dichloro-2-hydroxy-4-methoxy-6-methylphenyl)(2,4-dihydroxy-6-methylphenyl)-methanone

[39803-63-9]



$C_{16}H_{14}Cl_2O_5$ mol.wt. 357.19

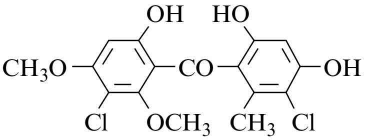
Synthesis

- Preparation by hydrogenolysis of 2,2′,4′-tris-(benzyloxy)-3,5-dichloro-4-methoxy-6,6′-dimethyl-benzophenone (SM) in the presence of 10% Pd/C in ethanol containing a few drops of trifluoroacetic acid for 3–4 h. SM was obtained by condensation of orcinol dibenzyl ether with 3,5-dichloroeverninic acid benzyl ether in the presence of a trifluoroacetic anhydride/trifluoroacetic acid mixture in refluxing chloroform for 6 h [1376].

m.p. and Spectra (NA).

(3-Chloro-4,6-dihydroxy-2-methylphenyl)(3-chloro-6-hydroxy-2,4-dimethoxyphenyl)-methanone

[68048-23-7] $C_{16}H_{14}Cl_2O_6$ mol.wt. 373.19



Synthesis

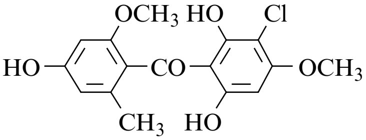
– Preparation by hydrogenolysis of 4,6,6′-tris-(benzyloxy)-3,3′-dichloro-2′,4′-dimethoxy-2-methyl-benzophenone (SM) in ethyl acetate/tetrahydrofuran in the presence of 10% Pd/C at 25° (29%). SM was obtained by condensation of 4,6-bis(benzyloxy)-3-chloro-2-methylbenzoic acid with 4-chloro-3,5-dimethoxyphenol benzyl ether in the presence of trifluoroacetic anhydride in methylene chloride under nitrogen for 20 min [1179].

m.p. 217–219° [1179]; ^{1}H NMR [1179], IR [1179], MS [1179].

(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)(4-hydroxy-2-methoxy-6-methylphenyl)-methanone

(Griseophenone B)

[3811-00-5] $C_{16}H_{15}ClO_6$ mol.wt. 338.74



Synthesis

– Not yet described.

Isolation from natural source

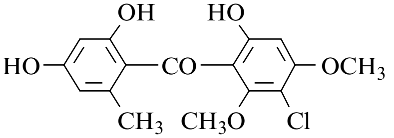
– From cultures of *Penicillium patulum* [1182,1350].

– Also refer to: [1348,1349,1378–1382].

m.p. 204°5–205°5 [1182]; IR [1182], UV [1182].

(3-Chloro-6-hydroxy-2,4-dimethoxyphenyl)(2,4-dihydroxy-6-methylphenyl) methanone

[68048-17-9] $C_{16}H_{15}ClO_6$ mol.wt. 338.74



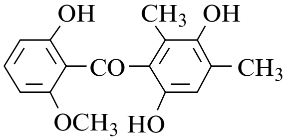
Synthesis

– Preparation by hydrogenolysis of 2′,4′,6-tris-(benzyloxy)-3-chloro-2,4-dimethoxy-6′-methyl-benzophenone (SM) in ethyl acetate/tetrahydrofuran in the presence of 10% Pd/C at 25°. SM was obtained by condensation of 2,4-bis(benzyloxy)-6-methylbenzoic acid with 4-chloro-3,5-dimethoxyphenol benzyl ether in the presence of trifluoroacetic anhydride in methylene chloride under nitrogen for 15 min (61%) [1179].

m.p. 181–182° [1179]; ^{1}H NMR [1179], IR [1179], MS [1179].

(3,6-Dihydroxy-2,4-dimethylphenyl)(2-hydroxy-6-methoxyphenyl)methanone

[42594-59-2] $C_{16}H_{16}O_5$ mol.wt. 288.30



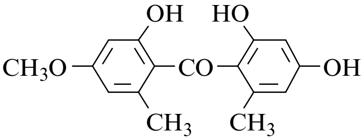
Synthesis

– Preparation by partial demethylation of 3-hydroxy-2′,6,6′-trimethoxy-2,4-dimethylbenzophenone with boron tribromide in methylene chloride (quantitative yield) [1181].

m.p. 188–191° [1181]; ^{1}H NMR [1181], IR [1181], UV [1181], MS [1181].

(2,4-Dihydroxy-6-methylphenyl)(2-hydroxy-4-methoxy-6-methylphenyl) methanone

[21147-34-2] $C_{16}H_{16}O_5$ mol.wt. 288.30



Syntheses

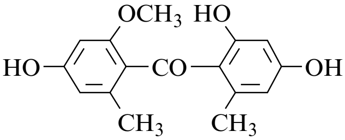
– Preparation by hydrogenolysis of 2, 2′,4′-tris-(benzyloxy)-4-methoxy-6,6′-dimethylbenzophenone (SM) in the presence of 10% Pd/C in ethyl acetate containing concentrated hydrochloric acid (5 drops) [1339]. SM was obtained by Friedel–Crafts acylation of 3,5-bis(benzyloxy)toluene with 2-(benzyloxy) -4- methoxy-6-methylbenzoic acid in the presence of trifluoroacetic anhydride in refluxing ethylene dichloride for 5 h (95%).

– Also obtained by photo-Fries rearrangement of 5-hydroxy-3-methylphenyl 2-hydroxy-4-methoxy-6-methylbenzoate in ethanol at r.t. for 80 h (21%) [754].

m.p. 158–160° [1339], 158–159° [754];
^{1}H NMR [754,1339], IR [754], UV [754], MS [114,1339].

(2,4-Dihydroxy-6-methylphenyl)(4-hydroxy-2-methoxy-6-methylphenyl) methanone

[23573-47-9] $C_{16}H_{16}O_5$ mol.wt. 288.30



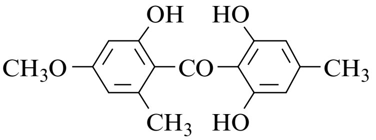
Synthesis

– Obtained by reaction of orcinol with 2-methoxy-4-O-methoxycarbonyl-6-methylbenzoic acid in trifluoroacetic anhydride, first at 0° for 30 min, then at 20° for 15 h (30%) [754].

m.p. 118–120° [754]; ^{1}H NMR [754], IR [754], UV [754].

(2,6-Dihydroxy-4-methylphenyl)(2-hydroxy-4-methoxy-6-methylphenyl) methanone

[21147-33-1] $C_{16}H_{16}O_5$ mol.wt. 288.30



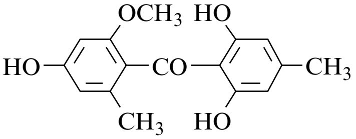
Syntheses

- Obtained (poor yields) by photo-Fries rearrangement of two esters in ethanol at r.t.,
  - from 3-hydroxy-5-methylphenyl 2-hydroxy-4-methoxy-6-methylbenzoate during 80 h (5%) [754];
  - from 3-methoxy-5-methylphenyl 2,6-dihydroxy-4-methylbenzoate during 60 h (<2%) [754].
- Preparation by hydrogenolysis of 3′,5′-dichloro-2,2′,6′-trihydroxy-4-methoxy-4′, 6-dimethyl-benzophenone in the presence of Pd/C in 1 N sodium hydroxide [754].

m.p. 56–58° [754]; ^{1}H NMR [754], IR [754], UV [754], MS [114].

(2,6-Dihydroxy-4-methylphenyl)(4-hydroxy-2-methoxy-6-methylphenyl) methanone

[23565-89-1] $C_{16}H_{16}O_5$ mol.wt. 288.30



Synthesis

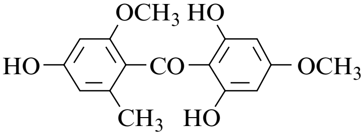
- Obtained (by-product) by reaction of orcinol with 2-methoxy-4-O-methoxy-carbonyl-6-methylbenzoic acid in trifluoroacetic anhydride, first at 0° for 30 min, then at 20° for 15 h (11%) [754].

m.p. 139–140° [754]; ^{1}H NMR [754], IR [754], UV [754].

(2,6-Dihydroxy-4-methoxyphenyl)(4-hydroxy-2-methoxy-6-methylphenyl) methanone

(Griseophenone C)

[3733-72-0] $C_{16}H_{16}O_6$ mol.wt. 304.30



Synthesis

- Not yet described.

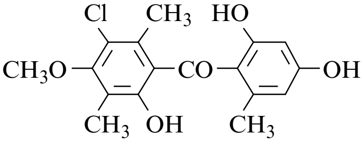
Isolation from natural source

- From cultures of *Penicillium patulum* [1182,1350,1383].
- Also refer to: [1349,1365,1378,1379,1382].

m.p. 183–185° [1350], 183–184° [1182], 175–178° [1383];
^{1}H NMR [1383], IR [1182], UV [1182].

(3-Chloro-6-hydroxy-4-methoxy-2,5-dimethylphenyl)(2,4-dihydroxy-6-methylphenyl)-methanone

[60138-98-9]



$C_{17}H_{17}ClO_5$ mol.wt. 336.77

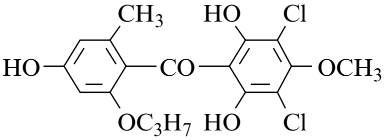
Synthesis

- Preparation by hydrogenolysis of 2,2′,4-tris-(benzyloxy)-5′-chloro-4′-methoxy-3′,6,6′-trimethyl-benzophenone in the presence of 10% Pd/C in ethyl acetate containing a small quantity of concentrated hydrochloric acid (89%) [1384].

m.p. 157–158° [1384]; ^{1}H NMR [1384].

(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)(4-hydroxy-2-methyl-6-propoxyphenyl)-methanone

[72614-88-1]



$C_{18}H_{18}Cl_2O_6$ mol.wt. 401.24

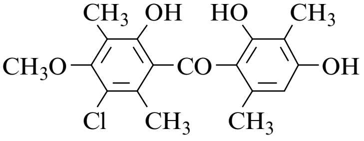
Synthesis

- Obtained by transformation of the 2-propoxy analog of griseophenone B by *Penicillium urticae* [1379], (10%) [1378].

m.p. (NA); MS [1378,1379].

(3-Chloro-6-hydroxy-4-methoxy-2,5-dimethylphenyl)(2,4-dihydroxy-3,6-dimethyl-phenyl)methanone

[61852-15-1]



$C_{18}H_{19}ClO_5$ mol.wt. 350,80

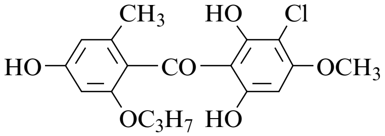
Synthesis

- Preparation by hydrogenolysis of 2,2′,-4′-tris-(benzyloxy)-5-chloro-4-methoxy-3,3′,6,6′-tetra-methylbenzophenone (SM) in the presence of 10% Pd/C in ethyl acetate containing concentrated hydrochloric acid (3 drops) (quantitative yield) [1385], (>90%) [1339]. SM was obtained by Friedel–Crafts acylation of 1,3-bis(benzyloxy)-2,5-dimethylbenzene with 2-(benzyloxy)-5-chloro-4-methoxy-3,6-dimethylbenzoic acid in the presence of trifluoroacetic anhydride in methylene chloride at r.t. for 5.5 h (40%) [1339].
- Also refer to: [1344].

m.p. 176–177° [1339,1385]; ^{1}H NMR [1385], IR [1385], MS [1385].

(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)(4-hydroxy-2-methyl-6-propoxyphenyl)-methanone

[69218-66-2]



$C_{18}H_{19}ClO_6$ mol.wt. 366.80

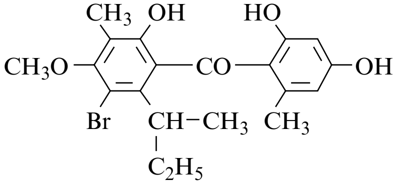
Synthesis

- Refer to: [1378,1379] (this compound is a 2-propoxy analog of griseophenone B).

m.p. and Spectra (NA).

[3-Bromo-6-hydroxy-4-methoxy-5-methyl-2-(1-methylpropyl)phenyl](2,4-dihydroxy-6-methylphenyl)methanone

[67097-17-0]



$C_{20}H_{23}BrO_5$ mol.wt. 423.30

Synthesis

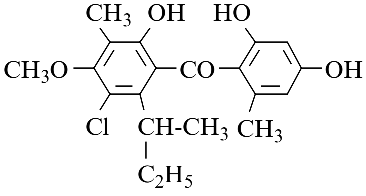
- Preparation by hydrogenolysis of 2,2′,4′-tris-(benzyloxy)-5-bromo-4-methoxy-3,6′-dimethyl-6-sec-butylbenzophenone in the presence of 10% Pd/C in ethyl acetate containing a small quantity of concentrated hydrochloric acid (89%) [1386].

- Also refer to: [1339,1344].

m.p. 185–188° [1386], hydrate: 97–101° [1386];
^{1}H NMR [1386], MS [1386].

[3-Chloro-6-hydroxy-4-methoxy-5-methyl-2-(1-methylpropyl)phenyl](2,4-dihydroxy-6-methylphenyl)methanone

[78023-64-0]



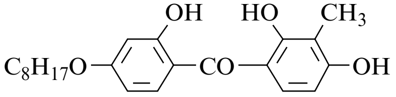
$C_{20}H_{23}ClO_5$ mol.wt. 378.85

Synthesis

- Preparation by hydrogenolysis of 2,2′,4′-tris-(benzyloxy)-5-chloro-4-methoxy-3,6′-dimethyl-6-sec-butylbenzophenone in the presence of 10% Pd/C in ethyl acetate containing concentrated hydrochloric acid (10 drops) (94%) [1387].

m.p. 177–179° [1387]; ^{1}H NMR [1387], MS [1387].

(2,4-Dihydroxy-3-methylphenyl)[2-hydroxy-4-(octyloxy)phenyl]methanone



$C_{22}H_{28}O_4$ mol.wt. 356.46

Synthesis

- Refer to: [224].

m.p. and Spectra (NA).

2.4 Tetrahydroxybenzophenones

2.4.1 *Hydroxy Groups Located on One Ring*

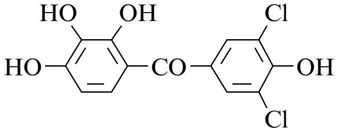
Not described till December 1999.

2.4.2 *Hydroxy Groups Located on Both Rings*

2.4.2.1 Substituents Located on One Ring

(3,5-Dichloro-4-hydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[105443-52-5]



$C_{13}H_8Cl_2O_5$ mol.wt. 315.11

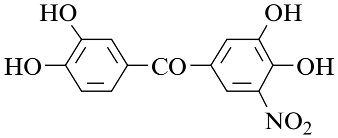
Synthesis

- Preparation by action of 3,5-dichloro-4-hydroxybenzoic acid on pyrogallol with boron trifluoride or its complexes [420].

m.p. and Spectra (NA).

(3,4-Dihydroxy-5-nitrophenyl)(3,4-dihydroxyphenyl)methanone

[134612-52-5]



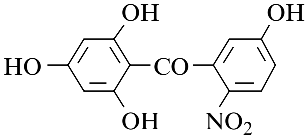
$C_{13}H_9NO_7$ mol.wt. 291.22

Synthesis

- Preparation by total demethylation of 4-hydroxy-5-nitro-3,3′,4′-trimethoxybenzophenone with hydrobromic acid in refluxing dilute acetic acid [1019].

m.p. 222–224° [1019]; Spectra (NA).

(5-Hydroxy-2-nitrophenyl)(2,4,6-trihydroxyphenyl)methanone



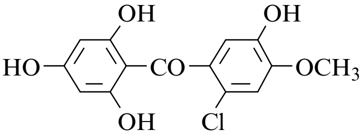
$C_{13}H_9NO_7$ mol.wt. 291.22

Synthesis

- Preparation by reaction of 5-hydroxy-2-nitrobenzoyl chloride with tris(trimethylsilyl) derivative of phloroglucinol in the presence of stannic chloride (or titanium tetrachloride or aluminium chloride) in refluxing methylene chloride for 2 h [921].

m.p. and Spectra (NA).

(2-Chloro-5-hydroxy-4-methoxyphenyl)(2,4,6-trihydroxyphenyl)methanone



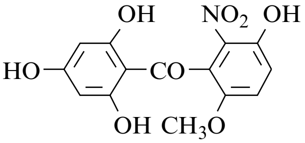
$C_{14}H_{11}ClO_6$ mol.wt. 310.69

Synthesis

- Preparation by reaction of 2-chloro-5-hydroxy-4-methoxybenzoyl chloride with tris-(trimethylsilyl) derivative of phloroglucinol in the presence of stannic chloride (or titanium tetrachloride or aluminium chloride) in refluxing methylene chloride for 2 h [921].

m.p. and Spectra (NA).

(3-Hydroxy-6-methoxy-2-nitrophenyl)(2,4,6-trihydroxyphenyl)methanone



$C_{14}H_{11}NO_8$ mol.wt. 321.24

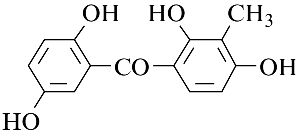
Synthesis

- Preparation by reaction of 3-hydroxy-6-methoxy-2-nitro-benzoyl chloride with tris-(trimethylsilyl) derivative of phloroglucinol in the presence of stannic chloride (or titanium tetrachloride or aluminium chloride) in refluxing methylene chloride for 2 h [921].

m.p. and Spectra (NA).

(2,4-Dihydroxy-3-methylphenyl)(2,5-dihydroxyphenyl)methanone

[61234-46-6]



$C_{14}H_{12}O_5$ mol.wt. 260.25

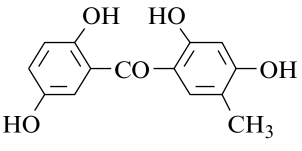
Synthesis

- Preparation by demethylation of 2,4,5′-trihydroxy-2′-methoxy-3-methylbenzophenone with hydrobromic acid in refluxing acetic acid during 2 h (79%) [413].

m.p. 238–240° [413]; Spectra (NA).

(2,4-Dihydroxy-5-methylphenyl)(2,5-dihydroxyphenyl)methanone

[61234-45-5]



$C_{14}H_{12}O_5$ mol.wt. 260.25

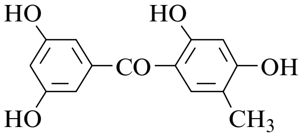
Synthesis

- Preparation by demethylation of 2,4,5′-trihydroxy-2′-methoxy-5-methylbenzophenone with hydrobromic acid in refluxing acetic acid during 2 h (77%) [413].

m.p. 244° [413]; Spectra (NA).

(2,4-Dihydroxy-5-methylphenyl)(3,5-dihydroxyphenyl)methanone

[61234-68-2] $C_{14}H_{12}O_5$ mol.wt. 260.25



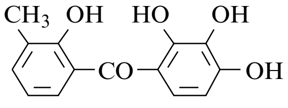
Synthesis

– Preparation by condensation of 3,5-diacetoxybenzonitrile with 4-methylresorcinol (80%) (Hoesch reaction) [413].

m.p. 263–265° [413]; Spectra (NA).

(2-Hydroxy-3-methylphenyl)(2,3,4-trihydroxyphenyl)methanone

[107412-94-2] $C_{14}H_{12}O_5$ mol.wt. 260.25



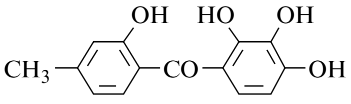
Synthesis

– Preparation by heating o-cresotic acid and pyrogallol with zinc chloride and phosphorous oxychloride (40%) [1367].

m.p. 137–138° [1367]; Spectra (NA).

(2-Hydroxy-4-methylphenyl)(2,3,4-trihydroxyphenyl)methanone

[109067-41-6] $C_{14}H_{12}O_5$ mol.wt. 260.25



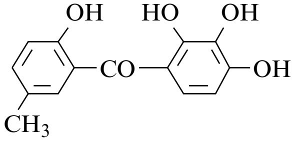
Synthesis

– Preparation by heating m-cresotic acid and pyrogallol with zinc chloride and phosphorous oxychloride (40%) [1367].

m.p. 122–123° [1367]; Spectra (NA).

(2-Hydroxy-5-methylphenyl)(2,3,4-trihydroxyphenyl)methanone

[105443-51-4] $C_{14}H_{12}O_5$ mol.wt. 260.25



Synthesis

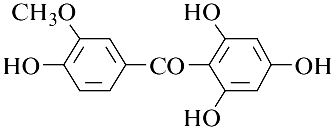
– Preparation by treatment of pyrogallol with 5-methylsalicylic acid,

- in tetrachloroethane bubbling boron trifluoride at 110° for 1 h [420];
- in the presence of Amberlyst-15 in refluxing toluene under azeotropical water removal for 21 h [419].

– Also refer to: [421].

m.p. and Spectra (NA).

(4-Hydroxy-3-methoxyphenyl)(2,4,6-trihydroxyphenyl)methanone



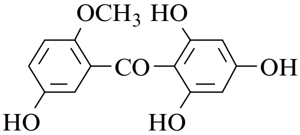
$C_{14}H_{12}O_6$ mol.wt. 276.25

Synthesis

- Preparation by reaction of vanillonitrile with phloro-glucinol in the presence of zinc chloride and hydrochloric acid in ethyl ether, first at r.t. for 1 h, then at 50° for 4 h, followed by hydrolysis of the ketimine hydrochloride so obtained with boiling water for 1–2 h (38%) [440].

monohydrate [440]; m.p. >200° (d) [440]; Spectra (NA).

(5-Hydroxy-2-methoxyphenyl)(2,4,6-trihydroxyphenyl)methanone



$C_{14}H_{12}O_6$ mol.wt. 276.25

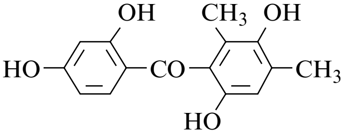
Synthesis

- Preparation by reaction of 5-hydroxy-2-methoxybenzoyl chloride with tris(trimethylsilyl) derivative of phloroglucinol in the presence of stannic chloride (or titanium tetrachloride or aluminium chloride) in refluxing methylene chloride for 2 h [921].

m.p. and Spectra (NA).

(3,6-Dihydroxy-2,4-dimethylphenyl)(2,4-dihydroxyphenyl)methanone

[42470-91-7]



$C_{15}H_{14}O_5$ mol.wt. 274.27

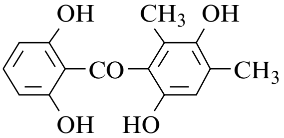
Synthesis

- Preparation by condensation of 3,6-dihydroxy-2,4-di-methylbenzoic acid with resorcinol in the presence of zinc chloride and phosphorous oxychloride at 40° for 1 h (69%) [1369].

m.p. 226° [1369]; ^{1}H NMR [1369], IR [1369], UV [1369], MS [1369].

(3,6-Dihydroxy-2,4-dimethylphenyl)(2,6-dihydroxyphenyl)methanone

[42594-60-5]



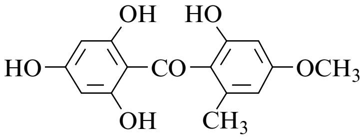
$C_{15}H_{14}O_5$ mol.wt. 274.27

Synthesis

- Obtained by demethylation of 2′,3,6-trihydroxy-6′-methoxy-2,4-dimethylbenzophenone with boron tribromide in refluxing methylene chloride for 90 h (58%) [1181].

m.p. 171–174° [1181]; ^{1}H NMR [1181], IR [1181], UV [1181], MS [1181].

(2-Hydroxy-4-methoxy-6-methylphenyl)(2,4,6-trihydroxyphenyl)methanone



$C_{15}H_{14}O_6$ mol.wt. 290.27

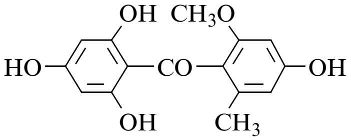
Synthesis

- Preparation by hydrogenolysis of 2,2′,4,6-tetrakis-(benzyloxy)-4′-methoxy-6′-methylbenzophenone (SM) in ethyl acetate/tetrahydrofuran mixture in the presence of 10% Pd/C at 25°. SM was obtained by condensation of 2-(benzyloxy)-4-methoxy-6-methyl-benzoic acid with phloroglucinol tribenzyl ether in the presence of trifluoroacetic anhydride in methylene chloride under nitrogen for 15 min (81%) [1179].

m.p. (NA); ^{1}H NMR [1179].

(4-Hydroxy-2-methoxy-6-methylphenyl)(2,4,6-trihydroxyphenyl)methanone

[60556-49-2]



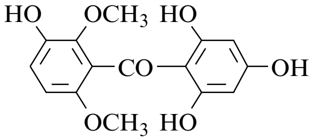
$C_{15}H_{14}O_6$ mol.wt. 290.27

Synthesis

- Preparation by catalytic hydrogenolysis of 2,4,4′,6-tetra-kis(benzyloxy)-2′-methoxy-6′-methylbenzophenone (SM) in the presence of 10% Pd/C in ethanol (72%). SM was obtained by condensation of 4-(benzyloxy)-6-methoxy-o-toluic acid with phloroglucinol tribenzyl ether in the presence of trifluoroacetic anhydride in methylene chloride (88%) [1349].
- Also refer to: [1348].

m.p. 177–178° [1349]; ^{1}H NMR [1349], IR [1349], UV [1349], MS [1349].

(3-Hydroxy-2,6-dimethoxyphenyl)(2,4,6-trihydroxyphenyl)methanone



$C_{15}H_{14}O_7$ mol.wt. 306.27

Synthesis

- Preparation by reaction of 3-hydroxy-2,6-dimethoxy-benzoyl chloride with tris(trimethylsilyl) derivative of phloroglucinol in the presence of stannic chloride (or titanium tetrachloride or aluminium chloride) in refluxing methylene chloride for 2 h [921].

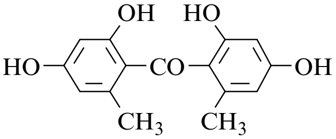
m.p. and Spectra (NA).

2.4.2.2 Substituents Located on Both Rings

Symmetrical ketones

Bis(2,4-Dihydroxy-6-methylphenyl)methanone

[39803-53-7] $C_{15}H_{14}O_5$ mol.wt. 274.27



Synthesis

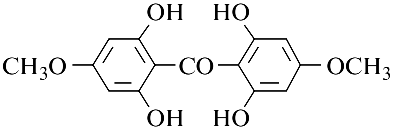
– Preparation by hydrogenolysis of 2,2′, 4,4′-tetrakis-(benzyloxy)-6,6′-dimethylbenzophenone (SM) in the presence of 10% Pd/C in ethyl acetate (90%) [1376]. SM was obtained by condensation of orcinol dibenzyl ether with orsellinic acid dibenzyl ether in methylene chloride in the presence of trifluoroacetic anhydride for 5 min (75%).

– Also refer to: [1388].

m.p. 195–198° [1376]; ^1H NMR [1376], IR [1376], UV [1376].

Bis(2,6-Dihydroxy-4-methoxyphenyl)methanone

$C_{15}H_{14}O_7$ mol.wt. 306.27



Synthesis

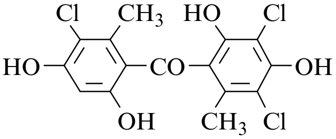
– Refer to: [1389].

m.p. and Spectra (NA).

Asymmetric ketones

(3-Chloro-4,6-dihydroxy-2-methylphenyl)(3,5-dichloro-2,4-dihydroxy-6-methylphenyl)-methanone

[69709-91-7] $C_{15}H_{11}Cl_3O_5$ mol.wt. 377.61



Synthesis

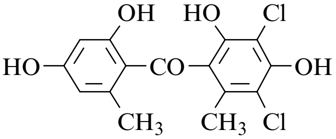
– Preparation by hydrogenolysis of 2,2′,4, 4′-tetrakis-(benzyloxy)-3,5,5′-trichloro-6,6′-dimethylbenzophenone (SM) in the presence of 10% Pd/C in ethyl acetate containing concentrated hydrochloric acid (5 drops) (>90%) [1339]. SM was obtained by Friedel–Crafts acylation of 3,5-bis(benzyloxy)-2-chlorotoluene with 2,4-bis(benzyloxy)-3,5-dichloro-6-methylbenzoic acid in the presence of trifluoroacetic anhydride in refluxing ethylene dichloride for 5 h (33%).

– Also refer to: [1344].

m.p. 201–203° [1339]; ^1H NMR [1339], MS [1339].

(3,5-Dichloro-2,4-dihydroxy-6-methylphenyl)(2,4-dihydroxy-6-methylphenyl)methanone

[39803-58-2] $C_{15}H_{12}Cl_2O_5$ mol.wt. 343.16



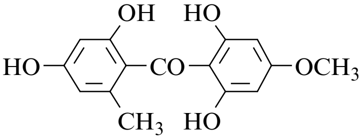
Synthesis

– Preparation by hydrogenolysis of 2,2′,4,4′-tetrakis-(benzyloxy)-3,5-dichloro-6,6′-dimethylbenzophenone (SM) in ethanol in the presence of 10% Pd/C for 3 h (51%). SM was obtained by condensation of 3,5-di-chloroorsellinic acid dibenzyl ether with orcinol dibenzyl ether in refluxing chloroform in the presence of trifluoroacetic anhydride/trifluoroacetic acid for 3 h [1376].

m.p. 213–216° [1376]; ^{1}H NMR [1376], UV [1376].

(2,6-Dihydroxy-4-methoxyphenyl)(2,4-dihydroxy-6-methylphenyl)methanone

[60556-46-9] $C_{15}H_{14}O_6$ mol.wt. 290.27



Synthesis

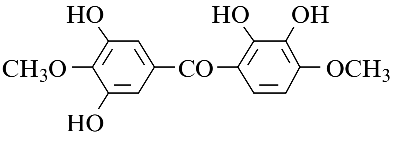
– Preparation by catalytic hydrogenolysis of 2,2′,4′,6-tetrakis(benzyloxy)-4-methoxy-6′-methyl-benzophenone (SM) in the presence of 10% Pd/C in ethyl acetate at r.t. (71%). SM was obtained by condensation of 2,6-bis(benzyloxy)-4-methoxybenzoic acid with 3,5-bis(benzyloxy)toluene in the presence of trifluoroacetic anhydride in methylene chloride for 5 min at r.t. (69%) [1349].

– Also refer to: [1378,1379].

m.p. 250–251° (first melting point at 115°) [1349];
^{1}H NMR [1349], IR [1349], UV [1349], MS [1349].

(2,3-Dihydroxy-4-methoxyphenyl)(3,5-dihydroxy-4-methoxyphenyl)methanone

$C_{15}H_{14}O_7$ mol.wt. 306.27



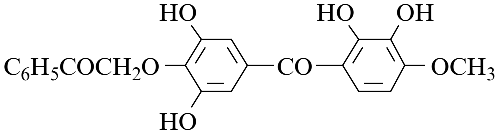
Synthesis

– Obtained by reaction of methyl iodide with exifone (2,3,3′,4,4′,5′-hexahydroxybenzophenone) in the presence of lithium carbonate in N,N-dimethylformamide at 30° for 15 h under nitrogen (30%) [369].

m.p. 216–218° [369]; ^{1}H NMR [369], ^{13}C NMR [369], MS [369]; pK_a [369].

[4-(Benzoylmethoxy)-3,5-dihydroxyphenyl](2,3-dihydroxy-4-methoxyphenyl) methanone

2-[4-(2,3-Dihydroxy-4-methoxybenzoyl)-2,6-dihydroxyphenoxy]-1-phenylethanone



$C_{22}H_{18}O_8$ mol.wt. 410.38

Synthesis

- Obtained by action of methyl iodide with 4′-phenacyl ether of exifone (2,3,3′,4,4′,5′-hexahydroxybenzophenone) in the presence of lithium carbonate in N,N-dimethylformamide at 30° for 15 h (30%) [369].

m.p. (NA); ^{1}H NMR [369], ^{13}C NMR [369].

2.5 Pentahydroxybenzophenones

2.5.1 Hydroxy Groups Located on One Ring

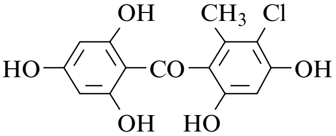
Not described till December 1999

2.5.2 Hydroxy Groups Located on Both Rings

2.5.2.1 Substituents Located on One Ring

(3-Chloro-4,6-dihydroxy-2-methylphenyl)(2,4,6-trihydroxyphenyl)methanone

[68048-30-6]



$C_{14}H_{11}ClO_6$ mol.wt. 310.69

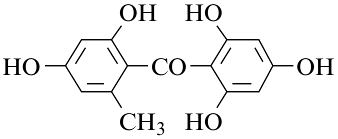
Synthesis

- Preparation by hydrogenolysis of 2′,4,4′,6,6′-pentakis-(benzyloxy)-3-chloro-2-methylbenzophenone (SM) in ethyl acetate/tetrahydrofuran in the presence of 10% Pd/C at 25°. SM was obtained by condensation of 4,6-bis-(benzyloxy)-3-chloro-2-methylbenzoic acid with phloroglucinol tribenzyl ether in the presence of trifluoroacetic anhydride in methylene chloride under nitrogen for 2 min (95%) [1179].

m.p. (NA); ^{1}H NMR [1179].

(2,4-Dihydroxy-6-methylphenyl)(2,4,6-trihydroxyphenyl)methanone

[55018-96-7]



$C_{14}H_{12}O_6$ mol.wt. 276.25

Syntheses

- Preparation by hydrogenolysis of 2,2′,4,4′,6-pentakis-(benzyloxy)-6′-methylbenzophenone (SM), in the presence of 10% Pd/C in ethyl acetate or in a ethanol/ethyl acetate mixture at atmospheric pressure and r.t. (>95%) [1179,1349,1390]. SM was obtained,
  - by condensation of 4,6-bis(benzyloxy)-o-toluic acid with phloroglucinol tribenzyl ether in the presence of trifluoroacetic anhydride (TFAA) in a chloroform/methylene chloride mixture at r.t. for 5 min (61%, colourless oil) [1349];
  - by condensation of 2,4,6-tris(benzyloxy)benzoic acid with orcinol dibenzyl ether in the presence of trifluoroacetic anhydride (TFAA) in methylene chloride for 10 min [1179], (83%, m.p. 33°) [1390].
- Also obtained (poor yield) by demethylation of 2,2′,4,4′,6-pentamethoxy-6′-methylbenzophenone (m.p. 126°5–127°) in methylene chloride in the presence of boron tribromide at r.t. for overnight (15%) [1390].
- Also obtained by Friedel–Crafts acylation of phloroglucinol with o-orsellinic acid [1391].
- Also obtained by Fries rearrangement of phloroglucinyl o-orsellinate (m.p. 185–188°) [1391].
- Also refer to: [1345,1378,1379,1389,1392].

N.B.: Discussion on hypothetical formation from various polyketones [1287]. This ketone is the biosynthetic precursor of the antibiotic griseofulvin and the various fungal and lichen xanthones [1287]. It was very unstable and underwent facile cyclization to norlichexanthone (1,3,6-tri-hydroxy-8-methylxanthen-9-one) [1179].

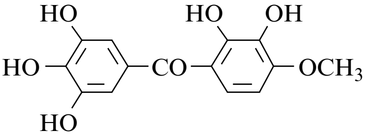
viscous, pale yellow oil [1349]; b.p. (NA);
pale yellow powder [1390]; m.p. (NA);
light yellow compound [1179];

N.B.: The description of the physical state of this ketone was imprecise.

^{1}H NMR [1349,1390], IR [1349,1390], UV [1349,1390],
MS [1349]; TLC [1390].

(2,3-Dihydroxy-4-methoxyphenyl)(3,4,5-trihydroxyphenyl)methanone

[177703-30-9] $C_{14}H_{12}O_7$ mol.wt. 292.25



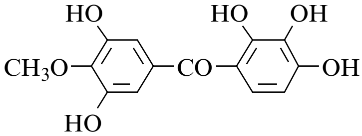
Synthesis

- Preparation from exifone (2,3,3′,4,4′,5′-hexahydroxy-benzophenone) in three steps: at first, phenacylation of exifone at the 4′-position, according to [365]. After, methylation of the 4′-phenacyl ether of exifone at the 4-position. Then, removal of the phenacyl protecting group of 4-methoxy-4′-phenacyl exifone derivative obtained in the presence of zinc dust in an acetic acid/methanol mixture at r.t. for 2 min (50%) [369].

m.p. 260–262° [369]; ^{1}H NMR [369], ^{13}C NMR [369], MS [369]; pK_a [369].

(3,5-Dihydroxy-4-methoxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[170630-11-2] $C_{14}H_{12}O_7$ mol.wt. 292.25



Synthesis

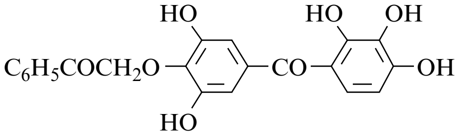
- Obtained by reaction of methyl iodide with exifone (2,3,3′,4,4′,5′-hexahydroxy-benzophenone) in the presence of lithium carbonate in N,N-dimethyl-formamide at 30° for 15 h under nitrogen (15%) [369].
- Also refer to: [1393].

m.p. 202–204° [369]; ^{1}H NMR [369], ^{13}C NMR [369], MS [369]; pK_a [369].

[4-(Benzoylmethoxy)-3,5-dihydroxyphenyl](2,3,4-trihydroxyphenyl) methanone

2-[2,6-Dihydroxy-4-(2,3,4-trihydroxybenzoyl)phenoxy]-1-phenylethanone

$C_{21}H_{16}O_8$ mol.wt. 396.35



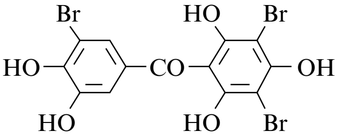
Synthesis

- Obtained by reaction of α-bromoacetophenone with exifone (2,3,3′,4,4′,5′-hexahydroxy-benzophenone) in the presence of lithium carbonate in N,N-dimethylformamide at r.t. for 15 h under nitrogen (40%) [365].

m.p. decomposes above 180° [365]; ^{1}H NMR [365], ^{13}C NMR [365], MS [365].

2.5.2.2 Substituents Located on Both Rings

(3-Bromo-4,5-dihydroxyphenyl)(3,5-dibromo-2,4,6-trihydroxyphenyl) methanone



$C_{13}H_7Br_3O_6$ mol.wt. 498.91

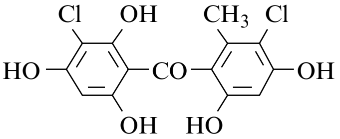
Synthesis

- Obtained by reaction of maclurin with bromine in boiling water [451].

N.B.: The formula proposed is the more likely.
monohydrate [451]; m.p. and Spectra (NA).

(3-Chloro-4,6-dihydroxy-2-methylphenyl)(3-chloro-2,4,6-trihydroxyphenyl) methanone

[68048-32-8]



$C_{14}H_{10}Cl_2O_6$ mol.wt. 345.14

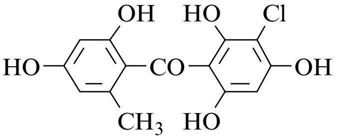
Synthesis

- Preparation by hydrogenolysis of 2,4,4′,6,6′-pentakis-(benzyloxy)-3,3′-dichloro-2′-methylbenzophenone (SM) in ethyl acetate/tetrahydrofuran mixture in the presence of 10% Pd/C at 25°. SM was obtained by condensation of 2,4,6-tris(benzyloxy)-3-chlorobenzoic acid with 3,5-bis (benzyloxy)-2-chlorotoluene in the presence of trifluoroacetic anhydride in methylene chloride under nitrogen for 20 min (18%) [1179].

m.p. (NA); ^{1}H NMR [1179].

(3-Chloro-2,4,6-trihydroxyphenyl)(2,4-dihydroxy-6-methylphenyl)methanone

[68048-31-7]



$C_{14}H_{11}ClO_6$ mol.wt. 310.69

Synthesis

- Preparation by hydrogenolysis of 2,2′,4, 4′,6-pentakis-(benzyloxy)-3-chloro-6′-methylbenzophenone (SM) in ethyl acetate/tetrahydrofuran mixture in the presence of 10% Pd/C at 25°. SM was obtained by condensation of 3-chloro-2,4,6-tris(benzyloxy)benzoic acid with orcinol dibenzyl ether in the presence of trifluoroacetic anhydride in methylene chloride under nitrogen for 15 min [1179].

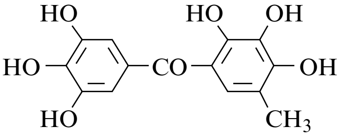
m.p. (NA); ^{1}H NMR [1179].

2.6 Hexahydroxybenzophenone

(2,3,4-Trihydroxy-5-methylphenyl)(3,4,5-trihydroxyphenyl)methanone

[112232-18-5]

$C_{14}H_{12}O_7$ mol.wt. 292.25



Synthesis

- Refer to: [438] (Japanese patent).

m.p. and Spectra (NA).

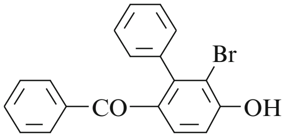
Chapter 3
Polyphenyl Phenyl Methanones
(Class of METHANONES)

3.1 Biphenyl Phenyl Methanones

3.1.1 Monohydroxylated Ketones

(6-Bromo-5-hydroxy[1,1′-biphenyl]-2-yl)phenylmethanone

[133721-72-9] $C_{19}H_{13}BrO_2$ mol.wt. 353.22



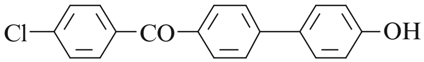
Synthesis

- Preparation by adding trimethylsilyl trifluoromethane-sulfonate to a solution of 5-bromo-2,3-dihydro-2,2-di-methyl-6-phenyl-4*H*-pyran-4-one, di-(tert-butyl)pyridine and 1-benzoylacetylene in chloroform at −30°. Then, the mixture was stirred at 20° for 24 h (59%) [833].

m.p. 155–156° [833]; ^{1}H NMR [833], IR [833], MS [833].

(4-Chlorophenyl)(4′-hydroxy[1,1′-biphenyl]-4-yl)methanone

[38304-24-4] $C_{19}H_{13}ClO_2$ mol.wt. 308.76



Syntheses

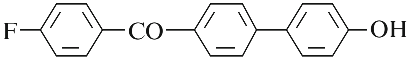
- Preparation by reaction of p-chlorobenzoic acid with 4-hydroxybiphenyl in the presence of trifluoromethanesulfonic acid, first at 50° for 23 h, then at 70° for 4 h (97%) [151].
- Preparation by Friedel–Crafts acylation of p-acetoxybiphenyl with p-chlorobenzoyl chloride, followed by hydrolysis of the keto ester so obtained (74%) [1394].

m.p. 196–197° [151,1394]; ^{1}H NMR [151,1394], IR [151,1394].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_3, © Springer Science+Business Media B.V. 2011

(4-Fluorophenyl)(4′-hydroxy[1,1′-biphenyl]-4-yl)methanone

[112782-46-4]



$C_{19}H_{13}FO_2$ mol.wt. 292.31

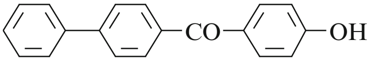
Synthesis

- Preparation by Fries rearrangement of 4-(4-fluorobenzoyloxy)biphenyl with aluminium chloride in o-dichlorobenzene at 120° for 8 h (good yield) [1395].

m.p. and Spectra (NA).

[1,1′-Biphenyl]-4-yl(4-hydroxyphenyl)methanone

[3558-83-6]



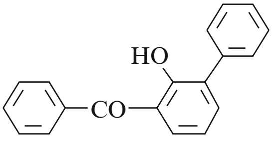
$C_{19}H_{14}O_2$ mol.wt. 274.32

Syntheses

- Preparation by demethylation of p-anisoylbiphenyl,
  - with pyridinium chloride at reflux a few min [1396];
  - with aluminium chloride in benzene [1141].
- Also obtained by reaction of EKONOL$^{(RM)}$, an aromatic polyester as Friedel–Crafts reagent, with biphenyl in triflic acid at 25° for 18 h (95%) [922]. Similar results can be obtained using hydrofluoric acid/boron trifluoride or aluminium chloride in place of triflic acid [922].
- Also refer to: Chem. Abstr., **127**, 34137f (1997).

m.p. 186° [1396], 185–187° [1141]; ^{13}C NMR [922], MS [922]; HPLC [922].

(2-Hydroxy[1,1′-biphenyl]-3-yl)phenylmethanone



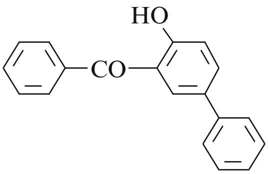
$C_{19}H_{14}O_2$ mol.wt. 274.32

Synthesis

- Obtained by irradiation of 2-biphenylyl benzoate with 254 nm light in benzene (23%) [1397].

m.p. and Spectra (NA).

(4-Hydroxy[1,1′-biphenyl]-3-yl)phenylmethanone



$C_{19}H_{14}O_2$ mol.wt. 274.32

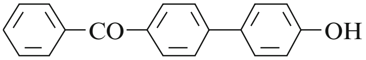
Synthesis

- Obtained by irradiation of 4-biphenylyl benzoate with 254 nm light in benzene (54%) [1397].

m.p. and Spectra (NA).

(4′-Hydroxy[1,1′-biphenyl]-4-yl)phenylmethanone

[5623-46-1] $C_{19}H_{14}O_2$ mol.wt. 274.32



Syntheses

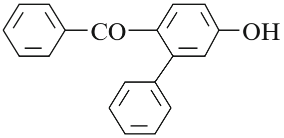
– Obtained by Fries rearrangement of 4-benzoyloxy-biphenyl in the presence of aluminium chloride without solvent for 30 min at 160° (22%) [1398] or in tetrachloroethane for 1 h at 140° [130]. There is an heteronuclear migration of the benzoyl group, since the Fries reaction catalysed by Lewis acids is not a true rearrangement, but more probably an intermolecular acylation [1399,1400].

– Preparation by Friedel–Crafts acylation of p-acetoxybiphenyl with benzoyl chloride, followed by hydrolysis of the resulting keto ester (94%) [1394].

m.p. 194–195°5 [1394], 193–195° [130,1398]; ^{1}H NMR [1394], IR [1394].

(5-Hydroxy[1,1′-biphenyl]-2-yl)phenylmethanone

[133721-67-2] $C_{19}H_{14}O_2$ mol.wt. 274.32



Syntheses

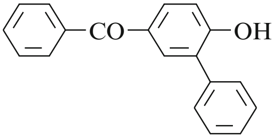
– Obtained by heating a solution of 5-[(triisopropylsilyl)oxy]-biphenyl-2-yl phenyl ketone in methanol with 2 N aqueous hydrochloric acid for 3 h at 80° in a sealed tube (93%) [833].

– Preparation by adding trimethylsilyl trifluoromethane-sulfonate to a solution of 2,3-dihydro-2,2-dimethyl-6-phenyl-*4H*-pyran-4-one, di-(tert-butyl)pyridine and 1-benzoylacetylene in chloroform at −30°. Then, the mixture was stirred at 0° for 30 min and at 20° for 24 h (84%) [833].

m.p. 179–180° [833]; ^{1}H NMR [833], IR [833], MS [833].

(6-Hydroxy[1,1′-biphenyl]-3-yl)phenylmethanone

[84627-07-6] $C_{19}H_{14}O_2$ mol.wt. 274.32



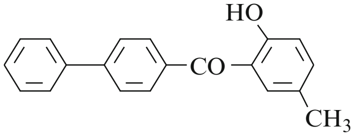
Syntheses

– Preparation by decarboxylation of 2-[(2′-hydroxy-5′-bi-phenylyl)carbonyl]benzoic acid in the presence of cupric acetate in refluxing quinoline for 30 min (96%) [1401].

– Also obtained by irradiation of 2-biphenylyl benzoate with 254 nm light in benzene (27%) [1397].

m.p. 195°2–197° [1401];
^{1}H NMR [1401], IR [1401], UV [1401]; TLC [1401].

[1,1′-Biphenyl]-4-yl(2-Hydroxy-5-methylphenyl)methanone]



$C_{20}H_{16}O_2$ mol.wt. 288.35

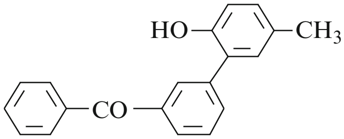
Syntheses

- Preparation by reaction of 4-biphenylcarbonyl chloride with p-cresol in the presence of aluminium chloride in trichlorobenzene at 190–200° for 2 h (64%) [1402].
- Preparation by Fries rearrangement of p-cresyl 4-bi-phenylcarboxylate with aluminium chloride in trichlorobenzene, first at 140° for 30 min, then between 140° and 200° for 30 min and at 200° for 3 h (82%) [1402].
- Also refer to: [235].

m.p. 79–80° [1402]; UV [1402].

(2′-Hydroxy-5′-methyl[1,1′-biphenyl]-3-yl)phenylmethanone

[132555-32-9]



$C_{20}H_{16}O_2$ mol.wt. 288.35

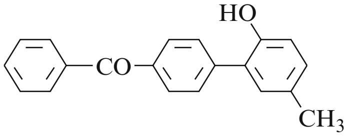
Synthesis

- Obtained by irradiation of a (*Z*)-(3-benzoylphenyl)azo tert-butyl sulfide and p-cresol mixture in the presence of potassium tert-butoxide in dimethyl sulfoxide for 3.5 h (53%) [1403].

glassy oil [1403]; b.p. (NA); ^{1}H NMR [1403].

(2′-Hydroxy-5′-methyl[1,1′-biphenyl]-4-yl)phenylmethanone

[132555-33-0]



$C_{20}H_{16}O_2$ mol.wt. 288.35

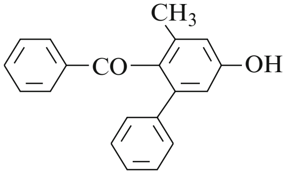
Synthesis

- Obtained by irradiation of a (*Z*)-(4-benzoylphenyl)azo tert-butyl sulfide and p-cresol mixture in the presence of potassium tert-butoxide in dimethyl sulfoxide for 2.5 h (54%) [1403].

m.p. 144°5–145°9 [1403]; ^{1}H NMR [1403], IR [1403].

(5-Hydroxy-3-methyl[1,1′-biphenyl]-2-yl)phenylmethanone

[133721-68-3]



$C_{20}H_{16}O_2$ mol.wt. 288.35

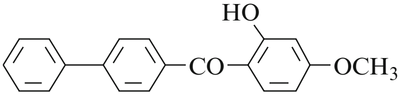
Synthesis

- Preparation by heating 3-methyl-5-[(triisopropylsilyl) oxy]-biphenyl-2-yl phenyl ketone in ethanol with 2 N aqueous hydrochloric acid for 4.5 h at 80° in a sealed tube (93%) [833].

m.p. 148–148°5 [833]; ^{1}H NMR [833], IR [833], MS [833].

[1,1′-Biphenyl]-4-yl(2-hydroxy-4-methoxyphenyl)methanone

[90986-69-9] $C_{20}H_{16}O_3$ mol.wt. 304.35



Synthesis

– Preparation by reaction of 4-biphenylcarbonyl chloride with resorcinol dimethyl ether in the presence of aluminium chloride [408],

- in nitrobenzene, first between 25° and 30° for 2 h, then at 80° for 4 h (56%) [1402];
- in refluxing carbon disulfide for 4 h (20–30%) [1141].

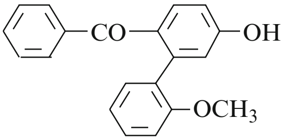
– Also refer to: [235].

m.p. 105–106° [1402], 104–105° [1141];

^{1}H NMR [1141], IR [1141], UV [1402], MS [1141].

(5-Hydroxy-2′-methoxy[1,1′-biphenyl]-2-yl)phenylmethanone

[133721-75-2] $C_{20}H_{16}O_3$ mol.wt. 304.35



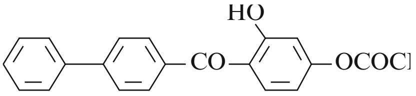
Synthesis

– Obtained by heating a solution of 5-[[dimethyl (1,1,2-tri-methylpropyl)silyl]oxy]-2′-methoxybiphenyl-2-yl phenyl ketone in ethanol with 2 N aqueous hydrochloric acid for 2.5 h at 80° in a sealed tube (93%) [833].

m.p. 177°5–178°5 [833]; ^{1}H NMR [833], IR [833], MS [833].

[4-(Acetyloxy)-2-hydroxyphenyl][1,1′-biphenyl]-4-ylmethanone

[36415-12-0] $C_{21}H_{16}O_4$ mol.wt. 332.36



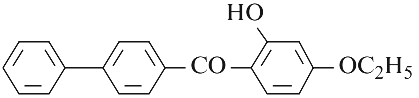
Synthesis

– Refer to: [235].

m.p. 108° [235]; UV [235].

[1,1′-Biphenyl]-4-yl(4-ethoxy-2-hydroxyphenyl)methanone

$C_{21}H_{18}O_3$ mol.wt. 318.37



Synthesis

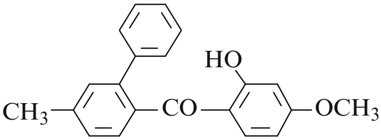
– Preparation by reaction of 4-biphenylcarbonyl chloride with resorcinoldiethyl ether in the presence of aluminium chloride in nitrobenzene, first between 25° and 30° for 2 h, then at 80° for 4 h [1402].

– Also refer to: [235].

m.p. 114–115° [1402]; UV [1402].

(2-Hydroxy-4-methoxyphenyl)(5-methyl[1,1′-biphenyl]-2-yl)methanone

[80988-17-6] $C_{21}H_{18}O_3$ mol.wt. 318.37



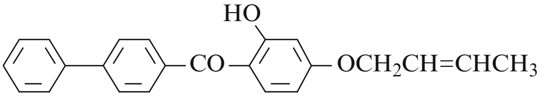
Synthesis

– Refer to: [1404,1405].

m.p. and Spectra (NA).

[1,1′-Biphenyl]-4-yl[4-(2-butenyloxy)-2-hydroxyphenyl]methanone

[36414-90-1] $C_{23}H_{20}O_3$ mol.wt. 344.41



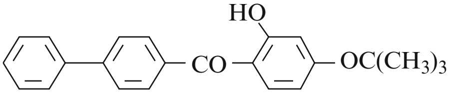
Synthesis

– Refer to: [235].

m.p. 117° [235]; UV [235].

[1,1′-Biphenyl]-4-yl[4-(1,1-dimethylethoxy)-2-hydroxyphenyl]methanone

[36488-90-1] $C_{23}H_{22}O_3$ mol.wt. 346.94



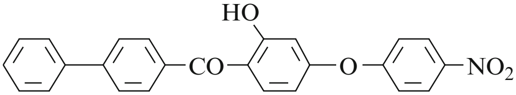
Synthesis

– Refer to: [235].

m.p. 80° [235]; UV [235].

[1,1′-Biphenyl]-4-yl[2-hydroxy-4-(4-nitrophenoxy)phenyl]methanone

[36469-48-4] $C_{25}H_{17}NO_5$ mol.wt. 411.41



Synthesis

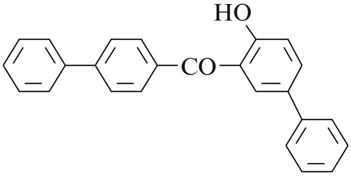
– Refer to: [235].

m.p. 234° [235]; UV [235].

[1,1′-Biphenyl]-4-yl(4-hydroxy[1,1′-biphenyl]-3-yl)methanone

4′,5-Diphenyl-2-hydroxybenzophenone

[95818-93-2] $C_{25}H_{18}O_2$ mol.wt. 350.42



Syntheses

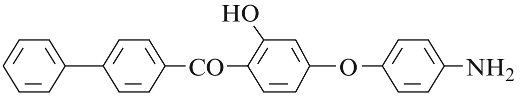
– Preparation by reaction of 4-biphenylcarbonyl chloride with 4-phenylphenol in the presence of aluminium chloride in trichlorobenzene at 190–200° for 2 h [1402].

– Also obtained by Fries rearrangement of 4-biphenylyl biphenyl-4-carboxylate, (4-biphenylyl 4-phenyl-benzoate), with aluminium bromide in chlorobenzene at 110° for 6 h [1406].

m.p. 135–136° [1402], 135° [1406]; IR [1406], UV [1402,1406]; GLC [1406].

[4-(4-Aminophenoxy)-2-hydroxyphenyl][1,1′-biphenyl]-4-ylmethanone

[36414-91-2] $C_{25}H_{19}NO_3$ mol.wt. 381.43



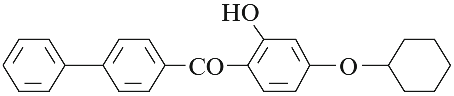
Synthesis

– Refer to: [235].

m.p. 163° [235]; UV [235].

[1,1′-Biphenyl]-4-yl[4-(cyclohexyloxy)-2-hydroxyphenyl]methanone

[36414-95-6] $C_{25}H_{24}O_3$ mol.wt. 372.46



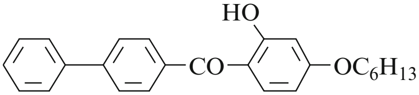
Synthesis

– Refer to: [235].

m.p. 123° [235]; UV [235].

[1,1′-Biphenyl]-4-yl[4-(hexyloxy)-2-hydroxyphenyl]methanone

[36419-22-4] $C_{25}H_{26}O_3$ mol.wt. 374.48



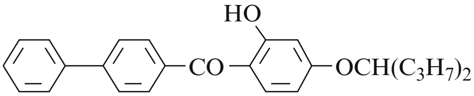
Synthesis

– Refer to: [235].

m.p. 89° [235]; UV [235].

[1,1′-Biphenyl]-4-yl[2-hydroxy-4-(1-propylbutoxy)phenyl]methanone

[36414-89-8] $C_{26}H_{28}O_3$ mol.wt. 388.51



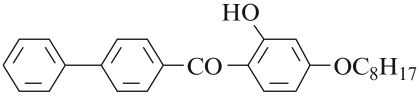
Synthesis

– Refer to: [235].

oil [235]; b.p. (NA); UV [235].

[1,1′-Biphenyl]-4-yl[2-hydroxy-4-(octyloxy)phenyl]methanone

[36130-58-2] $C_{27}H_{30}O_3$ mol.wt. 402.53



Synthesis

– Preparation by reaction of n-octyl chloride with 2,4-dihydroxy-4′-phenylbenzophenone in the presence of sodium hydroxide and antimony triiodide in diethylene glycol (diglycol) at 140° for 1 h (90%) [801].

– Also refer to: [235].

m.p. 69–70° [801], 67° [235]; UV [235].

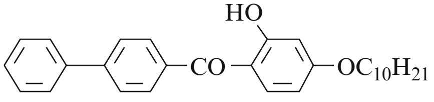
[1,1′-Biphenyl]-4-yl[4-(decyloxy)-2-hydroxyphenyl]methanone

[36419-24-6]

$C_{29}H_{34}O_3$ mol.wt. 430.59

Synthesis

– Refer to: [235].



m.p. 82° [235]; UV [235].

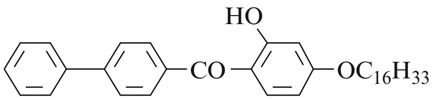
[1,1′-Biphenyl]-4-yl[4-(hexadecyloxy)-2-hydroxyphenyl]methanone

[36419-25-7]

$C_{35}H_{46}O_3$ mol.wt. 514.75

Synthesis

– Refer to: [235].



m.p. 88° [235]; UV [235].

3.1.2 Dihydroxylated Ketones

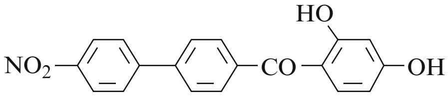
(2,4-Dihydroxyphenyl)(4′-nitro[1,1′-biphenyl]-4-yl)methanone

[36414-94-5]

$C_{19}H_{13}NO_5$ mol.wt. 335.32

Synthesis

– Refer to: [235].



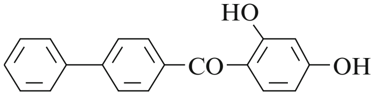
m.p. 299° [235]; UV [235].

[1,1′-Biphenyl]-4-yl(2,4-dihydroxyphenyl)methanone

[36130-57-1]

$C_{19}H_{14}O_3$ mol.wt. 290.32

Syntheses



– Preparation by reaction of 4-biphenylcarboxylic acid with resorcinol in the presence of boron trifluoride in tetrachloroethane at <45° for 5 h (67%) [1402].

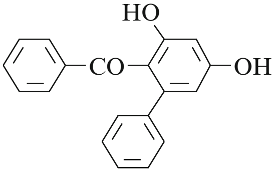
– Preparation by reaction of 4-biphenylcarbonyl chloride with resorcinol in the presence of aluminium chloride in nitrobenzene, first between 25° and 30° for 1 h, then at 55–60° for 4 h (91%) [1402].

– Also refer to: [235,801].

m.p. 188° [235], 183–184° [1402]; UV [235,1402].

(3,5-Dihydroxy[1,1′-biphenyl]-2-yl)phenylmethanone

[54439-82-6] $C_{19}H_{14}O_3$ mol.wt. 290.32



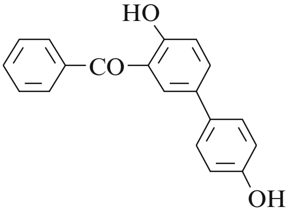
Synthesis

- Preparation from 3,3′-diphenyl-5,5′-diisoxazolylmethane by performing hydrogenolysis and subsequent hydrolysis with hydrochloric acid (55%) [693].

m.p. 148° [693]; ^{1}H NMR [693], MS [693].

(4,4′-Dihydroxy[1,1′-biphenyl]-3-yl)phenylmethanone

[52189-86-3] $C_{19}H_{14}O_3$ mol.wt. 290.32



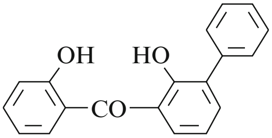
Syntheses

- Obtained by irradiation of [bi(cyclohexa-2,5-dienylidene)-4,4′-dione], so called 4,4′-diphenoquinone, in benzaldehyde for 2 days (21%) [1407].
- Also obtained by Fries rearrangement of 4,4′-biphenyldiyl dibenzoate with aluminium chloride in o-dichlorobenzene during 3 h at 180° (19%) [1408].

m.p. 142–144° [1408], 130° [1407];
^{1}H NMR [1407,1408], IR [1407,1408], UV [1408], MS [1407].

(2-Hydroxy[1,1′-biphenyl]-3-yl)(2-hydroxyphenyl)methanone

$C_{19}H_{14}O_3$ mol.wt. 290.32



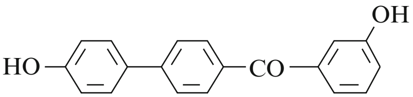
Synthesis

- Obtained by photo-Fries rearrangement of phenyl m-phenyl-salicylate (major product) [219].

m.p. and Spectra (NA).

(4′-Hydroxy[1,1′-biphenyl]-4-yl)(3-hydroxyphenyl)methanone

[75731-50-9] $C_{19}H_{14}O_3$ mol.wt. 290.32



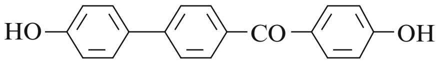
Synthesis

- Preparation by acylation of 4-hydroxybiphenyl with 3-hydroxybenzoic acid in hydrofluoric acid in the presence of boron trifluoride (pressure: 207 KPa) in an autoclave for 6 h at 0° (94%) [352].

m.p. 213–219° [352]; Spectra (NA).

(4′-Hydroxy[1,1′-biphenyl]-4-yl)(4-hydroxyphenyl)methanone

[86432-13-5] $C_{19}H_{14}O_3$ mol.wt. 290.32



Syntheses

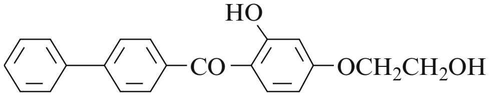
- Preparation by reaction of 4-hydroxybenzoic acid with 4-hydroxybiphenyl in the presence of trifluoromethanesulfonic acid at r.t. for overnight, then at 50° for 3.5 h and at 65° for 1.5 h (95%) [151].
- Preparation by Friedel–Crafts acylation of p-acetoxybiphenyl with p-hydroxybenzoyl chloride, followed by hydrolysis of the keto ester so obtained (65%) [1394].
- Preparation by heating a mixture of 4-hydroxybiphenyl-4′-carboxylic acid and phenol in nitrobenzene in the presence of boron trifluoride at 80° for 30 min (84%) [186].

m.p. 230–232° [1394], 190–198° [151]. There is discrepancy between the two melting points.

^{1}H NMR [151,1394], IR [1394].

[1,1′-Biphenyl]-4-yl[2-hydroxy-4-(2-hydroxyethoxy)phenyl]methanone

$C_{21}H_{18}O_4$ mol.wt. 334.37



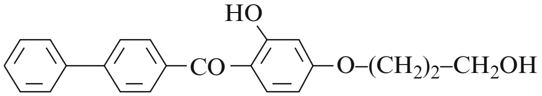
Synthesis

- Preparation by reaction of ethylene carbonate with 2,4-dihydroxy-4′-phenylbenzophenone in the presence of sodium methoxide in diisobutyl ketone for 2 h at 130° (84%) [700].

m.p. 157–158° [700]; Spectra (NA).

[1,1′-Biphenyl]-4-yl[2-hydroxy-4-(3-hydroxypropoxy)phenyl]methanone

[36469-47-3] $C_{22}H_{20}O_4$ mol.wt. 348.40



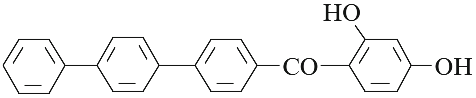
Synthesis

- Refer to: [235].

m.p. 145° [235]; UV [235].

3.2 Terphenyl Phenyl Methanones

(2,4-Dihydroxyphenyl)[1,1′;4′,1″]terphenyl-4″-ylmethanone



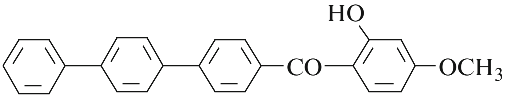
$C_{25}H_{18}O_3$ mol.wt. 366.42

Synthesis

- Preparation by Friedel–Crafts acylation of p-terphenyl with 2,4-dihydroxybenzoic acid or its derivatives [1409].

m.p. and Spectra (NA).

(2-Hydroxy-4-methoxyphenyl)[1,1′,4′,1″]terphenyl-4″-ylmethanone]



$C_{26}H_{20}O_3$ mol.wt. 380.44

Synthesis

- Preparation by Friedel–Crafts acylation of p-terphenyl with 2-hydroxy-4-methoxybenzoic acid or its derivatives [1409].

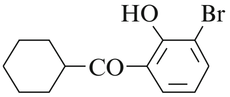
m.p. (NA); UV [1409].

Chapter 4
Cyclohexyl Phenyl Methanones
(Class of METHANONES)

4.1 Monohydroxylated Ketones

(3-Bromo-2-hydroxyphenyl)cyclohexylmethanone

[81066-14-0] $C_{13}H_{15}BrO_2$ mol.wt. 283.16



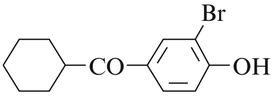
Syntheses

- Preparation by reaction of cyclohexanecarbonyl chloride with o-bromophenol in the presence of aluminium chloride without solvent at 140° for 1 h (48%) [1410].
- Preparation by Fries rearrangement of o-bromophenyl cyclohexanecarboxylate with aluminium chloride without solvent at 100° for 1 h (36%) [1411].

b.p.$_1$ 140–145° [1410]; m.p. 51–52° [1410]; Spectra (NA).

(3-Bromo-4-hydroxyphenyl)cyclohexylmethanone

[81066-15-1] $C_{13}H_{15}BrO_2$ mol.wt. 283.16



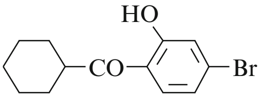
Syntheses

- Obtained by reaction of cyclohexanecarbonyl chloride with o-bromophenol in the presence of aluminium chloride without solvent at 120° for 2 h (16%) [1410].
- Also obtained by Fries rearrangement of o-bromophenyl cyclohexanecarboxylate with aluminium chloride without solvent at 120° for 1 h (19%) [1411].

b.p.$_1$ 160–170° [1410]; m.p. 138–139° [1410]; Spectra (NA).

(4-Bromo-2-hydroxyphenyl)cyclohexylmethanone

[81066-16-2] $C_{13}H_{15}BrO_2$ mol.wt. 283.16



Syntheses

- Preparation by reaction of cyclohexanecarbonyl chloride with m-bromophenol in the presence of aluminium chloride without solvent at 120° for 1–2 h (57–64%) [1410,1411].

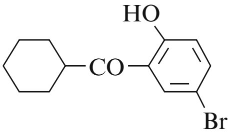
R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_4,

– Preparation by Fries rearrangement of m-bromophenyl cyclohexanecarboxylate with aluminium chloride without solvent at 100° for 1 h (71%) [1411].

b.p.$_1$ 165–175° [1410]; m.p. 56°5–57°5 [1410]; Spectra (NA).

(5-Bromo-2-hydroxyphenyl)cyclohexylmethanone

[81066-17-3] $C_{13}H_{15}BrO_2$ mol.wt. 283.16



Syntheses

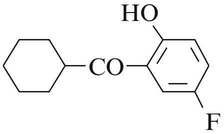
– Preparation by Fries rearrangement of p-bromophenyl cyclohexanecarboxylate with aluminium chloride without solvent at 120° for 1 h (55%) [1411].

– Also obtained by reaction of cyclohexanecarbonyl chloride with p-bromophenol in the presence of aluminium chloride without solvent at 120° for 2 h (32%) [1410] or between 100° and 140° for 1 h (12–22% yields) [1411].

b.p.$_1$ 150–170° [1410]; m.p. 98°5–99°5 [1410]; Spectra (NA).

Cyclohexyl(5-fluoro-2-hydroxyphenyl)methanone

[183280-18-4] $C_{13}H_{15}FO_2$ mol.wt. 222.26



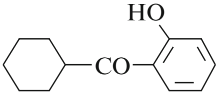
Synthesis

– Preparation by Fries rearrangement of p-fluorophenyl cyclohexanecarboxylate with aluminium chloride at 150–180° for 20 min (47%) [492].

m.p. 61°5 [492]; ^{1}H NMR [492], MS [492].

Cyclohexyl(2-hydroxyphenyl)methanone

[18066-52-9] $C_{13}H_{16}O_2$ mol.wt. 204.27



Syntheses

– Preparation by Fries rearrangement of phenyl cyclohexane-carboxylate with aluminium chloride,

- without solvent at 120° or at 160° for 2 h (45% and 27% yields, respectively) [1412] or at 150° for 3 h (70%) [1413];
- in nitrobenzene at 70° for 2 h [1414].

– Preparation by oxidative cleavage of 3-cyclohexylbenzofuran in methylene chloride with ozone at

– 78° for 15 min, then at r.t. for 1 h and saponification of the cyclohexyl 2-(formyloxy)phenyl ketone so obtained in methanol with 1 N sodium hydroxide at r.t. for 12 h [1415].

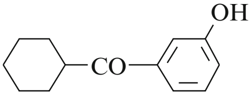
m.p. 104–105° [1412];

b.p.$_2$ 189–190° [1412], b.p.$_4$ 127–129° [1414]. There is discrepancy between the two boiling points.

^{1}H NMR [1415], IR [1415], MS [1415].

Cyclohexyl(3-hydroxyphenyl)methanone

[148493-08-7] $C_{13}H_{16}O_2$ mol.wt. 204.27



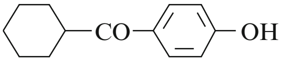
Synthesis

- Preparation by treatment of m-bromophenol in tetrahydrofuran with tert-butyllithium in pentane for 15 min at −78° under argon, after which a solution of cyclohexyl-N,O-dimethylhydroxamide in tetrahydrofuran was slowly added (60%). -Refer to: Chem. Abstr., **119**, 49010t (1993).

m.p. 70–72°; Spectra (NA).

Cyclohexyl(4-hydroxyphenyl)methanone

[38459-58-4] $C_{13}H_{16}O_2$ mol.wt. 204.27



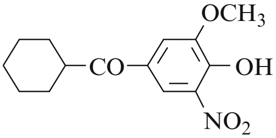
Syntheses

- Obtained (by-product) by Fries rearrangement of phenyl cyclohexanecarboxylate with aluminium chloride at 120° for 2 h (1%) [1412].
- Preparation by demethylation of 4-methoxyphenyl cyclohexyl ketone (SM) by treatment with boron tribromide in methylene chloride (76%). SM was obtained by Friede–Crafts acylation of anisole with cyclohexanecarboxylic acid chloride [1416].

m.p. and Spectra (NA).

Cyclohexyl(4-hydroxy-3-methoxy-5-nitrophenyl)methanone

[125629-27-8] $C_{14}H_{17}NO_5$ mol.wt. 279.29



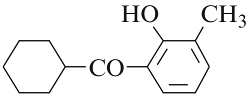
Synthesis

- Preparation by reaction of 65% nitric acid with cyclohexyl 4-hydroxy-3-methoxyphenyl ketone in acetic acid at r.t. (75%) [1084].

m.p. 135–141° [1084]; ^{1}H NMR [1084], MS [1084].

Cyclohexyl(2-hydroxy-3-methylphenyl)methanone

$C_{14}H_{18}O_2$ mol.wt. 218.30

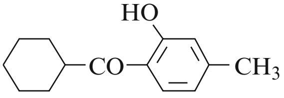


Synthesis

- Preparation by Fries rearrangement of o-cresyl cyclohexane-carboxylate with aluminium chloride at 120° for 2 h (56%) or at 160° for 2 h [1412].

m.p. 130° [1412]; Spectra (NA).

Cyclohexyl(2-hydroxy-4-methylphenyl)methanone



$C_{14}H_{18}O_2$ mol.wt. 218.30

Syntheses

– Obtained by Fries rearrangement of m-cresyl cyclohexane-carboxylate,

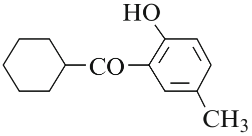
- with alumina in methanesulfonic acid for 25 min at 160° (86%). -Refer to: Chem. Abstr., **130**, 81248q (1999);
- with aluminium chloride without solvent at 120° or 160° for 2 h (poor yield) [1412].

– Preparation by Friede–Crafts acylation of m-cresol with cyclohexanecarboxylic acid in the presence of alumina in methanesulfonic acid for 5 min at 120° (87%).

– Refer to: Chem. Abstr., **130**, 81248q (1999).

b.p.$_2$ 115° [1412]; Spectra (NA).

Cyclohexyl(2-hydroxy-5-methylphenyl)methanone



$C_{14}H_{18}O_2$ mol.wt. 218.30

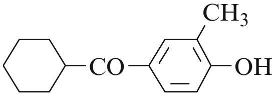
Syntheses

– Preparation by reaction of cyclohexanecarboxylic acid with p-cresol in the presence of aluminium chloride and phosphorous trichloride at 160° for 2 h (77%) [1417].

– Also obtained (poor yield) by Fries rearrangement of p-cresyl cyclohexanecarboxylate with aluminium chloride without solvent at 120° for 2 h (9%) or at 160° [1412].

m.p. 75–76° [1417];
b.p.$_2$ 120° [1412], b.p.$_5$ 160–170° [1417]. There is discrepancy between the two boiling points. Spectra (NA).

Cyclohexyl(4-hydroxy-3-methylphenyl)methanone



$C_{14}H_{18}O_2$ mol.wt. 218.30

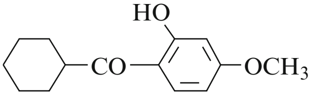
Synthesis

– Obtained (trace) by Fries rearrangement of o-cresyl cyclohexanecarboxylate with aluminium chloride at 120° or at 160° for 2 h (<1%) [1412].

m.p. 118–119° [1412]; Spectra (NA).

Cyclohexyl(2-hydroxy-4-methoxyphenyl)methanone

[69210-88-4] $C_{14}H_{18}O_3$ mol.wt. 234.30



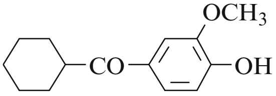
Syntheses

- Preparation by Fries rearrangement of m-methoxyphenyl cyclohexanecarboxylate in a nitromethane/toluene mixture in the presence of hafnium triflate/lithium perchlorate for 6 h at 50°.
- Preparation by Friede–Crafts acylation of m-methoxyphenol with cyclohexanecarbonyl chloride, cyclohexanecarboxylic acid or its anhydride in the same conditions.
- Refer to: Chem. Abstr., **127**, 278063v (1997).

m.p. and Spectra (NA).

Cyclohexyl(4-hydroxy-3-methoxyphenyl)methanone

[125629-26-7] $C_{14}H_{18}O_3$ mol.wt. 234.30



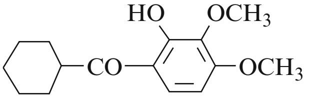
Synthesis

- Preparation by adding a solution of 30% hydrobromic acid in acetic acid to a solution of 4-(benzyloxy)-3-methoxy-phenyl cyclohexyl ketone in methylene chloride with stirring 2 h at r.t. (77%) [1084].

m.p. 113–115° [1084]; ^{1}H NMR [1084], MS [1084].

Cyclohexyl(2-hydroxy-3,4-dimethoxyphenyl)methanone

[121638-96-8] $C_{15}H_{20}O_4$ mol.wt. 264.32



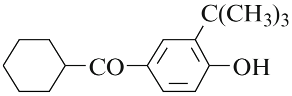
Synthesis

- Obtained (poor yield) by Friede–Crafts acylation of pyrogallol trimethyl ether with cyclo hexanecarbonyl chloride in the presence of aluminium chloride in refluxing methylene chloride for 2 h (7%) [756].

m.p. 85° [756]; Spectra (NA).

Cyclohexyl[3-(1,1-dimethylethyl)-4-hydroxyphenyl]methanone

[124979-10-8] $C_{17}H_{24}O_2$ mol.wt. 260.38



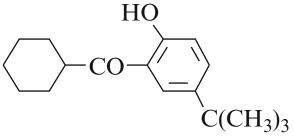
Synthesis

- Preparation by Friede–Crafts acylation of o-tert-butyl-phenol with cyclohexanecarbonyl chloride in ethylene dichloride in the presence of titanium tetrachloride, first at 0°, then at r.t. [816].

m.p. 142–143° [816]; Spectra (NA).

Cyclohexyl[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone

[18738-74-4] $C_{17}H_{24}O_2$ mol.wt. 260.38



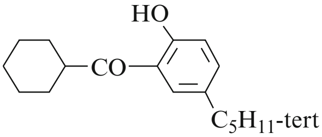
Syntheses

- Preparation by demethylation of 2-(cyclohexylcarbonyl)-4-tert-butylanisole with a mixture of 47% hydrobromic acid and 57% hydriodic acid in refluxing acetic acid for 2 h (95%) [817].
- Also obtained (poor yields) by Fries rearrangement of p-tert-butylphenyl cyclohexanecarboxylate with aluminium chloride at 120° for 2 h (5%) or at 160° for 2 h (8%) [1412].

b.p.$_6$ 219–220° [1412]; Spectra (NA).

Cyclohexyl[2-hydroxy-5-(1,1-dimethylpropyl)phenyl]methanone

$C_{18}H_{26}O_2$ mol.wt. 274.40



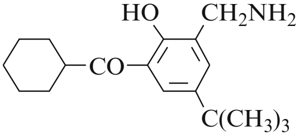
Synthesis

- Obtained (poor yields) by Fries rearrangement of p-tert-pentylphenyl cyclohexanecarboxylate with aluminium chloride at 120° for 2 h (16%) or at 160° for 2 h (7%) [1412].

oil [1412]; b.p.$_5$ 219–220° [1412]; Spectra (NA).

[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]cyclohexylmethanone

[75060-98-9] $C_{18}H_{27}NO_2$ mol.wt. 289.42



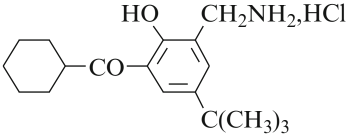
Synthesis

- Refer to: [817]; see the hydrochloride below.

m.p. and Spectra (NA).

[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]cyclohexylmethanone *(Hydrochloride)*

[75060-63-8] $C_{18}H_{27}NO_2 \cdot HCl$ mol.wt. 325.89



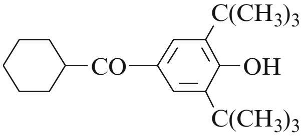
Synthesis

- Preparation by reaction of concentrated hydrochloric acid with 2-(cyclohexyl-carbonyl)-4-tert-butyl-6-(N-chloroacetylaminomethyl)phenol in refluxing ethanol for 30 h (67%) [817].

m.p. 205–209° [817]; Spectra (NA).

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]cyclohexylmethanone

[28440-98-4] $C_{21}H_{32}O_2$ mol.wt. 316.48



Synthesis

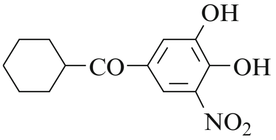
- Preparation by action of cyclohexanecarbonyl chloride with 2,6-di-tert-butylphenol in ethylene dichloride in the presence of titanium tetrachloride for 15–30 min at r.t.
- Refer to: Chem. Abstr., **90**, 121219v (1979)^{T}.

m.p. 125–127°^{T}; Spectra (NA).

4.2 Dihydroxylated Ketones

Cyclohexyl(3,4-dihydroxy-5-nitrophenyl)methanone

[125628-95-7] $C_{13}H_{15}NO_5$ mol.wt. 265.27



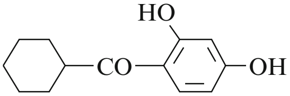
Synthesis

- Preparation by demethylation of cyclohexyl (4-hydroxy-3-methoxy-5-nitrophenyl) ketone with 48% hydrobromic acid in refluxing acetic acid for 20 h (55%) [1084].

m.p. 145–147° [1084]; ^{1}H NMR [1084], MS [1084].

Cyclohexyl(2,4-dihydroxyphenyl)methanone

[97231-21-5] $C_{13}H_{16}O_3$ mol.wt. 220.27



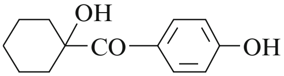
Synthesis

- Preparation by reaction of cyclohexanecarboxylic acid with resorcinol in the presence of zinc chloride at 125–135° (54%) (Nencki reaction) [1418].
- Also refer to: [298].

m.p. 115°5–116° [1418]; b.p.$_{4}$ 200–202° [1418]; Spectra (NA).

(1-Hydroxycyclohexyl)(4-hydroxyphenyl)methanone

[200420-24-2] $C_{13}H_{16}O_3$ mol.wt. 220.27



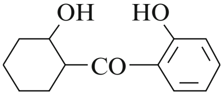
Synthesis

- Refer to: Chem. Abstr., **128**, 68498k (1998).

m.p. and Spectra (NA).

(2-Hydroxycyclohexyl)(2-hydroxyphenyl)methanone

[148077-95-6] $C_{13}H_{16}O_3$ mol.wt. 220.27



Synthesis

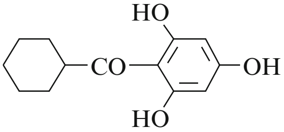
– Refer to: Chem. Abstr., **119**, 8806p (1993).

m.p. and Spectra (NA).

4.3 Trihydroxylated Ketones

Cyclohexyl(2,4,6-trihydroxyphenyl)methanone

[85602-45-5] $C_{13}H_{16}O_4$ mol.wt. 236.27



Synthesis

– Preparation by action of cyclohexanecarbonyl chloride with phloroglucinol in the presence of aluminium chloride in refluxing carbon disulfide/nitrobenzene mixture for 3 h.

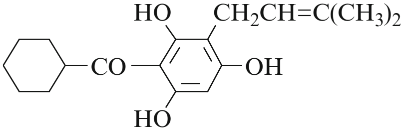
– Refer to: Chem. Abstr., **99**, 5339w (1983)^T.

– Also refer to: Chem. Abstr., **101**, 151650s (1984).

m.p. 110–113°^{T}; Spectra (NA).

Cyclohexyl[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl]methanone

[85602-18-2] $C_{18}H_{24}O_4$ mol.wt. 304.39



Synthesis

– Preparation by reaction of prenyl chloride with cyclohexyl 2,4,6-trihydroxyphenyl ketone in ethyl ether in the presence of a saturated aqueous sodium carbonate solution and a catalytic amount of cuprous chloride for 3 h at r.t. (48%).

– Refer to: Chem. Abstr., **101**, 151650s (1984)^T.

– Also refer to: Chem. Abstr., **101**, 221168s (1984)^{TT}.

m.p. 144–147°^{T}; ^{13}C NMR^T, ^{TT}, IR^T, MS^T.

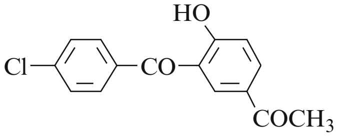
Part II
Diaroylphenols and Polyaroylphenols

Chapter 5
Phenols with One Benzoyl Group and One or Several Acetyl Groups
(Class of ETHANONES)

5.1 Monohydroxylated Ketones

1-[3-(4-Chlorobenzoyl)-4-hydroxyphenyl]ethanone

[108294-81-1] $C_{15}H_{11}ClO_3$ mol.wt. 274.70



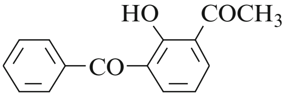
Synthesis

– Obtained (poor yield) by reaction of 5-acetyl-2-hydroxy-benzoyl chloride with chlorobenzene in the presence of aluminium chloride at 100° for 24 h (7%) [92].

m.p. 127–130° [92]; Spectra (NA).

1-(3-Benzoyl-2-hydroxyphenyl)ethanone

[85558-60-7] $C_{15}H_{12}O_3$ mol.wt. 240.26



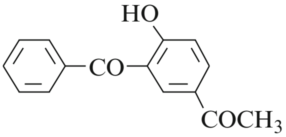
Synthesis

– Obtained by hydrolysis of 2-acetoxy-3-benzoyl-acetophenone [1419].

m.p. 108–109° [1419]; ^{1}H NMR [1419], IR [1419], MS [1419].

1-(3-Benzoyl-4-hydroxyphenyl)ethanone

[13043-37-3] $C_{15}H_{12}O_3$ mol.wt. 240.26



Synthesis

– Preparation by Fries rearrangement of p-acetylphenyl benzoate with aluminium chloride at 153° for 8 h (24%) [96].

m.p. 95° [96]; ^{1}H NMR [99], UV [99,109–111];
pK_a [96,115]; polarographic study [117]; TLC [116].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
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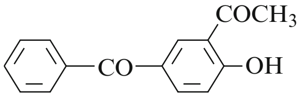
1-(5-Benzoyl-2-hydroxyphenyl)ethanone

[2589-80-2] $C_{15}H_{12}O_3$ mol.wt. 240.26



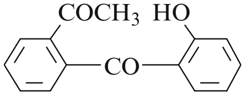
Synthesis

– Refer to: Chem. Abstr., **113**, 97396w (1990).

m.p. and Spectra (NA).

1-[2-(2-Hydroxybenzoyl)phenyl]ethanone

[17526-21-5] $C_{15}H_{12}O_3$ mol.wt. 240.26



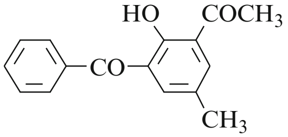
Syntheses

– Obtained by rearrangement of 10-methylanthronyl hydroperoxide (SM) with a mixture of acetic acid/4 N sulfuric acid (4:3). SM was obtained either by self-oxidation of 10-methylanthrone or by action of methylmagnesium iodide on 10-methylanthryl acetate [1420].
– Also obtained (poor yield) from meso-photoxide of 9-methylanthracene in refluxing o-dichloro-benzene for 3 h under nitrogen (3%) [1421].

m.p. 108° [1420], 107–108° [1421];
IR [1420], UV [1420], MS [1420].

(3-Benzoyl-2-hydroxy-5-methylphenyl)ethanone

[79877-07-9] $C_{16}H_{14}O_3$ mol.wt. 254.29



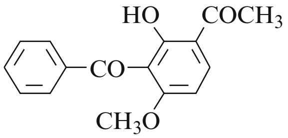
Syntheses

– Preparation by Fries rearrangement of 2-(benzoyloxy)-5-methylacetophenone with aluminium chloride at 130–140° for 2 h (60–65%) [616].
– Preparation by Fries rearrangement of 2-(acetyloxy)-5-methylbenzophenone with aluminium chloride at 140° for 1 h [573].
– Preparation by reaction of benzoyl chloride with 2-hydroxy-5-methylacetophenone in the presence of aluminium chloride in nitrobenzene at 80° for 8 h (40%) [1422].
– Also refer to: [1423].

m.p. 102–103° [573], 101–102° [1422];
^{1}H NMR [1422], IR [1422], UV [1422];
structural data [1424,1425].

1-(3-Benzoyl-2-hydroxy-4-methoxyphenyl)ethanone

[64857-84-7] $C_{16}H_{14}O_4$ mol.wt. 270.28



Syntheses

– Obtained by Friedel–Crafts acylation of paeonol with benzoyl chloride in the presence of aluminium chloride in nitrobenzene at r.t. or at 100° [270].

- Also obtained by Friedel–Crafts acylation of 2-hydroxy-6-methoxybenzophenone with acetic anhydride in the presence of aluminium chloride in nitrobenzene in a boiling water bath for 2 h [270].

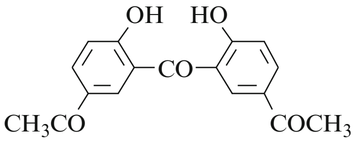
m.p. 169° [270]; Spectra (NA).

5.2 Dihydroxylated Ketones

5.2.1 Symmetrical Ketones

Bis(5-Acetyl-2-hydroxyphenyl)methanone

[33427-60-0]



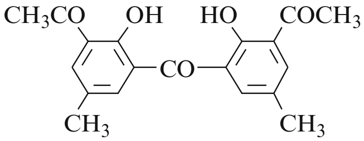
$C_{17}H_{14}O_5$ mol.wt. 298.30

Syntheses

- Obtained (poor yield) by Fries rearrangement of 2,2′-diacetoxybenzophenone with aluminium chloride without solvent at 160–180° for 20 min (10%) [1322].
- Also obtained by reaction of sodium hydroxide with 2,7-diacetylxanthone (94%) [1426].
- Also refer to: [1427].

m.p. >300° [1322], 175–177° [1426]. There is discrepancy between the two melting points.
^{1}H NMR [1322,1426], IR [1322], MS [1322,1426].

Bis(3-Acetyl-2-hydroxy-5-methylphenyl)methanone



$C_{19}H_{18}O_5$ mol.wt. 326.35

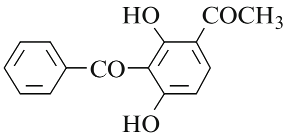
Synthesis

- Preparation by Fries rearrangement of 2,2′-diacetoxy-5,5′-dimethylbenzophenone with aluminium chloride without solvent, first at 120°, then at 170° for 20 min (80%) [1153].

m.p. 207–208° [1153]; Spectra (NA).

5.2.2 Asymmetric Ketones

1-(3-Benzoyl-2,4-dihydroxyphenyl)ethanone



$C_{15}H_{12}O_4$ mol.wt. 256.26

Syntheses

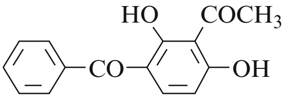
- Resacetophenone, by condensation with benzanilide imidochloride in the presence of aluminium chloride in nitrobenzene gave a keto anil.

This one was hydrolyzed with hydrochloric acid in refluxing ethanol and yielded the expected ketone (18%) [156].

– Also obtained by Fries rearrangement of 4-(benzoyloxy)-2-hydroxyacetophenone in the presence of aluminium chloride without solvent at 140° for 1.5 h (15%) [234].
– Also obtained (poor yield) by condensation of benzoyl chloride with resacetophenone in the presence of aluminium chloride in nitrobenzene in a water bath for 2 h (2%) [234].
– Also obtained by decarboxylation of 5-acetyl-3-benzoyl-2,4-dihydroxybenzoic acid on heating in a test-tube at 220–225° for 1 h [234].

m.p. 167–168° [156], 165° [234]; Spectra (NA).

1-(3-Benzoyl-2,6-dihydroxyphenyl)ethanone



$C_{15}H_{12}O_4$ mol.wt. 256.26

Syntheses

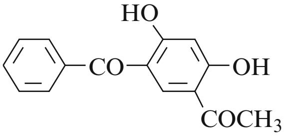
– Obtained by Friedel–Crafts acylation of resbenzophenone,
  - with acetic anhydride in the presence of aluminium chloride in nitrobenzene in a water bath for 6 h (30%) [234];
  - with acetyl chloride in the presence of aluminium chloride in o-dichlorobenzene [1428].

– Also obtained by Friedel–Crafts acylation of 2,6-dihydroxyacetophenone with benzoyl chloride in the presence of aluminium chloride in nitrobenzene, first at r.t., then in a water bath for 1 h [234].

m.p. 108° [234], 99°5–100° [1428]; Spectra (NA).

1-(5-Benzoyl-2,4-dihydroxyphenyl)ethanone

[64857-83-6]



$C_{15}H_{12}O_4$ mol.wt. 256.26

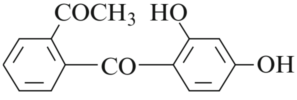
Synthesis

– Obtained by Friedel–Crafts acylation of paeonol with benzoyl chloride in the presence of aluminium chloride in nitro-benzene at r.t. or at 100° [270].

m.p. 138° [270]; Spectra (NA).

1-[2-(2,4-Dihydroxybenzoyl)phenyl]ethanone

[36414-93-4]



$C_{15}H_{12}O_4$ mol.wt. 256.26

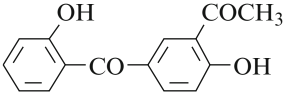
Synthesis

– Refer to: [235].

m.p. 184° [235]; UV [235].

1-[2-Hydroxy-5-(2-hydroxybenzoyl)phenyl]ethanone

[124208-69-1] $C_{15}H_{12}O_4$ mol.wt. 256.26



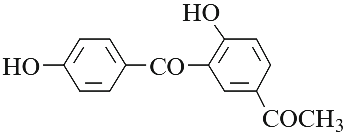
Syntheses

– Obtained by Fries rearrangement,
  - of 2,4′-diacetoxybenzophenone with aluminium chloride at 158–160° for 2 h (15%) [155];
  - of 4′-acetoxy-2-methoxybenzophenone with aluminium chloride at 153–155° for 2 h (60%) [155].

m.p. 130–131° [155]; Spectra (NA).

1-[4-Hydroxy-3-(4-hydroxybenzoyl)phenyl]ethanone

[124208-64-6] $C_{15}H_{12}O_4$ mol.wt. 256.26



Syntheses

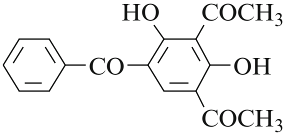
– Obtained by Fries rearrangement,
  - of phenyl 2-acetoxybenzoate with aluminium chloride at 180° for 3 h (38%) [155];
  - of phenyl 5-acetyl-2-hydroxybenzoate with aluminium chloride at 180° for 3 h (38%) [155].

m.p. 188° [155]; Spectra (NA).

1-(3-Acetyl-5-benzoyl-2,4-dihydroxyphenyl)ethanone

3,5-Diacetyl-2,4-dihydroxybenzophenone

[16832-72-7] $C_{17}H_{14}O_5$ mol.wt. 298.30



Syntheses

– Obtained by acetylation of 2,4-dihydroxybenzophenone,
  - with acetyl chloride in the presence of aluminium chloride in o-dichlorobenzene [1428];
  - with acetic anhydride in the presence of aluminium chloride in nitrobenzene in a water bath for 6 h (5%) [234].

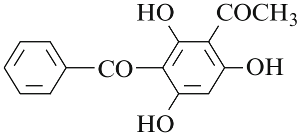
– Also obtained by condensation of 2-acetyl-4-benzoylresorcinol with acetic anhydride according to Friedel–Crafts [234].

m.p. 151–151°5 [1428], 151° [234]; Spectra (NA).

5.3 Trihydroxylated Ketone

1-(3-Benzoyl-2,4,6-trihydroxyphenyl)ethanone

[31188-65-5] $C_{15}H_{12}O_5$ mol.wt. 272.26



Synthesis

- Preparation by two successive Friedel–Crafts acylations of phloroglucinol, first with acetic acid, then with benzoic acid, always in the presence of boron trifluoride-etherate (compound 17).

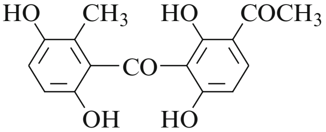
- Refer to: Chem. Abstr., **114**, 94606j (1991).

m.p. and Spectra (NA).

5.4 Tetrahydroxalated Ketone

1-[3-(3,6-Dihydroxy-2-methylbenzoyl)-2,4-dihydroxyphenyl]ethanone

[115834-34-9] $C_{16}H_{14}O_6$ mol.wt. 302.28



Synthesis

- Not yet described.

Isolation from natural source

- This ketone, named baishouwubenzophenone, is one of constituents of Baishouwu. The botanical source of this one is chiefly the tuber of *Cynanchum auriculatum* Royle ex Wight (Asclepiadaceae).

- Refer to: Chem. Abstr., **109**, 79560h (1988)^T.

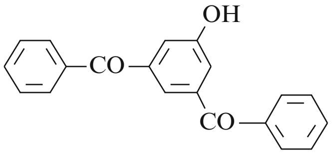
m.p. (NA); ^{1}H NMR^T, ^{13}C NMR^T, IR^T, UV^T, MS^T.

Chapter 6
Phenols with Two or Several Benzoyl Groups *(Class of METHANONES)*

6.1 Monohydroxylated Ketones

6.1.1 Symmetrical Ketones

(5-Hydroxy-1,3-phenylene)bis[phenylmethanone



$C_{20}H_{14}O_3$ mol.wt. 302.33

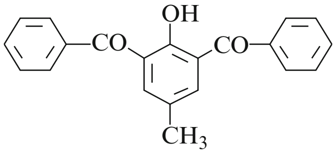
Synthesis

- Preparation by diazotization of 3,5-dibenzoylaniline, followed by hydrolysis of the resulting diazonium salt [1429].

m.p. 135° [1429]; Spectra (NA).

(2-Hydroxy-5-methyl-1,3-phenylene)bis[phenylmethanone

[77347-19-4]



$C_{21}H_{16}O_3$ mol.wt. 316.36

Syntheses

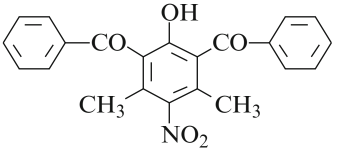
- Preparation by Fries rearrangement of 2-(benzoyl-oxy)-5-methylbenzophenone with aluminium chloride at 130–140° for 2 h (60–65%) [616].
- Preparation by reaction of benzoyl chloride with p-cresol in the presence of aluminium chloride in nitrobenzene at 80° for 8 h (30%) [1422].
- Preparation by reaction of benzotrichloride,
  - with 2-hydroxy-5-methylbenzophenone in the presence of aluminium chloride in nitrobenzene at 70° for 2 h [1430], (59%) [1423], (54%) [1431];
  - with p-cresol in the presence of aluminium chloride in carbon disulfide at 0° for 2 h (4%) [1423].
- Also refer to: [125,1432–1434].

m.p. 166–166°5 [1423], 164°2–165°6 [1431], 163–164° [1430], 160° [1422]; ^{1}H NMR [1422], IR [1422], UV[1422].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_6,

(2-Hydroxy-4,6-dimethyl-5-nitro-1,3-phenylene)bis[phenylmethanone

[85450-69-7] $C_{22}H_{17}NO_5$ mol.wt. 375.38



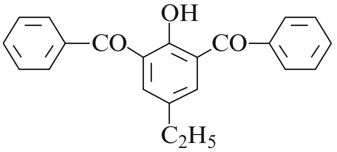
Synthesis

- Preparation by reaction of nitromethane with 1,3-dibenzoyl-4,6-dimethylpyrone in the presence of potassium tert-butoxide in tert-butanol at 60° for 4 h (77%) [1435].

m.p. 208–210° [1435]; Spectra (NA).

(5-Ethyl-2-hydroxy-1,3-phenylene)bis[phenylmethanone

$C_{22}H_{18}O_3$ mol.wt. 330.38



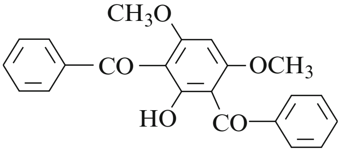
Synthesis

- Preparation by reaction of benzotrichloride with 5-ethyl-2-hydroxybenzophenone in the presence of aluminium chloride in nitrobenzene at 70° for 2 h [1431].
- Also refer to: [1432].

m.p. and Spectra (NA).

(2-Hydroxy-4,6-dimethoxy-1,3-phenylene)bis[phenylmethanone

[197169-08-7] $C_{22}H_{18}0_5$ mol.wt. 362.38



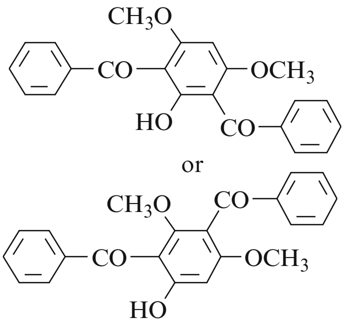
Synthesis

- Preparation from 1,3,5-trimethoxybenzene and benzoyl chloride by Friedel–Crafts acylation reaction.
- Refer to: Chem. Abstr., **127**, 292966y (1997).

m.p. and Spectra (NA).

(2-Hydroxy-4,6-dimethoxy-1,3-phenylene)bis[phenylmethanone
or
(6-Hydroxy-2,4-dimethoxy-1,3-phenylene)bis[phenylmethanone

$C_{22}H_{18}O_5$ mol.wt. 362.38



Synthesis

- Preparation by saponification of 2,4- or 2,6-di-benzoyl- 3,5-dimethoxyphenyl benzoate (SM) with potassium hydroxide in ethanol for 3 h at 100° in a sealed tube (90%) [220]. SM was obtained by Friedel–Crafts acylation of 3,5-dimethoxyphenyl benzoate with benzoyl chloride in the presence of zinc chloride in refluxing benzene for 45 min (70%, m.p. 194°). SM can also be obtained by Friedel–Crafts acylation of hydrocotoin (2-hydroxy-4,6-dimethoxy-benzophenone) with benzoyl chloride in the presence of zinc chloride in refluxing benzene for 90 min (72%, m.p. 194°).

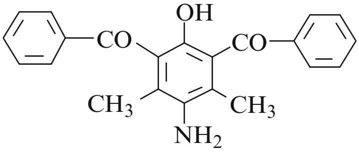
N.B.: K salt: [220].

m.p. 170° [220]; Spectra (NA).

(5-Amino-2-hydroxy-4,6-dimethyl-1,3-phenylene)bis[phenylmethanone

[85450-78-8]

$C_{22}H_{19}NO_3$ mol.wt. 345.40



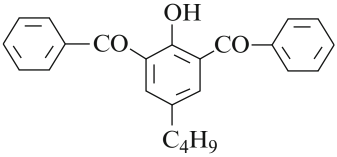
Synthesis

- Obtained (poor yield) by catalytic hydrogenation of 3-benzoyl-2-hydroxy-4,6-dimethyl-5-nitro-benzophenone in the presence of 10% Pd/C at 40° for 3 days (10%) [1435].

m.p. 208–211° [1435]; Spectra (NA).

(5-Butyl-2-hydroxy-1,3-phenylene)bis[phenylmethanone

$C_{23}H_{20}O_3$ mol.wt. 344.41



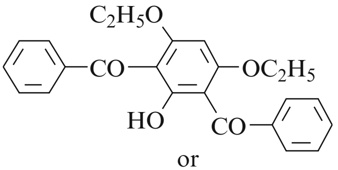
Synthesis

- Preparation by reaction of benzotrichloride with 5-butyl-2-hydroxybenzophenone in the presence of aluminium chloride in nitrobenzene at 70° for 2 h [1431].

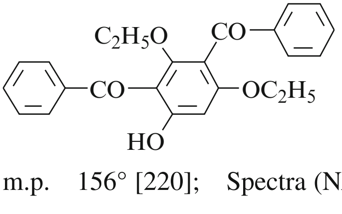
m.p. and Spectra (NA).

(2,4-Diethoxy-6-hydroxy-1,3-phenylene)bis[phenylmethanone
or
(4,6-Diethoxy-2-hydroxy-1,3-phenylene)bis[phenylmethanone

$C_{24}H_{22}O_5$ mol.wt. 390.44



or



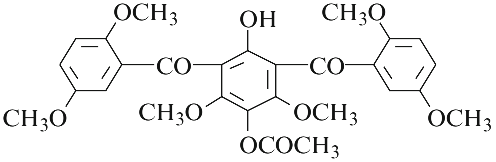
Synthesis

- Preparation by saponification of 2,4- or 2,6-di-benzoyl-3,5-diethoxyphenyl benzoate (SM) with potassium hydroxide in ethanol for 3 h at 100° in a sealed tube (90%). SM was obtained by Friedel–Crafts acylation of 3,5-diethoxyphenyl benzoate (m.p. 84°) with benzoyl chloride in the presence of zinc chloride in refluxing benzene for 45 min (72–81%, m.p. 163–164°) [220].

m.p. 156° [220]; Spectra (NA).

[5-(Acetyloxy)-2-hydroxy-4,6-dimethoxy-1,3-phenylene]bis [(2,5-dimethoxyphenyl)methanone

[129168-55-4]



$C_{28}H_{28}O_{11}$ mol.wt. 492.53

Synthesis

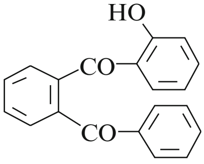
- Obtained (poor yield) by reaction of 2,5-dimethoxybenzoic acid with 2,6-dimethoxy-1,4-hydroquinone diacetate in the presence of trifluoroacetic anhydride for 2 weeks at r.t. (3%) [1160].

m.p. 114–115° [1160]; MS [1160].

6.1.2 *Asymmetric Ketones*

(2-Benzoylphenyl)(2-hydroxyphenyl)methanone

[14596-74-8]



$C_{20}H_{14}O_3$ mol.wt. 302.33

Syntheses

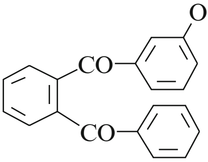
- *From photoxide of 9-phenylanthracen* (SM1):
  - SM1 in dilute acetic acid under nitrogen during 3 h isomerises into 1-(2-hydroxyphenyl)-3-phenylisobenzofuran (SM2). In this mixture, SM2, by self-oxidation in the presence of air and sunlight during 3 days gave the 2-(2-hydroxybenzoyl)benzophenone (22%) [1436].
  - Also obtained (poor yield) by action of sulfuric acid with SM1 in acetic acid/acetic anhydride at r.t. for 5 min (8%) [1436].
  - Also obtained (poor yield) from SM1 in refluxing toluene (at 110°) or in o-dichlorobenzene (at 180°) for 3 h under nitrogen (2–5%, yields respectively). The reaction carried out in the presence of N-maleimide in refluxing toluene in the same conditions gave a 3% yield [1421].
- *From 1-(2-hydroxyphenyl)-3-phenylisobenzofuran* (SM2):
  - Obtained by self-oxidation of SM2 in chloroformic solution in the presence of air and sunlight during 3 days (quantitative yield) [1436].
  - Also obtained by action of sulfuric acid with SM2 in acetic acid/acetic anhydride for 5 min (51%) [1436].
  - Also obtained by action of N-methylmaleimide with SM2 in methylene chloride at 0° (20%) [1436].
- *From photoxide of 9-methoxy-10-phenylanthracene* (SM3): Obtained by action of concentrated sulfuric acid (d = 1.83) with SM3 in acetone for 24 h at r.t. (25%) [1437].
- *From 3,3-diphenyl-1,2-indanedione* (SM4): Obtained by irradiation of SM4 in the presence of air in benzene during 70 h at r.t. (10%) [86,1438].

- *From 10-phenylanthrone* (SM5): Obtained by irradiation of SM5 in the presence of air in benzene for 2.5 h at r.t. (20%) [1438], (30%) [86].
- *From 10-phenylanthronyl hydroperoxide* (SM6): Obtained by photochemical degradation of SM6 in benzene for 5.5 h (29%) [1438]. Also obtained by chemical degradation of SM6 with sulfuric acid in acetone for 1 to 1.5 h (21%) [1438].
- *From photoxide of 1,3-diphenylisobenzofuran* (SM7): Obtained by isomerization of SM7 in acetic acid solution [1439].

m.p. 139–140° [86,1421,1436,1438], 138–139° [1437];
^{1}H NMR [1437], IR [86,1437,1438], UV [1438].

(2-Benzoylphenyl)(3-hydroxyphenyl)methanone

[57436-75-6] $C_{20}H_{14}O_3$ mol.wt. 302.33



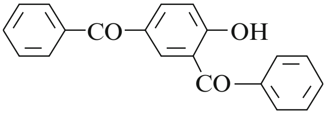
Synthesis

- Obtained by isomerization of photoxide of 1,3-diphenylisobenzofuran, via an arene oxide formation, in acetic acid solution [1439].

m.p. 135–137° [1439]; Spectra (NA).

(4-Hydroxy-1,3-phenylene)bis[phenylmethanone

[2589-81-3] $C_{20}H_{14}O_3$ mol.wt. 302.33



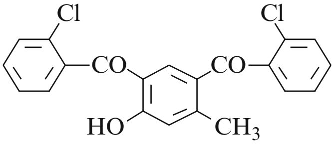
Syntheses

- Preparation by Friedel–Crafts acylation,
  - of phenol with benzoyl chloride in the presence of aluminium chloride at 200° for 15 min (69%) [24];
  - of 4-hydroxybenzophenone with benzoyl chloride in the presence of aluminium chloride from 100° up to 150° [145].
- Also obtained by Fries rearrangement of 4-benzoyloxybenzophenone with aluminium chloride at 160–165° for 1 h [1440].

m.p. 105–106° [1440], 105° [145], 101° [24], 98° [96];
1H NMR (Sadtler: standard n° 20032 M) [99],
IR (Sadtler: standard n° 47041) [24], UV [24,99,109];
pK_a [96]; TLC [116].

(4-Hydroxy-6-methyl-1,3-phenylene)bis[(2-chlorophenyl)methanone

[147167-72-4] $C_{21}H_{14}Cl_2O_3$ mol.wt. 385.25



Syntheses

- Preparation by reaction of o-chlorobenzoic with 2′-chloro-2-hydroxy-4-methylbenzophenone in the presence of phosphoric acid and phosphorous pentoxide at 70° for 20 h (76%) [1089].

- Also obtained by reaction of o-chlorobenzoic acid with m-cresol in the presence of phosphoric acid and phosphorous pentoxide at 130° for 2 h (15%) [1089].
- Also obtained by Fries rearrangement of m-cresyl o-chlorobenzoate with phosphoric acid and phosphorous pentoxide at 130° for 1 h (16%) [1089].
- Also obtained by isomerization of 2′-chloro-4-hydroxy-2-methylbenzophenone in the presence of phosphoric acid and phosphorous pentoxide at 130° for 4 h (15%) [1089].

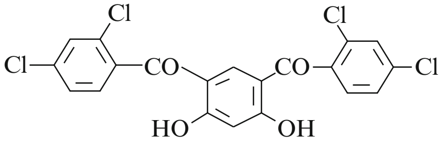
m.p. 105–107° [1089]; ^{1}H NMR [1089], IR [1089].

6.2 Di- and Polyhydroxylated Ketones

6.2.1 Symmetrical Ketones

(4,6-Dihydroxy-1,3-phenylene)bis[(2,4-dichlorophenyl)methanone

[13340-61-9] $C_{20}H_{10}Cl_4O_4$ mol.wt. 456.11



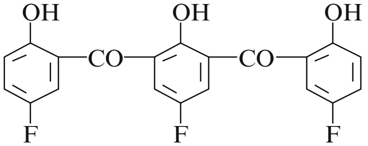
Synthesis

- Preparation by Friedel–Crafts acylation of resorcinol with 2,4-dichlorobenzoyl chloride in the presence of trifluoromethanesulfonic acid and methanesulfonic acid, via a Fries rearrangement,
  - without solvent, at 180° for 3 h (92%) [1441];
  - in o-dichlorobenzene at 100° for 12 h (76%) [1441].
- Also refer to: [1442].

m.p. 209° [1441]; ^{13}C NMR [1441].

(5-Fluoro-2-hydroxy-1,3-phenylene)bis[(5-fluoro-2-hydroxyphenyl) methanone

[78563-16-3] $C_{20}H_{11}F_3O_5$ mol.wt. 388.30



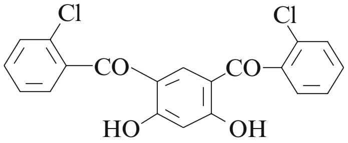
Synthesis

- Preparation by treatment of the corresponding triacetate (SM) with 15% aqueous sodium hydroxide solution (90%). SM was obtained by oxidation of the methylene groups in 1-acetoxy-2,6-bis (2′-acetoxy-5′-fluoro-α-tolyl)-4-fluorobenzene to carbonyl groups by means of chromium trioxide in acetic anhydride (97%) [1443].

m.p. 154–155° [1443]; IR [1443], MS [1443].

(4,6-Dihydroxy-1,3-phenylene)bis[(2-chlorophenyl)methanone

[152383-56-7] $C_{20}H_{12}Cl_2O_4$ mol.wt. 387.22



Synthesis

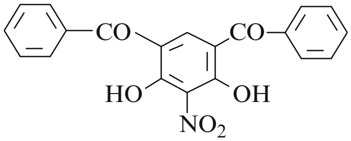
– Preparation by reaction of o-chlorobenzoic acid with resorcinol diacetate in the presence of hexafluoro-isopropylsulfonic acid in methanesulfonic acid first at 160° for 1 h, then at 180° for 12 h [1441].

– Also refer to: [1442].

m.p. (NA); ^{13}C NMR [1441].

(4,6-Dihydroxy-5-nitro-1,3-phenylene)bis[phenylmethanone

[102160-16-7] $C_{20}H_{13}NO_6$ mol.wt. 363.33



Syntheses

– Obtained by condensation of benzoyl chloride with 2-nitroresorcinol in the presence of aluminium chloride in nitrobenzene at 100° [1196,1207].

– Preparation by Fries rearrangement of 2-nitro-resorcinol dibenzoate with aluminium chloride in nitrobenzene at 100–110° for 3 h (40%) [1207].

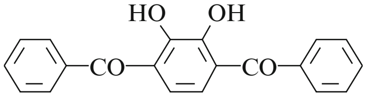
– Preparation by nitration of 4,6-dibenzoylresorcinol with nitric acid in a sulfuric acid and acetic acid solution at 0° for 1 h [1207].

– Also refer to: [203].

m.p. 215–216° [1196], 215° [1207]; Spectra (NA).

(2,3-Dihydroxy-1,4-phenylene)bis[phenylmethanone

[31709-42-9] $C_{20}H_{14}O_4$ mol.wt. 318.33



Syntheses

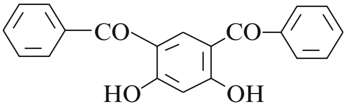
– Obtained by hydrogenolysis of 3,6-diphenyl-benzo[1,2-d:4,3-d′]diisoxazole (SM) (m.p. 206–207°) in the presence of Raney nickel at r.t., and treating the intermediate with 20% sulfuric acid (21%) [1444]. SM was prepared through a four-steps synthesis: first, reaction of benzonitrile oxide with 1,3-cyclohexadiene in boiling ethyl ether. Second, bromination of the 5,10-diphenyl-3,12-dioxa-4,11-diazatricyclo[7,3,0,0 ^{2,6}]dodeca- 4,10-diene (m.p. 186–187°) obtained with N-bromosuccinimide in carbon tetrachloride. After which, dehydrohalogenation of the intermediate compound in boiling triethylamine and aromatization with N-bromosuccinimide in carbon tetrachloride.

– Preparation by hydrolysis of 2,3-dibenzoyloxy-1,4-dibenzoylbenzene (SM) in concentrated sulfuric acid at r.t. for 15 min (75%) [1445]. SM (m.p. 144°) was obtained by oxidation of 2,3,6,7-tetraphenylbenzo[2,1-b:3,4-b′]difuran (o-benzotetraphenyldifurfuran) with chromium trioxide in boiling acetic acid for 75 min.

m.p. 159°5 [1445], 156–158° [1444]; IR [1444].

(4,6-Dihydroxy-1,3-phenylene)bis[phenylmethanone

[3088-15-1]



$C_{20}H_{14}O_4$ mol.wt. 318.33

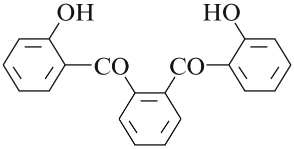
Syntheses

- Preparation by saponification of 4,6-di-(benzoyloxy)-1,3-dibenzoylbenzene (SM) with potassium hydroxide in refluxing ethanol [203,210]. The keto ester SM was obtained by acylation of resorcinol dibenzoate with benzoyl chloride in the presence of zinc chloride at 100–120° during several days [210].
- Preparation by Fries rearrangement of resorcinol dibenzoate,
  - with aluminium chloride [1207];
  - with zinc chloride (25%) [1446].
- Also obtained by photo-Fries rearrangement of resorcinol dibenzoate in benzene for 8 h under nitrogen (15%) [843].
- Also refer to: [226,230,1447].

oily liquid [843]; b.p. (NA). This product is impure or in a metastable state.
m.p. 149–150° [203], 149° [210], 145° [113];
^{1}H NMR [843], UV [113].

1,2-Phenylenebis[(2-hydroxyphenyl)methanone

[119838-11-8]



$C_{20}H_{14}O_4$ mol.wt. 318.33

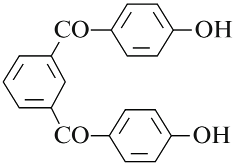
Synthesis

- Preparation by demethylation of bis(2-methoxybenzoyl)-benzene with 47% hydrobromic acid at reflux for 5 h (95%) [1448].

m.p. 160–161° [1448];
^{1}H NMR [1448], IR [1448], MS [1448].

1,3-Phenylenebis[(4-hydroxyphenyl)methanone

[5436-05-5]



$C_{20}H_{14}O_4$ mol.wt. 318.33

Syntheses

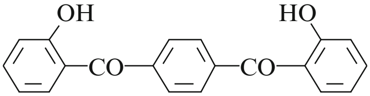
- Preparation by demethylation of 1,3-di-(p-methoxy-benzoyl)- benzene (SM) with hydrobromic acid (d = 1.49) in refluxing acetic acid for 6 h (68%) [1449] or for 10 h [1450]. SM was obtained by Friedel–Crafts acylation of anisole with isophthaloyl chloride [1450].
- Preparation by Friedel–Crafts acylation of anisole with isophthaloyl chloride in the presence of aluminium chloride in boiling carbon disulfide (major product) [1449], (<75%) [1451].

- Preparation by Fries rearrangement of diphenyl isophthalate with aluminium chloride at 185–195° for 25 min [1451].
- Obtained by action of isophthalic acid with phenol in the presence of trifluoromethanesulfonic acid, first at 50° for 23 h, then at 70° for 4 h (50%) [151].
- Also refer to: [956,1452–1455].

m.p. 215° [1449], 207–209° [1451], 194° [151]. There is discrepancy between the melting points.
^{1}H NMR [151], IR [151].

1,4-Phenylenebis[(2-hydroxyphenyl)methanone

[66832-95-9] $C_{20}H_{14}O_4$ mol.wt. 318.33



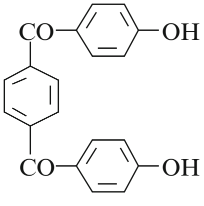
Synthesis

- Preparation by total demethylation of 1,4-bis (2-anisoyl)-benzene. This one was obtained by condensation of 2-anisoyl magnesium bromide with terephthalonitrile [1456].
- Also refer to: [1457].

m.p. and Spectra (NA).

1,4-Phenylenebis[(4-hydroxyphenyl)methanone

[15517-46-1] $C_{20}H_{14}O_4$ mol.wt. 318.33



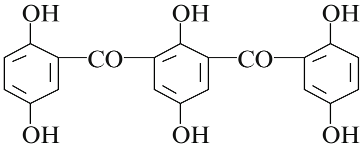
Syntheses

- Obtained by Fries rearrangement of phenyl terephthalate (diester) with aluminium chloride at 185–195° for 25 min [1451].
- Preparation by Friedel–Crafts acylation of anisole (2 mol) with terephthaloyl chloride (1 mol) in the presence of aluminium chloride in refluxing carbon disulfide for 1 h [1451].
- Preparation by reaction of terephthalic acid with phenol in the presence of trifluoromethanesulfonic acid at 50° for 23 h, then at 70° for 4 h (34%) [151].
- Also refer to: Chem. Abstr., **101**, 15450z (1984).

m.p. 298–299° [1451], 292° [151];
^{1}H NMR [151], IR [151].

(2,5-Dihydroxy-1,3-phenylene)bis[(2,5-dihydroxyphenyl)methanone

[78563-18-5] $C_{20}H_{14}O_8$ mol.wt. 382.33



Synthesis

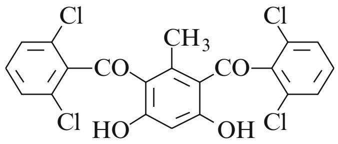
- Preparation by saponification of the corresponding hexaacetate (SM) with 6 N sodium hydroxide in methanol or with 1%

sodium hydroxide in aqueous solution (15% yield). SM was obtained by oxidation of the methylene groups of 1,4-diacetoxy-2,6-bis (2′,5′-diacetoxy-α-tolyl)benzene to carbonyl groups by means of chromium trioxide in acetic anhydride (80% yield) [1443].

m.p. >300° [1443]; Spectra (NA).

(4,6-Dihydroxy-2-methyl-1,3-phenylene)bis[(2,6-dichlorophenyl)methanone

[152383-58-9]



$C_{21}H_{12}Cl_4O_4$ mol.wt. 470.13

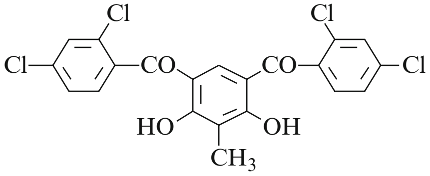
Synthesis

- Refer to: [1442] (compound **6**).

m.p. and Spectra (NA).

(4,6-Dihydroxy-5-methyl-1,3-phenylene)bis[(2,4-dichlorophenyl)methanone

[153167-54-5]



$C_{21}H_{12}Cl_4O_4$ mol.wt. 470.13

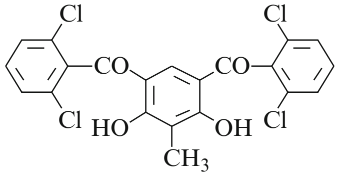
Synthesis

- Preparation by Fries rearrangement of 2-methylresorcinol 2,4-dichlorobenzoate (diester) with fluoroalcanesulfonic acid and alcanesulfonic acid mixture [1441].

m.p. (NA); ^{13}C NMR [1441].

(4,6-Dihydroxy-5-methyl-1,3-phenylene)bis[(2,6-dichlorophenyl)methanone

[153167-55-6]



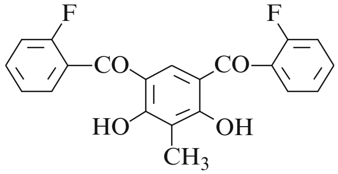
$C_{21}H_{12}Cl_4O_4$ mol.wt. 470.13

Synthesis

- Preparation by Fries rearrangement of 2-methyl-resorcinol 2,6-dichlorobenzoate (diester) with trifluoromethanesulfonic acid or hexafluoro-isopropanesulfonic acid in methanesulfonic acid [1441].

m.p. (NA); ^{13}C NMR [1441].

(4,6-Dihydroxy-5-methyl-1,3-phenylene)bis[(2-fluorophenyl)methanone



$C_{21}H_{14}F_2O_4$ mol.wt. 368.34

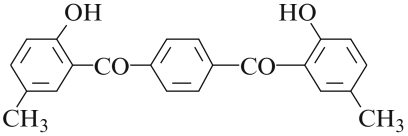
Synthesis

- Refer to: [1442] (compound **3**).

m.p. and Spectra (NA).

1,4-Phenylenebis[(2-hydroxy-5-methylphenyl)methanone

[4084-62-2]



$C_{22}H_{18}O_4$ mol.wt. 346.38

Syntheses

– Preparation by Fries rearrangement of p-tolyl terephthalate (diester) in the presence of aluminium chloride,

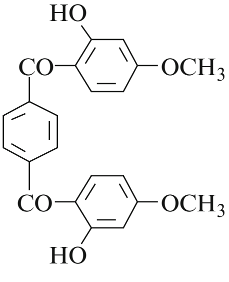
- *without solvent*
  at 210° (90%) [1458] or at 220–230° for 15 min, then a short time at 250° (27%) [1459],
- *with solvent*
  in chlorobenzene at 132° for 5 h (31%) [1460] or at reflux for 4 h (68%) [1457] or for 38 h (25%) [1461]. The diester was obtained by heating p-cresol and terephthaloyl chloride at 200° for 90 min [1457]. in nitrobenzene at 100° for 3 h [1462].

– Also obtained by photo-Fries rearrangement of p-tolyl terephthalate (diester) in benzene for 20 h under nitrogen (10%) [1459].

– Preparation by reaction of 1,4-bis(trichloromethyl)benzene with p-cresol in the presence of 20% or 30% sodium hydroxide, first at 50°, then 95° for 1 h (69% and 77% yields, respectively) [1457].

m.p. 190–190°5 [1461], 187–189° [1459], 165–168° [1457]. There is discrepancy between the melting points. IR [1457,1461], UV [1457].

1,4-Phenylenebis[(2-hydroxy-4-methoxyphenyl)methanone



$C_{22}H_{18}O_6$ mol.wt. 378.38

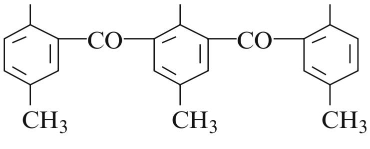
Synthesis

– Preparation by reaction of 1,4-bis(trichloromethyl) benzene with resorcinol monomethyl ether in the presence of 30% sodium hydroxide, first at 70°, then at 95° for 1 h (71%) [1457].
glassy brown compound [1457];

m.p. (NA); IR [1457], UV [1457].

(2-Hydroxy-5-methyl-1,3-phenylene)bis[(2-hydroxy-5-methylphenyl) methanone

[27404-61-1]



$C_{23}H_{20}O_5$ mol.wt. 376.41

Synthesis

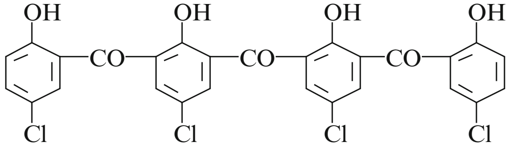
– Preparation by Friedel–Crafts acylation of 4-methoxytoluene with 2-methoxy-5-methyl-benzene-1,3-dicarbonyl dichloride in carbon disulfide in the presence of aluminium chloride, first at r.t. for overnight, then at reflux for 4 h (40%). The same reaction, carried out in nitrobenzene at 120° for 2 h, gave a 37% yield [1153].

- Also refer to: [431] (compound VII).

m.p. 149° [1153], 122–123° [1329]. There is discrepancy between the two melting points.
MS [1329].

Bis[5-chloro-3-(5-chloro-2-hydroxybenzoyl)-2-hydroxyphenyl]methanone

[78563-33-4] $C_{27}H_{14}Cl_4O_7$ mol.wt. 592.21



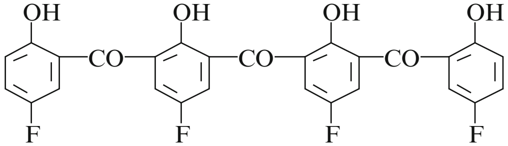
Synthesis

- Preparation by saponification of the corresponding tetraacetate with 15% aqueous sodium hydroxide solution at 40° for 1 h (90%) [1443].

m.p. 224° [1443]; MS [1443].

Bis[5-fluoro-3-(5-fluoro-2-hydroxybenzoyl)-2-hydroxyphenyl]methanone

[78563-35-6] $C_{27}H_{14}F_4O_7$ mol.wt. 526.40



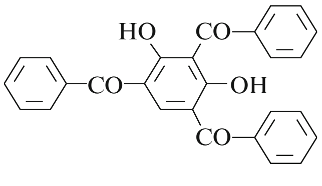
Synthesis

- Preparation by saponification of the corresponding tetraacetate with 15% aqueous sodium hydroxide solution at 40° for 1 h (95%) [1443].

m.p. 165–167° [1443]; IR [1443], UV [1443], MS [1443].

(2,4-Dihydroxy-1,3,5-benzenetriyl)tris[phenylmethanone

[82-67-7] $C_{27}H_{18}O_5$ mol.wt. 422.44



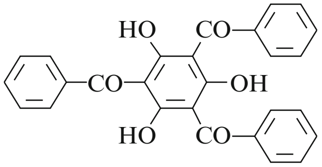
Synthesis

- Obtained by action of benzoyl chloride with resorcinol dibenzoate in the presence of aluminium chloride at 100° for 8 h, then at 150° [145].

m.p. 185° [113,145]; UV [113].

(2,4,6-Trihydroxy-1,3,5-benzenetriyl)tris[phenylmethanone

[1818-24-2] $C_{27}H_{18}O_6$ mol.wt. 438.44



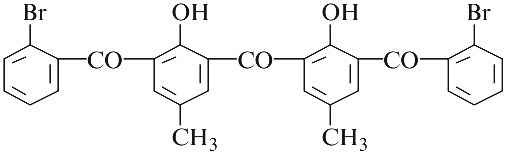
Syntheses

- Obtained by Fries rearrangement of phloroglucinol tribenzoate with aluminium chloride at 130–140° for 30 min (30%) [271].
- Also obtained by action of benzoyl chloride with phloro-glucinol in the presence of boron trifluoride at 50° (72–78%) [1463].

m.p. 185° [271], 134–136° [1463]. There is discrepancy between the two melting points.
Spectra (NA).

Bis[3-(2-bromobenzoyl)-2-hydroxy-5-methylphenyl]methanone

$C_{29}H_{20}Br_2O_5$ mol.wt. 608.28



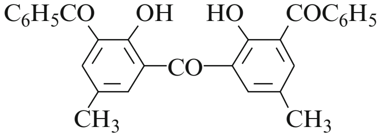
Synthesis

- Preparation by Fries rearrangement of 2,2′-di-(o-bromobenzoyloxy)-5,5′-dimethylbenzophenone with aluminium chloride without solvent for 30 min at 160° (50%) [1153].

m.p. 170° [1153]; Spectra (NA).

Bis(3-benzoyl-2-hydroxy-5-methylphenyl)methanone

$C_{29}H_{22}O_5$ mol.wt. 450.49



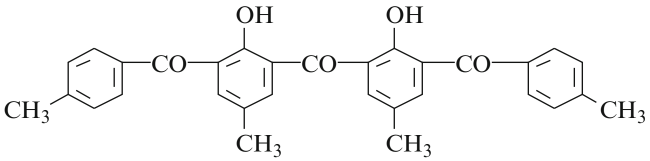
Synthesis

- Preparation by Fries rearrangement of 2, 2′-di-(benzoyloxy)-5,5′-dimethylbenzophenone with aluminium chloride without solvent at 160° for 30 min (50%) [1153].

m.p. 204° [1153]; Spectra (NA).

Bis[3-(4-methylbenzoyl)-2-hydroxy-5-methylphenyl]methanone

$C_{31}H_{26}O_5$ mol.wt. 478.54

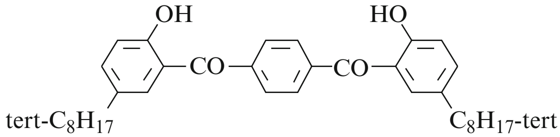


Synthesis

- Preparation by Fries rearrangement of 2,2′-di-(p-toluoyloxy)-5, 5′-dimethyl-benzophenone with aluminium chloride without solvent for 30 min at 160° (81%) [1153].

m.p. 184–185° [1153]; Spectra (NA).

1,4-Phenylenebis[2-hydroxy-5-(1,1,3,3-tetramethylbutyl)phenyl]methanone



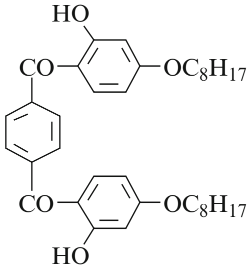
$C_{36}H_{46}O_4$ mol.wt. 542.76

Syntheses

- Preparation by reaction of 1,4-bis-(trichloromethyl)benzene with p-(1,1,3,3-tetramethylbutyl)phenol in the presence of 30% sodium hydroxide, first at 70°, then at 95° for 1 h (71%) [1457].
- Preparation by reaction of terephthaloyl chloride with p-(1,1,3,3-tetramethylbutyl)phenol in ethylene dichloride in the presence of aluminium chloride, first at 0°, then between 5° and 10° for 7 h (58%) [1457].

glassy brown compound [1457]; m.p. (NA);
IR [1457], UV [1457].

1,4-Phenylenebis[2-hydroxy-4-(octyloxy)phenyl]methanone



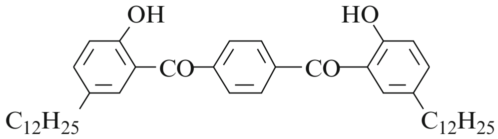
$C_{36}H_{46}O_6$ mol.wt. 574.76

Synthesis

- Preparation by reaction of 1,4-bis(trichloromethyl) benzene with resorcinol monooctyl ether in the presence of 30% sodium hydroxide, first at 70°, then at 95° for 1 h (74%) [1457].

glassy light brown compound [1457]; m.p. (NA);
IR [1457], UV [1457].

1,4-Phenylenebis[(2-hydroxy-5-dodecylphenyl)methanone



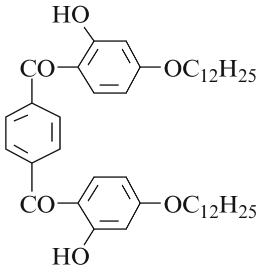
$C_{44}H_{62}O_4$ mol.wt. 654.97

Synthesis

- Preparation by reaction of 1,4-bis-(trichloromethyl) benzene with 4-do-decylphenol in the presence of 30% sodium hydroxide, first at 70°, then at 95° for 1 h (68%) [1457].

glassy light brown compound [1457]; m.p. (NA);
IR [1457], UV [1457].

1,4-Phenylenebis[2-hydroxy-4-(dodecyloxy)phenyl]methanone



$C_{44}H_{62}O_6$ mol.wt. 686.97

Synthesis

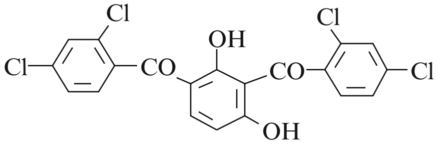
- Preparation by reaction of 1,4-bis(trichloromethyl) benzene with resorcinol monododecyl ether in the presence of 30% sodium hydroxide, first at 70°, then at 95° for 1 h (69%) [1457].

glassy dark brown compound [1457];
m.p. (NA);
IR [1457], UV [1457].

6.2.2 Asymmetric Ketones

(2,4-Dihydroxy-1,3-phenylene)bis[(2,4-dichlorophenyl)methanone

[153167-57-8]



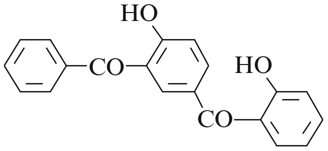
$C_{20}H_{10}Cl_4O_4$ mol.wt. 456.11

Synthesis

- Preparation by reaction of 2,4-dichloro-benzoyl chloride with resorcinol in the presence of trifluoromethanesulfonic acid in methanesulfonic acid, at 180° for 3 h (by-product) or in o-dichlorobenzene at 100° for 12 h (24%) [1441].
- Also refer to: [1442] (compound **4**).

m.p. (NA); ^{13}C NMR [1441].

(3-Benzoyl-4-hydroxyphenyl)(2-hydroxyphenyl)methanone



$C_{20}H_{14}O_4$ mol.wt. 318.33

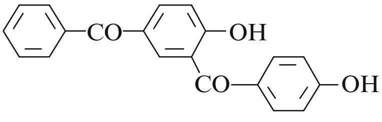
Synthesis

- Obtained (by-product) by heating a mixture of o-anisoyl chloride, benzene and aluminium chloride between 79° and 90° for 2.25 h (14%) [12].

m.p. 127°9–130°1 [12]; Spectra (NA).

(5-Benzoyl-2-hydroxyphenyl)(4-hydroxyphenyl)methanone

[124208-66-8] $C_{20}H_{14}O_4$ mol.wt. 318.33



Syntheses

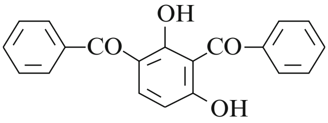
– Obtained by Fries rearrangement of phenyl 5-benzoyl-2-hydroxybenzoate with aluminium chloride at 180° for 3 h (20%); the same reaction, in the presence of phenol, gave a 36% yield [155].

– Also obtained by Fries rearrangement of phenyl 2-benzoyloxybenzoate with aluminium chloride at 180° for 3 h (10%). The same reaction, in the presence of phenol, gave a 21% yield [155].

m.p. 162° [155]; Spectra (NA).

(2,4-Dihydroxy-1,3-phenylene)bis[phenylmethanone

[82-64-4] $C_{20}H_{14}O_4$ mol.wt. 318.33



Syntheses

– Obtained by reaction of chromium trioxide with angular meta benzotetraphenyldifurfuran in boiling acetic acid for 1 h, followed by saponification of the non-isolated keto ester formed with 10% sodium hydroxide in refluxing ethanol for 1.5 h [203].

– Also obtained on decarboxylation of 2,4-dihydroxy-3,5-dibenzoylbenzoic acid [208].

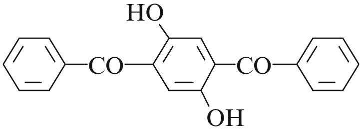
– Also obtained by photo-Fries rearrangement of resorcinol dibenzoate in benzene for 8 h under nitrogen (10%) [843].

– Also refer to: [1464,1465] (Japanese patents).

oil [843]. This product is impure or in a metastable state.
m.p. 105° [113], 103–104° [203], 102° [208];
^{1}H NMR [98,843], UV [113,241,242].

(2,5-Dihydroxy-1,4-phenylene)bis[phenylmethanone

[97971-75-0] $C_{20}H_{14}O_4$ mol.wt. 318.33



Syntheses

– Preparation from lin-parabenzotetraphenyldifurfuran by oxidation with chromium trioxide in refluxing acetic anhydride for >1 h, followed by saponification of the 2,5-dibenzoylhydroquinone dibenzoate so formed with potassium hydroxide in ethanol in a water bath for 30 min (43%) [254].

– Also obtained by reaction of benzoyl chloride with hydroquinone in the presence of aluminium chloride at 190–200° [146,169,1466] or at 200–205° [256]

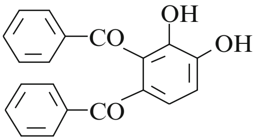
for 2 days, followed by saponification of the keto ester so formed (2,5-dibenzoylhydroquinone dibenzoate) with potassium hydroxide in ethanol (2%) [1466], (12–15%) [256].

- Also obtained by photo-Fries rearrangement of hydroquinone dibenzoate in benzene for 8 h under nitrogen (10%) [843].

m.p. 210°5–211°1 [256], 207° [146,1466], 203° [254], 172–173° [843]. There is discrepancy between the melting points.
^{1}H NMR [843].

(3,4-Dihydroxy-1,2-phenylene)bis[phenylmethanone

[97971-73-8] $C_{20}H_{14}O_4$ mol.wt. 318.33



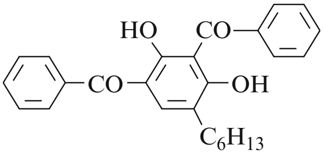
Synthesis

- Obtained by photo-Fries rearrangement of 1,2-di-(benzoyloxy)benzene in benzene for 8 h under nitrogen (28%) [843].

m.p. 134–136° [843]; ^{1}H NMR [843].

(2,4-Dihydroxy-5-hexyl-1,3-phenylene)bis[phenylmethanone

$C_{26}H_{26}O_4$ mol.wt. 402.49



Synthesis

- Preparation by reaction of benzoyl chloride with 4-hexylresorcinol in the presence of aluminium chloride in nitrobenzene at 80–90° for 3 h (52%) [113].

m.p. 68–69° [113]; Spectra (NA).

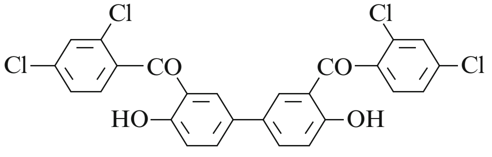
Part III
Miscellaneous Related Compounds (*Class of METHANONES*)

Chapter 7
Miscellaneous Related Compounds

7.1 Diphenyl Derivatives

(4,4′-Dihydroxy[1,1′-biphenyl]-3,3′-diyl)bis[(2,4-dichorophenyl)methanone

[152383-57-8] $C_{26}H_{14}Cl_4O_4$ mol.wt. 532.21



Synthesis

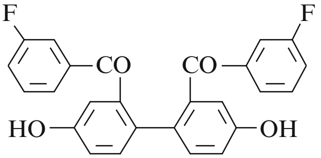
– Preparation by Fries rearrangement of 4,4′-dihydroxybiphenyl di-(2,4-di-chlorobenzoate) in the presence of tri-fluoromethanesulfonic acid and ethane-sulfonic acid in refluxing dichlorobenzene for 16 h (94%) [1441].

N.B.: Di-Na salt [1442].

m.p. (NA); ^{13}C NMR [1441].

(4,4′-Dihydroxy[1,1′-biphenyl]-2,2′-diyl)bis[(3-fluorophenyl)methanone

[176548-03-1] $C_{26}H_{16}F_2O_4$ mol.wt. 430.41



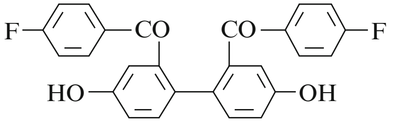
Synthesis

– Preparation by demethylation of 2,2′-bis-(m-fluoro-benzoyl)-4,4′-dimethoxybiphenyl with boron tribromide in methylene chloride, first at –70°, then at 25° for 8 h (80%) [258].

m.p. 239–241° [258]; ^{1}H NMR [258].

(4,4′-Dihydroxy[1,1′-biphenyl]-2,2′-diyl)bis[(4-fluorophenyl)methanone

[162658-02-8] $C_{26}H_{16}F_2O_4$ mol.wt. 430.41



Synthesis

– Preparation by total demethylation of 2,2′-bis(p-fluorobenzoyl)-4,4′-di-methoxybiphenyl with boron

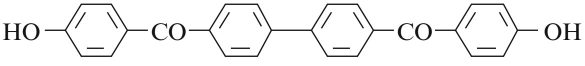
R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_7, © Springer Science+Business Media B.V. 2011

tribromide in methylene chloride under nitrogen first at −70°, then at 25° for 8 h (94%) [258,680].

m.p. 256–258° [680]; ^{1}H NMR [680], ^{13}C NMR [680], MS [680].

[1,1′-Biphenyl]-4,4′-diylbis[(4-hydroxyphenyl)methanone

[106647-50-1] $C_{26}H_{18}O_4$ mol.wt. 394.43



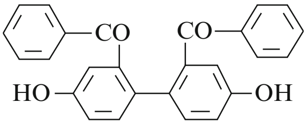
Synthesis

– Preparation by Fries rearrangement of 4,4′-bis-(phenoxycarbonyl)diphenyl in hydrofluoric acid between −10° and 0° for 20 h (96%) [1467].

m.p. and Spectra (NA).

(4,4′-Dihydroxy[1,1′-biphenyl]-2,2′-diyl)bis[phenylmethanone

[162658-01-7] $C_{26}H_{18}O_4$ mol.wt. 394.43



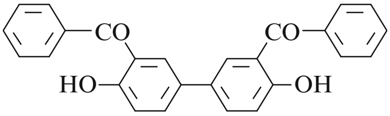
Synthesis

– Preparation by total demethylation of 2,2′-dibenzoyl-4,4′-dimethoxybiphenyl with boron tribromide in methylene chloride under nitrogen first at −70°, then at 25° for 8 h (83%) [258,680].

m.p. (NA); ^{1}H NMR [680].

(4,4′-Dihydroxy[1,1′-biphenyl]-3,3′-diyl)bis[phenylmethanone

[71182-85-9] $C_{26}H_{18}O_4$ mol.wt. 394.43



Synthesis

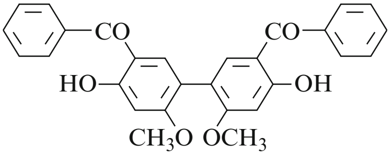
– Preparation by Fries rearrangement of 4,4′-bi-phenyldiyl dibenzoate,

- with aluminium chloride in o-dichlorobenzene during 3 h at 180° (70%) [1408] or in refluxing chlorobenzene for 3 days (30%) [1468];
- with aluminium chloride and sodium chloride mixture, first at 140°, then at 200° for 20 min (51%) [1469].

m.p. 187°5–189°5 [1408], 184–185° [1468];
^{1}H NMR [1408], IR [1408], UV [1408].

(4,4′-Dihydroxy-6,6′-dimethoxy[1,1′-biphenyl]-3,3′-diyl)bis[phenylmethanone

[42045-62-5] $C_{28}H_{22}O_6$ mol.wt. 454.48



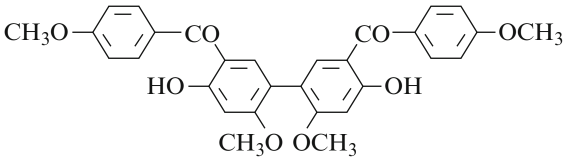
Synthesis

– Obtained by reaction of 2-hydroxy-4-methoxy-benzophenone with lead tetraacetate in acetic acid at 100° for 5 h (15%). The same reaction using manganic acetate gave 7% yield [650].

m.p. 199–200° [650];
^{1}H NMR [650], IR [650], UV [650].

(4,4′-Dihydroxy-6,6′-dimethoxy[1,1′-biphenyl]-3,3′-diyl)bis[(4-methoxyphenyl)-methanone

[42045-61-4] $C_{30}H_{26}O_8$ mol.wt. 514.53



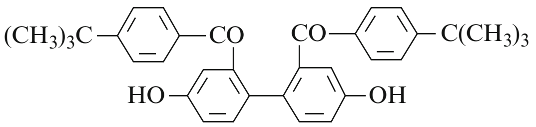
Synthesis

- Obtained by reaction of 2-hydroxy-4,4′-dimethoxybenzophenone with lead tetraacetate in acetic acid at 100° for 5 h (21%) [1082].

m.p. 232–234° [1082]; ^{1}H NMR [1082], IR [1082], UV [1082].

(4,4′-Dihydroxy[1,1′-biphenyl]-2,2′-diyl)bis[4-(1,1-dimethylethyl)phenyl] methanone

[162658-03-9] $C_{34}H_{34}O_4$ mol.wt. 506.64



Synthesis

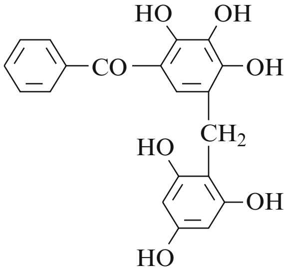
- Preparation by total demethylation of 2,2′-bis(p-tert-butylbenzoyl)-4,4′-dimethoxybiphenyl with boron tribromide in methylene chloride under nitrogen first at −70°, then at 25° for 8 h (77%) [258,680].

m.p. 236–238° [680]; ^{1}H NMR [680], ^{13}C NMR [680], MS [680].

7.2 Diphenylmethane Derivatives

Phenyl[2,3,4-trihydroxy-5-[(2,4,6-trihydroxyphenyl)methyl]phenyl]methanone

[138250-29-0] $C_{20}H_{16}O_7$ mol.wt. 368.34

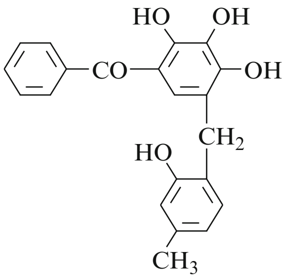


Synthesis

- Refer to: [1355–1357].

m.p. and Spectra (NA).

Phenyl[2,3,4-trihydroxy-5-[(2-hydroxy-4-methylphenyl)methyl]phenyl] methanone



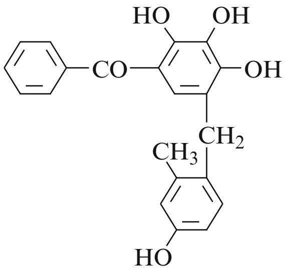
$C_{21}H_{18}O_5$ mol.wt. 350.37

Synthesis

- Refer to: [1356].

m.p. and Spectra (NA).

Phenyl[2,3,4-trihydroxy-5-[(4-hydroxy-2-methylphenyl)methyl]phenyl]methanone



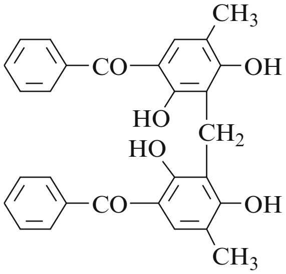
$C_{21}H_{18}O_5$ mol.wt. 350.37

Synthesis

- Refer to: [1356].

m.p. and Spectra (NA).

[Methylenebis(2,4-dihydroxy-5-methyl-3,1-phenylene)]bis[phenylmethanone



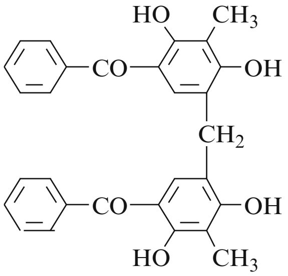
$C_{29}H_{24}O_6$ mol.wt. 468.51

Synthesis

- Preparation by reaction of 40% aqueous formaldehyde solution with 2,4-dihydroxy-5-methylbenzophenone in ethanol in the presence of concentrated sulfuric acid for 24 h at r.t. (73%) [805].

m.p. 240° [805]; Spectra (NA).

[Methylenebis(4,6-dihydroxy-5-methyl-3,1-phenylene)]bis[phenylmethanone



$C_{29}H_{24}O_6$ mol.wt. 468.51

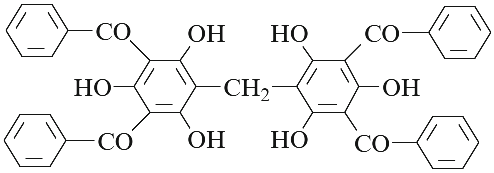
Synthesis

- Preparation by reaction of 40% aqueous formaldehyde solution with 2,4-dihydroxy-3-methylbenzophenone in ethanol in the presence of concentrated sulfuric acid for 3 days at r.t. (68%) [805].

m.p. 207–208° [805]; Spectra (NA).

[Methylenebis(2,4,6-trihydroxy-5,1,3-benzenetriyl)]tetrakis[phenylmethanone

[98149-22-5]



$C_{41}H_{28}O_{10}$ mol.wt. 680.67

Synthesis

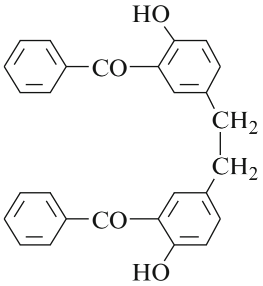
- Preparation by condensation of 2,4-di-benzoylphloroglucinol with 40% aqueous formaldehyde solution (58–64%) [1463].

m.p. 215–217° [1463]; Spectra (NA).

7.3 Diphenylethane Derivative

[1,2-Ethanediylbis(6-hydroxy-3,1-phenylene)]bis[phenylmethanone

[76346-16-2]



$C_{28}H_{22}O_4$ mol.wt. 422.48

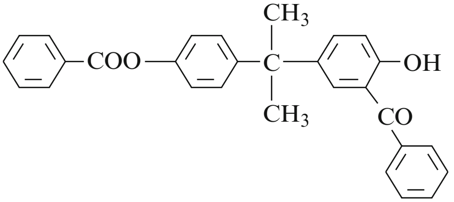
Synthesis

- Obtained (poor yield) by reaction of benzoyl peroxide with 2-hydroxy-5-methylbenzophenone in refluxing chloroform for 6 h (<4%) [278].

m.p. 172–173° [278]; ^{1}H NMR [278], IR [278], UV [278].

7.4 Diphenylpropane Derivatives

3-Benzoyldiphenylolpropane 4′-monobenzoate



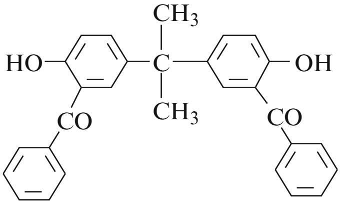
$C_{29}H_{24}O_4$ mol.wt. 436.51

Synthesis

- Obtained by photo-Fries rearrangement of diphenylolpropane dibenzoate (Bisphenol A, dibenzoate) in benzene during 96 h under nitrogen (22%) [1470].

b.p. $_{0.15}$ 220° [1470]; IR [1470], UV [1470].

2,2-Bis(3-benzoyl-4-hydroxyphenyl)propane



$C_{29}H_{24}O_4$ mol.wt. 436.51

Synthesis

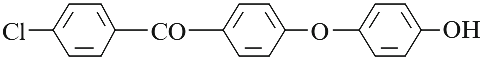
- Obtained by reaction of benzoyl chloride with diphenylolpropane dimethyl ether in the presence of aluminium chloride in ethylene dichloride, first at 0° for 45 min, then at r.t. overnight and at 45° for 90 min (14%) [1470].

m.p. 159°5–160°5 [1470]; IR [1470], UV [1470].

7.5 Diphenyl Oxide Derivatives

(4-Chlorophenyl)[4-(4-hydroxyphenoxy)phenyl]methanone

[86405-16-5]



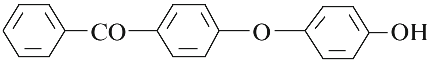
$C_{19}H_{13}ClO_3$ mol.wt. 324.76

Synthesis

- Refer to: Chem. Abstr., **127**, 278690x (1997).

m.p. and Spectra (NA).

[4-(4-Hydroxyphenoxy)phenyl]phenylmethanone



$C_{19}H_{14}O_3$ mol.wt. 290.22

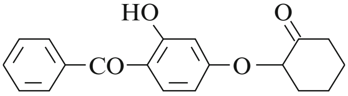
Synthesis

- Preparation by demethylation of 4-(4-methoxyphenoxy)benzophenone (m.p. 104–105°) with aluminium chloride in boiling benzene during some hours [1471].

m.p. 109° [1471]; Spectra (NA).

2-(4-Benzoyl-3-hydroxyphenoxy)cyclohexanone

[125426-75-7] $C_{19}H_{18}O_4$ mol.wt. 310.35



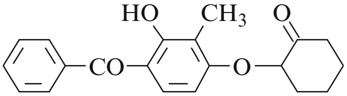
Synthesis

– Preparation by reaction of α-bromocyclohexanone with resbenzophenone in the presence of potassium carbonate in refluxing acetone for 6 h (70%) [221].

m.p. 132–133° [221]; ^{1}H NMR [221].

2-(4-Benzoyl-3-hydroxy-2-methylphenoxy)cyclohexanone

[125426-85-9] $C_{20}H_{20}O_4$ mol.wt. 324.38



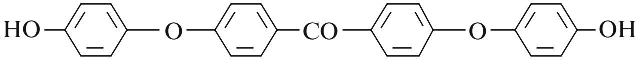
Synthesis

– Preparation by reaction of α-bromocyclohexanone with 2,4-dihydroxy-3-methylbenzophenone in the presence of potassium carbonate in refluxing acetone for 6 h [221].

m.p. 121–122° [221]; ^{1}H NMR [221].

Bis[4-(4-hydroxyphenoxy)phenyl]methanone

$C_{25}H_{18}O_5$ mol.wt. 398.41



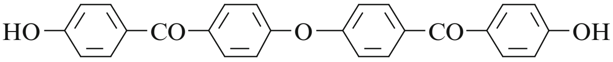
Synthesis

– Preparation by demethylation of 4,4′-(4-methoxyphenoxy)benzophenone (m.p. 198–199°) with excess aluminium chloride in boiling benzene (30%) [1471].

m.p. 214° [1471]; Spectra (NA).

(Oxydi-4,1-phenylene)bis(4-hydroxyphenyl)methanone

[86432-12-4] $C_{26}H_{18}O_5$ mol.wt. 258.23



Syntheses

– Obtained by reaction of 4,4′-Oxybis(benzoic acid) with phenol in the presence of trifluoromethanesulfonic acid at r.t. for 6 days [151].
– Preparation by Fries rearrangement of 4,4′-bis(phenoxycarbonyl)diphenyl ether,

  - with methanesulfonic acid at 120° for 2 h (70%) [1472];
  - with trifluoromethanesulfonic acid (high yield) [922];
  - with hydrofluoric acid at −10° to 0° for 20 h (96%) [1473].

– Also obtained by reaction of EKONOL$^{(TM)}$, an aromatic polyester as Friedel–Crafts reagent, with diphenyl ether in triflic acid solution at 25° for 18 h (98%) [922].

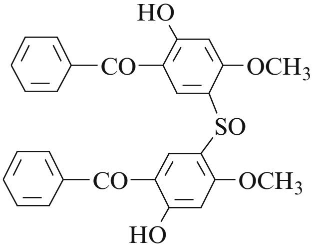
Similar results can be obtained using hydrofluoric acid/boron trifluoride or aluminium chloride in place of triflic acid [922].

m.p. (NA); ^{1}H NMR [151], ^{13}C NMR [922], MS [922]; HPLC [922].

7.6 Diphenyl Sulfoxide Derivatives

[Sulfinylbis(6-hydroxy-4-methoxy-3,1-phenylene)]bis[phenylmethanone

[35839-46-4]



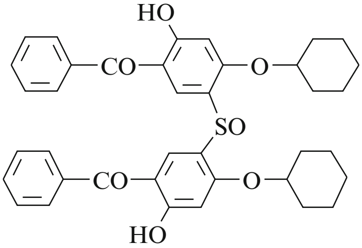
$C_{28}H_{22}O_7S$ mol.wt. 502.54

Synthesis

- Obtained by action of thionyl chloride with 2-hydroxy-4-methoxybenzophenone in the presence of aluminium chloride in nitrobenzene at r.t. for 24 h [1474].

m.p. 202–203° [1474]; Spectra (NA).

[Sulfinylbis[4-(cyclohexyloxy)-6-hydroxy-3,1-phenylene]]bis[phenylmethanone



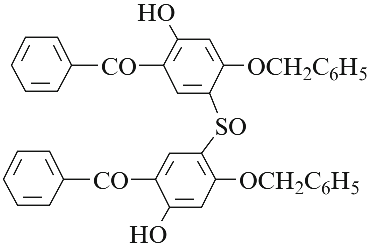
$C_{38}H_{38}O_7S$ mol.wt. 638.79

Synthesis

- Obtained by action of thionyl chloride with 2-hydroxy-4-(cyclohexyloxy)benzophenone in the presence of aluminium chloride in nitrobenzene at r.t. for 24 h [1474].

m.p. and Spectra (NA).

[Sulfinylbis[4-(benzyloxy)-6-hydroxy-3,1-phenylene]]bis[phenylmethanone



$C_{40}H_{30}O_7S$ mol.wt. 654.74

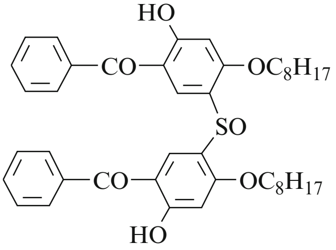
Synthesis

- Obtained by action of thionyl chloride with 2-hydroxy-4-(benzyloxy)benzophenone in the presence of aluminium chloride in nitrobenzene at r.t. for 24 h [1474].

m.p. and Spectra (NA).

[Sulfinylbis[6-hydroxy-4-(octyloxy)-3,1-phenylene]]bis[phenylmethanone

[35839-47-5] $C_{42}H_{50}O_7S$ mol.wt. 698.92



Synthesis

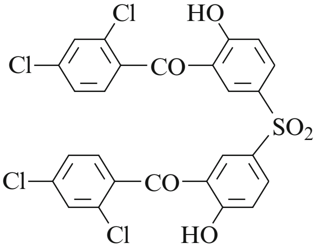
– Obtained by action of thionyl chloride with 2-hydroxy-4-(octyloxy)benzophenone in the presence of aluminium chloride in nitrobenzene at r.t. for 24 h [1474].

m.p. 131°5–132° [1474]; Spectra (NA).

7.7 Diphenyl Sulfone Derivatives

[Sulfonylbis(6-hydroxy-3,1-phenylene)]bis[(2,4-dichlorophenyl)methanone

[153167-56-7] $C_{26}H_{14}Cl_4O_6S$ mol.wt. 596.27



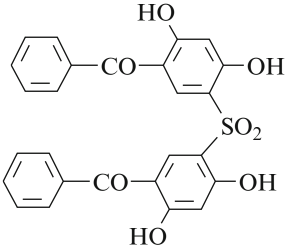
Synthesis

– Preparation by Fries rearrangement of bis (4-hydroxy-phenyl)sulfonedi-(2,4-dichlorobenzoate) with a mixture of fluoroalcanesulfonic acid and alcanesulfonic acid [1441].

m.p. (NA); ^{13}C NMR [1441].

[Sulfonylbis(4,6-dihydroxy-3,1-phenylene)]bis[phenylmethanone

[35839-48-6] $C_{26}H_{18}O_8S$ mol.wt. 490.49



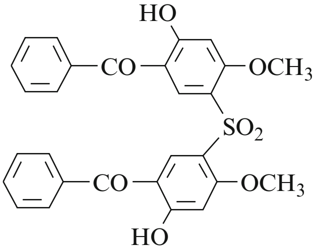
Synthesis

– Obtained by reaction of a 30% aqueous solution of hydrogen peroxide with 5,5′-thiobis(2,4-dihydroxy-benzophenone) in acetic acid. The mixture was then heated on a steam bath for 6 h [1474].

m.p. 217–219° [1474]; Spectra (NA).

[Sulfonylbis(6-hydroxy-4-methoxy-3,1-phenylene)]bis[phenylmethanone

[35698-04-5]



$C_{28}H_{22}O_8S$ mol.wt. 518.54

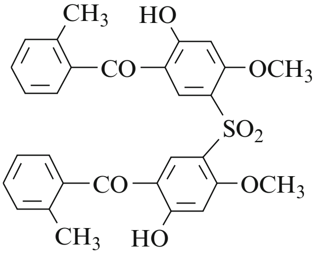
Synthesis

- Obtained by reaction of a 30% aqueous solution of hydrogen peroxide with 5,5′-thiobis(2-hydroxy-4-methoxybenzophenone) in acetic acid. The mixture was then heated on a steam bath for 4 h [1474].

m.p 264–267° [1474]; Spectra (NA).

[Sulfonylbis(6-hydroxy-4-methoxy-3,1-phenylene)]bis[(2-methylphenyl) methanone

[35698-06-7]



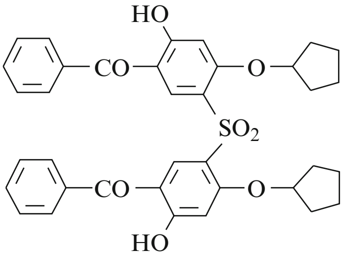
$C_{30}H_{26}O_8S$ mol.wt. 546.60

Synthesis

- Obtained by reaction of a 30% aqueous solution of hydrogen peroxide with 5,5′-thiobis(2-hydroxy-4-methoxy-2′-methylbenzophenone) in acetic acid. The mixture was then heated on a steam bath for 6 h [1474].

m.p. 268–269° [1474]; Spectra (NA).

[Sulfonylbis[4-(cyclopentyloxy)-6-hydroxy-3,1-phenylene]]bis[phenylmethanone



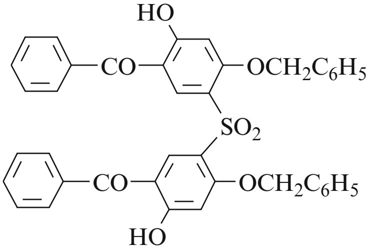
$C_{36}H_{34}O_8S$ mol.wt. 626.73

Synthesis

- Obtained by reaction of a 30% aqueous solution of hydrogen peroxide with 5,5′-thiobis[2-hydroxy-4-(cyclopentyloxy) benzophenone] in acetic acid. The mixture was then heated on a steam bath for 6 h [1474].

m.p. and Spectra (NA).

[Sulfonylbis[4-(benzyloxy)-6-hydroxy-3,1-phenylene]]bis[phenylmethanone



$C_{40}H_{30}O_8S$ mol.wt. 670.74

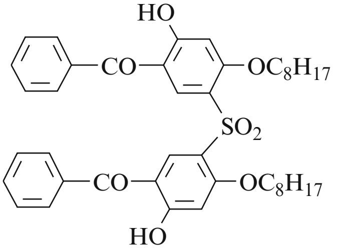
Synthesis

- Obtained by reaction of a 30% solution of hydrogen peroxide with 5,5′-sulfinylbis(2-hydroxy-4-benzyloxybenzophenone) in acetic acid. The mixture was then heated on a water bath for 6 h [1474].

m.p. and Spectra (NA).

[Sulfonylbis[6-hydroxy-4-(octyloxy)-3,1-phenylene]]bis[phenylmethanone

[35698-05-6]



$C_{42}H_{50}O_8S$ mol.wt. 714.92

Synthesis

- Obtained by reaction of a 30% solution of hydrogen peroxide with 5,5′-sulfinylbis(2-hydroxy-4-octyloxybenzophenone) in acetic acid. The mixture was then heated on a steam bath for 6 h [1474].

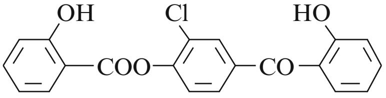
m.p. and Spectra (NA).

7.8 Other Acylated Compounds

2-Chloro-4-(2-hydroxybenzoyl)phenyl 2-hydroxybenzoate

2-Hydroxybenzoic acid, 2-chloro-4-(2-hydroxybenzoyl)phenyl ester

[123861-93-8]



$C_{20}H_{13}ClO_5$ mol.wt. 368.77

Syntheses

- Obtained by Fries rearrangement,

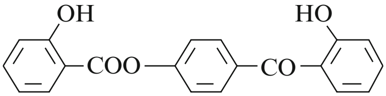
- of o-chlorophenyl 2-(nicotinoyloxy)benzoate in the presence of aluminium chloride without solvent at 150–152° for 2 h (26%) [346];
- of o-chlorophenyl salicylate in the presence of aluminium chloride without solvent at 180–183° for 3 h (20%) [506] according to [1475].

m.p. 151–152° [346,506]; Spectra (NA).

4-(2-Hydroxybenzoyl)phenyl 2-hydroxybenzoate

2-Hydroxybenzoic acid 4-(2-hydroxybenzoyl)phenyl ester

[124011-55-8] $C_{20}H_{14}O_5$ mol.wt. 334.33



Syntheses

- Preparation by total demethylation of 4-(2-methoxybenzoyl)phenyl 2-methoxybenzoate with aluminium chloride in refluxing benzene for 1 h (50%) [346].
- Also obtained by Fries rearrangement of phenyl salicylate (salol) with aluminium chloride [1475], without solvent at 180–182° for 3 h (10%) [155].
- Also obtained (by-product) by Fries rearrangement of phenyl 2-(nicotinoyloxy) benzoate with aluminium chloride without solvent at 140–145° for 2 h (16%) [346].

m.p. 110° [155], 109–110° [346]; Spectra (NA).

Part IV
Addendum to Volume 1

Chapter 8
Addendum 2000–2008

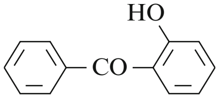
Part I Monoaroylphenols

Chapter 1. Unsubstituted Hydroxybenzophenones *(Class of METHANONES)*

1.1 Monohydroxybenzophenones [1476] p. 3

(2-Hydroxyphenyl)phenylmethanone

[117-99-7] $C_{13}H_{10}O_2$ mol.wt. 198.22



Described [1476] **p. 3**

Methyl ether [2553-04-0] $C_{14}H_{12}O_2$ mol.wt. 212.25

- Obtained by action of etheral diazomethane on 2-hydroxybenzophenone in the presence of methanol [1477].
- Also obtained by reaction of bromoethane with 2-hydroxybenzophenone in the presence of sodium ethoxide in ethanol [1478].
- Also obtained by reaction of 2-methoxybenzoyl chloride with benzene in the presence of aluminium chloride [1479].
- Also obtained by reaction of benzonitrile with anisole (Hoesch reaction) (36%) [1480].
- Also obtained by heating its oxime with pyridinium chloride containing 10% of water (72%) [1481].
- Also obtained by reaction of 2-methoxyphenylmagnesium bromide,
  - with benzonitrile (50%) [1482];
  - with benzaldehyde [1483].
- Also refer to: [1484–1488].

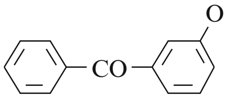
R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_8,

b.p.$_{0.9}$ 126–128° [1486], b.p.$_{0.8}$ 136° [1489], b.p.$_{10}$ 190° [1481], b.p.$_{27}$ 210° [1478];

m.p. 39° [1478,1481], 36–37° [1483], 35.5–36.5° [1482];
^{1}H NMR [1486,1487,1489,1490], ^{13}C NMR [1487–1489],
IR [1485,1487,1489,1490],
UV [1479,1487,1490,1491], MS [1486,1488].

(3-Hydroxyphenyl)phenylmethanone

[13020-57-0] $C_{13}H_{10}O_2$ mol.wt. 198.22



Described [1476] **p. 6**

Syntheses

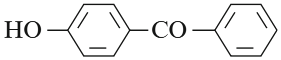
– Also refer to: [1492–1494].

Methyl ether [6136-67-0] $C_{14}H_{12}O_2$ mol.wt. 212.25

– Obtained by photolysis of 2-bromo-3′-methoxybenzophenone in acetonitrile under nitrogen at 350 nm for 36 min (18%) [1495].

(4-Hydroxyphenyl)phenylmethanone

[1137-42-4] $C_{13}H_{10}O_2$ mol.wt. 198.22



Described [1476] **p. 7**

Methyl ether [611-94-9] $C_{14}H_{12}O_2$ mol.wt. 212.25

– Obtained by reaction of 4-methoxybenzoyl chloride with benzene in the presence of aluminium chloride [1496].
– Also obtained by reaction of benzoyl chloride with anisole [1497].
– Also obtained by photocatalytic oxidation of 4-methoxybenzhydrol using silica-encapsulated $H_3PW_{12}O_{40}$ as photocatalyst in acetonitrile for 1.25 h at r.t. under oxygen gas as the sole reoxidant of the catalyst, (90%) [1498].
– Also obtained by photolysis of 2-bromo-4′-methoxybenzophenone in acetonitrile under nitrogen at 350 nm for 14 min (39%) [1495].
– Also obtained by reaction of benzonitrile with anisole (Hoesch reaction) (34%) [1480].
– Also refer to: [1482,1499,1500], (94%) [1501].

m.p. 58–60° [1501];
^{1}H NMR [1498], IR [1498], UV [1501], MS [1498]; TLC [1498].

Ethyl ether [27982-06-5] $C_{15}H_{14}O_2$ mol.wt. 226.27 (m.p. 42–44°) [1502]

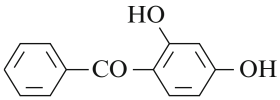
– Refer to: [1502].

1.2 Dihydroxybenzophenones [1476] p. 11

1.2.1 Hydroxy Groups Located on One Ring [1476] p. 11

(2,4-Dihydroxyphenyl)phenylmethanone

[131-56-6] $C_{13}H_{10}O_3$ mol.wt. 214.22



Described [1476] **p. 11**

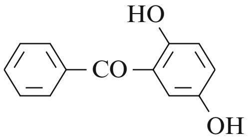
Dimethyl ether [3555-84-8] $C_{15}H_{14}O_3$ mol.wt. 242.27

- Obtained by reaction of benzoic acid with 1,3-dimethoxybenzene in the presence of PPA at 90° for 8 h (90%) [1503].
- Also refer to: [1485,1504].

white solid [1503]; m.p. 87° [1503], 86–88° [1504];
^{1}HNMR [1503], IR [1485,1503].

(2,5-Dihydroxyphenyl)phenylmethanone

[2050-37-5] $C_{13}H_{10}O_3$ mol.wt. 214.22



Described [1476] **p. 14**

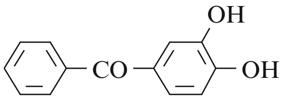
Dimethyl ether [4038-13-5] $C_{15}H_{14}O_3$ mol.wt. 242.27

- Obtained by reaction of benzoyl chloride with 1,4-dimethoxybenzene in methylene chloride in the presence of stannic chloride at 0° (82%) [1503].
- Also refer to: [1504].

m.p. 51° [1503], 49–50° [1504];
^{1}H NMR [1503], IR [1485,1503].

(3,4-Dihydroxyphenyl)phenylmethanone

[10425-11-3] $C_{13}H_{10}O_3$ mol.wt. 214.22



Described [1476] **p. 15**

Syntheses

- Also obtained by Friedel–Crafts reaction between benzoyl chloride and pyrocatechol dimethyl ether, followed by demethylation of the product so obtained [1505].
- Also refer to: [1506–1508].

m.p. 132° [1505].

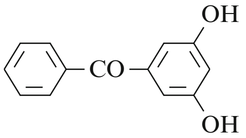
Dimethyl ether [4038-14-6] $C_{15}H_{14}O_3$ mol.wt. 242.27
(4-Benzoylveratrole)

- Obtained by reaction of benzoyl chloride with veratrole,
  - in the presence of aluminium chloride in carbon disulfide (68%) [1509];
  - in the presence of zinc chloride [1510].
- Also obtained by reaction of benzoic acid with veratrole in the presence of PPA at 90° for 8 h (90%) [1503].
- Also obtained by reaction of phenylcarbonyl benzoate with veratrole in the presence of $TiCl(OTf)_3$ and TfOH in acetonitrile at r.t. for 12 h (94%) [1511].
- Also obtained by photolysis of 2-bromo-3′,4′-dimethoxybenzophenone in acetonitrile under nitrogen at 350 nm for 72 min (2%) [1495].
- Also obtained by oxidation of (3,4-dimethoxyphenyl)phenylmethanol with chromic acid [1512].
- Also refer to: [1479,1504,1513–1518].

m.p. 103–104° [1512], 102° [1503], 101–102° [1510], 99–100° [1504], 99° [1513], 98–100° [1509];
^{1}H NMR [1503,1515], IR [1503,1504,1518],
UV [1479,1503,1515–1517], MS [1514].

(3,5-Dihydroxyphenyl)phenylmethanone

[38009-30-2] $C_{13}H_{10}O_3$ mol.wt. 214.22



Described [1476] **p. 16**

Dimethyl ether [628263-26-3] $C_{15}H_{14}O_3$ mol.wt. 242.27

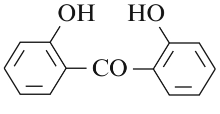
- Obtained by irradiation of 2-bromo-3′,5′-dimethoxybenzophenone at 350 nm in acetonitrile under nitrogen atmosphere for 72 h at r.t. (3%) [1495].

1.2.2 Hydroxy Groups Located on Both Rings [1476] p. 17

Symmetrical ketones [1476] p. 17

Bis(2-hydroxyphenyl)methanone

[835-11-0] $C_{13}H_{10}O_3$ mol.wt. 214.22



Described [1476] **p. 17**

Syntheses

- Also refer to: [1519–1521].

N.B.: Thermal and dielectric studies [1521].

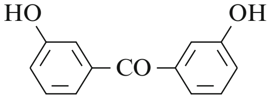
Dimethyl ether [13102-33-5] $C_{15}H_{14}O_3$ mol.wt. 242.27

- Obtained by reaction of anisole with N,N-dimethylamide chlorocarbonic acid (77%) [1522].
- Refer to: [1485].

^{1}H NMR [1522], ^{13}C NMR [1522], IR [1485,1522].

Bis(3-hydroxyphenyl)methanone

[611-80-3] $C_{13}H_{10}O_3$ mol.wt. 214.22



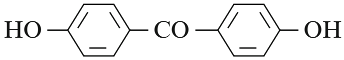
Described [1476] **p. 17**

Synthesis

- Also refer to: [1523].

Bis(4-hydroxyphenyl)methanone

[611-99-4] $C_{13}H_{10}O_3$ mol.wt. 214.22



Described [1476] **p. 18**

Synthesis

- Also obtained by Friedel–Crafts reaction between p-anisoyl chloride and anisole, followed by demethylation of the obtained product [1505].

m.p. 210° [1505].

Dimethyl ether [90-96-0] $C_{15}H_{14}O_3$ mol.wt. 242.27

- Obtained by photocatalytic oxidation of 4,4′-dimethoxybenzhydrol (m.p. 69–71°) catalyzed by $H_3PW_{12}O_{40}/SiO_2$ in acetonitrile under oxygen atmosphere at r.t. for 1 h (90%) [1498].
- Also obtained by microwave irradiation of 4-iodoanisole in the presence of $Co_2(CO)_8$ in acetonitrile under air in sealed vessel for 10 s (57%) [1524].
- Also obtained by reaction of 4-methoxyphenylglyoxylonitrile with 4-methoxyphenylmagnesium bromide in the presence of $Fe(acac)_3$ (5 mol%) in THF at −10° for 30 min under argon (98%) [1525].
- Also obtained by reaction of p-anisoyl chloride with anisole [1526] in the presence of MoO_2Cl_2 (20 mol%) at reflux for 20 h (81%) [1527].
- Obtained by photolysis of 2-bromo-4′,4′-dimethoxybenzophenone in acetonitrile under nitrogen at 350 nm for 24 min (34%) [1495].
- Also refer to: [1501] (93%).

m.p. 146–147.5° [1501], 143° [1526];
^{1}H NMR [1498], IR [1498], UV [1501], MS [1498];
TLC [1498]; GC-MS [1524].

Diethyl ether $C_{17}H_{18}O_3$ mol.wt. 270.33

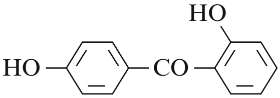
- Preparation by reaction of 4-ethoxybenzoyl chloride with phenetole in the presence of aluminium chloride in carbon disulfide at 55° (80%) [1497].

b.p.$_{15}$ 258° [1497]; m.p. 132° [1497].

Asymmetric ketones [1476] p. 19

(2-Hydroxyphenyl)(4-hydroxyphenyl)methanone

[606-12-2] $C_{13}H_{10}O_3$ mol.wt. 214.22



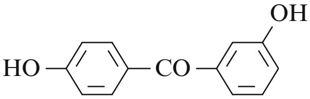
Described [1476] **p. 20**

Dimethyl ether [5449-69-4] $C_{15}H_{14}O_3$ mol.wt. 242.27

- Refer to: [1485]; IR [1485].

(3-Hydroxyphenyl)(4-hydroxyphenyl)methanone

[611-81-4] $C_{13}H_{10}O_3$ mol.wt. 214.22



Described [1476] **p. 21**

Synthesis

- Preparation by treatment of 3,4′-dimethoxybenzophenone with pyridinium chloride at reflux for 30 min (95%) [1528].

white solid; m.p. 188–189° [1528];
^{1}H NMR [1528], ^{13}C NMR [1528], IR [1528], MS [1528].

Dimethyl ether [75731-44-1] $C_{15}H_{14}O_3$ mol.wt. 242.27

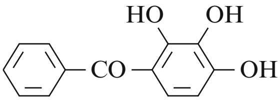
- Obtained by photolysis of 2-bromo-3′,4-dimethoxybenzophenone in acetonitrile under nitrogen at 350 nm for 48 min (18%) [1495].

1.3 Trihydroxybenzophenones [1476] p. 22

1.3.1 Hydroxy Groups Located on One Ring [1476] p. 22

Phenyl(2,3,4-trihydroxyphenyl)methanone

[1143-72-2] $C_{13}H_{10}O_4$ mol.wt. 230.22



Described [1476] **p. 22**

Syntheses

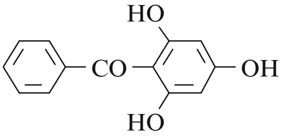
- Also obtained by Friedel–Crafts reaction between benzoyl chloride and pyrogallol trimethyl ether, followed by demethylation of the obtained product [1505].

- Also refer to: [1529,1530] (Japanese patent) [1531–1534] (Chinese patent).

m.p. 118° [1505].

Phenyl(2,4,6-trihydroxyphenyl)methanone *(Phlorbenzophenone)*

[3555-86-0] $C_{13}H_{10}O_4$ mol.wt. 230.22



Described [1476] **p. 23**

Syntheses

- Also obtained by treatment of 2,4,6-trimethoxybenzophenone,
  - with boron tribromide (5 equiv) in methylene chloride, first at −78°, then at r.t. (92%) [1535] for 66 h (80%) [1536];
  - with aluminium chloride in methylene chloride at 20° for 12 h (96%) [1535].
- Also refer to: [1537,1538].

m.p. 167–169° [1535], 164–165° [1537];
^{1}H NMR [1535,1539–1541], ^{13}C NMR [1541],
IR [1535,1540], MS [1541].

BIOLOGICAL ACTIVITY: Antimicrobial activity against Bacillus subtilis [1542]; activity against Escherichia coli [1542].

USE: Stabilization PVC [1543].

Trimethyl ether [3770-80-7] $C_{16}H_{16}O_4$ mol.wt. 272.30 (58%) [1536]
(Methylhydrocotoin)

- Obtained by reaction of benzoyl chloride with 1,3,5-trimethoxybenzene in the presence of aluminium chloride in ethyl ether, first between 0° and 5° for 1 h, then at r.t. for 15 h (46%) [1544].
- Also obtained by reaction of benzoyl chloride with 2,4,6-trimethoxyphenyllithium in ethyl ether at 0° for 1 h under nitrogen (50%) [1545].
- Also obtained by reaction of benzonitrile with phloroglucinol trimethyl ether (Hoesch reaction) (66%) [1480].

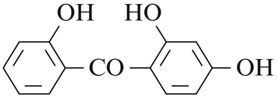
m.p. 113–115° [1535,1545], 113° [1544];
^{1}H NMR [1535,1544,1545], ^{13}C NMR [1535],
IR [1545], UV [1545].

Tribenzoate $C_{34}H_{22}O_7$ mol.wt. 542.54 (m.p. 125–126°) [1537].

1.3.2 Hydroxy Groups Located on Both Rings [1476] p. 24

(2,4-Dihydroxyphenyl)(2-hydroxyphenyl)methanone

[13087-18-8] $C_{13}H_{10}O_4$ mol.wt. 230.22



Described [1476] **p. 25**

Synthesis

- Also obtained by action of salicylic acid with resorcinol in the presence of zinc chloride for 45 min at 125–140° [1505].

m.p. 138° [1505].

Trimethyl ether [33077-87-1] $C_{16}H_{16}O_4$ mol.wt. 272.30

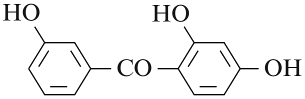
- Obtained by reaction of 2-methoxybenzoic acid with 1,3-dimethoxybenzene in the presence of PPA at 90° for 8 h (85%) [1503].
- Also refer to: [1515,1546,1547].

m.p. 61–62° [1515,1546,1547], 59–60° [1503];
^{1}H NMR [1503], IR [1503].

(2,4-Dihydroxyphenyl)(3-hydroxyphenyl)methanone

[837-60-5] $C_{13}H_{10}O_4$ mol.wt. 230.22

Described [1476] **p. 25**



Trimethyl ether [844-38-2] $C_{16}H_{16}O_4$ mol.wt. 272.30

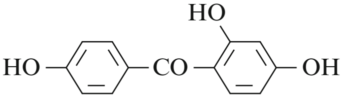
- Obtained by reaction of 3-methoxybenzoic acid with 1,3-dimethoxybenzene in the presence of PPA at 90° for 8 h (85%) [1503].
- Also refer to: [1548].

m.p. 79–80° [1503]; ^{1}H NMR [1503], IR [1503].

(2,4-Dihydroxyphenyl)(4-hydroxyphenyl)methanone

[1470-79-7] $C_{13}H_{10}O_4$ mol.wt. 230.22

Described [1476] **p. 25**



Syntheses

- Obtained by treatment of its trimethyl ether with aluminium chloride in toluene at 120° [1505].
- Also obtained by Friedel–Crafts reaction between p-anisoyl chloride and resorcinol dimethyl ether, followed by demethylation of the obtained product [1505].
- Also refer to: [1531,1532].

m.p. 198° [1505].

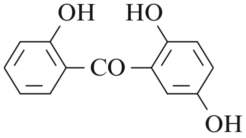
Trimethyl ether [4038-15-7] $C_{16}H_{16}O_4$ mol.wt. 272.30

- Obtained by heating of its oxime with pyridinium chloride containing 10% of water [1481].
- Also obtained by reaction of p-anisoyl chloride with resorcinol dimethyl ether in the presence of aluminium chloride [1549].
- Also obtained by reaction of 2,4-dimethoxybenzoyl chloride with anisole in the presence of aluminium chloride [1550].
- Also obtained by reaction of 4-methoxybenzoic acid with 1,3-dimethoxybenzene in the presence of PPA at 90° for 8 h (85%) [1503].
- Also refer to: [1551–1556].

m.p. 145° [1503], 73–74° [1481,1550], 70–71° [1549], 67° [1557];
One of the reported melting points is obviously wrong.
^{1}H NMR [1503,1556], IR [1503,1557], UV [1551].

(2,5-Dihydroxyphenyl)(2-hydroxyphenyl)methanone

[183106-13-0] $C_{13}H_{10}O_4$ mol.wt. 230.22



Described [1476] **p. 26**

Synthesis

- Also obtained by action of salicylic acid with hydroquinone in the presence of zinc chloride for 45 min at 125–140° [1505].

m.p. 98° [1505].

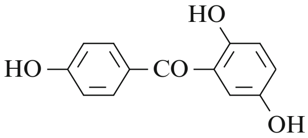
Trimethyl ether [32938-33-3] $C_{16}H_{16}O_4$ mol.wt. 272.30

- Obtained by reaction of 2-methoxybenzoyl chloride with 1,4-dimethoxybenzene in methylene chloride in the presence of stannic chloride (82%) [1503].
- Also refer to: [1558].

m.p. 48° [1503]; ^{1}H NMR [1503], IR [1503].

(2,5-Dihydroxyphenyl)(4-hydroxyphenyl)methanone

[120506-56-1] $C_{13}H_{10}O_4$ mol.wt. 230.22



Described [1476] **p. 26**

Synthesis

- Also obtained by Friedel–Crafts reaction between p-anisoyl chloride and hydroquinone dimethyl ether, followed by demethylation of the obtained product [1505].

m.p. 162° [1505].

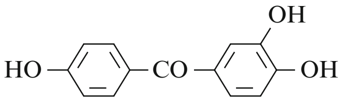
Trimethyl ether [80427-23-2] $C_{16}H_{16}O_4$ mol.wt. 272.30

- Obtained by reaction of 4-methoxybenzoyl chloride with 1,4-dimethoxybenzene in methylene chloride in the presence of stannic chloride (80%) [1503].
- Also refer to: [1559].

white solid; m.p. 70° [1503]; ^{1}H NMR [1503], IR [1503].

(3,4-Dihydroxyphenyl)(4-hydroxyphenyl)methanone

[74697-54-4] $C_{13}H_{10}O_4$ mol.wt. 230.22



Described [1476] **p. 27**

Syntheses

- Also obtained by Friedel–Crafts reaction between p-anisoyl chloride and pyrocatechol dimethyl ether, followed by demethylation of the obtained product [1505].

- Also obtained by reaction of 3,4-dimethoxybenzoyl chloride with anisole in the presence of aluminium chloride in carbon disulfide. The obtained trimethoxybenzophenone was converted to the trihydroxybenzophenone by heating with an equal weight of aluminium chloride in toluene for 1 h at 120° [1505].

  m.p. 205° [1505].

Trimethyl ether [2898-54-6] $C_{16}H_{16}O_4$ mol.wt. 272.30.

- Obtained by reaction of 4-methoxybenzoic acid with veratrole in the presence of PPA at 90° for 8 h (80%) [1503].
- Also refer to: [1560].

  white solid; m.p. 96–97° [1503]; ^{1}H NMR [1503], IR [1503], UV [1503].

1.4 Tetrahydroxybenzophenones [1476] p. 28

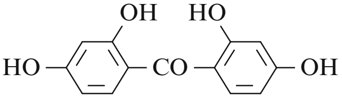
1.4.1 Hydroxy Groups Located on One Ring [1476] p. 28

1.4.2 Hydroxy Groups Located on Both Rings [1476] p. 28

Symmetrical ketones [1476] p. 28

Bis(2,4-dihydroxyphenyl)methanone

[131-55-5] $C_{13}H_{10}O_5$ mol.wt. 246.22



Described [1476] **p. 29**

Syntheses

- Also prepared from 2,4-dihydroxybenzoic acid and resorcinol in sulfolane using anhydrous zinc chloride and phosphorous oxychloride as catalysts for 2.2 h at 70° (87%) [1561].
- Also obtained by action of β-resorcylic acid with resorcinol in the presence of zinc chloride for 45 min at 125–140° [1505].
- Also refer to: [1562] (**THBP**), [1534] (Chinese patent).

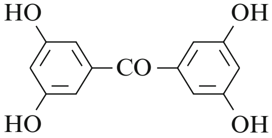
  m.p. 180° [1505].

Tetramethyl ether [3555-85-9] $C_{17}H_{18}O_5$ mol.wt. 302.33.

- Refer to: [1562] (**TMBP**).

Bis(3,5-dihydroxyphenyl)methanone

[873220-56-5] $C_{13}H_{10}O_5$ mol.wt. 246.22



New compound

Synthesis

- Preparation by treatment of its tetramethyl ether with boron tribromide in methylene chloride at 0° [1563].

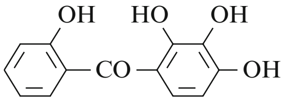
Tetramethyl ether [184090-09-3] $C_{17}H_{18}O_5$ mol.wt. 302.33.

- Obtained by oxidation of 3,3′,5,5′-tetramethoxybenzhydrol with manganese dioxide in methylene chloride at r.t. [1563].

Asymmetric ketones [1476] p. 30

(2-Hydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[42204-63-7] $C_{13}H_{10}O_5$ mol.wt. 246.22



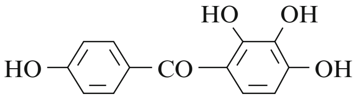
Described [1476] **p. 32**

Synthesis

- Also refer to: [1534] (Chinese patent).

(4-Hydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[31127-54-5] $C_{13}H_{10}O_5$ mol.wt. 246.22



Described [1476] **p. 34**

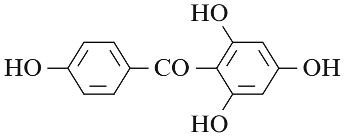
Syntheses

- Also obtained by Friedel–Crafts reaction between p-anisoyl chloride and pyrogallol trimethyl ether, followed by demethylation of the product obtained [1505].
- Also refer to: [1564,1565] (Japanese patent), [1534,1566,1567] (Chinese patents).

m.p. 219° [1505].

(4-Hydroxyphenyl)(2,4,6-trihydroxyphenyl)methanone *(Iriflophenone)*

[52591-10-3] $C_{13}H_{10}O_5$ mol.wt. 246.22



Described [1476] **p. 34**

Monohydrate [880877-63-4] $C_{13}H_{10}O_5, H_2O$ mol.wt. 264.24

Isolation from natural sources

- From the rhizomes of *Iris germanica* Linn. [1568].

m.p. 209–210° [1568]; X-ray Data [1568].

1.5 Pentahydroxybenzophenones [1476] p. 35

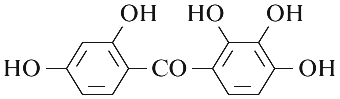
1.5.1 Hydroxy Groups Located on One Ring [1476] p. 35

1.5.2 Hydroxy Groups Located on Both Rings [1476] p. 35

(2,4-Dihydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[92379-42-5]

$C_{13}H_{10}O_6$ mol.wt. 262.22



Described [1476] **p. 35**

Synthesis

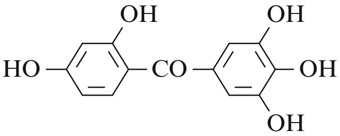
- Also obtained by action of β-resorcylic acid with pyrogallol in the presence of zinc chloride for 45 min at 125–140° [1505].

m.p. 200° [1505].

(2,4-Dihydroxyphenyl)(3,4,5-trihydroxyphenyl)methanone

[10425-09-9]

$C_{13}H_{10}O_6$ mol.wt. 262.22



Described [1476] **p. 36**

Synthesis

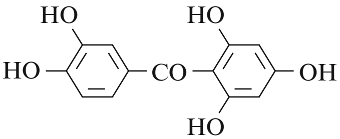
- Also obtained by action of β-resorcylic acid with pyrogallol in the presence of zinc chloride for 45 min at 125–140° [1505].

m.p. 253° [1505].

(3,4-Dihydroxyphenyl)(2,4,6-trihydroxyphenyl)methanone

[519-34-6]

$C_{13}H_{10}O_6$ mol.wt. 262.22



Described [1476] **p. 37**

Syntheses

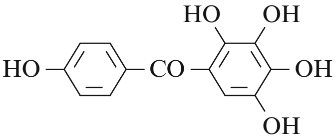
- Also obtained by Friedel–Crafts reaction between veratryl chloride and phlroglucinol dimethyl ether, followed by demethylation of the obtained product [1505].
- Also refer to: [1569].

m.p. 220° [1505].

(4-Hydroxyphenyl)(2,3,4,5-tetrahydroxyphenyl)methanone

[112232-17-4]

$C_{13}H_{10}O_6$ mol.wt. 262.22



Described [1476] **p. 39**

Synthesis

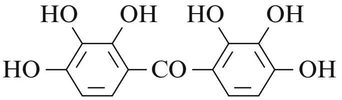
- Also refer to: [1534] (Chinese patent).

1.6 Hexahydroxybenzophenones [1476] p. 39

Symmetrical ketones [1476] p. 39

Bis(2,3,4-trihydroxyphenyl)methanone *(Exifone, Adlone)*

[75440-84-5] $C_{13}H_{10}O_7$ mol.wt. 278.22



Described [1476] **p. 39**

Synthesis

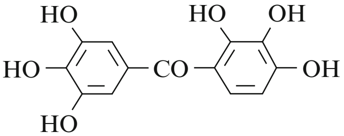
- Also obtained by action of 2,3,4-trihydroxybenzoic-acid with pyrogallol in the presence of zinc chloride for 45 min at 125–140° [1505].

m.p. 240° [1505].

Asymmetric ketones [1476] p. 40

(2,3,4-Trihydroxyphenyl)(3,4,5-trihydroxyphenyl)methanone *(Exifone, Adlone)*

[52479-85-3] $C_{13}H_{10}O_7$ mol.wt. 278.22



Described [1476] **p. 40**

Syntheses

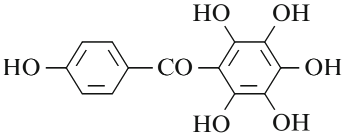
- Also obtained by action of gallic acid with pyrogallol in the presence of zinc chloride for 45 min at 125–140° [1505].
- Also refer to: [1529,1533,1570].

m.p. 276° [1505].

BIOLOGICAL ACTIVITY: Synergistic antimalarial activity of exifone and rufigallol. [1571]; synergistic antimalarial activity of exifone and vitamin C [1571]; pharmacology [1572]; animal studies in memory improvement [1573]; in antagonism of benzodiazepine-induced amnesia [1574]; clinical evaluation in senile dementia [1575]; in Parkinson's disease [1576].

(4-Hydroxyphenyl)(2,3,4,5,6-pentahydroxyphenyl)methanone

[960294-81-9] $C_{13}H_{10}O_7$ mol.wt. 278.22



New compound

Synthesis

- Refer to: [1534] (Chinese patent).

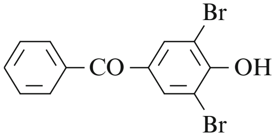
Chapter 2. Substituted Hydroxybenzophenones *(Class of METHANONES)*

2.1 Monohydroxybenzophenones [1476] p. 43

2.1.1 Substituents Located on the Hydroxylated Ring [1476] p. 43

(3,5-Dibromo-4-hydroxyphenyl)phenylmethanone

[26733-16-4] $C_{13}H_8Br_2O_2$ mol.wt. 356.01



Described [1476] **p. 44**

Syntheses

- Also obtained by adding HMTAB (hexamethylene tetramine-bromine complex) to 4-hydroxybenzophenone in methylene chloride at r.t. for 5 min (40%) [1577].
- Also obtained by reaction of bromine with 4-hydroxybenzophenone in dilute acetic acid [1497].

m.p. 153.5° [1497], 148–150° [1577].

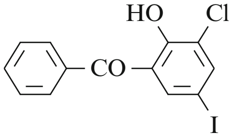
Ethyl ether $C_{15}H_{12}Br_2O_2$ mol.wt. 384.07

- Obtained by reaction of ethyl iodide with 2-hydroxy-3,5-dibromobenzophenone in the presence of potassium hydroxide in boiling ethanol [1497].

b.p.$_{11}$ 244° [1497]; m.p. 83.5° [1497].

(3-Chloro-2-hydroxy-5-iodophenyl)phenylmethanone

[883566-15-2] $C_{13}H_8ClIO_2$ mol.wt. 358.56



New compound

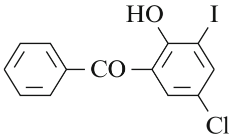
Synthesis

- Obtained by reaction of iodine and iodic acid with 3-chloro-2-hydroxybenzophenone at 35–40° in 95% ethyl alcohol (82%) [1578].

m.p. 172° [1578]; ^{1}H NMR [1578], IR [1578], MS [1578].

(5-Chloro-2-hydroxy-3-iodophenyl)phenylmethanone

[58878-51-6] $C_{13}H_8ClIO_2$ mol.wt. 358.56



New compound

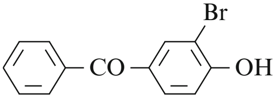
Syntheses

- Obtained by reaction of iodine and iodic acid with 5-chloro-2-hydroxybenzophenone at 35–40° in 95% ethyl alcohol (80%) [1578].
- Also refer to: [1579,1580].

m.p. 118° [1578], 110–111° [1579,1580];
^{1}H NMR [1578], IR [1578], MS [1578].

(3-Bromo-4-hydroxyphenyl)phenylmethanone

[89899-44-5] $C_{13}H_9BrO_2$ mol.wt. 277.11



Described [1476] **p. 50**

Syntheses

- Also obtained by adding HMTAB (hexamethylene tetramine-bromine complex) to 4-hydroxybenzophenone in methylene chloride at −5° for 145 min (97%) [1577].
- Also obtained by reaction of bromine with 4-hydroxybenzophenone in anhydrous acetic acid [1497].

m.p. 183° [1497], 181–183° [1577].

Methyl ether $C_{14}H_{11}BrO_2$ mol.wt. 294.14

- Refer to: [1514]; MS [1514].

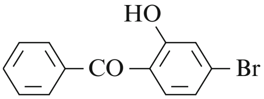
Ethyl ether $C_{15}H_{13}BrO_2$ mol.wt. 305.17

- Obtained by reaction of ethyl iodide with 3-bromo-4-hydroxybenzophenone in the presence of potassium hydroxide in boiling ethanol for 3 h [1497].
- Also obtained by reaction of bromine with 4-ethoxybenzophenone in dilute acetic acid at r.t. for 1 h [1497].

m.p. 102° [1497].

(4-Bromo-2-hydroxyphenyl)phenylmethanone

[6723-04-2] $C_{13}H_9BrO_2$ mol.wt. 277.11



Described [1476] **p. 50**

Syntheses

- Obtained by treatment of 6-bromo-2,3-diphenylbenzofuran with chromium trioxide [1581].
- Also refer to: [1582–1584].

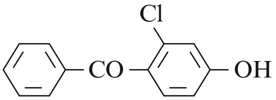
m.p. 83° [1582]; UV [1582].

Oxime (1*E*) [335195-33-0] $C_{13}H_{10}BrNO_2$ mol.wt. 292.13 (75%) [1581]

m.p. 115° [1581]; ^{1}H NMR [1581], IR [1581], MS [1581].

(2-Chloro-4-hydroxyphenyl)phenylmethanone

[81375-00-0] $C_{13}H_9ClO_2$ mol.wt. 232.67



Described [1476] **p. 51**

Methyl ether [67601-27-8] $C_{14}H_{11}ClO_2$ mol.wt. 246.69

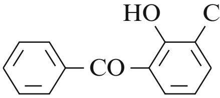
– Obtained by heating for 4 h a mixture of 3-chloroanisole and benzoyl chloride,

- in the presence of ferric chloride (75%) [1585];
- in the presence of aluminium chloride (80%) [1585].

b.p.$_{12}$ 183° [1585].

(3-Chloro-2-hydroxyphenyl)phenylmethanone

[35582-86-6] $C_{13}H_9ClO_2$ mol.wt. 232.67



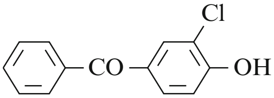
Described [1476] **p. 52**

Synthesis

– Also refer to: [1578].

(3-Chloro-4-hydroxyphenyl)phenylmethanone

[55191-20-3] $C_{13}H_9ClO_2$ mol.wt. 232.67



Described [1476] **p. 52**

Synthesis

– Also refer to: [1514]; MS [1514].

Methyl ether [10547-61-2] $C_{14}H_{11}ClO_2$ mol.wt. 246.69

– Obtained by heating for 4 h a mixture of 2-chloroanisole and benzoyl chloride,

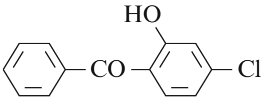
- in the presence of ferric chloride (70%) [1585];
- in the presence of aluminium chloride (81%) [1585].

– Also refer to: [1514].

b.p.$_{12}$ 181° [1585]; m.p. 99° [1585]; MS [1514].

(4-Chloro-2-hydroxyphenyl)phenylmethanone

[2985-80-0] $C_{13}H_9ClO_2$ mol.wt. 232.67



Described [1476] **p. 53**

Synthesis

– Obtained by heating 4-chloro-2-hydroxybenzoyl chloride with benzene in the presence of aluminium chloride (81%) [1586].

m.p. 70–71° [1586]; ^{1}H NMR [1586].

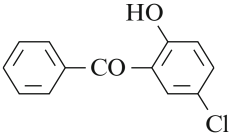
Methyl ether [78589-12-5] $C_{14}H_{11}ClO_2$ mol.wt. 246.69

– Obtained by treatment of 4-chloro-2-hydroxybenzophenone with methyl iodide in DMSO in the presence of potassium hydroxide at 40° (25%) [1586].

m.p. 91–93° [1586]; ^{1}H NMR [1586], ^{13}C NMR [1586], IR [1586].

(5-Chloro-2-hydroxyphenyl)phenylmethanone

[85-19-8] $C_{13}H_9ClO_2$ mol.wt. 232.67



Described [1476] **p. 53**

Synthesis

- Also refer to: [1584].

Methyl ether [4072-28-0] $C_{14}H_{11}ClO_2$ mol.wt. 246.69

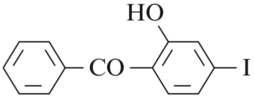
- Obtained by heating for 4 h a mixture of 4-chloroanisole and benzoyl chloride,
  - in the presence of ferric chloride (70%) [1585];
  - in the presence of aluminium chloride (81%) [1585].
- Obtained by reaction of dimethyl sulfate with 5-chloro-2-hydroxybenzophenone potassium salt in toluene [1587].
- Preparation from 1-(5-chloro-2-methoxyphenyl)-1-phenylethylene [1588].
- Preparation from 5-chloro-2-methoxyphenylboronic acid [1588].
- Also refer to: [1557,1589,1590].

b.p.$_{12}$ 180° [1585]: m.p. 103–104° [1590], 101° [1585], 100.5–101° [1587], 100° [1557];

^{1}H NMR [1588], ^{13}C NMR [1588], IR [1557,1588].

(2-Hydroxy-4-iodophenyl)phenylmethanone

[335195-30-7] $C_{13}H_9IO_2$ mol.wt. 324.12



New compound

Synthesis

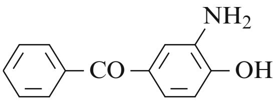
- Obtained by treatment of 6-iodo-2,3-diphenylbenzofuran with chromium trioxide [1581].

Oxime (1*E*) [335195-36-3] $C_{13}H_{10}INO_2$ mol.wt. 339.13 (82%) [1581]

m.p. 143–144° [1581]; ^{1}H NMR [1581], IR [1581], MS [1581].

(3-Amino-4-hydroxyphenyl)phenylmethanone

[42404-41-1] $C_{13}H_{11}NO_2$ mol.wt. 213.24



Described [1476] **p. 61**

Syntheses

- Also obtained by treatment of 4-hydroxy-3-nitrobenzophenone with hydrazine hydrate in the presence of Raney nickel in methanol at 60–60° for 1.5 h [1591].
- Also refer to: [1533,1592–1595].

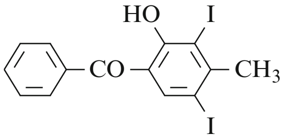
UV [1595].

Hydrochloride $C_{13}H_{11}NO_2$, HCl mol.wt. 249.70.

- Refer to: [1596] (91%); (m.p. 185–196°) [1596].

(2-Hydroxy-3,5-diiodo-4-methylphenyl)phenylmethanone

[883566-14-1] $C_{14}H_{10}I_2O_2$ mol.wt. 464.04



New compound

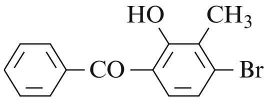
Synthesis

- Obtained by reaction of iodine and iodic acid with 2-hydroxy-4-methylbenzophenone at 35–40° in 95% ethyl alcohol (84%) [1578].

m.p. 121° [1578]; ^{1}H NMR [1578], IR [1578], MS [1578].

(4-Bromo-2-hydroxy-3-methylphenyl)phenylmethanone

[6758-89-0] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



Described [1476] **p. 65**

Synthesis

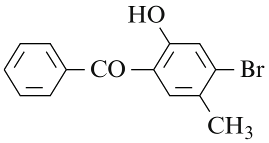
- Obtained by treatment of 6-bromo-7-methyl-2,3-diphenyl-benzofuran with chromium trioxide [1581].

Oxime (1*E*) [335195-35-2] $C_{14}H_{12}BrNO_2$ mol.wt. 306.16 (69%) [1581]

m.p. 140–142° [1581]; ^{1}H NMR [1581], IR [1581], MS [1581].

(4-Bromo-2-hydroxy-5-methylphenyl)phenylmethanone

[6723-07-5] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



Described [1476] **p. 65**

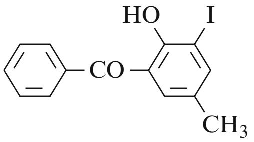
Synthesis

- Obtained by treatment of 6-bromo-5-methyl-2,3-diphenyl-benzofuran with chromium trioxide [1581].

Oxime (1*E*) [335195-34-1] $C_{14}H_{12}BrNO_2$ mol.wt. 306.16 (78%) [1581], m.p. 135–137° [1581]; ^{1}H NMR [1581], IR [1581], MS [1581].

(2-Hydroxy-3-iodo-5-methylphenyl)phenylmethanone

[883566-13-0] $C_{14}H_{11}IO_2$ mol.wt. 338.14



New compound

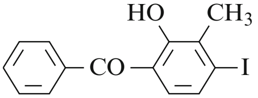
Synthesis

- Obtained by reaction of iodine and iodic acid with 2-hydroxy-5-methylbenzophenone at 35–40° in 95% ethyl alcohol (82%) [1578].

m.p. 80° [1578];
^{1}H NMR [1578], ^{13}C NMR [1578], IR [1578], MS [1578].

(2-Hydroxy-4-iodo-3-methylphenyl)phenylmethanone

[194784-86-6] $C_{14}H_{11}IO_2$ mol.wt. 338.14



New compound

Synthesis

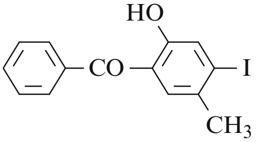
- Obtained by treatment of 6-iodo-7-methyl-2,3-diphenylbenzofuran with chromium trioxide [1581].

Oxime (1*E*) [335195-38-5] $C_{14}H_{12}INO_2$ mol.wt. 353.16 (83%) [1581].

m.p. 161° [1581]; ^{1}H NMR [1581], IR [1581], MS [1581].

(2-Hydroxy-4-iodo-5-methylphenyl)phenylmethanone

[335195-31-8] $C_{14}H_{11}IO_2$ mol.wt. 338.14



New compound

Synthesis

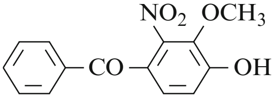
- Obtained by treatment of 6-iodo-5-methyl-2,3-diphenylbenzofuran with chromium trioxide [1581].

Oxime (1*E*) [335195-37-4] $C_{14}H_{12}INO_2$ mol.wt. 353.16 (85%) [1581].

m.p. 152–155° [1581]; ^{1}H NMR [1581], IR [1581], MS [1581].

(4-Hydroxy-3-methoxy-2-nitrophenyl)phenylmethanone

[383382-98-7] $C_{14}H_{11}NO_5$ mol.wt. 273.25



New compound

Synthesis

- Obtained by hydrolysis of 4-acetoxy-3-methoxy-2-nitro-benzophenone with 3 N sodium hydroxide in dilute methanol at r.t. for 15 min (93%) [1597].

m.p. 168.9–170° [1597]; ^{1}H NMR [1597], ^{13}C NMR [1597].

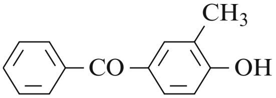
Acetate [383382-96-5] $C_{16}H_{13}NO_6$ mol.wt. 315.22

- Obtained by adding copper (II) nitrate trihydrate in one portion to a solution of 4-(acetyloxy)-3-methoxybenzophenone (m.p. 103–104°) in acetic anhydride at r.t. (52%) [1597].

m.p. 84.5–86° [1597]; ^{1}H NMR [1597], ^{13}C NMR [1597].

(4-Hydroxy-3-methylphenyl)phenylmethanone

[5326-42-1] $C_{14}H_{12}O_2$ mol.wt. 212.25



Described [1476] **p. 78**

Syntheses

- Also obtained by Friedel–Crafts acylation of o-cresol with benzoic acid in a mixture of graphite and methanesulfonic acid at 120° for 3 h (90%) [1598].
- Also refer to: [1514,1599].

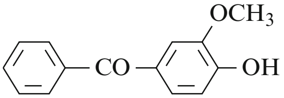
^{1}H NMR [1598], ^{13}C NMR [1598], MS [1514].

Methyl ether [30090-97-2] $C_{15}H_{14}O_2$ mol.wt. 226.27

- Refer to: [1514]; MS [1514].

(4-Hydroxy-3-methoxyphenyl)phenylmethanone

[51439-89-5] $C_{14}H_{12}O_3$ mol.wt. 228.25



Described [1476] **p. 83**

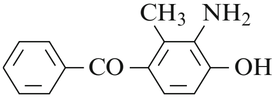
Acetate [383382-97-6] $C_{16}H_{14}O_4$ mol.wt. 270.28

- Preparation by reaction of acetic anhydride with 4-hydroxy-3-methoxybenzophenone in the presence of pyridine in methylene chloride at r.t. for 15 min (94%) [1597].

m.p. 103–104° [1597]; ^{1}H NMR [1597], ^{13}C NMR [1597].

(3-Amino-4-hydroxy-2-methylphenyl)phenylmethanone

[909255-30-7] $C_{14}H_{13}NO_2$ mol.wt. 227.26



New compound

Synthesis

- Obtained by treatment of (3-amino-4-methoxy-2-methyl-phenyl)phenylmethanone with aluminium chloride in toluene at 100° for 20 min (86%) [1595].

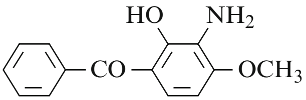
m.p. 145–147° [1595]; ^{1}H NMR [1595], ^{13}C NMR [1595].

Methyl ether $C_{15}H_{15}NO_2$ mol.wt. 241.29

- Obtained by reaction of benzoic acid with 2-methoxy-6-methylaniline in the presence of PPA at 100° for 3 h (40%) [1595].

(3-Amino-2-hydroxy-4-methoxyphenyl)phenylmethanone

[253681-30-0] $C_{14}H_{13}NO_3$ mol.wt. 243.26



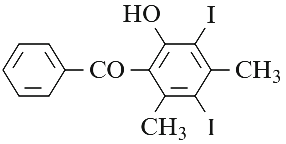
New compound

Synthesis

- Obtained by treatment of 3-amino-2,4-dimethoxy-benzophenone with aluminium chloride in refluxing methylene chloride for 1 h (70–90%) [1529].

(2-Hydroxy-3,5-diiodo-4,6-dimethylphenyl)phenylmethanone

[883566-16-3] $C_{15}H_{12}I_2O_2$ mol.wt. 478.07



New compound

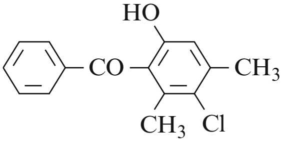
Synthesis

- Obtained by reaction of iodine and iodic acid with 2-hydroxy-4,6-dimethylbenzophenone at 35–40° in 95% ethyl alcohol (75%) [1578].

m.p. 133° [1578]; ^1H NMR [1578], IR [1578], MS [1578].

(3-Chloro-6-hydroxy-2,4-dimethylphenyl)phenylmethanone

[34174-02-2] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



Described [1476] **p. 88**

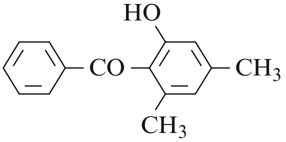
Synthesis

- Obtained by [3 + 3] cyclisation of 3-chloro-4-(tri-methylsilyloxy)-3-penten-2-one (**3a**) with 2,4-bis (trimethylsilyloxy)-2-phenyl-2-butene (**4c**) in the presence of titanium tetrachloride in methylene chloride, first at −78°, then to 20° for 20 h (44%) [1600].

m.p. 199° [1600]; ^1H NMR [1600], ^{13}C NMR [1600], IR [1600], MS [1600].

(2-Hydroxy-4,6-dimethylphenyl)phenylmethanone

[2929-45-5] $C_{15}H_{14}O_2$ mol.wt. 226.27



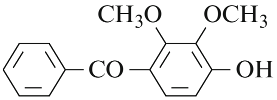
Described [1476] **p. 93**

Synthesis

- Also obtained by Fries rearrangement of 3,5-dimethylphenyl benzoate in the presence of 1-butyl-3-methylimidazolium chloroaluminate $[BMIm]^+Al_2Cl_7^-$ melt at 120° for 2 h (94%) [1601].

(4-Hydroxy-2,3-dimethoxyphenyl)phenylmethanone

[872881-75-9] $C_{15}H_{14}O_4$ mol.wt. 258.27



New compound

Isolation from natural sources

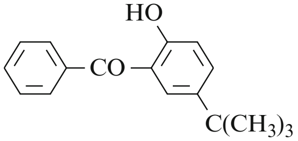
- From the roots of *Securidaca inappendiculata* Hassk (Polygalaceae) [1602].

Colourless powder; m.p. 62.5° [1602]; $(\alpha)_D^{20}$ = 0 (0.50 $CHCl_3$) [1602]; ^1H NMR [1602], ^{13}C NMR [1602], MS [1602], IR [1602], UV [1602], MS [1602].

BIOLOGICAL ACTIVITY: Used as antiinflammatory, antibacterial and anti-rheumatism agent in China [1602].

[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]phenylmethanone

[10425-05-5]



$C_{17}H_{18}O_2$ mol.wt. 254.33

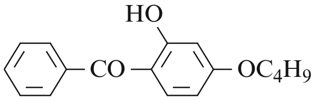
Described [1476] **p. 115**

Synthesis

- Obtained by palladium diacetate catalyzed reaction of benzonitrile with p-tert-butylphenol in the presence of TFA in DMSO at 90° (71%) [1480].

(4-Butoxy-2-hydroxyphenyl)phenylmethanone

[15131-43-8]



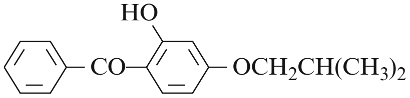
$C_{17}H_{18}O_3$ mol.wt. 270.33

Described [1476] **p. 119**

Synthesis

- Also refer to: [1603].

[4-(1,1-Dimethylethoxy)-2-hydroxyphenyl]phenylmethanone



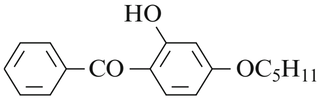
$C_{17}H_{18}O_3$ mol.wt. 270.33

New compound

- Polymer with 1,2-ethanediol [1020077-60-4].
- Refer to: [1603].

[2-Hydroxy-4-(pentyloxy)phenyl]phenylmethanone

[83937-21-7]



$C_{18}H_{20}O_3$ mol.wt. 284.36

Described [1476] **p. 124**

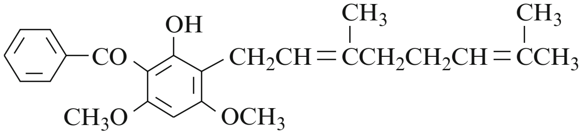
Synthesis

- Also refer to: [1604].

[3-(3,7-Dimethyl-2,6-octadienyl)-2-hydroxy-4,6-dimethoxyphenyl]phenylmethanone *(E) (Marupone monomethyl ether)*

[53948-16-6]

$C_{25}H_{30}O_4$ mol.wt. 394.51



New compound

Synthesis

- Obtained by treatment of marupone with diazomethane in ethyl ether at r.t. for 24 h [1605].

oil [1605]; ^{1}H NMR [1605], IR [1605], UV [1605].

Acetate (*E*) [53948-17-7] $C_{27}H_{32}O_5$ mol.wt. 436.55

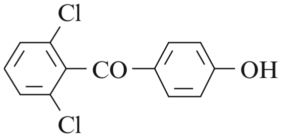
- Obtained by treatment of marupone monomethyl ether with acetic anhydride in the presence of pyridine at r.t. for 24 h [1605].

oil [1605]; ^{1}H NMR [1605], IR [1605].

2.1.2 Substituents Located on the Other Ring [1476] p. 143

(2,6-Dichlorophenyl)(4-hydroxyphenyl)methanone

[61002-53-7] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



Described [1476] **p. 146**

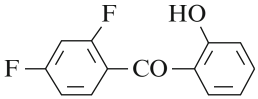
Syntheses

- Obtained by reaction of 2,6-dichlorobenzoyl chloride with anisole in the presence of aluminium chloride [1606].
- Also refer to: [1607].

m.p. 208° [1606].

(2,4-Difluorophenyl)(2-hydroxyphenyl)methanone

[46795-44-2] $C_{13}H_8F_2O_2$ mol.wt. 234.20



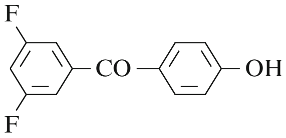
New compound

Synthesis

- Obtained by an intramolecular acyl radical *ipso* substitution (68%) [1608].

(3,5-Difluorophenyl)(4-hydroxyphenyl)methanone *(Homopolymer)*

[148253-50-3] $C_{13}H_8F_2O_2$ mol.wt. 234.20



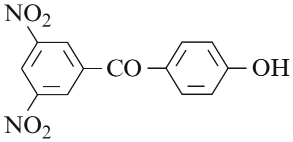
New compound

Synthesis

- Refer to: [1609] (Japanese patent).

(3,5-Dinitrophenyl)(4-hydroxyphenyl)methanone

[51339-44-7] $C_{13}H_8N_2O_6$ mol.wt. 288.22

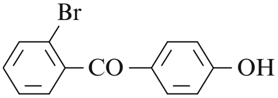


Described [1476] **p. 147**

Synthesis

- Also refer to: [1610] (Japanese patent).

(2-Bromophenyl)(4-hydroxyphenyl)methanone



$C_{13}H_9BrO_2$ mol.wt. 277.11

Described [1476] **p. 148**

Syntheses

- Also obtained by dealkylation of 2-bromo-4′-ethoxy-benzophenone by treatment with hydrobromic acid (d = 1.49) in acetic acid [1611].
- Also obtained by Fries rearrangement of phenyl 2-bromobenzoate with aluminium chloride [1612].

b.p.$_{10}$ 260° [1611]; m.p. 114° [1611], 100–103° [1612].

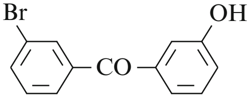
Methyl ether [59142-63-1] $C_{14}H_{11}BrO_2$ mol.wt. 291.14

- Obtained by reaction of p-anisoyl chloride with o-bromophenylzinc iodide in the presence of $Pd(PPh_3)_4$ in THF at 20° for 1 h [1613].
- Also obtained by reaction of o-bromobenzoyl chloride with anisole in the presence of aluminium chloride in chloroform at 20° for 12 h (89%) [1614].
- Also obtained by treatment of 2-bromo-α-(4-methoxyphenyl)benzyl alcohol with PCC in methylene chloride at 20° for 2 h (90%) [1615].
- Preparation [1616] according to [1617].
- Also refer to: [1495,1618,1619].

m.p. 90–92° [1495];
^{1}H NMR [1495,1614,1615], ^{13}C NMR [1495,1614,1615], IR [1495].

(3-Bromophenyl)(3-hydroxyphenyl)methanone

[62810-50-8]



$C_{13}H_9BrO_2$ mol.wt. 277.11

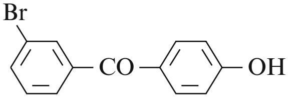
Described [1476] **p. 148**

Methyl ether [750633-66-0] $C_{14}H_{11}BrO_2$ mol.wt. 291.14

- Obtained by reaction of 3-bromobenzoyl chloride with 3-methoxyphenylmagnesium bromide [1620], (98%) [1621].
- Also refer to: [1622].

colourless oil [1621]; ^{1}H NMR [1621], ^{13}C NMR [1621].

(3-Bromophenyl)(4-hydroxyphenyl)methanone



$C_{13}H_9BrO_2$ mol.wt. 277.11

Described [1476] **p. 149**

Synthesis

- Also obtained by dealkylation of its ethyl ether by treatment with hydrobromic acid (d = 1.49) in boiling acetic acid for 2 days [1497].

m.p. 171° [1497].

Methyl ether [54118-76-2] $C_{14}H_{11}BrO_2$ mol.wt. 291.14

- Refer to: [1501] (78%).

  m.p. 80–81° [1501]; UV [1501].

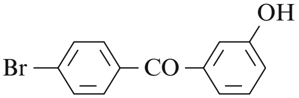
Ethyl ether $C_{15}H_{13}BrO_2$ mol.wt. 277.11

- Preparation by reaction of 3-bromobenzoyl chloride with phenetole in the presence of aluminium chloride in carbon disulfide at 55° (80%) [1497].

  b.p.$_{11}$ 232° [1497]; m.p. 79.5° [1497].

(4-Bromophenyl)(3-hydroxyphenyl)methanone

[62810-46-2] $C_{13}H_9BrO_2$ mol.wt. 277.11



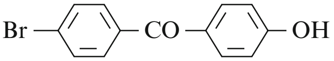
Described [1476] **p. 149**

Methyl ether [151239-47-3] $C_{14}H_{11}BrO_2$ mol.wt. 291.14

- Obtained by reaction of 4-bromophenylmagnesium iodide with 3-methoxybenzoyl chloride in the presence of bis[2-(N,N-dimethylamino)ethyl] ether in THF at −5° to 0° (80%) [1623].

(4-Bromophenyl)(4-hydroxyphenyl)methanone

[4369-50-0] $C_{13}H_9BrO_2$ mol.wt. 277.11



Described [1476] **p. 150**

Syntheses

- Also refer to: [1624–1626].

  m.p. 191° [1626]; ^{1}H NMR [1624,1625].

Methyl ether [54118-75-1] $C_{14}H_{11}BrO_2$ mol.wt. 291.14

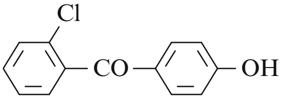
- Refer to: (79%) [1627], (62%) [1501].

  m.p. 155–157° [1501], 147–149° [1627];
  ^{1}H NMR [1627], ^{13}C NMR [1627], IR [1627], UV [1501], MS [1627].

- Complex preparation: Tandem catalysis access to ketones from aldehydes and arylboronic acids via rhodium-catalyzed addition/oxidation, (52%) [1628].

(2-Chlorophenyl)(4-hydroxyphenyl)methanone

[55270-71-8] $C_{13}H_9ClO_2$ mol.wt. 232.67



Described [1476] **p. 151**

Methyl ether [54118-74-0] $C_{14}H_{11}ClO_2$ mol.wt. 246.69

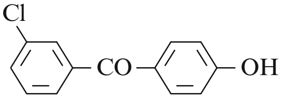
- Obtained by reaction of 4-methoxybenzoyl chloride with chlorobenzene in the presence of aluminium chloride in carbon disulfide at r.t. for 3 h (90%) [1501].

– Also refer to: [1629].

m.p. 79–80.5° [1501]; UV [1501].

(3-Chlorophenyl)(4-hydroxyphenyl)methanone

[61002-52-6] $C_{13}H_9ClO_2$ mol.wt. 232.67



Described [1476] **p. 152**

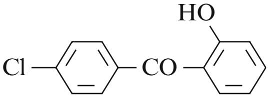
Methyl ether [13389-51-0] $C_{14}H_{11}ClO_2$ mol.wt. 246.69

– Refer to: (96%) [1501].

m.p. 71.5–72° [1501]; UV [1501].

(4-Chlorophenyl)(2-hydroxyphenyl)methanone

[2985-79-7] $C_{13}H_9ClO_2$ mol.wt. 232.67



Described [1476] **p. 152**

Methyl ether [78589-10-3] $C_{14}H_{11}ClO_2$ mol.wt. 246.69

– Obtained by reaction of 4-chlorobenzoyl chloride with anisole [1630] in the presence of MoO_2Cl_2 (20 mol%) at reflux for 20 h (5%) [1527].

– Also obtained by reaction of 4-chlorobenzoic acid with anisole,

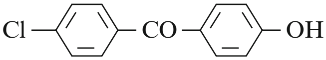
- in the presence of difluorophosphoric anhydride in methylene chloride at 20° for 4 h (46%) [1631];
- in the presence of boron trifluoride etherate at 80° [1632].

– Also refer to: [1557,1633].

m.p. 78° [1557]; IR [1557].

(4-Chlorophenyl)(4-hydroxyphenyl)methanone

[42019-78-3] $C_{13}H_9ClO_2$ mol.wt. 232.67



Described [1476] **p. 153**

Synthesis

– Also refer to: [1634].

Methyl ether [10547-60-1] $C_{14}H_{11}ClO_2$ mol.wt. 246.69

– Obtained by reaction of 4-chlorobenzoyl chloride with anisole in the presence of MoO_2Cl_2 (20 mol%) at reflux for 20 h (75%) [1527].

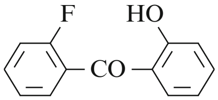
– Also obtained by reaction of 4-chlorophenylglyoxylonitrile with 4-methoxyphenylmagnesium bromide in the presence of $Fe(acac)_3$ (5 mol%) in THF at −10° for 30 min under argon (89%) [1525].

- Also refer to: [1571], (90%) [1627], (70%) [1501].

m.p. 126–127.5° [1501], 116–118° [1627];
^{1}H NMR [1627], ^{13}C NMR [1627], IR [1627], UV [1501], MS [1627].

(2-Fluorophenyl)(2-hydroxyphenyl)methanone

[329235-41-8] $C_{13}H_9FO_2$ mol.wt. 216.21



New compound

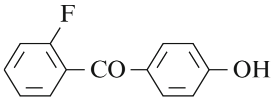
Synthesis

- Obtained by an intramolecular acyl radical *ipso* substitution (57%) [1608].

(2-Fluorophenyl)(4-hydroxyphenyl)methanone

$C_{13}H_9FO_2$ mol.wt. 216.21

Described [1476] **p. 155**

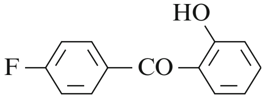


Methyl ether [66938-29-2] $C_{14}H_{11}FO_2$ mol.wt. 230.24

- Refer to: [1635].

(4-Fluorophenyl)(2-hydroxyphenyl)methanone

[62666-37-9] $C_{13}H_9FO_2$ mol.wt. 216.21



Described [1476] **p. 155**

Syntheses

- Also obtained by demethylation of 4-fluoro-2′-methoxy-benzophenone with boron tribromide in methylene chloride at 0° [1484].
- Also obtained by an intramolecular acyl radical *ipso* substitution (53%) [1608].

^{1}H NMR [1484], ^{13}C NMR [1484], IR [1484], MS [1484].

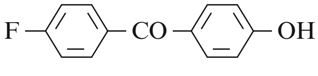
Methyl ether [750633-46-6] $C_{14}H_{11}FO_2$ mol.wt. 230.24

- Obtained by reaction of 2-methoxyphenylmagnesium bromide with 4-fluorobenzaldehyde in THF at 0° for 3 h [1484].

^{1}H NMR [1484], ^{13}C NMR [1484], IR [1484], MS [1484].

(4-Fluorophenyl)(4-hydroxyphenyl)methanone

[25913-05-7] $C_{13}H_9FO_2$ mol.wt. 216.21



Described [1476] **p. 156**

Synthesis

- Also refer to: [1636].

Methyl ether [345-89-1] $C_{14}H_{11}FO_2$ mol.wt. 230.24

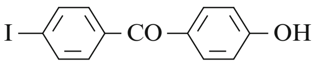
- Obtained by reaction of p-fluorobenzoyl chloride with anisole in the presence of aluminium chloride in carbon disulfide at r.t. for 1 h (75%) [1637].
- Also refer to: (85%) [1627]; (61%) [1501].

m.p. 96° [1637], 95–96° [1501], 89–91° [1627];
^{1}H NMR [1627,1637], ^{13}C NMR [1627], ^{19}F NMR [1637],
IR [1627,1637], UV [1501,1637], MS [1627].

(4-Hydroxyphenyl)(4-iodophenyl)methanone

[113275-52-8] $C_{13}H_{9}IO_2$ mol.wt. 324.12

Described [1476] **p. 157**



Methyl ether [54118-73-9] $C_{14}H_{11}IO_2$ mol.wt. 338.14

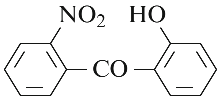
- Obtained by reaction of 4-methoxybenzoyl chloride with iodoanisole in the presence of aluminium chloride in carbon disulfide at r.t. for 3 h (57%) [1501].

m.p. 187–189° [1501]; UV [1501].

(2-Hydroxyphenyl)(2-nitrophenyl)methanone

[22293-32-9] $C_{13}H_{9}NO_4$ mol.wt. 243.22

Described [1476] **p. 157**



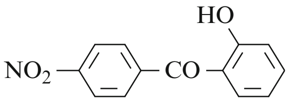
Synthesis

- Also obtained by an intramolecular acyl radical *ipso* substitution (40%) [1608].

(2-Hydroxyphenyl)(4-nitrophenyl)methanone

[68223-20-1] $C_{13}H_{9}NO_4$ mol.wt. 243.22

Described [1476] **p. 158**



Syntheses

- Also obtained by demethylation of 2-methoxy-4′-nitro-benzophenone with boron tribromide in methylene chloride at 0° [1484].
- Also obtained by an intramolecular acyl radical *ipso* substitution (52%) [1608].

^{1}H NMR [1484], ^{13}C NMR [1484], IR [1484], MS [1484].

Methyl ether [42495-50-1] $C_{14}H_{11}NO_4$ mol.wt. 257.24

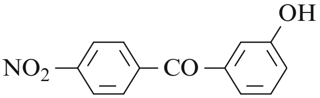
- Obtained by reaction of p-nitrobenzoyl chloride with anisole,
  - in the presence of MoO_2Cl_2 (20 mol%) at reflux for 20 h (3%) [1527];
  - in the presence of perfluorobutane sulfonic acid [1630].
- Also obtained by reaction of 2-methoxyphenylmagnesium bromide with 4-nitrobenzaldehyde in THF at 0° for 3 h [1484].

- Also obtained by a Pd-catalyzed coupling-type reaction of 4-nitrobenzaldehyde and 2-methoxy-phenylboronic acid in the presence of P(1-nap)$_3$, using Cs_2CO_3 in toluene with air (65%) [1638].
- Also obtained by reaction of p-nitrobenzoic acid with anisole in the presence of TFAA at 80° [1632].
- Also refer to: [1639,1640].

m.p. 117–119° [1639,1640];
^{1}H NMR [1484,1638,1640], ^{13}C NMR [1484,1638,1640], IR [1484], MS [1484].

(3-Hydroxyphenyl)(4-nitrophenyl)methanone

[147029-77-4] $C_{13}H_9NO_4$ mol.wt. 243.22



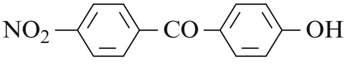
Described [1476] **p. 158**

Methyl ether [62507-47-5] $C_{14}H_{11}NO_4$ mol.wt. 257.24

- Also obtained by adding under an argon atmosphere, 60% sodium hydride in oil to a mixture of 4-nitrofluorobenzene, 3-methoxybenzaldehyde and 1,3,4,5-tetramethylimidazolium iodide in DMF. The mixture was stirred at −15° for 15 min; then at −5° for 2 h (70%) [1641].

(4-Hydroxyphenyl)(4-nitrophenyl)methanone

[18920-70-2] $C_{13}H_9NO_4$ mol.wt. 243.22



Described [1476] **p. 159**

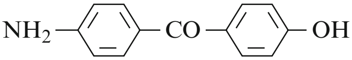
Methyl ether [1151-94-6] $C_{14}H_{11}NO_4$ mol.wt. 257.24

- Obtained by reaction of p-nitrobenzoyl chloride with anisole,
  - in the presence of aluminium chloride in carbon disulfide for 1 h at r.t. (72%) [1637];
  - in the presence of MoO_2Cl_2 (20 mol%) at reflux for 20 h (31%) [1527].
- Also obtained by adding under an argon atmosphere, 60% sodium hydride in oil to a mixture of 4-nitrofluorobenzene, 4-methoxybenzaldehyde and 1,3,4,5-tetramethylimidazolium iodide in DMF. The mixture was stirred at −15° for 15 min; then at −5° for 2 h (85%) [1641].
- Also obtained by reaction of dimethyl sulfate with 4-hydroxy-4′-nitrobenzophenone in the presence of aqueous sodium hydroxide [1642].
- Also refer to: [1643–1650], (73%) [1501].

b.p.$_{0.9}$ 192–194° [1646];
m.p. 126–128° [1646], 124° [1501], 123° [1637], 121–122° [1642];
^{1}H NMR [1637,1643,1645], ^{13}C NMR [1647,1650],
IR [1637,1642], UV [1501,1637,1642], MS [1648].

(4-Aminophenyl)(4-hydroxyphenyl)methanone

[14963-34-9] $C_{13}H_{11}NO_2$ mol.wt. 213.24



Described [1476] **p. 162**

Methyl ether [4834-72-4] $C_{14}H_{13}NO_2$ mol.wt. 227.26

- Obtained by reduction of 4-nitro-4′-methoxybenzophenone with sodium hydrosulfite in refluxing ethanol overnight (62%) [1637].
- Also obtained by treatment of its hydrochloride with 20% ammonia [1651].
- Also refer to: [1501] (92%).

m.p. 110° [1637], 108–109.5° [1501], 108° [1651];
^{1}H NMR [1637], IR [1637], UV [1501,1637].

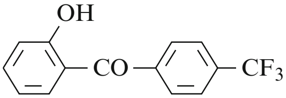
Hydrochloride $C_{14}H_{13}NO_2$, HCl mol.wt. 263.72

- Obtained by heating 4-hydroxy-4′-nitrobenzophenone with stannous chloride in the presence of concentrated hydrochloric acid for 15 min [1651].

m.p. 193° [1651].

(2-Hydroxyphenyl)[4-(trifluoromethyl)phenyl]methanone

[34450-48-1] $C_{14}H_9F_3O_2$ mol.wt. 266.22



New compound

Synthesis

- Obtained by demethylation of 2-methoxy-4-(trifluoro-methyl)benzophenone with boron tribromide in methylene chloride at 0° for 4 h [1484].

^{1}H NMR [1484], ^{13}C NMR [1484], IR [1484], MS [1484].

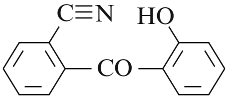
Methyl ether [256475-07-7] $C_{15}H_{11}F_3O_2$ mol.wt. 280.25

- Obtained by reaction of 2-methoxyphenylmagnesium bromide with 4-(trifluoromethyl)-benzaldehyde in THF at 0° for 3 h [1484].
- Also refer to: [1485].

^{1}H NMR [1484], ^{13}C NMR [1484], IR [1484,1485], MS [1484].

2-(2-Hydroxybenzoyl)benzonitrile

[131118-03-1] $C_{14}H_9NO_2$ mol.wt. 223.23



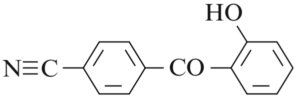
New compound

Synthesis

- Obtained by an intramolecular acyl radical *ipso* substitution (30%) [1608].

4-(2-Hydroxybenzoyl)benzonitrile

[131117-91-4] $C_{14}H_9NO_2$ mol.wt. 223.23



New compound

Synthesis

- Obtained by demethylation of 4-(2-methoxybenzoyl)-benzonitrile with boron tribromide in methylene chloride at 0° [1484].

^{1}H NMR [1484], ^{13}C NMR [1484], IR [1484], MS [1484].

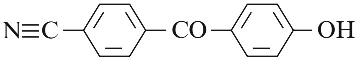
Methyl ether [131117-90-3] $C_{15}H_{11}NO_2$ mol.wt. 237.26

- Obtained by reaction of 2-methoxyphenylmagnesium bromide with 4-cyanobenzaldehyde in THF at 0° for 3 h [1484].

^{1}H NMR [1484], ^{13}C NMR [1484], IR [1484], MS [1484].

4-(4-Hydroxybenzoyl)benzonitrile

[27645-61-0] $C_{14}H_9NO_2$ mol.wt. 223.23



New compound

Syntheses

- Obtained by demethylation of 4-cyano-4′-methoxybenzophenone with aluminium chloride in boiling methylene chloride for 20 h [1652].
- Also obtained from 4-(4-methoxymethoxybenzoyl)benzonitrile by heating with aqueous hydrochloric acid in THF for 3 h [1625].

^{1}H NMR [1625].

Metthyl ether [27645-60-9] $C_{15}H_{11}NO_2$ mol.wt. 237.26

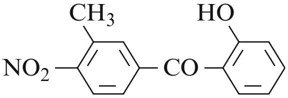
- Obtained by reaction of 4-methoxybenzoyl chloride with acetonitrile in the presence of aluminium chloride in carbon disulfide at r.t. for 3 h (35%) [1501].
- Also obtained by reaction of 4-methoxyphenylglyoxylonitrile with 4-cyanophenylmagnesium bromide in the presence of Fe(acac)$_3$ (5 mol%) in THF at −10° for 30 min under argon (84%) [1525].
- Also obtained by reaction of p-cyanobenzoyl chloride with anisole in the presence of aluminium chloride in benzene [1653].
- Also obtained by treatment of (4-bromophenyl)(4-methoxyphenyl)methanone with copper (I) cyanide [1654].

m.p. 131–132° [1654], 130–132° [1525], 125–128° [1501], 106–109° [1653], 92–93° [1652];

^{1}H NMR [1525], ^{13}C NMR [1525], IR [1525], UV [1501], MS [1525].

(2-Hydroxyphenyl)(3-methyl-4-nitrophenyl)methanone

[1018668-99-9] $C_{14}H_{11}NO_4$ mol.wt. 257.25



New compound

Synthesis

– Obtained by demethylation of 2′-methoxy-3-methyl-4-nitrobenzophenone with boron tribromide in methylene chloride at 0° for 4 h [1484].

^{1}H NMR [1484], ^{13}C NMR [1484], IR [1484], MS [1484].

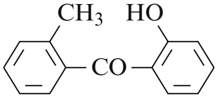
Methyl ether $C_{15}H_{13}NO_4$ mol.wt. 271.27

– Obtained by reaction of 2-methoxyphenylmagnesium bromide with 3-methyl-4-nitrobenzaldehyde in THF at 0° for 3 h [1484].

^{1}H NMR [1484], ^{13}C NMR [1484], IR [1484], MS [1484].

(2-Hydroxyphenyl)(2-methylphenyl)methanone

[51974-19-7] $C_{14}H_{12}O_2$ mol.wt. 212.25



Described [1476] **p. 166**

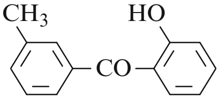
Methyl ether [142256-62-0] $C_{15}H_{14}O_2$ mol.wt. 226.27

– Obtained by reaction of pyridinium chlorochromate (PCC) with (2-methoxyphenyl)(2-methyl-phenyl)methanol in the presence of Celite in methylene chloride at r.t. (70%) [1655].
– Also obtained by reaction of (2-methoxyphenyl)lithium with N-methoxy-2,N-dimethylbenzamide [1485].
– Also refer to: [1485].

m.p. 71–72° [1655];
^{1}H NMR [1655], IR [1485,1655], MS [1655].

(2-Hydroxyphenyl)(3-methylphenyl)methanone

[33785-66-9] $C_{14}H_{12}O_2$ mol.wt. 212.25



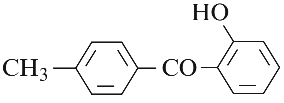
Described [1476] **p. 167**

Synthesis

– Also obtained by an intramolecular acyl radical *ipso* substitution (76%) [1608].

(2-Hydroxyphenyl)(4-methylphenyl)methanone

[19434-30-1] $C_{14}H_{12}O_2$ mol.wt. 212.25



Described [1476] **p. 167**

Syntheses

- Also obtained by demethylation of 4-methyl-2′-methoxy-benzophenone with boron tribromide in methylene chloride at 0° [1484].
- Also obtained by an intramolecular acyl radical *ipso* substitution (80%) [1608].

^1H NMR [1484], ^{13}C NMR [1484], IR [1484], MS [1484].

Methyl ether [28137-36-2] $C_{15}H_{14}O_2$ mol.wt. 226.27

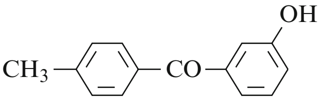
- Obtained by reaction of p-toluoyl chloride with anisole in the presence of MoO_2Cl_2 (20 mol%),
  - in refluxing acetonitrile for 20 h (3%) [1527];
  - in refluxing methylene chloride for 20 h (1%) [1527].
- Also obtained by reaction of 2-methoxybenzoyl chloride with toluene, in the presence of MoO_2Cl_2 (20 mol%) at reflux for 20 h (3%) [1527].
- Also obtained by reaction of O-methylsalicylic acid with toluene in the presence of CF_3SO_3H in various solvents at 25° [1656].
- Also obtained by reaction of 2-methoxyphenylmagnesium bromide with 4-methylbenzaldehyde in THF at 0° for 3 h [1484].
- Also obtained by methylation of 2-hydroxy-4′-methylbenzophenone [1557].
- Also refer to: [1632,1640,1657].

m.p. 64–65° [1657], 61° [1557];
^1H NMR [1484,1640,1657], ^{13}C NMR [1484,1640,1657,1658], IR [1484,1557,1657], MS [1484].

(3-Hydroxyphenyl)(4-methylphenyl)methanone

[62810-49-5] $C_{14}H_{12}O_2$ mol.wt. 212.25

Described [1476] **p. 168**



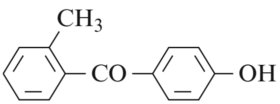
Methyl ether [82520-37-4] $C_{15}H_{14}O_2$ mol.wt. 226.27

- Complex preparation: Tandem catalysis access to ketones from aldehydes and arylboronic acids via rhodium-catalyzed addition/oxidation, (64%) [1628].

(4-Hydroxyphenyl)(2-methylphenyl)methanone

[52981-01-8] $C_{14}H_{12}O_2$ mol.wt. 212.25

Described [1476] **p. 168**



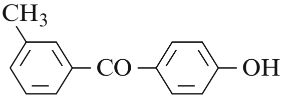
Methyl ether [41204-59-5] $C_{15}H_{14}O_2$ mol.wt. 226.27

- Refer to: [1501] (94%).
  b.p.$_{1.5}$ 147–148° [1501]; UV [1501].

(4-Hydroxyphenyl)(3-methylphenyl)methanone

[71372-37-7] $C_{14}H_{12}O_2$ mol.wt. 212.25

Described [1476] **p. 169**



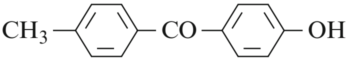
Methyl ether [53039-63-7] $C_{15}H_{14}O_2$ mol.wt. 226.27

- Refer to: [1501] (91%).
  m.p. 55–56° [1501]; UV [1501].

(4-Hydroxyphenyl)(4-methylphenyl)methanone

[134-92-9] $C_{14}H_{12}O_2$ mol.wt. 212.25

Described [1476] **p. 169**



Methyl ether [23886-71-7] $C_{15}H_{14}O_2$ mol.wt. 226.27

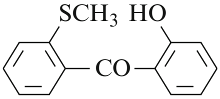
- Also obtained by reaction of p-toluoyl chloride with anisole in the presence of MoO_2Cl_2 (20 mol%),
  - in refluxing bromobenzene for 20 h (82%) [1527];
  - in refluxing acetonitrile for 20 h (59%) [1527];
  - in refluxing methylene chloride for 20 h (49%) [1527].
- Also obtained by reaction of 4-methoxybenzoyl chloride with toluene in the presence of MoO_2Cl_2 (20 mol%) at reflux for 20 h (6%) [1527].
- Complex preparation: Tandem catalysis access to ketones from aldehydes and arylboronic acids via rhodium-catalyzed addition/oxidation, (74%) [1628].
- Also refer to: [1632,1640,1659–1664], (93%) [1665], (82%) [1501], (81%) [1627], (73%) [1666,1667], (60%) [1657].

  m.p. 105–107° [1663], 91–91.2° [1661], 90–92° [1668], 88.5–89° [1665], 88–89° [1657], 87.5–88.5° [1501];
  ^1H NMR [1627,1657,1659,1664,1667], ^{13}C NMR [1627,1640,1657,1659,1664], IR [1627,1663,1664], UV [1501,1660,1662], MS [1666,1667].

(2-Hydroxyphenyl)[2-(methylthio)phenyl]methanone

[1004540-28-6] $C_{14}H_{12}O_2S$ mol.wt. 244.31

New compound



Synthesis

- Refer to: [1669].

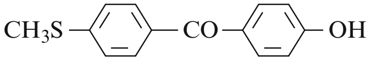
Methyl ether [746652-03-9] $C_{15}H_{14}O_2S$ mol.wt. 258.34

- Refer to: [1669].

(4-Hydroxyphenyl)[4-(methylthio)phenyl]methanone

(4-Hydroxyphenyl)(4-methylsulfanylphenyl)methanone

[83888-61-3] $C_{14}H_{12}O_2S$ mol.wt. 244.31



Described [1476] **p. 169**

Syntheses

- Obtained from its methyl ether [1670].
- Also obtained by reaction of p-methylthiobenzoyl chloride with phenol in 1,2-dichloroethane [1670].
- Also refer to: [1671,1672].

m.p. 133–134° [1671].

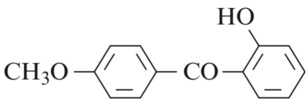
Methyl ether [54118-72-8] $C_{15}H_{14}O_2S$ mol.wt. 258.34

- Obtained by reaction of 4-methoxybenzoyl chloride with thioanisole in the presence of aluminium chloride in carbon disulfide at r.t. for 3 h (37%) [1501].
- Also obtained by reaction of 4-methylthiobenzoyl chloride with anisole in the presence of aluminium chloride in 1,2-dichloroethane [1670].
- Also obtained by reaction of 4-methylthiobenzoic acid with anisole in the presence of boron trifluoride etherate at 80° [1632].
- Obtained by reaction of 4-methoxybenzoyl chloride with thioanisole in the presence of MoO_2Cl_2 (20 mol%) at reflux for 20 h (56%) [1527].

m.p. 126–127° [1501]; UV [1501].

(2-Hydroxyphenyl)(4-methoxyphenyl)methanone

[18733-07-8] $C_{14}H_{12}O_3$ mol.wt. 228.25



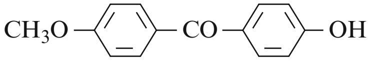
Described [1476] **p. 171**

Synthesis

- Also obtained by an intramolecular acyl radical *ipso* substitution (56%) [1608].

(4-Hydroxyphenyl)(4-methoxyphenyl)methanone

[61002-54-8] $C_{14}H_{12}O_3$ mol.wt. 228.25



Described [1476] **p. 172**

Synthesis

- Complex preparation: Tandem catalysis access to ketones from aldehydes and arylboronic acids via rhodium-catalyzed addition/oxidation, (82%) [1628].

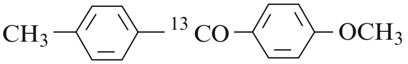
Ethyl ether [52886-92-7] $C_{16}H_{16}O_3$ mol.wt. 256.30

- Obtained by reaction of p-anisoyl chloride with phenetole in the presence of aluminium chloride in carbon disulfide [1673].
- Also obtained by reaction of ethyl iodide with 4-hydroxy-4′-methoxybenzophenone in the presence of sodium ethoxide [1674].
- Also refer to: (17%) [1526,1675,1676], (85%) [1501].

m.p. 112–113° [1501], 112° [1674], 111° [1676], 106–107° [1526,1673], 105° [1675];
1H NMR [1675], IR [1675], UV [1501,1675], MS [1675].
BIOLOGICAL DATA: Radioprotective activity, intraperitoneally [1677]

(4-Methoxyphenyl)(4-methylphenyl)methanone ^{13}C

[112379-67-6] $C_{14}{}^{13}CH_{14}O_2$ mol.wt. 227.26



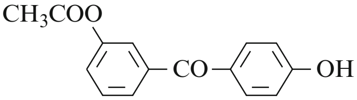
New compound

Synthesis

- Obtained from (E)-2-anisyl-1,2-ditolyl (2-^{13}C)vinyl bromide and 1,1,1-trifluoroethanol [1678].

[3-(Acetyloxy)phenyl](4-hydroxyphenyl)methanone

[263395-60-4] $C_{15}H_{12}O_4$ mol.wt. 256.07



New compound

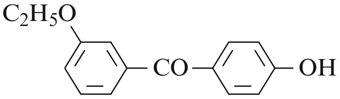
Synthesis

- Obtained by adding acetic anhydride to a solution of the sodium salt of 3,4′-dihydroxybenzophenone in DMF at −10° (59%) [1528].

white solid [1528]; m.p. 118–120° [1528];
^{1}H NMR [1528], ^{13}C NMR [1528], IR [1528], MS [1528].

(3-Ethoxyphenyl)(4-hydroxyphenyl)methanone

[263395-63-7] $C_{15}H_{14}O_3$ Mol.wt. 242.27



New compound

Synthesis

- Obtained in two steps: first, treatment of 3,4′-dihydroxy-benzophenone with sodium hydride (2.2 equiv) in DMF, then reaction of ethyl iodide (1.1 equiv) with the sodium salt previously obtained, at −10° (50%) [1528].

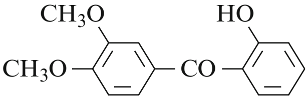
white solid; m.p. 98–100° [1528];
^{1}H NMR [1528], ^{13}C NMR [1528], IR [1528], MS [1528].

Ethyl ether $C_{17}H_{18}O_3$ mol.wt. 270.33

- Also obtained in the above reaction (25–30%) [1528].

(3,4-Dimethoxyphenyl)(2-hydroxyphenyl)methanone

[183106-12-9] $C_{15}H_{14}O_4$ mol.wt. 258.27



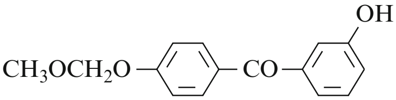
Described [1476] **p. 179**

Synthesis

- Also obtained by an intramolecular acyl radical *ipso* substitution (49%) [1608].

(3-Hydroxyphenyl)[4-(methoxymethoxy)phenyl]methanone

[263395-55-7] $C_{15}H_{14}O_4$ mol.wt. 258.27



New compound

Synthesis

- MOMCl was added to a solution of 3,4′-dihydroxybenzophenone and DIPEA in THF. After removing the cooling bath, the solution was stirred for 4 h (51%) [1528].

white solid; m.p. 94–95° [1528];
^{1}H NMR [1528], ^{13}C NMR [1528], IR [1528], MS [1528].

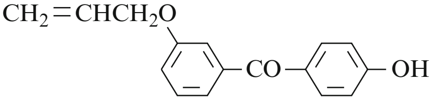
Methoxymethyl ether $C_{17}H_{18}O_5$ mol.wt. 302.33

- The diprotected compound was isolated in this reaction (31%) [1528].
yellow oil [1528];

^{1}H NMR [1528], ^{13}C NMR [1528], IR [1528], MS [1528].

(4-Hydroxyphenyl)[3-(2-propenyloxy)phenyl]methanone

[263395-62-6] $C_{16}H_{14}O_3$ mol.wt. 254.29



New compound

Synthesis

- Obtained by adding allyl bromide to a solution of the sodium salt of 3,4′-dihydroxybenzophenone in DMF at −10° (52%) [1528].

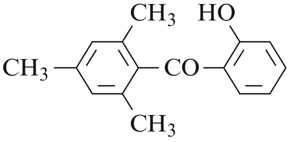
white solid [1528]; m.p. 63–65° [1528];
^{1}H NMR [1528], ^{13}C NMR [1528], IR [1528], MS [1528].

4-(2-propenyl)ether $C_{19}H_{18}O_3$ mol.wt. 294.35

- Also obtained in the above reaction (25–30%) [1528].

(2-Hydroxyphenyl)(2,4,6-trimethylphenyl)methanone

[46863-20-1] $C_{16}H_{16}O_2$ mol.wt. 240.30



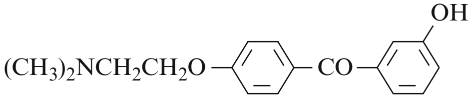
Described [1476] **p. 182**

Synthesis

- Also obtained by an intramolecular acyl radical *ipso* substitution (38%) [1608].

[4-[2-(Dimethylamino)ethoxy]phenyl](3-hydroxyphenyl)methanone

[263395-65-9] $C_{17}H_{19}NO_3$ mol.wt. 285.33



New compound

Synthesis

– Obtained by treatment of its pivalic ester below with 2 N KOH in ethanol at r.t. for 5 h (86%) [1528].

white solid; m.p. 96–98° [1528];
^1H NMR [1528], ^{13}C NMR [1528], IR [1528], MS [1528].

Pivalic ester $C_{22}H_{27}NO_4$ mol.wt. 369.19

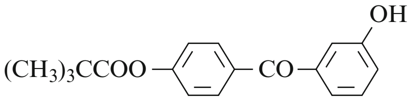
– Obtained by treatment of 4′-hydroxy-3-(trimethylacetoxy)benzophenone with 2-(dimethylamino)-ethyl chloride in the presence of potassium carbonate in refluxing acetone-water (19:1) for 5 h in the dark (61%) [1528].

yellow solid; m.p. 60–62° [1528];

^1H NMR [1528], ^{13}C NMR [1528], IR [1528], MS [1528].

(3-Hydroxyphenyl)[4-(trimethylacetoxy)phenyl]methanone

$C_{18}H_{18}O_4$ mol.wt. 298.34



New compound

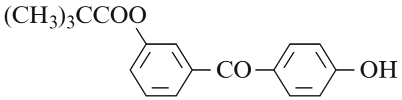
Synthesis

– Obtained by adding pivaloyl chloride to a solution of the sodium salt of 3, 4′-dihydroxybenzophenone in DMF at 0°. Then, the solution was stirred at r.t. for 2 h (47%) [1528].

white solid; m.p. 104–105° [1528];
^1H NMR [1528], ^{13}C NMR [1528], IR [1528], MS [1528].

(4-Hydroxyphenyl)[3-(trimethylacetoxy)phenyl]methanone

$C_{18}H_{18}O_4$ mol.wt. 298.34



New compound

Syntheses

– Obtained by heating a solution of 4′-methoxy-methoxy-3-(trimethylacetoxy)benzophenone in 15% acetic acid at 80° for 20 h (76%) [1528].

– Also obtained by adding pivaloyl chloride to a solution of 3,4′-dihydroxybenzophenone sodium salt in DMF at −10° (85%) [1528].

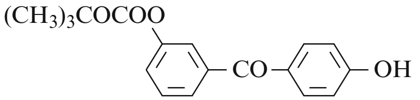
white solid; m.p. 117–119° [1528];
^1H NMR [1528], ^{13}C NMR [1528], IR [1528], MS [1528].

Methoxymethyl ether $C_{20}H_{22}O_5$ mol.wt. 342.39

- Obtained by treatment of 3-hydroxy-4′-(methoxymethoxy)benzophenone in THF at 0° with triethylamine and pivaloyl chloride, then stirring the solution obtained at r.t. for 3 h (quantitative yield) [1528].

yellow oil [1528];
^{1}H NMR [1528], ^{13}C NMR [1528], IR [1528], MS [1528].

[(3-tert-Butoxycarbonyloxy)phenyl](4-hydroxyphenyl)methanone



$C_{18}H_{18}O_5$ mol.wt. 314.34

New compound

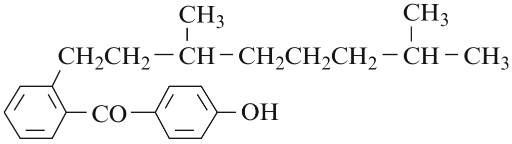
Synthesis

- Obtained by adding $(BOC)_2O$ to a solution of 3,4′-dihydroxybenzophenone sodium salt in DMF at −10° (62%) [1528].

white solid; m.p. 142–144° [1528];
^{1}H NMR [1528], ^{13}C NMR [1528], IR [1528], MS [1528].

[2-(3,7-Dimethyloctyl)phenyl](4-hydroxyphenyl)methanone

[908368-59-2]



$C_{23}H_{30}O_2$ mol.wt. 338.49

New compound

Synthesis

- According to a procedure of [1679], a mixture of 2-(3,7-dimethyloctyl)-4′-methoxybenzophenone, 57% HI solution and acetic acid was heated at reflux for 5 h (84%) [1621].

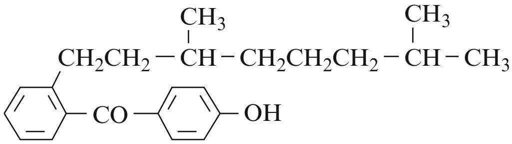
colourless oil; $(\alpha)_D^{28} = -4.29°$ ($CHCl_3$) [1621];
^{1}H NMR [1621], ^{13}C NMR [1621].

Methyl ether [908368-68-3] $C_{24}H_{32}O_2$ mol.wt. 352.52

- Refer to: [1621].

[2-[(3*R*)-3,7-Dimethyloctyl]phenyl](4-hydroxyphenyl)methanone

[908368-61-6]



$C_{23}H_{30}O_2$ mol.wt. 338.49

New compound

Synthesis

- According to a procedure of [1679], a mixture of 2-(3,7-dimethyloctyl)-4′-methoxybenzophenone, 57% HI solution and acetic acid was heated at reflux for 3.5 h (50%) [1621].

clear oil [1621]; ^{1}H NMR [1621], ^{13}C NMR [1621].

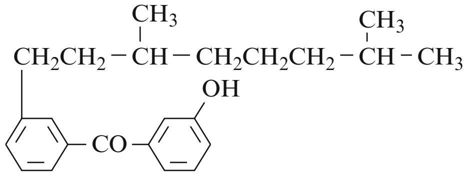
Methyl ether [908368-60-5] $C_{24}H_{32}O_2$ mol.wt. 352.52

- According to the procedure of [1680], to a solution of (*R*)-3,7-dimethyloctene (6.4 mmol) in THF was added dropwise 0.5 M 9-BBN (7.25 mmol) in THF. The resulting mixture was stirred at r.t. for 4 h, then transferred via a syringe to a mixture of Pd(dppf)Cl_2 (1.60 mmol), Cs_2CO_3 (24.1 mmol), $AsPh_3$ (1.61 mmol) and 2-iodo-4′-methoxybenzophenone in a mixture of THF, DMF and H_2O. The resulting mixture was heated at reflux under N_2 overnight (65%) [1621].

colourless oil; $(\alpha)_D^{28} = -5.03°$ ($CHCl_3$) [1621];
^1H NMR [1621], ^{13}C NMR [1621].

[3-[(3*R*)-3,7-Dimethyloctyl]phenyl](3-hydroxyphenyl)methanone

[908368-64-9] $C_{23}H_{30}O_2$ mol.wt. 338.49



New compound

Synthesis

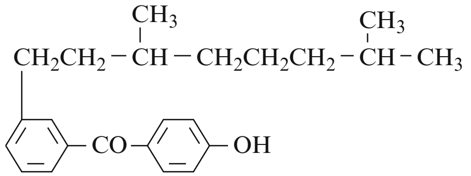
- Refer to: [1621] (multi-step reaction).

Methyl ether [908368-75-2] $C_{24}H_{32}O_2$ mol.wt. 352.52

- Refer to: [1621].

[3-[(3*R*)-3,7-Dimethyloctyl]phenyl](4-hydroxyphenyl)methanone

$C_{23}H_{30}O_2$ mol.wt. 338.49



New compound

Synthesis

- Obtained from its methyl ether (74%) [1621].

Colourless oil [1621]; ^1H NMR [1621], ^{13}C NMR [1621].

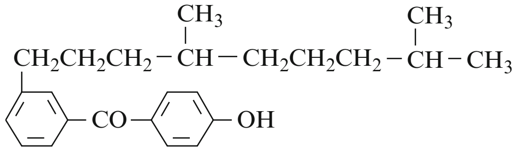
Methyl ether $C_{24}H_{32}O_2$ mol.wt. 352.52

- Refer to: [1621] (69%).

Colourless oil [1621]; ^1H NMR [1621], ^{13}C NMR [1621].

[3-[(4*R*)-4,8-Dimethylnonyl]phenyl](4-hydroxyphenyl)methanone

[908368-62-7] $C_{24}H_{32}O_2$ mol.wt. 352.52



New compound

Synthesis

- Obtained by refluxing a mixture of 3-(4,8-dimethylnonyl)-4′-methoxybenzophenone, 57% HI solution and acetic acid for 3.5 h (81%) [1621].

  colourless oil [1621]; ^{1}H NMR [1621], ^{13}C NMR [1621].

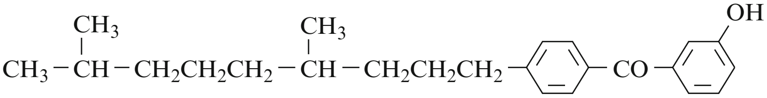
Methyl ether [908368-70-7] $C_{25}H_{34}O_2$ mol.wt. 366.54

- Obtained by reaction of (*R*)-4,8-dimethylnonene and 3-iodo-4′-methoxybenzophenone (Multi-step reaction) (72%) [1621].

  colourless oil [1621]; ^{1}H NMR [1621], ^{13}C NMR [1621].

[4-[(4*R*)-4,8-Dimethylnonyl]phenyl](3-hydroxyphenyl)methanone

[908368-63-8] $C_{24}H_{32}O_2$ mol.wt. 352.52



New compound

Synthesis

- Obtained by refluxing a mixture of (R)-4-(4,8-dimethylnonyl)-3′-methoxybenzophenone, 57% HI solution and acetic acid for 5 h (81%) [1621].

  colourless oil [1621]; ^{1}H NMR [1621], ^{13}C NMR [1621].

Methyl ether [908368-73-0] $C_{25}H_{34}O_2$ mol.wt. 366.54

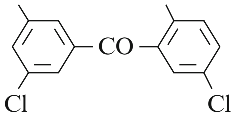
- According to the method [1681], it was obtained by Friedel–Crafts reaction of m-anisoyl chloride with (4,8-dimethylnonyl)phenyl in the presence of aluminium chloride in refluxing carbon disulfide for 5 h (94%) [1621].

  golden oil [1621]; ^{1}H NMR [1621], ^{13}C NMR [1621].

2.1.3 Substituents Located on Both Rings [1476] p. 190

(3-Bromo-5-chlorophenyl)(5-chloro-2-hydroxyphenyl)methanone

[329944-61-8] $C_{13}H_7BrCl_2O_2$ mol.wt. 346.01



New compound

Synthesis

- Obtained by treatment of its methyl ether with boron tribromide in methylene chloride (quantitative yield) [1682].

  yellow crystals [1682]; ^{1}H NMR [1682], ^{13}C NMR [1682].

Methyl ether [329944-59-4] $C_{14}H_9BrCl_2O_2$ mol.wt. 360.03

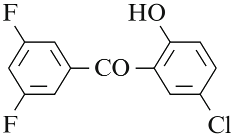
- A solution of 1,3-dibromo-5-chlorobenzene (1 mmol) in ethyl ether was cooled to −78° and n-butyllithium in hexane (1.1 mmol) was added over 2 min.

The reaction mixture was stirred at −78° for an additional 10 min, and then 5-chloro-N,2-dimethoxy-N-methylbenzamide (1 mmol) was added in small portions over 4 min. The reaction mixture was stirred at −78° for 1.25 h and then allowed to warm to r.t. and stirred for 14 h (97%) [1682].

^{1}H NMR [1682], ^{13}C NMR [1682], MS [1682].

(5-Chloro-2-hydroxyphenyl)(3,5-difluorophenyl)methanone

[329941-82-4] $C_{13}H_7ClF_2O_2$ mol.wt. 268.65



New compound

Synthesis

- Obtained by treatment of its methyl ether with boron tribromide in methylene chloride (98%) [1682].

^{1}H NMR [1682], MS [1682].

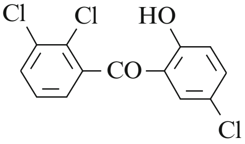
Methyl ether [874889-35-7] $C_{14}H_9ClF_2O_2$ mol.wt. 282.67

- A solution of n-butyllithium in hexanes (18.2 mmol) was added over 4 min to a solution of 2-bromo-4-chloroanisole (15.8 mmol) in ethyl ether cooled to −78°. The resulting mixture was stirred at −78° for 20 min, and then a solution of 3,5-difluoro-N-methoxy-N-methylbenzamide (17.3 mmol) in ethyl ether was added over 5 min. The reaction mixture was stirred at −78° for 1 h and then allowed to warm at r.t. over 3.25 h (42%) [1682].

white solid [1682]; ^{1}H NMR [1682], ^{13}C NMR [1682].

(5-Chloro-2-hydroxyphenyl)(2,3-dichlorophenyl)methanone

[842169-21-5] $C_{13}H_7Cl_3O_2$ mol.wt. 301.56



New compound

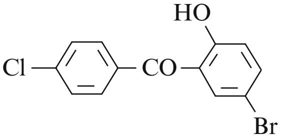
Synthesis

- Preparation by Fries rearrangement of 4-chlorophenyl 2,3-dichlorobenzoate in the presence of aluminium chloride in nitrobenzene at 80–90° for 45 min (85%) [1683].

m.p. 78–80° [1683]; ^{1}H NMR [1683], IR [1683], MS [1683].

(5-Bromo-2-hydroxyphenyl)(4-chlorophenyl)methanone

[939382-98-6] $C_{13}H_8BrClO_2$ mol.wt. 311.56



New compound

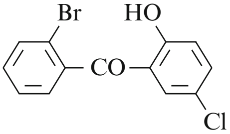
Synthesis

- Preparation by Fries rearrangement of 4-bromophenyl-p-chlorobenzoate in the presence of aluminium chloride in nitrobenzene at 80–90° for 45 min (81%) [1683].

m.p. 82–84° [1683]; ^{1}H NMR [1683], IR [1683], MS [1683].

(2-Bromophenyl)(5-chloro-2-hydroxyphenyl)methanone

[92739-90-7] $C_{13}H_8BrClO_2$ mol.wt. 311.56



Described [1476] **p. 201**

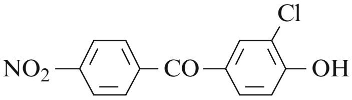
Synthesis

- Preparation by reaction of 2-bromobenzoyl chloride with 4-chloroanisole in the presence of aluminium chloride in refluxing methylene chloride overnight (96%) [1682].

tan solid [1682]; ^{1}H NMR [1682].

(3-Chloro-4-hydroxyphenyl)(4-nitrophenyl)methanone

[99768-27-1] $C_{13}H_8ClNO_4$ mol.wt. 277.66



Described [1476] **p. 205**

Synthesis

- Also refer to: [1684].

Methyl ether [76442-91-6] $C_{14}H_{10}ClNO_4$ mol.wt. 291.69

- Obtained by reaction of p-nitrobenzoyl chloride with 2-chloroanisole in the presence of aluminium chloride in carbon disulfide [1684].
- Also obtained by reaction of dimethyl sulfate with 3-chloro-4-hydroxy-4′-nitrobenzophenone [1684].
- Also obtained by heating for 4 h a mixture of 2-chloroanisole and 4-nitrobenzoyl chloride,
  - in the presence of ferric chloride (89%) [1585];
  - in the presence of aluminium chloride (85%) [1585].

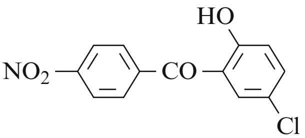
b.p.$_{12}$ 263° [1585]; m.p. 166° [1585,1684].

2,4-Dinitrophenylhydrazone (of the methyl ether)

$C_{20}H_{14}ClN_5O_7$ mol.wt. 471.81
m.p. 237° [1684].

(5-Chloro-2-hydroxyphenyl)(4-nitrophenyl)methanone

[84443-36-7] $C_{13}H_8ClNO_4$ mol.wt. 277.66



Described [1476] **p. 206**

Methyl ether [76442-97-2] $C_{14}H_{10}ClNO_4$ mol.wt. 291.69

- Obtained by heating for 4 h a mixture of 4-chloroanisole and 4-nitrobenzoyl chloride,

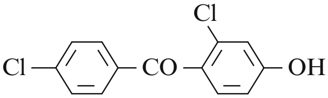
  - in the presence of ferric chloride (89%) [1585];
  - in the presence of aluminium chloride (85%) [1585] and in carbon disulfide [1684].

- Also obtained by reaction of dimethyl sulfate with 5-chloro-2-hydroxy-4′-nitrobenzophenone in the presence of aqueous sodium hydroxide [1684].

b.p.$_{12}$ 265° [1585]; m.p. 114° [1684, 1585].

(2-Chloro-4-hydroxyphenyl)(4-chlorophenyl)methanone

[949492-38-0] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



New compound

Synthesis

- Obtained by treatment of its methyl ether with aqueous hydrobromic acid in acetic acid [1685].

Methyl ether [76442-93-8] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14

- Obtained by heating for 4 h a mixture of 3-chloroanisole and 4-chlorobenzoyl chloride,
  - in the presence of ferric chloride (91%) [1585];
  - in the presence of aluminium chloride (89%) [1585].
- Also obtained by reaction of 2-chloro-4-methoxyphenylmagnesium iodide with 4-chloro-benzaldehyde in refluxing ethyl ether for 50 min [1686].
- Also obtained by reaction of 2-chloro-4-methoxybenzoyl chloride with chlorobenzene in the presence of aluminium chloride in carbon disulfide [1589].
- Also refer to: [1687].

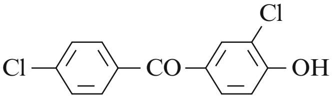
b.p.$_{12}$ 251° [1585]; m.p. 74° [1585], 73–74.5° [1686], 68–70° [1589].

2,4-Dinitrophenylhydrazone (of the methyl ether) $C_{20}H_{14}Cl_2N_4O_5$ mol. wt. 461.26

m.p. 213–217° [1686].

(3-Chloro-4-hydroxyphenyl)(4-chlorophenyl)methanone

[34189-58-7] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



Described [1476] **p. 207**

Methyl ether $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14

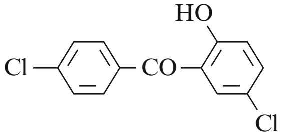
- Obtained by heating for 4 h a mixture of 2-chloroanisole and 4-chlorobenzoyl chloride,

- in the presence of ferric chloride (89%) [1585];
- in the presence of aluminium chloride (84%) [1585].

b.p.$_{12}$ 250° [1585]; m.p. 126° [1585].

(5-Chloro-2-hydroxyphenyl)(4-chlorophenyl)methanone

[61785-37-3] $C_{13}H_8Cl_2O_2$ mol.wt. 267.11



Described [1476] **p. 209**

Synthesis

– Preparation by Fries rearrangement of 4-chlorophenyl 4-chlorobenzoate in the presence of aluminium chloride in nitrobenzene at 80–90° for 45 min (94%) [1683].

m.p. 85–87° [1683]; ^{1}H NMR [1683], IR [1683], MS [1683].

Methyl ether [76442-96-1] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.14

– Obtained by heating for 4 h a mixture of 4-chloroanisole and 4-chlorobenzoyl chloride,

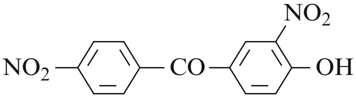
- in the presence of ferric chloride (89%) [1585];
- in the presence of aluminium chloride [1688], (84%) [1585].

– Also refer to: [1557].

b.p.$_{12}$ 245° [1585]; m.p. 131° [1688, 1585], 90° [1557]; IR [1557].

(4-Hydroxy-3-nitrophenyl)(4-nitrophenyl)methanone

[37567-41-2] $C_{13}H_8N_2O_6$ mol.wt. 288.22



Described [1476] **p. 212**

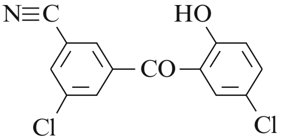
Methyl ether [873987-05-4] $C_{14}H_{10}N_2O_6$ mol.wt. 302.24

– Obtained by Friedel–Crafts acylation of anisole with 4-nitrobenzoyl chloride and subsequent nitration of the obtained ketone [1649].

– Also obtained by nitration of 4-methoxy-4′-nitrobenzophenone with potassium nitrate in concentrated sulfuric acid at 20° for 5 h [1644].

3-Chloro-5-(5-chloro-2-hydroxybenzoyl)benzonitrile

[329944-65-2] $C_{14}H_7Cl_2NO_2$ mol.wt. 292.07



New compound

Synthesis

– Obtained by treatment of its methyl ether with boron tribromide in methylene chloride, from −78° to r.t. (quantitative yield) [1682].

yellow solid [1682]; ^{1}H NMR [1682], ^{13}C NMR [1682].

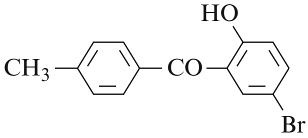
Methyl ether [329944-63-0] $C_{15}H_9Cl_2NO_2$ mol.wt. 306.15

- Obtained by treatment of (3-bromo-5-chlorophenyl)(5-chloro-2-methoxyphenyl) methanone with sodium cyanide and cuprous iodide in the presence of $Pd(PPh_3)_4$ in refluxing acetonitrile for 45 min (56%) [1682].

^{1}H NMR [1682], ^{13}C NMR [1682].

(5-Bromo-2-hydroxyphenyl)(4-methylphenyl)methanone

[215380-62-4] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



New compound

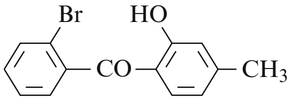
Synthesis

- Obtained by reaction of 2-(1*H*-benzotriazol-1-ylcarbonyl)-4-bromophenol with 4-methylphenylmagnesium bromide at 25° for 4 h (66%) [1689].

white crystals; m.p. 85–87° [1689];
^{1}H NMR [1689], ^{13}C NMR [1689].

(2-Bromophenyl)(2-hydroxy-4-methylphenyl)methanone

[1023758-43-1] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



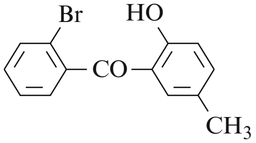
New compound

Syntheses

- Obtained by Fries rearrangement of m-cresyl 2-bromobenzoate in the presence of PPA [1690].
- Also obtained by Friedel–Crafts acylation of m-cresol with o-bromobenzoic acid in the presence of PPA [1690].

(2-Bromophenyl)(2-hydroxy-5-methylphenyl)methanone

[55270-73-0] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



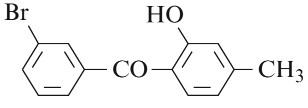
Described [1476] **p. 231**

Synthesis

- Also refer to: [1691].

(3-Bromophenyl)(2-hydroxy-4-methylphenyl)methanone

[218784-25-9] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



Described [1476] **p. 231**

Syntheses

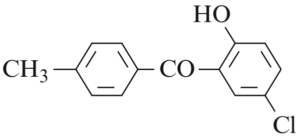
- Also obtained by Friedel–Crafts acylation of m-cresol with m-bromobenzoic acid in a mixture of graphite and methane sulfonic acid at 120° for 5.5 h (63%) [1598].
- Also refer to: [1692].

m.p. 88° [1598]; ^{1}H NMR [1598], ^{13}C NMR [1598].

(5-Chloro-2-hydroxyphenyl)(4-methylphenyl)methanone

[116544-78-6] $C_{14}H_{11}ClO_2$ mol.wt. 246.69

Described [1476] **p. 234**



Methyl ether [52980-99-1] $C_{15}H_{13}ClO_2$ mol.wt. 260.72

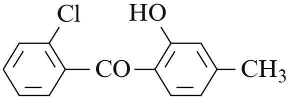
- Obtained by heating for 4 h a mixture of 4-chloroanisole and 4-methybenzoyl chloride,
  - in the presence of ferric chloride (87%) [1585];
  - in the presence of aluminium chloride (81%) [1585].
- Also obtained by methylation of 5-chloro-2-hydroxy-4′-methylbenzophenone [1557].

b.p.$_{12}$ 215° [1585]; m.p. 131° [1557], 103° [1585]; IR [1557].

(2-Chlorophenyl)(2-hydroxy-4-methylphenyl)methanone

[107623-97-2] $C_{14}H_{11}ClO_2$ mol.wt. 246.69

Described [1476] **p. 235**



Syntheses

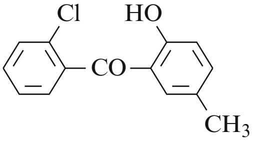
- Obtained by Fries rearrangement of m-cresyl 2-chlorobenzoate in the presence of PPA [1690].
- Also obtained by Friedel–Crafts acylation of m-cresol with o-chlorobenzoic acid,
  - in the presence of PPA [1690];
  - in a mixture of graphite and methanesulfonic acid at 120° for 3.5 h (80%) [1598].

^{1}H NMR [1598], ^{13}C NMR [1598], IR [1598].

(2-Chlorophenyl)(2-hydroxy-5-methylphenyl)methanone

[6280-52-0] $C_{14}H_{11}ClO_2$ mol.wt. 246.69

Described [1476] **p. 235**

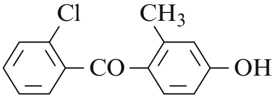


Synthesis

- Also refer to: [1691].

(2-Chlorophenyl)(4-hydroxy-2-methylphenyl)methanone

[92103-15-6] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



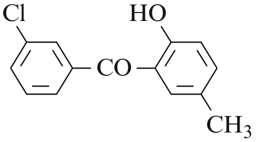
Described [1476] **p. 235**

Syntheses

- Obtained by Fries rearrangement of m-cresyl 2-chlorobenzoate in the presence of PPA [1690].
- Also obtained by Friedel–Crafts acylation of m-cresol with o-chlorobenzoic acid in the presence of PPA [1690].

(3-Chlorophenyl)(2-hydroxy-5-methylphenyl)methanone

[6280-54-2] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



Described [1476] **p. 237**

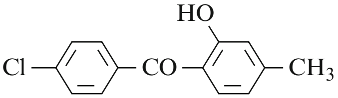
Syntheses

- Preparation by Fries rearrangement of p-cresyl m-chloro-benzoate in the presence of aluminium chloride in nitrobenzene at 80–90° for 45 min (90%) [1683].
- Also refer to: [1584,1691].

m.p. 71–73° [1683]; ^1H NMR [1683], IR [1683], MS [1683].

(4-Chlorophenyl)(2-hydroxy-4-methylphenyl)methanone

[107622-28-6] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



Described [1476] **p. 238**

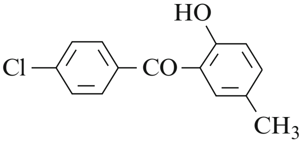
Synthesis

- Also obtained by Friedel–Crafts acylation of m-cresol with p-toluic acid in a mixture of graphite and methanesulfonic acid at 120° for 4 h (66%) [1598].

white crystals; m.p. 81° [1598];
^1H NMR [1598], ^{13}C NMR [1598], IR [1598].

(4-Chlorophenyl)(2-hydroxy-5-methylphenyl)methanone

[6279-05-6] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



Described [1476] **p. 238**

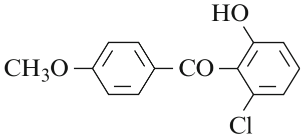
Synthesis

- Preparation by Fries rearrangement of p-cresyl p-chlorobenzoate in the presence of aluminium chloride in nitrobenzene at 80–90° for 45 min (87%) [1683].

m.p. 85–87° [1683]; ^1H NMR [1683], IR [1683], MS [1683].

(2-Chloro-6-hydroxyphenyl)(4-methoxyphenyl)methanone

[1005486-58-7] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



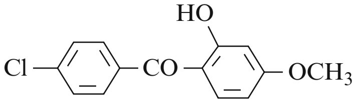
New compound

Synthesis

- Refer to: [1693].

(4-Chlorophenyl)(2-hydroxy-4-methoxyphenyl)methanone

[85-28-9] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



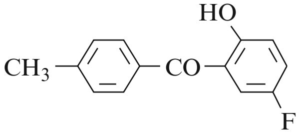
Described [1476] **p. 242**

Synthesis

- Also refer to: [1694].

(5-Fluoro-2-hydroxyphenyl)(4-methylphenyl)methanone

[62433-29-8] $C_{14}H_{11}FO_2$ mol.wt. 230.24



Described [1476] **p. 243**

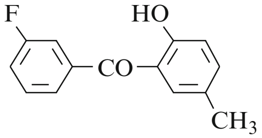
Synthesis

- Preparation by Fries rearrangement of 4-fluorophenyl p-toluate in the presence of aluminium chloride in nitrobenzene at 80–90° for 45 min (89%) [1683].

m.p. 80–82° [1683]; ^1H NMR [1683], IR [1683], MS [1683].

(3-Fluorophenyl)(2-hydroxy-5-methylphenyl)methanone

[55270-80-9] $C_{14}H_{11}FO_2$ mol.wt. 230.24



Described [1476] **p. 243**

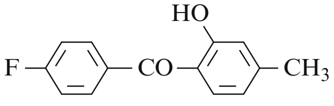
Synthesis

- Preparation by Fries rearrangement of 4-methylphenyl 3-fluorobenzoate in the presence of aluminium chloride in nitrobenzene at 80–90° for 45 min (85%) [1683].

m.p. 45–47° [1683]; ^1H NMR [1683], IR [1683], MS [1683].

(4-Fluorophenyl)(2-hydroxy-4-methylphenyl)methanone

[108294-71-9] $C_{14}H_{11}FO_2$ mol.wt. 230.24



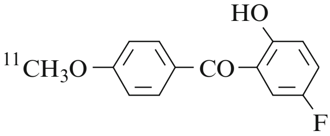
Described [1476] **p. 244**

Synthesis

- Also refer to: [1695].

(5-Fluoro-2-hydroxyphenyl)[4-methoxy-(^{11}C)phenyl]methanone

[161585-22-4] $C_{14}H_{11}FO_3$ mol.wt. 245.23



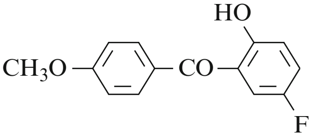
New compound

Synthesis

- Obtained by reaction of ^{11}C methyl iodide with 5-fluoro-2,4′-dihydroxybenzophenone in the presence of 0.8 M aqueous sodium hydroxide in DMF at 150° [1696].

(5-Fluoro-2-hydroxyphenyl)(4-methoxyphenyl)methanone

[727-93-5] $C_{14}H_{11}FO_3$ mol.wt. 246.24



Described [1476] **p. 244**

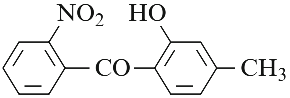
Syntheses

- Obtained by Fries rearrangement of 4-fluorophenyl 4-methoxybenzoate with titanium tetrachloride, first at 120°, then at 160° for 20 min (46%) [1696].
- Also refer to: [1697].

yellow powder; m.p. 79.5° [1696]; b.p.$_{30}$ 260° [1697]; ^{1}H NMR [1696], MS [1696].

(2-Hydroxy-4-methylphenyl)(2-nitrophenyl)methanone

[1023758-44-2] $C_{14}H_{11}NO_4$ mol.wt. 257.25



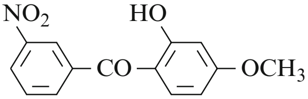
New compound

Syntheses

- Obtained by Fries rearrangement of m-cresyl 2-nitrobenzoate in the presence of PPA [1690].
- Also obtained by Friedel–Crafts acylation of m-cresol with o-nitrobenzoic acid in the presence of PPA [1690].

(2-Hydroxy-4-methoxyphenyl)(3-nitrophenyl)methanone

[126077-53-0] $C_{14}H_{11}NO_5$ mol.wt. 273.25



Described [1476] **p. 250**

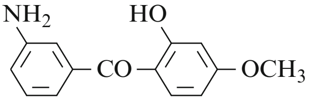
Synthesis

- Also obtained by Friedel–Crafts acylation of 1,3-dimethoxybenzene with m-nitrobenzoyl chloride in the presence of aluminium chloride in methylene chloride at 40° for 3 h (72%) [1698].

^{1}H NMR [1698], IR [1698], MS [1698].

(3-Aminophenyl)(2-hydroxy-4-methoxyphenyl)methanone

[126346-90-5]



$C_{14}H_{13}NO_3$ mol.wt. 243.26

New compound

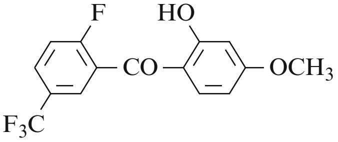
Synthesis

- Obtained by catalytic hydrogenation of 2-hydroxy-4-methoxy-3′-nitrobenzophenone over 5% Pd/C (90%) [1698].

^{1}H NMR [1698], IR [1698], MS [1698].

[2-Fluoro-5-(trifluoromethyl)phenyl](2-hydroxy-4-methoxyphenyl)methanone

[885481-51-6]



$C_{15}H_{10}F_4O_3$ mol.wt. 314.24

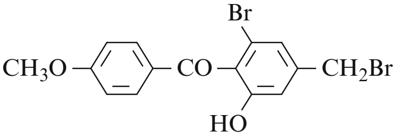
New compound

Synthesis

- Refer to: [1699].

[2-Bromo-4-(bromomethyl)-6-hydroxyphenyl](4-methoxyphenyl)methanone

[1005488-92-5]



$C_{15}H_{12}Br_2O_3$ mol.wt. 400.07

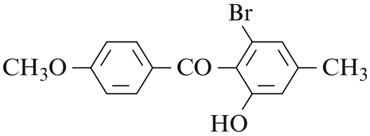
New compound

Synthesis

- Refer to: [1693].

(2-Bromo-6-hydroxy-4-methylphenyl)(4-methoxyphenyl)methanone

[1005488-89-0]



$C_{15}H_{13}BrO_3$ mol.wt. 321.17

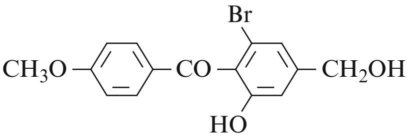
New compound

Synthesis

- Refer to: [1693].

[2-Bromo-6-hydroxy-4-(hydroxymethyl)phenyl](4-methoxyphenyl)methanone

[1005488-91-4]



$C_{15}H_{13}BrO_4$ mol.wt. 337.17

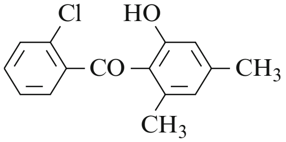
New compound

Synthesis

- Refer to: [1693].

(2-Chlorophenyl)(2-hydroxy-4,6-dimethylphenyl)methanone

[1019637-56-9] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



New compound

Synthesis

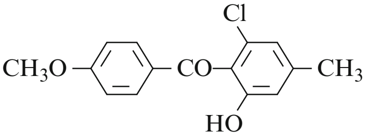
- Obtained by Friedel–Crafts acylation of 3,5-dimethylphenol with 2-chlorobenzoyl chloride in the presence of aluminium chloride in chlorobenzene [1700].

Methyl ether [1019637-58-1] $C_{16}H_{15}ClO_2$ mol.wt. 274.75

- Obtained by reaction of dimethyl sulfate with 2′-chloro-2-hydroxy-4,6-dimethylbenzophenone in the presence of potassium hydroxide in acetonitrile [1700].

(2-Chloro-6-hydroxy-4-methylphenyl)(4-methoxyphenyl)methanone

[1005486-64-5] $C_{15}H_{13}ClO_3$ mol.wt. 276.72



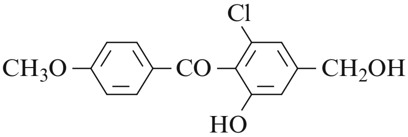
New compound

Synthesis

- Refer to: [1693].

[2-Chloro-6-hydroxy-4-(hydroxymethyl)phenyl](4-methoxyphenyl)methanone

[1005486-69-0] $C_{15}H_{13}ClO_4$ mol.wt. 292.72



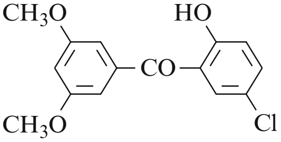
New compound

Synthesis

- Refer to: [1693].

(5-Chloro-2-hydroxyphenyl)(3,5-dimethoxyphenyl)methanone

[329944-55-0] $C_{15}H_{13}ClO_4$ mol.wt. 292.72



New compound

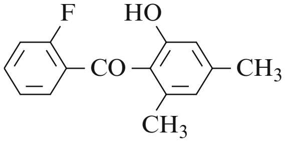
Synthesis

- A solution of 2-bromo-4-chlorophenol (4 mmol) in THF was cooled to −78°. A solution of *n*-butyllithium in hexanes (8.8 mmol) was added over 5 min, and the resulting mixture was stirred at −78° for 1 h. A solution of N-methyl-N-3,5-trimethoxybenzamide (4 mmol) in THF was added over 4 min and the resulting mixture was stirred at −78° for 1.25 h and then at r.t. for 14 h (20%) [1682].

yellow crystals [1682]; ^1H NMR [1682], ^{13}C NMR [1682].

(2-Fluorophenyl)(2-hydroxy-4,6-dimethylphenyl)methanone

[1019637-57-0] $C_{15}H_{13}FO_2$ mol.wt. 244.27



New compound

Synthesis

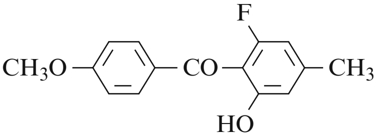
- Obtained by Friedel–Crafts acylation of 3,5-dimethylphenol with 2-fluorobenzoyl chloride in the presence of aluminium chloride in chlorobenzene [1700].

Methyl ether [1019637-59-2] $C_{16}H_{15}FO_2$ mol.wt. 258.29

- Obtained by reaction of dimethyl sulfate with 2′-fluoro-2-hydroxy-4,6-dimethylbenzophenone in the presence of potassium hydroxide in acetonitrile [1700].

(2-Fluoro-6-hydroxy-4-methylphenyl)(4-methoxyphenyl)methanone

[1005488-56-1] $C_{15}H_{13}FO_3$ mol.wt. 260.26



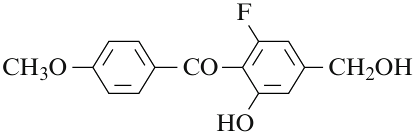
New compound

Synthesis

- Refer to: [1693].

[2-Fluoro-6-hydroxy-4-(hydroxymethyl)phenyl](4-methoxyphenyl)methanone

[1005488-58-3] $C_{15}H_{13}FO_4$ mol.wt. 276.26



New compound

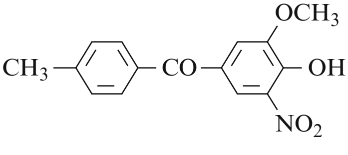
Synthesis

- Refer to: [1693].

(4-Hydroxy-3-methoxy-5-nitrophenyl)(4-methylphenyl)methanone

(Tolcapone, 3-O-methyltolcapone) (Ro 40-7591)

[134612-80-9] $C_{15}H_{13}NO_5$ mol.wt. 287.27



Described [1476] **p. 277**

Syntheses

- Also obtained by reaction of 70% nitric acid with (4-hydroxy-3-methoxyphenyl)(4-methylphenyl)-methanone in acetic acid at 0° for 30 min (88%) [1701].
- A plasma metabolite of tolcapone in human [1702].

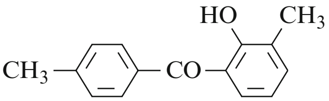
  N.B.: Conjugaison (O-methylation by COMT).
- Also refer to: [1703,1704].

  white solid [1701];

m.p. 137–139° [1703,1704], 135–137° [1701];
^{1}H NMR [1701], ^{13}C NMR [1701], MS [1701].

(2-Hydroxy-3-methylphenyl)(4-methylphenyl)methanone

[887344-78-7] $C_{15}H_{14}O_2$ mol.wt. 226.27



New compound

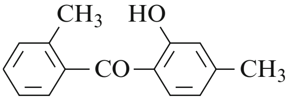
Synthese

- Obtained by reaction of 2-(1*H*-benzotriazol-1-ylcarbonyl)-6-methylphenol with 4-methylphenylmagnesium bromide at 25° for 4 h (74%) [1689].
- Also refer to: [1705].

white crystals [1689]; m.p. 62–63° [1689,1705];
^{1}H NMR [1689], ^{13}C NMR [1689].

(2-Hydroxy-4-methylphenyl)(2-methylphenyl)methanone

[1023758-42-0] $C_{15}H_{14}O_2$ mol.wt. 226.27



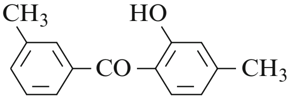
New compound

Syntheses

- Obtained by Fries rearrangement of m-cresyl o-toluate in the presence of PPA [1690].
- Also obtained by Friedel–Crafts acylation of m-cresol with o-toluic acid in the presence of PPA [1690].
- Also refer to: [1695].

(2-Hydroxy-4-methylphenyl)(3-methylphenyl)methanone

[92548-90-8] $C_{15}H_{14}O_2$ mol.wt. 226.27



Described [1476] **p. 279**

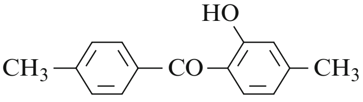
Syntheses

- Also obtained by Friedel–Crafts acylation of m-cresol with m-toluic acid in a mixture of graphite and methanesulfonic acid at 120° for 2 h (86%) [1598].
- Also refer to: [1692,1695].

^{1}H NMR [1598], ^{13}C NMR [1598], IR [1598].

(2-Hydroxy-4-methylphenyl)(4-methylphenyl)methanone

[81652-53-1] $C_{15}H_{14}O_2$ mol.wt. 226.27



Described [1476] **p. 279**

Syntheses

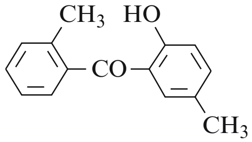
- Also obtained by Friedel–Crafts acylation of m-cresol with p-toluic acid in a mixture of graphite and methanesulfonic acid at 120° for 2 h (50%) [1598].

- Also refer to: [1584,1692,1695].

white crystals; m.p. 72° [1598]; ^{1}H NMR [1598], ^{13}C NMR [1598].

(2-Hydroxy-5-methylphenyl)(2-methylphenyl)methanone

[147029-79-6] $C_{15}H_{14}O_2$ mol.wt. 226.27



Described [1476] **p. 280**

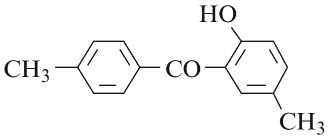
Synthesis

- Preparation by Fries rearrangement of 5-methylphenyl 2-methylbenzoate in the presence of aluminium chloride in nitrobenzene at 80–90° for 45 min (90%) [1683].

m.p. 71–73° [1683]; ^{1}H NMR [1683], IR [1683], MS [1683].

(2-Hydroxy-5-methylphenyl)(4-methylphenyl)methanone

[26880-95-5] $C_{15}H_{14}O_2$ mol.wt. 226.27



Described [1476] **p. 280**

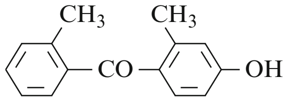
Syntheses

- Preparation by Fries rearrangement of p-tolyl p-toluate in the presence of aluminium chloride in nitrobenzene at 80–90° for 45 min (92%) [1683].

m.p. 75–77° [1683]; ^{1}H NMR [1683], IR [1683], MS [1683].

(4-Hydroxy-2-methylphenyl)(2-methylphenyl)methanone

[1023758-40-8] $C_{15}H_{14}O_2$ mol.wt. 226.27



New compound

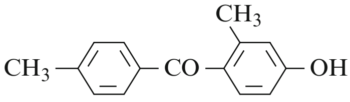
Syntheses

- Obtained by Fries rearrangement of m-cresyl o-toluate in the presence of PPA [1690].
- Also obtained by Friedel–Crafts acylation of m-cresol with o-toluic acid in the presence of PPA [1690].

(4-Hydroxy-2-methylphenyl)(4-methylphenyl)methanone

$C_{15}H_{14}O_2$ mol.wt. 226.27

Described [1476] **p. 281**

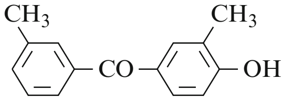


Methyl ether [41295-26-5] $C_{16}H_{16}O_2$ mol.wt. 240.30

- Refer to: [1706] (Japanese patent).

(4-Hydroxy-3-methylphenyl)(3-methylphenyl)methanone

[62064-85-1] $C_{15}H_{14}O_2$ mol.wt. 226.27



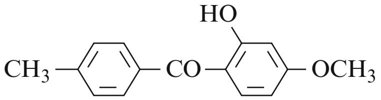
Described [1476] **p. 282**

Methyl ether [41295-28-7] $C_{16}H_{16}O_2$ mol.wt. 240.30

- Refer to: [1707,1708].

(2-Hydroxy-4-methoxyphenyl)(4-methylphenyl)methanone

[1641-17-4] $C_{15}H_{14}O_3$ mol.wt. 242.27



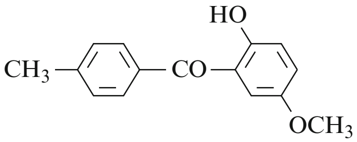
Described [1476] **p. 282**

Synthesis

- Also obtained by an intramolecular acyl radical *ipso* substitution (33%) [1608].

(2-Hydroxy-5-methoxyphenyl)(4-methylphenyl)methanone

[4998-50-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



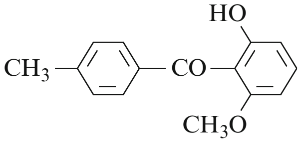
New compound

Synthesis

- Also obtained by an intramolecular acyl radical *ipso* substitution (28%) [1608].

(2-Hydroxy-6-methoxyphenyl)(4-methylphenyl)methanone

[329235-51-0] $C_{15}H_{14}O_3$ mol.wt. 242.27



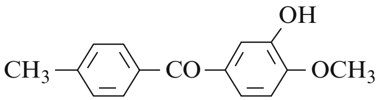
New compound

Synthesis

- Also obtained by an intramolecular acyl radical *ipso* substitution (37%) [1608].

(3-Hydroxy-4-methoxyphenyl)(4-methylphenyl)methanone

[210704-43-1] $C_{15}H_{14}O_3$ mol.wt. 242.27



New compound

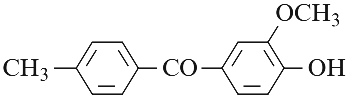
Synthesis

- Obtained by reaction of p-methylbenzoyl chloride with veratrole in the presence of aluminium chloride in methylene chloride at 32–35° [1709].

Li salt	[210704-41-9]	$C_{15}H_{13}O_3Li$	mol.wt. 248.21
Na salt	[210704-39-5]	$C_{15}H_{13}O_3Na$	mol.wt. 264.26

(4-Hydroxy-3-methoxyphenyl)(4-methylphenyl)methanone

[134612-39-8] $C_{15}H_{14}O_3$ mol.wt. 242.27



Described [1476] **p. 283**

Syntheses

- Also obtained by refluxing a mixture of its benzyl ether, 10% Pd/C and ammonium formate in methanol under nitrogen for 30 min (92%) [1701].
- Also obtained by reaction of p-methylbenzoyl chloride with veratrole in the presence of aluminium chloride in methylene chloride at 32–35° [1709].
- Also refer to: [1703,1704].

white solid [1701];
m.p. 103–105° [1703,1704], 98–100° [1701];
^{1}H NMR [1701], ^{13}C NMR [1701], MS [1701].

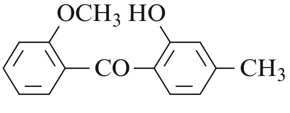
Benzyl ether [134612-29-6] $C_{22}H_{20}O_3$ mol.wt. 332.40

- Obtained by adding sodium tert-butoxide to a stirred suspension of (4-benzyloxy-3-methoxyphenyl)(4-methylphenyl)methanol in toluene at r.t. under nitrogen, followed by cyclohexanone. Then, the mixture was refluxed for 1 h (89%) [1701].
- Also refer to: [1703,1704].

white solid [1701];
m.p. 79–81° [1703,1704], 70–72° [1701];
^{1}H NMR [1701], ^{13}C NMR [1701], MS [1701].

(2-Hydroxy-4-methylphenyl)(2-methoxyphenyl)methanone

[1023758-41-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



New compound

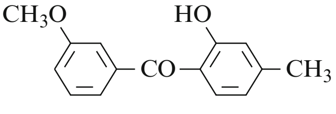
Synthesis

- Obtained by reaction of o-methoxybenzoic acid with m-cresol in the presence of PPA at 70° for 1 h (30%) [1690].

orange needles; m.p. 75–80° [1690];
^{1}H NMR [1690], IR [1690], UV [1690].

(2-Hydroxy-4-methylphenyl)(3-methoxyphenyl)methanone

[1023758-48-6] $C_{15}H_{14}O_3$ mol.wt. 242.27



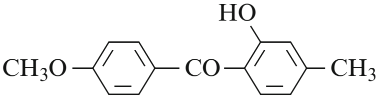
New compound

Synthesis

- Obtained by reaction of m-methoxybenzoic acid with m-cresol in the presence of PPA at 70° for 1 h [1690].

(2-Hydroxy-4-methylphenyl)(4-methoxyphenyl)methanone

[108478-27-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



Described [1476] **p. 283**

Syntheses

– Also obtained by reaction of p-methoxybenzoic acid with m-cresol,

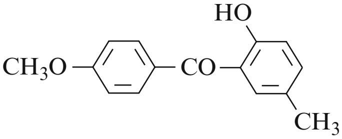
- in the presence of methanesulfonic acid at 140° (90%) [1692];
- in the presence of PPA at 70° for 1 h [1690];
- in a mixture of graphite and methanesulfonic acid at 120° for 2 h (51%) [1598].

– Also refer to: [1584].

white crystals; m.p. 97° [1598];
^{1}H NMR [1598], ^{13}C NMR [1598], IR [1598].

(2-Hydroxy-5-methylphenyl)(4-methoxyphenyl)methanone

[26880-96-6] $C_{15}H_{14}O_3$ mol.wt. 242.27



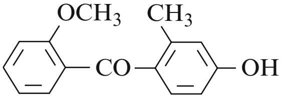
Described [1476] **p. 284**

Synthesis

– Also refer to: [1691].

(4-Hydroxy-2-methylphenyl)(2-methoxyphenyl)methanone

[1023758-39-5] $C_{15}H_{14}O_3$ mol.wt. 242.27



New compound

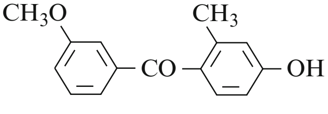
Synthesis

– Obtained by reaction of o-methoxybenzoic acid with m-cresol in the presence of PPA at 70° for 1 h (20%) or for 6 h (70%) [1690].

white crystals; m.p. 105–110° [1690];
^{1}H NMR [1690], IR [1690], UV [1690].

(4-Hydroxy-2-methylphenyl)(3-methoxyphenyl)methanone

[1023758-47-5] $C_{15}H_{14}O_3$ mol.wt. 242.27

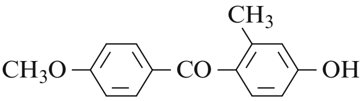


New compound

Synthesis

– Obtained by reaction of m-methoxybenzoic acid with m-cresol in the presence of PPA at 70° for 1 h [1690].

(4-Hydroxy-2-methylphenyl)(4-methoxyphenyl)methanone



$C_{15}H_{14}O_3$ mol.wt. 242.27

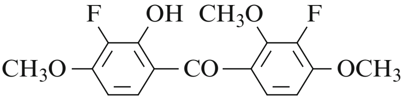
New compound

Synthesis

- Obtained by partial demethylation of 4,4′-dimethoxy-2-methylbenzophenone by boiling for 15 min with pyridinium chloride (75%) [1710].

(3-Fluoro-2,4-dimethoxyphenyl)(3-fluoro-2-hydroxy-4-methoxyphenyl)methanone

[1018451-13-2]



$C_{16}H_{14}F_2O_5$ mol.wt. 324.28

New compound

Synthesis

- Obtained by partial demethylation of 2,2′,4,4′-tetra-methoxy-3,3′-difluorobenzophenone with boron trichloride in methylene chloride at 0° for 1 h [1711].

MS [1711].

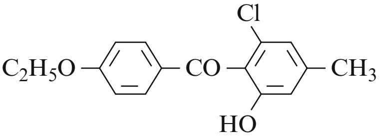
Methyl ether [1018451-12-1] $C_{17}H_{16}F_2O_5$ mol.wt. 338.31

- Obtained by heating a mixture of methyl 3-fluoro-2,4-dimethoxybenzoate (3 mmol), 2-fluoro-1,3-dimethoxybenzene (3 mmol) and Eaton's acid (a 9% solution of P_2O_5 in methanesulfonic acid) (20 ml) at 80° for 3 h (97%) [1711].

  yellow orange liquid [1711]; ^1H NMR [1711], ^{19}F NMR [1711], MS [1711].

(2-Chloro-6-hydroxy-4-methylphenyl)(4-ethoxyphenyl)methanone

[1005486-84-9]



$C_{16}H_{15}ClO_3$ mol.wt. 290.75

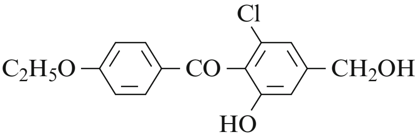
New compound

Synthesis

- Refer to: [1693].

[2-Chloro-6-hydroxy-4-(hydroxymethyl)phenyl](4-ethoxyphenyl)methanone

[1005486-86-1]



$C_{16}H_{15}ClO_4$ mol.wt. 306.74

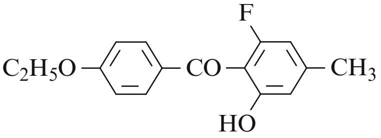
New compound

Synthesis

- Refer to: [1693].

(4-Ethoxyphenyl)(2-fluoro-6-hydroxy-4-methylphenyl)methanone

[1005486-93-0] $C_{16}H_{15}FO_3$ mol.wt. 274.29



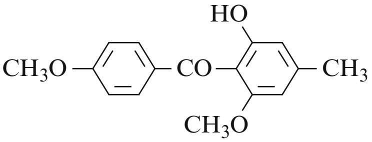
New compound

Synthesis

- Refer to: [1693].

(2-Hydroxy-6-methoxy-4-methylphenyl)(4-methoxyphenyl)methanone

[1005488-48-1] $C_{16}H_{16}O_4$ mol.wt. 272.30



New compound

Synthesis

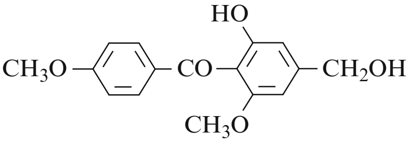
- Refer to: [1693].

Acetate [1005488-49-2] $C_{18}H_{18}O_5$ mol.wt. 314.34

- Refer to: [1693].

[2-Hydroxy-4-(hydroxymethyl)-6-methoxyphenyl](4-methoxyphenyl)methanone

[1005488-50-5] $C_{16}H_{16}O_5$ mol.wt. 288.30



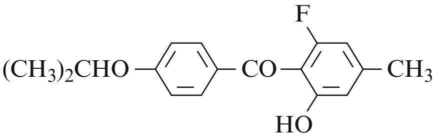
New compound

Synthesis

- Refer to: [1693].

(2-Fluoro-6-hydroxy-4-methylphenyl)[4-(1-methylethoxy)phenyl]methanone

[1005488-15-2] $C_{17}H_{17}FO_3$ mol.wt. 288.32



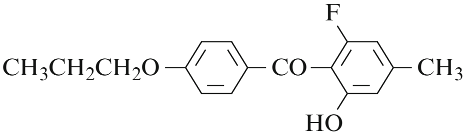
New compound

Synthesis

- Refer to: [1693].

(2-Fluoro-6-hydroxy-4-methylphenyl)(4-propoxyphenyl)methanone

[1005487-98-8] $C_{17}H_{17}FO_3$ mol.wt. 288.32



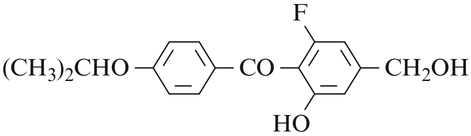
New compound

Synthesis

- Refer to: [1693].

[2-Fluoro-6-hydroxy-4-(hydroxymethyl)phenyl][4-(1-methylethoxy)phenyl] methanone

[1005488-17-4]



$C_{17}H_{17}FO_4$ mol.wt. 304.32

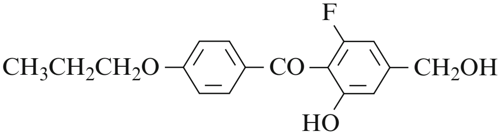
New compound

Synthesis

- Refer to: [1693].

[2-Fluoro-6-hydroxy-4-(hydroxymethyl)phenyl](4-propoxyphenyl)methanone

[1005487-99-9]



$C_{17}H_{17}FO_4$ mol.wt. 304.32

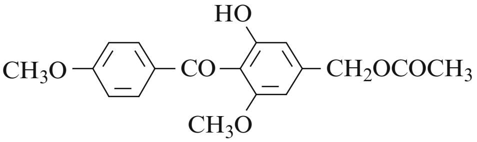
New compound

Synthesis

- Refer to: [1693].

[4-[(Acetyloxy)methyl]-2-hydroxy-6-methoxyphenyl](4-methoxyphenyl) methanone

[1005488-51-6]



$C_{18}H_{18}O_6$ mol.wt. 330.34

New compound

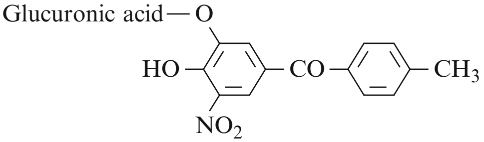
Synthesis

- Refer to: [1693].

[2-Hydroxy-5-(4-methylbenzoyl)-3-nitrophenyl]-β-D-glucopyranosiduronic acid

(Tolcapone, 3-O-β,D-glucuronide) (Ro 61-1448)

[204853-33-8]



$C_{20}H_{19}NO_{11}$ mol.wt. 449.37

New compound

Isolation from natural sources

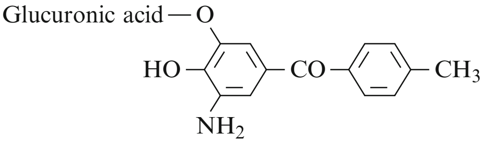
- A plasma metabolite of tolcapone in human [1702].

N.B.: Conjugaison (glucuronidation by glucuronyltransferase).

[3-Amino-2-hydroxy-5-(4-methylbenzoyl)phenyl]-β-D-glucopyranosiduronic acid

(Tolcapone, Amine glucuronide)

[254902-30-2]



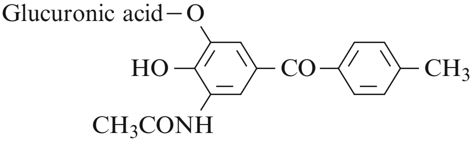
$C_{20}H_{21}NO_9$ mol.wt. 419.39

New compound

Synthesis

- From Tolcapone via reductive metabolism in human [1702].

[3-Acetamido-5-(glucuronyloxy)-4-hydroxyphenyl](4-methylphenyl)methanone
(Tolcaponne, N-acetylamino glucuronide)



$C_{22}H_{20}NO_{10}$ mol.wt. 449.37

New compound

Synthesis

– From Tolcapone via reductive metabolism in human [1702].

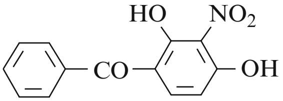
2.2 Dihydroxybenzophenones [1476] p. 364

2.2.1 Hydroxy Groups Located on the Same Ring [1476] p. 364

2.2.1.1 Substituents Located on the Hydroxylated Ring [1476] p. 364

(2,4-Dihydroxy-3-nitrophenyl)phenylmethanone

[59746-91-7]



$C_{13}H_9NO_5$ mol.wt. 259.22

Described [1476] **p. 371**

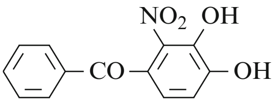
Synthesis

– Also refer to: [1543].

m.p. 228° [1543].
USE: Stabilization PVC [1543].

(3,4-Dihydroxy-2-nitrophenyl)phenylmethanone

[383382-84-1]



$C_{13}H_9NO_5$ mol.wt. 259.22

New compound

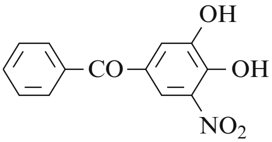
Synthesis

– Obtained from 4-hydroxy-3-methoxy-2-nitrobenzophenone by ether cleavage with aluminium chloride in a pyridine/1, 2-dichloroethane mixture at 100° for 30 min (81%) [1597].

orange crystals [1597]; ^{1}H NMR [1597], ^{13}C NMR [1597], IR [1597].

(3,4-Dihydroxy-5-nitrophenyl)phenylmethanone

[125628-96-8]



$C_{13}H_9NO_5$ mol.wt. 259.22

Described [1476] **p. 373**

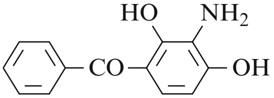
Synthesis

– Also refer to: [1712] (compound **7a**, n = 0).

m.p. 134–136° [1712]; ^{1}H NMR [1712], ^{13}C NMR [1712], IR [1712].

(3-Amino-2,4-dihydroxyphenyl)phenylmethanone

[87119-03-7] $C_{13}H_{11}NO_3$ mol.wt. 229.24



Described [1476] **p. 373**

Syntheses

– Obtained by treatment of its dimethyl ether,

- with aluminium chloride in refluxing toluene for 2 h (45–60%) [1529];
- with boron tribromide at r.t. for 3 h (60%) [1529].

– Also obtained by reaction of iso-amylamine with 2,3,4-trihydroxybenzophenone in the presence of lithium perchlorate in methanol (30%) or by electrochemical oxidation in acetic acid buffer (pH ca. 4.5) at r.t. [1713].

– Also obtained by reaction of 1-isopropyl-2-methylpropylamine with 2,3,4-trihydroxy-benzophenone in the presence of lithium perchlorate in methanol (55%) or by electrochemical oxidation in acetic acid buffer (pH ca. 4.5) at r.t. [1713].

– Also obtained by reaction of diisopropylamine with 2,3,4-trihydroxybenzophenone in the presence of lithium perchlorate in methanol or by electrochemical oxidation in acetic acid buffer (pH ca. 4.5) at r.t. [1713].

– Also obtained by reaction of 1,2-dimethylpropylamine with 2,3,4-trihydroxybenzophenone in the presence of lithium perchlorate in methanol (40%) or by electrochemical oxidation in acetic acid buffer (pH ca. 4.5) at r.t. [1713].

– Also obtained by reaction of α-phenylbenzylamine with 2,3,4-trihydroxybenzophenone in the presence of lithium perchlorate in methanol at 25° (72%) or by electrolysis [1714].

– Also obtained by reaction of phenylmethylamine with 2,3,4-trihydroxybenzophenone in the presence of lithium perchlorate in methanol at 20° (62%) or by electrolysis [1715].

– Also refer to: [1533,1595,1716–1718].

m.p. 195–197° [1714], 186–188° [1715];
^{1}H NMR [1713], ^{13}C NMR [1713].

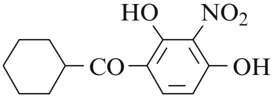
BIOLOGICAL ACTIVITY: Toxicity [1529]; potent neuroprotective agent in vitro [1529].

Dimethyl ether [253681-20-8] $C_{15}H_{15}NO_3$ mol.wt. 257.29

– Obtained by Friedel–Crafts acylation of 2,6-dimethoxyaniline with benzoic acid in the presence of PPA at 80° for 8 h (45–55%) [1529].

Cyclohexyl(2,4-dihydroxy-3-nitrophenyl)methanone

[909255-41-0] $C_{13}H_{15}NO_5$ mol.wt. 265.27



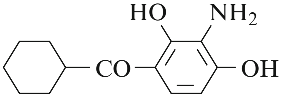
New compound

Synthesis

– Obtained by reaction of cyclohexanecarboxylic acid with 2-nitroresorcinol in the presence of PPA at 70–80° for 5 h (60%) [1595].

(3-Amino-2,4-dihydroxyphenyl)cyclohexylmethanone

[909255-14-7] $C_{13}H_{17}NO_3$ mol.wt. 235.28



New compound

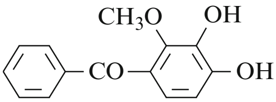
Synthesis

– Obtained by treatment of cyclohexyl(2,4-dihydroxy-3-nitrophenyl)methanone with zinc in acetic acid at 20° (76%) [1595].

m.p. 124–126° [1595]; ^{1}H NMR [1595], ^{13}C NMR [1595].

(3,4-Dihydroxy-2-methoxyphenyl)phenylmethanone

[177703-29-6] $C_{14}H_{12}O_4$ mol.wt. 244.25



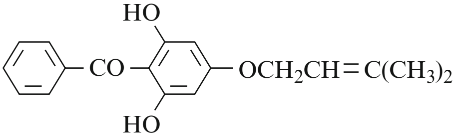
Described [1476] **p. 380**

Synthesis

– Also refer to: [1719].

[2,6-Dihydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]phenylmethanone

[70219-83-9] $C_{18}H_{18}O_4$ mol.wt. 298.34



Described [1476] **p. 386**

Synthesis

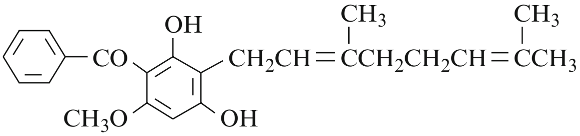
– Also obtained by treatment of 2,4,6-trihydroxy-benzophenone with prenyl di-n-cyclohexyl-sulfonium tetrafluoroborate salt (1 equiv) in the presence of Hünig's base (1.1 equiv) in methylene chloride at r.t. for 7 h (10%) [1536].

Isolation from natural sources

– From the aerial parts of *Helichrysum asperum* (Thunb.) Hilliard et Burtt. var. *albidulum* Hilliard (DC) [1720].

[3-(3,7-Dimethyl-2,6-octadienyl)-2,4-dihydroxy-6-methoxyphenyl]phenylmethanone *(E) (Marupone)*

[53948-11-1] $C_{24}H_{28}O_4$ mol.wt. 380.48



New compound

Isolation from natural sources

– From *Moronobea pulchra* Ducke (Guttiferae) [1536,1605].

Yellow crystals; m.p. 125–127° [1605]; ^{1}H NMR [1605], IR [1605], UV [1605], MS [1605].

Diacetate [53948-15-5] $C_{28}H_{32}O_6$ mol.wt. 464.56

- Obtained by treatment of marupone with acetic anhydride in the presence of pyridine at r.t. for 24 h [1605].

oil [1605]; ^{1}H NMR [1605], IR [1605].

Dimethyl ether *(E)* [53948-14-4] $C_{26}H_{32}O_4$ mol.wt. 408.54

- Obtained by treatment of marupone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 6 h [1605].
- Also obtained by reaction of 2,4,6-trimethoxybenzophenone with geranyl di-n-butyl-sulfonium tetrafluoroborate salt (2.5 equiv) in the presence of Hunig's base (5 equiv) in toluene at 80° for 16 h (15%) [1536].

oil [1605]; ^{1}H NMR [1605], IR [1605], UV [1605].

[3-(3,7-dimethyloctyl)-2,4-Dihydroxy-6-methoxyphenyl]phenylmethanone

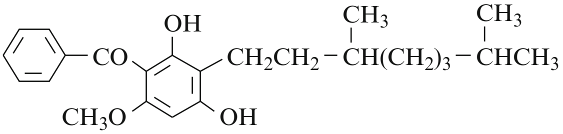
[53948-13-3]

$C_{24}H_{32}O_4$

mol.wt. 384.52

New compound

Synthesis



- Obtained by catalytic hydrogenation of marupone with hydrogen in the presence of Pd/C in ethanol at r.t. and pressure [1605].

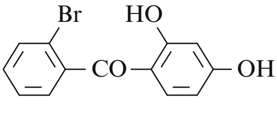
m.p. 140–143° [1605]; ^{1}H NMR [1605], IR [1605], UV [1605], MS [1605].

2.2.1.2 Substituents Located on the Other Ring [1476] p. 392

(2-Bromophenyl)(2,4-dihydroxyphenyl)methanone

$C_{13}H_9BrO_3$ mol.wt. 293.12

Described [1476] **p. 393**



Dimethyl ether [183106-10-7] $C_{15}H_{13}BrO_3$ mol.wt. 321.17

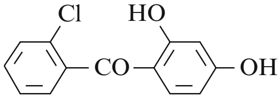
- Obtained by reaction of 2-bromobenzoic acid with 1,3-dimethoxybenzene in the presence of PPA at 90° for 8 h (80%) [1503].

m.p. 62° [1503];
^{1}H NMR [1503], IR [1503], UV [1503], MS [1503]; TLC [1503].

(2-Chlorophenyl)(2,4-dihydroxyphenyl)methanone

[50685-40-0] $C_{13}H_9ClO_3$ mol.wt. 248.67

Described [1476] **p. 394**



Dimethyl ether [34702-01-7] $C_{15}H_{13}ClO_3$ mol.wt. 276.72

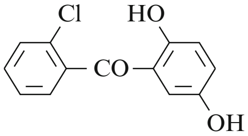
- Obtained by reaction of 2-chlorobenzoic acid with 1,3-dimethoxybenzene in the presence of PPA at 90° for 8 h (90%) [1503].
- Also refer to: [1721].

m.p. 60° [1503]; ^{1}H NMR [1503], IR [1503].

(2-Chlorophenyl)(2,5-dihydroxyphenyl)methanone

[37883-99-1] $C_{13}H_9ClO_3$ mol.wt. 248.67

Described [1476] **p. 394**



Dimethyl ether [37883-94-6] $C_{15}H_{13}ClO_3$ mol.wt. 276.72

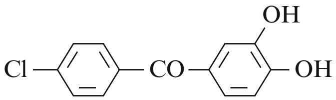
- Obtained by reaction of 2-chlorobenzoyl chloride with 1,4-dimethoxybenzene in methylene chloride in the presence of stannic chloride (80%) [1503].
- Also refer to: [1722].

m.p. 55° [1503]; ^{1}H NMR [1503], IR [1503].

(4-Chlorophenyl)(3,4-dihydroxyphenyl)methanone

[134612-84-3] $C_{13}H_9ClO_3$ mol.wt. 248.67

Described [1476] **p. 397**



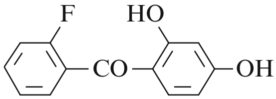
Synthesis

- Also refer to: [1534] (Chinese patent).

(2,4-Dihydroxyphenyl)(2-fluorophenyl)methanone

[19390-38-6] $C_{13}H_9FO_3$ mol.wt. 232.21

Described [1476] **p. 397**



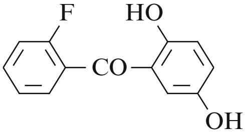
Dimethyl ether [7396-80-7] $C_{15}H_{13}FO_3$ mol.wt. 260.26

- Obtained by reaction of 2-fluorobenzoic acid with 1,3-dimethoxybenzene in the presence of PPA at 90° for 8 h (85%) [1503].

white solid; m.p. 70° [1503];
^{1}H NMR [1503], IR [1503], UV [1503], MS [1503]; TLC [1503].

(2,5-Dihydroxyphenyl)(2-fluorophenyl)methanone

[176547-98-1] $C_{13}H_9FO_3$ mol.wt. 232.21



Described [1476] **p. 397**

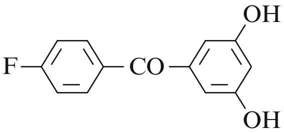
Dimethyl ether [176547-97-0] $C_{15}H_{13}FO_3$ mol.wt. 260.26

- Obtained by reaction of 2-fluorobenzoyl chloride with 1,4-dimethoxybenzene in methylene chloride in the presence of stannic chloride at 0° (83%) [1503].

m.p. 50–51° [1503];
^{1}H NMR [1503], IR [1503], UV [1503], MS [1503]; TLC [1503].

(3,5-Dihydroxyphenyl)(4-fluorophenyl)methanone

[148253-51-4] $C_{13}H_9FO_3$ mol.wt. 232.21
[148253-52-5] *(Polymer)*



Described [1476] **p. 398**

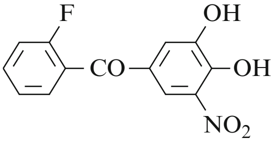
Synthesis

- Also refer to: [1609].

2.2.1.3 Substituents Located on Both Rings [1476] p. 409

(3,4-Dihydroxy-5-nitrophenyl)(2-fluorophenyl)methanone

[125628-97-9] $C_{13}H_8FNO_5$ mol.wt. 277.21



Described [1476] **p. 413**

Synthesis

- Also refer to: [1723].
- BIOLOGICAL ACTIVITY: Inhibition of dopamine uptake [1724]; cytotoxicity [1724].
- Also refer to: [1725,1726].

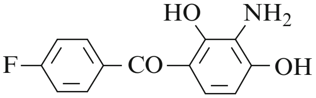
Dimethyl ether $C_{15}H_{12}FNO_5$ mol.wt. 305.26

- Obtained by reaction of dimethyl sulfate with 2′-fluoro-3,4-dihydroxy-5-nitrobenzophenone in the presence of dilute sodium hydroxide and tetrabutylammonium bromide in methylene chloride at r.t. for 17 h (91%) [1725].
- Also obtained by adding sodium hydride and dimethyl sulfate to a solution of 4-hydroxy-3-methoxy-5-nitro-2′-fluorobenzophenone in DMF, and refluxing 30 min (73%) [1727].

^{1}H NMR [1727].

(3-Amino-2,4-dihydroxyphenyl)(4-fluorophenyl)methanone

[909255-18-1] $C_{13}H_{10}FNO_3$ mol.wt. 247.23



New compound

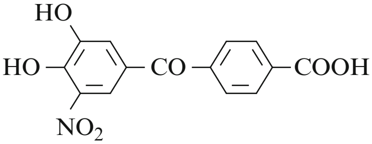
Syntheses

- Obtained by total demethylation of (3-amino-2,4-dimethoxyphenyl)(4-fluorophenyl)methanone in the presence of aluminium chloride (70%) [1595].
- Also obtained from (p-fluorophenyl)benzoic acid [1595].
- Also obtained from 2,6-dimethoxyaniline [1595].
- Also refer to: [1728].

light-yellow crystals [1595], yellow crystals [1728];
m.p. 179–181° [1595];
^{1}H NMR [1595], ^{13}C NMR [1595]; X-ray Data [1595,1728].

4-(3,4-Dihydroxy-5-nitrobenzoyl)benzoic acid *(Tolcapone, carboxylic acid) (Ro 47-1669)*

[212902-63-1] $C_{14}H_9NO_7$ mol.wt. 303.23



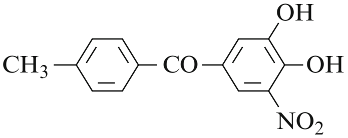
New compound

Isolation from natural sources

- A plasma metabolite of tolcapone in human [1702].

(3,4-Dihydroxy-5-nitrophenyl)(4-methylphenyl)methanone *(Tolcapone) (Ro-40-7592)*

[134308-13-7] $C_{14}H_{11}NO_5$ mol.wt. 273.25



Described [1476] **p. 418**

Syntheses

- Also obtained by adding aluminium chloride to a stirred suspension of (4-hydroxy-3-methoxy-5-nitrophenyl) (4-methylphenyl)methanone in ethyl acetate at r.t. under nitrogen, followed by pyridine. The red solution obtained was refluxed for 2 h (91%) [1701].
- Also refer to: [1702–1704,1712,1729–1735].

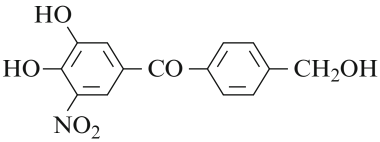
yellow solid [1701];
m.p. 146–148° [1703,1704], 141–143° [1701]; ^{1}H NMR [1701], ^{13}C NMR [1701], MS [1701].

BIOLOGICAL ACTIVITY: Hepatotoxicity [1712,1729,1731]; a novel inhibitor of catechol-O-methyltransferase [1702].

(3,4-Dihydroxy-5-nitrophenyl)[4-(hydroxymethyl)phenyl]methanone

(Tolcapone, primary alcohol metabolite) (Ro 47-1868)

[254912-15-7] $C_{14}H_{11}NO_6$ mol.wt. 289.25



New compound

Isolation from natural sources

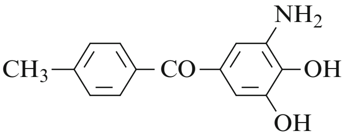
- A plasma metabolite of tolcapone in human [1702].

N.B.: Oxidation CYP3A4.

(3-Amino-4,5-dihydroxyphenyl)(4-methylphenyl)methanone

(Tolcapone, amine derivative) (Ro 61-3662)

[254912-17-9] $C_{14}H_{13}NO_3$ mol.wt. 243.26



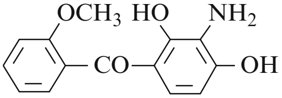
New compound

Synthesis

- From Tolcapone via reductive metabolism in human [1702].

(3-Amino-2,4-dihydroxyphenyl)(2-methoxyphenyl)methanone

[909255-19-2] $C_{14}H_{13}NO_4$ mol.wt. 259.26



New compound

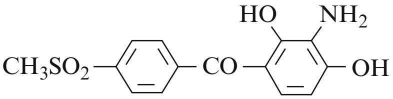
Synthesis

- Obtained by treatment of (2,4-dihydroxy-3-nitrophenyl)-(2-methoxyphenyl)methanone with zinc in acetic acid at 20° (76%) [1595].

m.p. 153–155° [1595]; ^{1}H NMR [1595], ^{13}C NMR [1595].

(3-Amino-2,4-dihydroxyphenyl)[(4-methylsulfonyl)phenyl]methanone

[909255-17-0] $C_{14}H_{13}NO_5S$ mol.wt. 307.33



New compound

Synthesis

- Preparation by treatment of (3-amino-2,4-dimethoxyphenyl)(4-methylsulfonyl)methanone with aluminium chloride in toluene at 100° for 20 min (72%) [1595].

m.p. 233–235° [1595]; ^{1}H NMR [1595], ^{13}C NMR [1595].

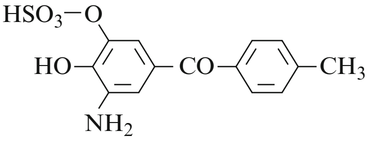
Dimethyl ether $C_{16}H_{17}NO_5S$ mol.wt. 335.38

- Obtained by reaction of p-(methylsulfonyl)benzoic acid with 2,6-dimethoxyaniline in the presence of PPA at 100° for 7 h (42%) [1595].

[3-Amino-4-hydroxy-5-(sulfooxy)phenyl](4-methylphenyl)methanone

(Tolcapone, amine sulphate)

[254902-29-9] $C_{14}H_{13}NO_6S$ mol.wt. 323.32



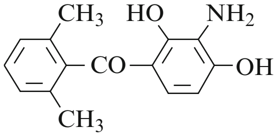
New compound

Synthesis

- From Tolcapone via reductive metabolism in human [1702].

(3-Amino-2,4-dihydroxyphenyl)(2,6-dimethylphenyl)methanone

[909255-20-5] $C_{15}H_{15}NO_3$ mol.wt. 257.29



New compound

Synthesis

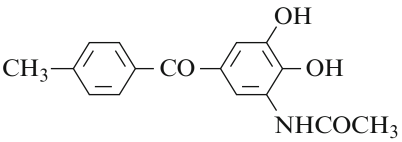
- Obtained by treatment of (2,4-dihydroxy-3-nitrophenyl)-(2,6-dimethylphenyl)methanone with zinc in acetic acid at 20° (72%) [1595].

m.p. 187–189° [1595]; ^{1}H NMR [1595], ^{13}C NMR [1595]; X-ray Data [1595,1728].

(3-Acetamido-4,5-dihydroxyphenyl)(4-methylphenyl)methanone

(Tolcapone, N-acetylamino metabolite) (Ro 48-2485)

$C_{16}H_{15}NO_4$ mol.wt. 285.30



New compound

Synthesis

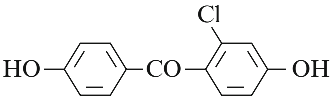
- From Tolcapone via reductive metabolism in human [1702].

2.2.2 *Hydroxy Groups Located on Both Rings [1476] p. 423*

2.2.2.1 Substituents Located on One Ring [1476] p. 423

(2-Chloro-4-hydroxyphenyl)(4-hydroxyphenyl)methanone

[98155-77-2] $C_{13}H_9ClO_3$ mol.wt. 248.67



Described [1476] **p. 425**

Dimethyl ether [30457-41-1] $C_{15}H_{13}ClO_3$ mol.wt. 276.72

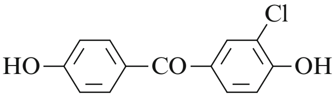
- Obtained by heating for 4 h a mixture of 3-chloroanisole and 4-methoxybenzoyl chloride,
  - in the presence of ferric chloride (92%) [1585];
  - in the presence of aluminium chloride (80%) [1585].

b.p.$_{12}$ 232° [1585]; m.p. 81° [1585].

(3-Chloro-4-hydroxyphenyl)(4-hydroxyphenyl)methanone

[92005-17-9] $C_{13}H_9ClO_3$ mol.wt. 248.67

Described [1476] **p. 426**



Dimethyl ether [74697-33-9] $C_{15}H_{13}ClO_3$ mol.wt. 276.72

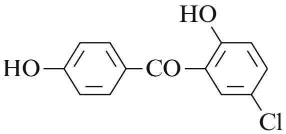
- Obtained by heating for 4 h a mixture of 2-chloroanisole and 4-methoxybenzoyl chloride,
  - in the presence of ferric chloride (90%) [1585];
  - in the presence of aluminium chloride (85%) [1585].

b.p.$_{12}$ 234° [1585]; m.p. 121° [1585].

(5-Chloro-2-hydroxyphenyl)(4-hydroxyphenyl)methanone

[126165-40-0] $C_{13}H_9ClO_3$ mol.wt. 248.67

Described [1476] **p. 428**



Dimethyl ether [76442-95-0] $C_{15}H_{13}ClO_3$ mol.wt. 276.72

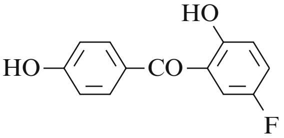
- Obtained by heating for 4 h a mixture of 4-chloroanisole and 4-methoxybenzoyl chloride,
  - in the presence of ferric chloride (90%) [1585];
  - in the presence of aluminium chloride (85%) [1585].
- Also refer to: [1557].

b.p.$_{12}$ 231° [1585]; m.p. 113° [1585], 77° [1557]; IR [1557].

(5-Fluoro-2-hydroxyphenyl)(4-hydroxyphenyl)methanone

[159300-38-6] $C_{13}H_9FO_3$ mol.wt. 232.21

Described [1476] **p. 428**



Dimethyl ether [60972-10-3] $C_{15}H_{13}FO_3$ mol.wt. 260.27

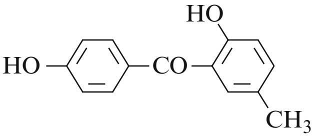
- Refer to: [1736].

  m.p. 135° [1736].
  BIOLOGICAL DATA: Fungicide [1736].

(2-Hydroxy-5-methylphenyl)(4-hydroxyphenyl)methanone

[25148-21-4] $C_{14}H_{12}O_3$ mol.wt. 228.25

Described [1476] **p. 430**



Dimethyl ether [54118-71-7] $C_{16}H_{16}O_3$ mol.wt. 256.30

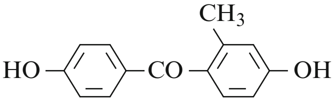
- Refer to: (78%) [1501].

  m.p. 65–67° [1501]; UV [1501].

(4-Hydroxy-2-methylphenyl)(4-hydroxyphenyl)methanone

[98155-72-7] $C_{14}H_{12}O_3$ mol.wt. 228.25

Described [1476] **p. 431**



Dimethyl ether [51974-20-0] $C_{16}H_{16}O_3$ mol.wt. 256.30

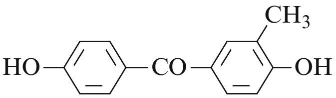
- Obtained by reaction of 4-methoxybenzoyl chloride with 3-methylanisole in the presence of aluminium chloride in carbon disulfide at r.t. for 3 h (96%) [1501].

  m.p. 82–83.5° [1501]; UV [1501].

(4-Hydroxy-3-methylphenyl)(4-hydroxyphenyl)methanone

[92005-11-3] $C_{14}H_{12}O_3$ mol.wt. 228.25

Described [1476] **p. 432**



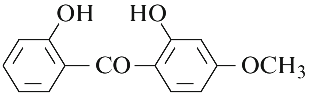
Dimethyl ether [54118-70-6] $C_{16}H_{16}O_3$ mol.wt. 256.30

- Obtained by reaction of 4-methoxybenzoyl chloride with 2-methylanisole in the presence of aluminium chloride in carbon disulfide at r.t. for 3 h (97%) [1501].

  m.p. 65–66° [1501]; UV [1501].

(2-Hydroxy-4-methoxyphenyl)(2-hydroxyphenyl)methanone

[131-53-3] $C_{14}H_{12}O_4$ mol.wt. 244.25



Described [1476] **p. 432**

Synthesis

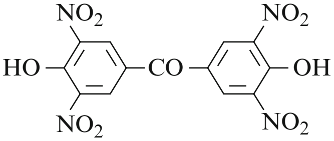
- Also refer to: [1534] (Chinese patent).

2.2.2.2 Substituents Located on Both Rings [1476] p. 441

Symmetrical ketones [1476] p. 441

Bis(4-hydroxy-3,5-dinitrophenyl)methanone

$C_{13}H_6N_4O_{11}$ mol.wt. 394.21



New compound

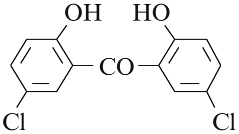
Synthesis

- Obtained by treatment of 4,4′-dichloro-3,3′,5,5′-tetranitro-benzophenone with ammonia in ethanol in a sealed tube at 100° for 8 h (44%) [1737].

m.p. 203° [1737].

Bis(5-chloro-2-hydroxyphenyl)methanone

[6178-89-8] $C_{13}H_8Cl_2O_3$ mol.wt. 283.11



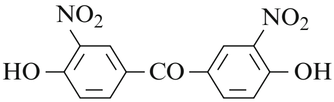
Described [1476] **p. 444**

Synthesis

- Also refer to: [1738].

Bis(4-hydroxy-3-nitrophenyl)methanone

[37567-35-4] $C_{13}H_8N_2O_7$ mol.wt. 304.22



Described [1476] **p. 445**

Syntheses

- Obtained by heating its dimethyl or diethyl ether with potassium hydroxide in dilute ethanol under pressure at 175° (55–59%) [1737].
- Also obtained by heating at 135° during 5 h in a sealed tube, 4,4′-dichloro-3,3′-dinitro-benzophenone with an aqueous solution of sodium carbonate (67%) [1737].

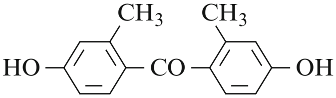
- Preparation by heating a solution of 1,1,1-trichloro-2,2-bis(4-chloro-3-nitrophenyl) ethane and potassium nitrite in DMAA (dimethylacetamide) at 100° for 4 h (95%) [1739].
- Preparation by heating a solution of 1,1,1-trichloro-2,2-bis(4-hydroxy-3-nitrophenyl)ethane and sodium nitrite in DMF between 120° and 130° for 10 h (93–95%) [1740].
- Also refer to: [1741].

m.p. 197–198° [1740], 196–198° [1739], 175° [1737];
IR [1739,1740]; TLC [1739,1740].

Dimethyl ether $C_{15}H_{12}N_2O_7$ mol.wt. 332.27 [1737].
Diethyl ether $C_{17}H_{16}N_2O_7$ mol.wt. 360.32 [1737].

Bis(4-hydroxy-2-methylphenyl)methanone

[98155-74-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



Described [1476] **p. 447**

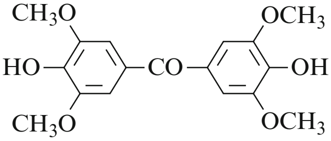
Dimethyl ether [5191-70-8] $C_{17}H_{18}O_3$ mol.wt. 270.33

- Obtained by oxidation of bis(4-methoxy-2-methylphenyl)methane with chromic acid [1742].
- Also refer to: [1485].

m.p. 72° [1742]; IR [1485].

Bis(4-hydroxy-3,5-dimethoxyphenyl)methanone

[34007-64-2] $C_{17}H_{18}O_7$ mol.wt. 334.33



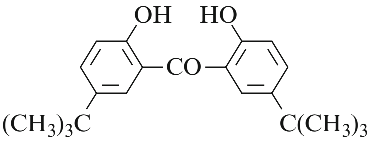
Described [1476] **p. 450**

Synthesis

- Also refer to: [1743] (Chinese patent).

Bis[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone

[25446-98-4] $C_{21}H_{26}O_3$ mol.wt. 326.44



Described [1476] **p. 450**

Dimethyl ether [98085-85-9] $C_{23}H_{30}O_3$ mol.wt. 354.49

- Obtained by reaction of 4-tert-butylanisole with N,N-dimethylamide chlorocarbonic acid (60%) [1522].

- Also refer to: [1485]; IR [1485].

^{1}H NMR [1522], ^{13}C NMR [1522], IR [1522].

Asymmetric ketones [1476] p. 452

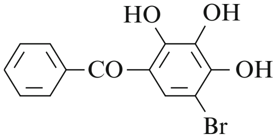
2.3 Trihydroxybenzophenones [1476] p. 464

2.3.1 Hydroxy Groups Located on the Same Ring [1476] p. 464

2.3.1.1 Substituents Located on the Hydroxylated Ring [1476] p. 464

(5-Bromo-2,3,4-trihydroxyphenyl)phenylmethanone

[870652-41-8] $C_{13}H_9BrO_4$ mol.wt. 309.12



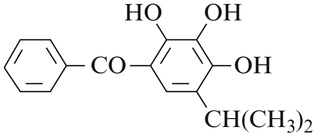
New compound

Synthesis

- Refer to: [1719].

Phenyl[2,3,4-trihydroxy-5-(1-methylethyl)phenyl]methanone

[1011708-91-0] $C_{16}H_{16}O_4$ mol.wt. 272.30



New compound

Synthesis

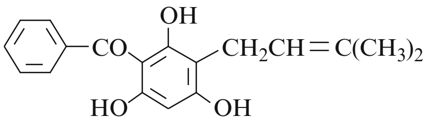
- Obtained by treatment of its trimethyl ether with boron tribromide (6 equiv) in methylene chloride, first at −78°, then at −20° (>72%) [1658].

Trimethyl ether [1011708-96-5] $C_{19}H_{22}O_4$ mol.wt. 314.38

- Obtained by Dess–Martin oxidation of 2,3,4-trimethoxy-5-isopropylbenzhydrol employing the triacetoxyperiodinane (the “Dess–Martin Periodinane” reagent) in methylene chloride at r.t. for 30 min (>76%) [1658].

Phenyl[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl]methanone

[93796-20-4] $C_{18}H_{18}O_4$ mol.wt. 298.34



New compound

Syntheses

- Obtained by reaction of 2,4,6-trihydroxy-benzophenone with prenyl-di-n-hexylsulfonium tetrafluoroborate salt (1 equiv) in the presence of Hünig’s base (1.1 equiv) in chloroform at r.t. for 7 h (42%) [1536].

- Also obtained (by-product) by reaction of α,α-dimethylallyl alcohol with 2,4,6-trihydroxy-benzophenone in the presence of boron trifluoride etherate in dioxane at 20° for 5.5 h (2%) [1744].

Isolation from natural sources

- From the aerial parts of *Helichrysum asperum* (Thunb.) Hilliard et Burtt var. *albidulum* (DC) Hilliard (Compositae) [1720].

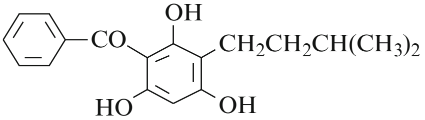
m.p. 102–103° [1720];
^{1}H NMR [1720,1744], IR [1720], UV [1744], MS [1720,1744].

Trimethyl ether $C_{21}H_{24}O_4$ mol.wt. 340.42 [1536].

- Obtained by reaction of 2,4,6-trimethoxybenzophenone with prenyl-di-n-hexyl-sulfonium tetrafluoroborate salt (2.5 equiv) in the presence of Hünig's base (5 equiv) in toluene at 100° for 16 h (51%) [1536].

Phenyl[2,4,6-trihydroxy-3-(3-methylbutyl)phenyl]methanone

[93796-23-7] $C_{18}H_{20}O_4$ mol.wt. 300.35



New compound

Synthesis

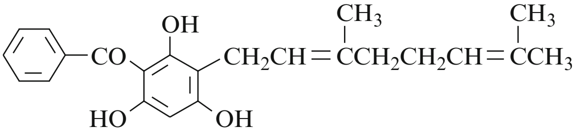
- Obtained by hydrogenation of 2,4,6-trihydroxy-3-(3-methyl-2-butenyl)benzophenone with hydrogen in the presence of Pd/C in methanol at 20° for 4 h (59%) [1744].

m.p. 94–97° [1744]; ^{1}H NMR [1744], UV [1744], MS [1744].

[3-(3,7-Dimethyl-2,6-octadienyl)-2,4,6-trihydroxyphenyl]phenylmethanone

[70219-87-3] *(E)*

[76015-48-0] *(Z)* $C_{23}H_{26}O_4$ mol.wt. 366.46



Described [1476] **p. 465**

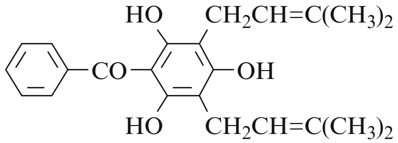
Isolation from natural sources

- From *Helichrysum monticola* Hilliard (Compositae) [1720].

Phenyl[2,4,6-trihydroxy-3,5-bis(3-methyl-2-butenyl)phenyl]methanone

(Clusiaphenone B)

[70219-84-0]



$C_{23}H_{26}O_4$ mol.wt. 366.46

Described [1476] **p. 466**

Syntheses

- Preparation by C-prenylation of 2,4,6-trihydroxy-benzophenone,
  - with prenyl bromide in the presence of aqueous potassium hydroxide at 0° (45%) [1535,1538,1745];
  - with 2-methyl-3-buten-2-ol in the presence of boron trifluoride etherate in dioxane at 20° for 5.5 h (11%) [1744].

Isolation from natural sources

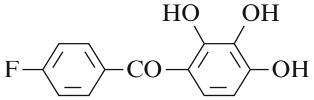
- From the fruits of *Clusia sandiensis* (Guttiferae) [1746].

oil [1746]; m.p. 93–94° [1744];
^{1}H NMR [1744,1746], ^{13}C NMR [1746], UV [1744], MS [1744,1746].

2.3.1.2 Substituents Located on the Other Ring [1476] p. 466

(4-Fluorophenyl)(2,3,4-trihydroxyphenyl)methanone

[84795-00-6]



$C_{13}H_9FO_4$ mol.wt. 248.21

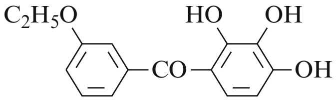
Described [1476] **p. 468**

Synthesis

- Also refer to: [1719].

(3-Ethoxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[674786-33-5]



$C_{15}H_{14}O_5$ mol.wt. 274.27

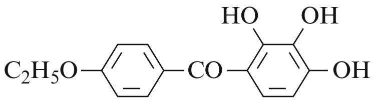
New compound

Synthesis

- Refer to: [1747].

(4-Ethoxyphenyl)(2,3,4-trihydroxyphenyl)methanone

[69471-29-0]



$C_{15}H_{14}O_5$ mol.wt. 274.27

New compound

Synthesis

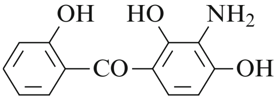
- Refer to: [1747].

2.3.2 Hydroxy Groups Located on Both Rings [1476] p. 471

2.3.2.1 Substituents Located on One Ring [1476] p. 471

(3-Amino-2,4-dihydroxyphenyl)(2-hydroxyphenyl)methanone

[1021955-12-3] $C_{13}H_{11}NO_4$ mol.wt. 245.23



New compound

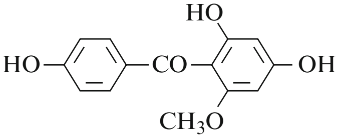
Synthesis

– Obtained by demethylation of (3-amino-2, 4-dihydroxy-phenyl)(2-methoxyphenyl)methanone by heating at 50° for 1.5 h with aluminium chloride in toluene (50%) [1728].

yellow solid; m.p. 176° [1728];
^{1}H NMR [1728]; X-ray analysis [1728].

(2,4-Dihydroxy-6-methoxyphenyl)(4-hydroxyphenyl)methanone

[56308-11-3] $C_{14}H_{12}O_5$ mol.wt. 260.25



Described [1476] **p. 475**

Isolation from natural sources

- Obtained by treatment of *Mahkoside A* (N.B.) with 30% aqueous hydrochloric acid in refluxing ethanol (95%) [1748].

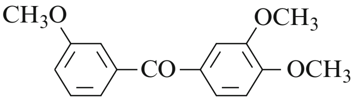
  N.B.: *Mahkoside A* is the 2-β-D-galactopyranoside derivative of the title compound. It is isolated from *Mahkota Dewa* (*Phaleria macrocarpa* [Scheff.] Boerl) [1748].
- Also refer to: [1749–1751].

yellow solid; m.p. 83–85° [1748];
^{1}H NMR [1748], ^{13}C NMR [1748], IR [1748], MS [1748].

BIOLOGICAL DATA: Cytotoxicity [1748,1749]; UV absorber, photo sensitizer and medical intermediate [1750].

(3,4-Dimethoxyphenyl)(3-methoxyphenyl)methanone

[792-57-4] $C_{16}H_{16}O_4$ mol. wt. 272.30



New compound

Syntheses

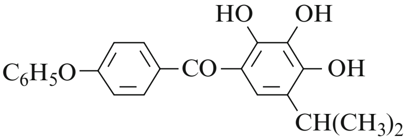
– Obtained by reaction of 3-methoxybenzoic acid with veratrole in the presence of PPA at 90° for 8 h (85%) [1503].
- Also refer to: [1560].

white solid; m.p. 79–80° [1503]; ^{1}H NMR [1503], IR [1503], UV [1503].

2.3.2.2 Substituents Located on Both Rings [1476] p. 480

(4-Phenoxyphenyl)[2,3,4-trihydroxy-5-(1-methylethyl)phenyl]methanone

[1011708-92-1] $C_{22}H_{20}O_5$ mol.wt. 364.40



New compound

Synthesis

- Obtained by treatment of its trimethyl ether with boron tribromide (6 equiv) in methylene chloride, first at −78°, then at −20° (>72%) [1658].

Trimethyl ether [1011708-90-9] $C_{25}H_{26}O_5$ mol.wt. 406.48.

- Obtained by Dess–Martin oxidation of (2,3,4-trimethoxy-5-isopropyl)(4-phenoxy)benzhydrol employing the triacetoxyperiodinane (the "Dess–Martin Periodinane" reagent) in methylene chloride at r.t. for 30 min (>76%) [1658].

2.4 Tetrahydroxybenzophenones [1476] p. 489

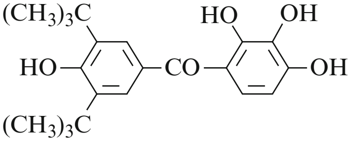
2.4.1 Hydroxy Groups Located on One Ring [1476] p. 489

2.4.2 Substituents Located on Both Rings [1476] p. 489

2.4.2.1 Substituents Located on One Ring [1476] p. 489

[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](2,3,4-trihydroxyphenyl)methanone

[251562-02-4] $C_{21}H_{26}O_5$ mol.wt. 358.43



New compound

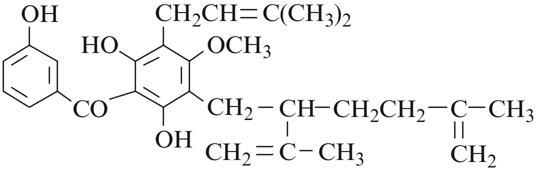
Synthesis

- Refer to: [1719].

[2,6-Dihydroxy-4-methoxy-3-(3-methyl-2-butenyl)-5-[5-methyl-2-(1-methylethenyl)-5-hexenyl]phenyl](3-hydroxyphenyl)methanone (-) *(Tovophenone A)*

[91387-68-7] $C_{29}H_{36}O_5$
mol.wt. 464.60

New compound



Isolation from natural sources

- From *Tovomita brevistaminea* (Guttiferae) [1536,1752].
- From *Tovomita mangle* G. Maritz (Guttiferae – sub-family Clusioideae) [1753].

yellow oil [1752,1753];
$(\alpha)_D = -20°$ ($CHCl_3$) [1753], −18.4° (MeOH) [1752];
^1H NMR [1752,1753], ^{13}C NMR [1752,1753], IR [1752,1753], UV [1752,1753], MS [1752,1753].

BIOLOGICAL ACTIVITY: Cytotoxicity [1752]; KB, human oral epidermoid carcinoma [1752].

Triacetate $C_{35}H_{42}O_8$ mol.wt. 590.71

- Obtained by reaction of acetic anhydride with Tovophenone A in the presence of pyridine at r.t. overnight [1753].

oil [1753]; ^1H NMR [1753], IR [1753].

Trimethyl ether $C_{32}H_{42}O_5$ mol.wt. 506.68

- Obtained by reaction of dimethyl sulfate with Tovophenone A in the presence of potassium carbonate in acetone (quantitative yield) [1753].

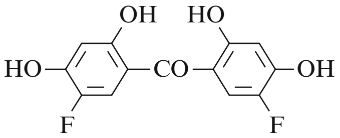
oil [1753]; $(\alpha)_D = -14°$ ($CHCl_3$) [1753];
^1H NMR [1753], ^{13}C NMR [1753], IR [1753].

2.4.2.2 Substituents Located on Both Rings [1476] p. 494

Symmetrical ketones [1476] p. 494

Bis(5-fluoro-2,4-dihydroxyphenyl)methanone

[430459-44-2] $C_{13}H_8F_2O_5$ mol.wt. 282.20



New compound

Syntheses

- Obtained by total demethylation of its tetramethyl ether with boron tribromide in methylene chloride at 40° (72%) [1754].
- Also obtained via the Friedel–Crafts acylation of 1-fluoro-2,4-dimethoxybenzene with 5-fluoro- 2,4-dimethoxybenzoyl chloride in the presence of aluminium chloride (44%) [1755].

yellow crystals [1755];
^1H NMR [1755], ^{13}C NMR [1755], ^{19}F NMR [1755].

Tetramethyl ether [879288-16-1] $C_{17}H_{16}F_2O_5$ mol.wt. 338.31

- The 4-fluoro-6-iodoresorcinol dimethyl ether was converted to the bis(5-fluoro-2,4-dimethoxyphenyl)methanone using Larhed's microwave and cobalt octacarbonyl-mediated synthesis of diaryl ketones [1524], (96%) [1754].

Asymmetric ketones [1476] p. 494

2.5 Pentahydroxybenzophenones [1476] p. 496

2.5.1 Hydroxy Groups Located on One Ring [1476] p. 496

2.5.2 Hydroxy Groups Located on Both Rings [1476] p. 496

2.5.2.1 Substituents Located on One Ring [1476] p. 496

2.5.2.2 Substituents Located on Both Rings [1476] p. 499

2.6 Hexahydroxybenzophenone [1476] p. 500

Chapter 3. Polyphenyl Phenyl Methanones *(Class of METHANONES)*

3.1 Biphenyl Phenyl Methanones [1476] p. 501

3.1.1 Monohydroxylated Ketones [1476] p. 501

3.1.2 Dihydroxylated Ketones [1476] p. 508

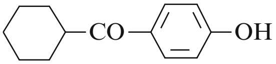
3.2 Terphenyl Phenyl Methanones [1476] p. 511

Chapter 4. Cyclohexyl Phenyl Methanones *(Class of METHANONES)*

4.1.1 Monohydroxylated Ketones [1476] p. 513

Cyclohexyl(4-hydroxyphenyl)methanone

[38459-58-4] $C_{13}H_{16}O_2$ mol.wt. 204.27



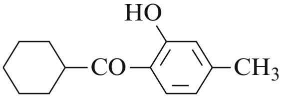
Described [1476] **p. 515**

Methyl ether [7469-80-9] $C_{14}H_{18}O_2$ mol.wt. 218.30

- Obtained by Friedel–Crafts acylation of anisole with cyclohexanecarbonyl chloride on mesoporous silica catalyst MCM-41 (73%) [1756].
- Also refer to: [1757,1758].

Cyclohexyl(2-hydroxy-4-methylphenyl)methanone

[20112-74-7] $C_{14}H_{18}O_2$ mol.wt. 218.30



Described [1476] **p. 516**

Syntheses

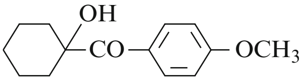
– Also obtained by Friedel–Crafts acylation of m-cresol with cyclohexanecarboxylic acid in a mixture of graphite and methanesulfonic acid at 120° for 2 h (90%) [1598].

– Also obtained by treatment of 3-methylphenyl cyclohexanecarboxylate in the presence of alumina in methanesulfonic acid for 25 min at 160° (86%) [1692].

^{1}H NMR [1598], ^{13}C NMR [1598], IR [1598].

(1-Hydroxycyclohexyl)(4-methoxyphenyl)methanone

[7469-82-1] $C_{14}H_{18}O_3$ mol.wt. 234.20



New compound

Synthesis

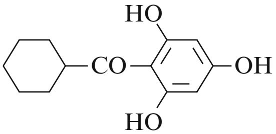
– Refer to: [1758].

4.1.2 Dihydroxylated Ketones [1476] p. 519

4.1.3 Trihydroxylated Ketones [1476] p. 520

Cyclohexyl(2,4,6-trihydroxyphenyl)methanone

[85602-45-5] $C_{13}H_{16}O_4$ mol.wt. 236.27



Described [1476] **p. 520**

Syntheses

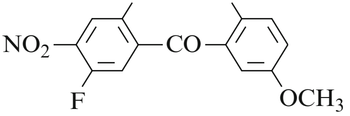
– Also refer to: [1759], (27%) [1760].

oily solid [1760];
^{1}H NMR [1760], ^{13}C NMR [1760], IR [1760], UV [1760], MS [1760]; TLC [1760].

Chapter 4.2. Ketones Without Hydroxy Groups *(Class of METHANONES)*

(2,5-Difluoro-4-nitrophenyl)(2-fluoro-5-methoxyphenyl)methanone

[1015414-82-0] $C_{14}H_8F_3NO_4$ mol.wt. 311.22



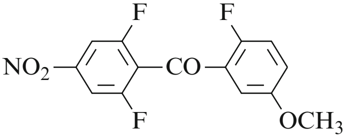
New compound

Synthesis

– Obtained by adding under an argon atmosphere, 60% sodium hydride in oil to a mixture of 2,4,5-trifluoro-nitrobenzene, 2-fluoro-5-methoxybenzaldehyde and 1,3-dimethylimidazolium iodide in DMF. The mixture was stirred at −15° for 10 min; then at r.t. for 2 h (72%) [1641].

(2,6-Difluoro-4-nitrophenyl)(2-fluoro-5-methoxyphenyl)methanone

[1015414-83-1] $C_{14}H_8F_3NO_4$ mol.wt. 311.22



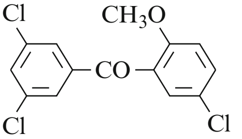
New compound

Synthesis

- Obtained by adding under an argon atmosphere, 60% sodium hydride in oil to amixture of 3,4,5-trifluoro-nitrobenzene, 2-fluoro-5-methoxybenzaldehyde and 1,3-dimethylimidazolium iodide in DMF. The mixture was stirred at 0° for 50 min (89%) [1641].

(5-Chloro-2-methoxyphenyl)(3,5-dichlorophenyl)methanone

[872088-11-4] $C_{14}H_9Cl_3O_2$ mol.wt. 315.58



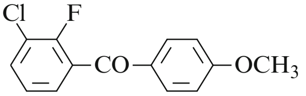
New compound

Synthesis

- Obtained by reaction of 3,5-dichlorophenylmagnesium bromide with 5-chloro-2-methoxybenzoyl chloride in the presence of bis[2-(N,N-dimethylamino)ethyl] ether in THF at −5 to 0° (85%) [1623].

(3-Chloro-2-fluorophenyl)(4-methoxyphenyl)methanone

[680610-55-3] $C_{14}H_{10}ClFO_2$ mol.wt. 264.68



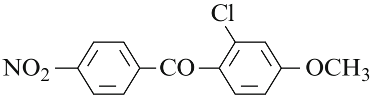
New compound

Syntheses

- Obtained by reaction of 4-methoxyphenylmagnesium bromide with 3-chloro-2-fluoro-N-methoxy-N-methylbenzamide [1761].
- Also refer to: [1699].

(2-Chloro-4-methoxyphenyl)(4-nitrophenyl)methanone

[76442-94-9] $C_{14}H_{10}ClNO_4$ mol.wt. 291.69



New compound

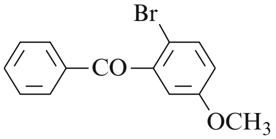
Syntheses

- Obtained by heating for 4 h a mixture of 3-chloroanisole and 4-nitrobenzoyl chloride,
  - in the presence of ferric chloride (85%) [1585];
  - in the presence of aluminium chloride (83%) [1585].
- Obtained by reaction of p-nitrobenzoyl chloride with 3-chloroanisole in the presence of aluminium chloride in carbon disulfide [1684].

b.p.$_{12}$ 264° [1585]; m.p. 84° [1585,1684].

(2-Bromo-5-methoxyphenyl)phenylmethanone

[60080-98-0] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



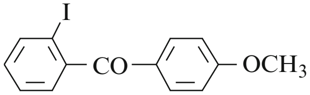
New compound

Syntheses

- Refer to: [1616,1762].

(2-Iodophenyl)(4-methoxyphenyl)methanone

[138504-32-2] $C_{14}H_{11}IO_2$ mol.wt. 338.14



New compound

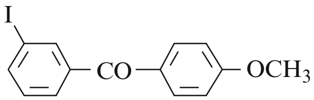
Synthesis

- Obtained from 2-iodobenzoic acid [1621].

^{1}H NMR [1621], ^{13}C NMR [1621].

(3-Iodophenyl)(4-methoxyphenyl)methanone

[908368-69-4] $C_{14}H_{11}IO_2$ mol.wt. 338.14



New compound

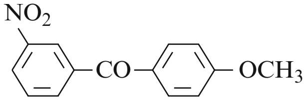
Synthesis

- Obtained from 3-iodobenzoic acid [1621].

^{1}H NMR [1621], ^{13}C NMR [1621].

(4-Methoxyphenyl)(3-nitrophenyl)methanone

[54118-78-4] $C_{14}H_{11}NO_4$ mol.wt. 257.24



New compound

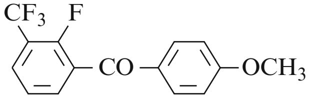
Synthesis

- Refer to: (52%) [1501].

m.p. 96.5–98.5° [1501]; UV [1501].

[2-Fluoro-3-(trifluoromethyl)phenyl](4-methoxyphenyl)methanone

[680610-53-1] $C_{15}H_{10}F_4O_2$ mol.wt. 298.24



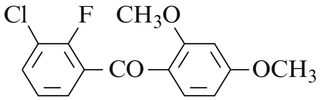
New compound

Syntheses

- Obtained by reaction of 4-methoxyphenylmagnesium bromide with 2-fluoro-N-methoxy-N-methyl-3-trifluoromethylbenzamide [1761].
- Also refer to: [1699].

(3-Chloro-2-fluorophenyl)(2,4-dimethoxyphenyl)methanone

[680610-70-2] $C_{15}H_{12}ClFO_3$ mol.wt. 294.71



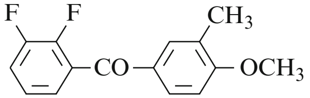
New compound

Synthesis

- Refer to: [1699].

(2,3-Difluorophenyl)(4-methoxy-3-methylphenyl)methanone

[680610-61-1] $C_{15}H_{12}F_2O_2$ mol.wt. 262.26



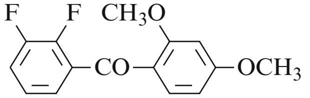
New compound

Synthesis

- Refer to: [1699].

(2,3-Difluorophenyl)(2,4-dimethoxyphenyl)methanone

[680610-71-3] $C_{15}H_{12}F_2O_3$ mol.wt. 278.26



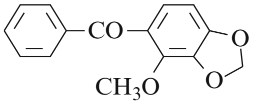
New compound

Synthesis

- Refer to: [1699].

(4-Methoxy-1,3-benzodioxol-5-yl)phenylmethanone

[872881-74-8] $C_{15}H_{12}O_4$ mol.wt. 256.26



New compound

Isolation from natural sources

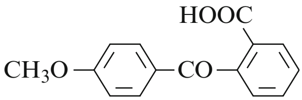
- From the roots of *Securidaca inappendiculata* Hassk (Polygalaceae) [1602].

Colourless crystals; m.p. 81.5° [1602]; $(\alpha)_D^{28} = 0$ (0.50 $CHCl_3$) [1602]; ^1H NMR [1602], ^{13}C NMR [1602], UV [1602], MS [1602]; X ray data [1602].

BIOLOGICAL ACTIVITY: Used as antiinflammatory, antibacterial and antirheumatism agent in China [1602].

2-(4-Methoxybenzoyl)benzoic acid

[1151-15-1] $C_{15}H_{12}O_4$ mol.wt. 256.26



New compound

Synthesis

- Refer to: [1501] (31%).

m.p. 144–145° [1501]; UV [1501].

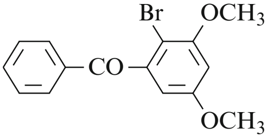
Na salt [54118-77-3] $C_{15}H_{11}O_4Na$ mol.wt. 278.24

- Refer to: [1501] (65%).

m.p. 260° [1501]; UV [1501].

(2-Bromo-3,5-dimethoxyphenyl)phenylmethanone

[1000990-08-8] $C_{15}H_{13}BrO_3$ mol.wt. 321.17



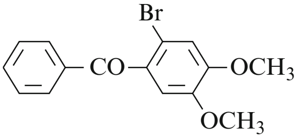
New compound

Synthesis

– Refer to: [1495].

(2-Bromo-4,5-dimethoxyphenyl)phenylmethanone

[59142-61-9] $C_{15}H_{13}BrO_3$ mol.wt. 321.17



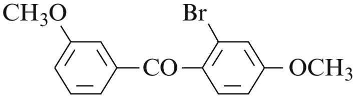
New compound

Synthesis

– Refer to: [1495].

(2-Bromo-4-methoxyphenyl)(3-methoxyphenyl)methanone

[1000990-04-4] $C_{15}H_{13}BrO_3$ mol.wt. 321.17



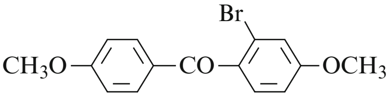
New compound

Synthesis

– Refer to: [1495].

(2-Bromo-4-methoxyphenyl)(4-methoxyphenyl)methanone

[30457-39-7] $C_{15}H_{13}BrO_3$ mol.wt. 321.17



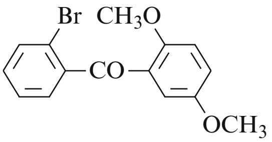
New compound

Synthesis

– Refer to: [1495].

(2-Bromophenyl)(2,5-dimethoxyphenyl)methanone

[137327-31-2] $C_{15}H_{13}BrO_3$ mol.wt. 321.17



New compound

Syntheses

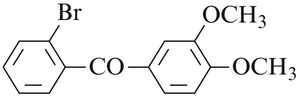
– Obtained by reaction of 2-bromobenzoyl chloride with 1,4-dimethoxybenzene in methylene chloride in the presence of stannic chloride (80%) [1503].

– Also refer to: [1763].

m.p. 54° [1503]; ^{1}H NMR [1503], IR [1503].

(2-Bromophenyl)(3,4-dimethoxyphenyl)methanone

[23346-79-4] $C_{15}H_{13}BrO_3$ mol.wt. 321.17



New compound

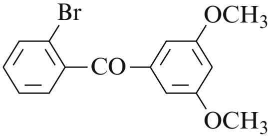
Syntheses

- Obtained by reaction of 2-bromobenzoic acid with veratrole in the presence of PPA at 90° for 8 h (80%) [1503].
- Also refer to: [1763].

m.p. 155° [1503];
^{1}H NMR [1503], IR [1503], UV [1503].

(2-Bromophenyl)(3,5-dimethoxyphenyl)methanone

[951892-04-9] $C_{15}H_{13}BrO_3$ mol.wt. 321.17



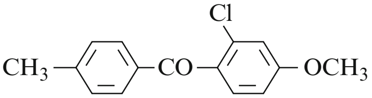
New compound

Synthesis

- Refer to: [1495].

(2-Chloro-4-methoxyphenyl)(4-methylphenyl)methanone

[76442-92-7] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



New compound

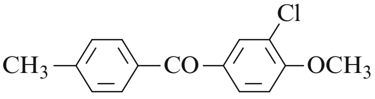
Syntheses

- Obtained by heating for 4 h a mixture of 3-chloroanisole and 4-methylbenzoyl chloride,
  - in the presence of ferric chloride (90%) [1585];
  - in the presence of aluminium chloride (85%) [1585].

b.p.$_{12}$ 216° [1585]; m.p. 63° [1585].

(3-Chloro-4-methoxyphenyl)(4-methylphenyl)methanone

[41295-44-7] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



New compound

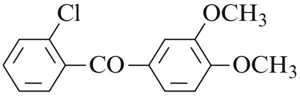
Syntheses

- Obtained by heating for 4 h a mixture of 2-chloroanisole and 4-methylbenzoyl chloride,
  - in the presence of ferric chloride (87%) [1585];
  - in the presence of aluminium chloride [1764], (91%) [1585].

b.p.$_{12}$ 214° [1585]; m.p. 107–108° [1764], 107° [1585].

(2-Chlorophenyl)(3,4-dimethoxyphenyl)methanone

[34702-00-6] $C_{15}H_{13}ClO_3$ mol.wt. 276.72



New compound

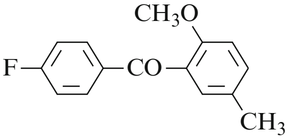
Syntheses

- Obtained by reaction of 2-chlorobenzoic acid with veratrole in the presence of PPA at 90° for 8 h (82%) [1503].
- Also refer to: [1721].

m.p. 141–142° [1721], 140° [1503];
^{1}H NMR [1503], IR [1503], UV [1503].

(4-Fluorophenyl)(2-methoxy-5-methylphenyl)methanone

[1000604-04-5] $C_{15}H_{13}FO_2$ mol.wt. 244.27



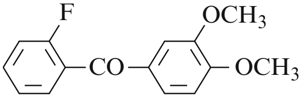
New compound

Synthesis

- Refer to: [1765].

(3,4-Dimethoxyphenyl)(2-fluorophenyl)methanone

[116412-86-3] $C_{15}H_{13}FO_3$ mol.wt. 260.26



New compound

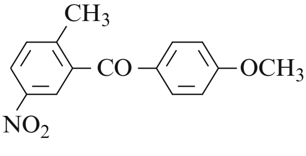
Syntheses

- Obtained by reaction of 2-fluorobenzoic acid with veratrole in the presence of PPA at 90° for 8 h (85%) [1503].
- Also refer to: [1766].

m.p. 76° [1503]; ^{1}H NMR [1503], IR [1503]; TLC [1503].

(4-Methoxyphenyl)(2-methyl-5-nitrophenyl)methanone

[1022080-07-4] $C_{15}H_{13}NO_4$ mol.wt. 271.27



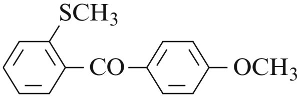
New compound

Synthesis

- Refer to: [1767].

(4-Methoxyphenyl)[2-(methylthio)phenyl]methanone

[760192-84-5] $C_{15}H_{14}O_2S$ mol.wt. 258.34



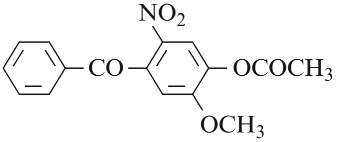
New compound

Synthesis

- Refer to: [1669].

[4-(Acetyloxy)-5-methoxy-2-nitrophenyl]phenylmethanone

[873296-36-7] $C_{16}H_{13}NO_6$ mol.wt. 315.18



New compound

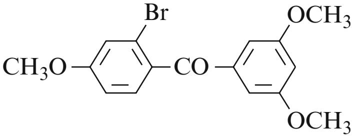
Synthesis

- Obtained by adding copper (II) nitrate trihydrate in one portion to a solution of 4-(acetyloxy)-3-methoxy-benzophenone (m.p. 103–104°) in acetic anhydride at r.t. (20%) [1597].

m.p. 210.2–211.1° [1597]; ^{1}H NMR [1597], ^{13}C NMR [1597].

(2-Bromo-4-methoxyphenyl)(3,5-dimethoxyphenyl)methanone

[1000990-05-5] $C_{16}H_{15}BrO_4$ mol.wt. 351.20



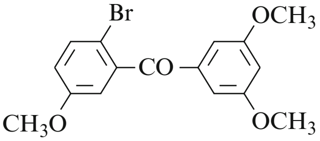
New compound

Synthesis

- Refer to: [1495].

(2-Bromo-5-methoxyphenyl)(3,5-dimethoxyphenyl)methanone

[1000990-06-6] $C_{16}H_{15}BrO_4$ mol.wt. 351.20



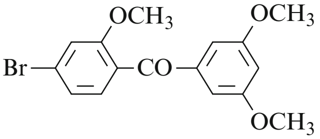
New compound

Synthesis

- Refer to: [1495].

(4-Bromo-2-methoxyphenyl)(3,5-dimethoxyphenyl)methanone

[1000990-15-7] $C_{16}H_{15}BrO_4$ mol.wt. 351.20



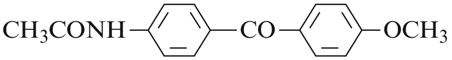
New compound

Synthesis

- Refer to: [1495].

[4-(Acetylamino)phenyl](4-methoxyphenyl)methanone

[97732-63-3] $C_{16}H_{15}NO_3$ mol.wt. 269.30



New compound

Syntheses

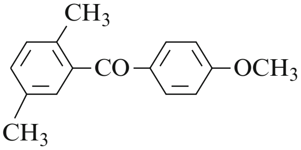
- Obtained by reaction of acetic anhydride with (4-methoxyphenyl)(4-nitrophenyl)methanone in the presence of pyridine at 110° for 5 min (60%) [1637].
- Also obtained by reaction of acetic anhydride with 4-amino-4′-methoxybenzophenone in benzene [1768].

– Also refer to: [1769].

m.p. 172° [1637], 170–171° [1768];
^{1}H NMR [1637], IR [1637], UV [1637,1769], MS [1637].

(2,5-Dimethylphenyl)(4-methoxyphenyl)methanone

[22996-47-0] $C_{16}H_{16}O_2$ mol.wt. 240.30



New compound

Syntheses

– Obtained by reaction of p-anisoyl chloride with p-xylene,
  - in the presence of aluminium chloride in methylene chloride between 10° and 20° (83%) [1770];
  - in the presence of MoO_2Cl_2 (20 mol%) at reflux for 20 h (31%) [1527].

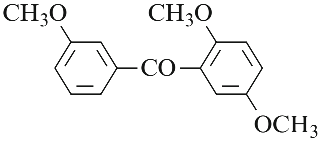
– Also obtained by reaction of p-anisic anhydride with p-xylene [1631].

– Also obtained by reaction of 2,5-dimethylbenzoyl chloride with anisole in the presence of aluminium chloride in methylene chloride at 20° for 1 h [1771].

m.p. 88° [1631]; ^{1}H NMR [1770,1771], ^{13}C NMR [1771], IR [1770,1771].

(2,5-Dimethoxyphenyl)(3-methoxyphenyl)methanone

[183106-11-8] $C_{16}H_{16}O_4$ mol.wt. 272.30



New compound

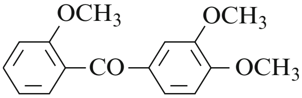
Synthesis

– Obtained by reaction of 3-methoxybenzoyl chloride with 1,4-dimethoxybenzene in methylene chloride in the presence of stannic chloride (80%) [1503].

white needles; m.p. 71–72° [1503];
^{1}H NMR [1503], IR [1503], UV [1503], MS [1503]; TLC [1503].

(3,4-Dimethoxyphenyl)(2-methoxyphenyl)methanone

[131252-46-5] $C_{16}H_{16}O_4$ mol.wt. 272.30



New compound

Syntheses

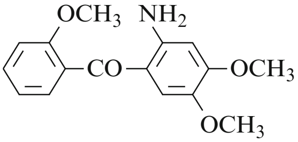
– Obtained by reaction of 2-methoxybenzoic acid with veratrole in the presence of PPA at 90° for 8 h (82%) [1503].

– Also refer to: [1772].

m.p. 80–82° [1560], 78° [1503]; ^{1}H NMR [1503], IR [1503], UV [1503].

(2-Amino-4,5-dimethoxyphenyl)(2-methoxyphenyl)methanone

[882531-40-0] $C_{16}H_{17}NO_4$ mol.wt. 287.32



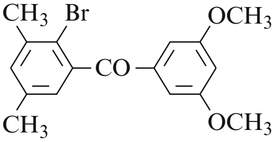
New compound

Synthesis

– Refer to: [1773].

(2-Bromo-3,5-dimethylphenyl)(3,5-dimethoxyphenyl)methanone

[1000990-11-3] $C_{17}H_{17}BrO_3$ mol.wt. 349.22



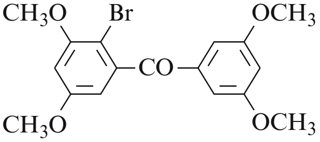
New compound

Synthesis

– Refer to: [1495].

(2-Bromo-3,5-dimethoxyphenyl)(3,5-dimethoxyphenyl)methanone

[1000990-09-9] $C_{17}H_{17}BrO_5$ mol.wt. 381.22



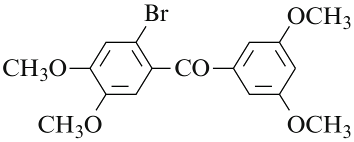
New compound

Synthesis

– Refer to: [1495].

(2-Bromo-4,5-dimethoxyphenyl)(3,5-dimethoxyphenyl)methanone

[1000990-07-7] $C_{17}H_{17}BrO_5$ mol.wt. 381.22



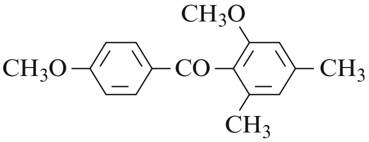
New compound

Synthesis

– Refer to: [1495].

(2-Methoxy-4,6-dimethylphenyl)(2-methoxyphenyl)methanone

[1019637-60-5] $C_{17}H_{18}O_3$ mol.wt. 270.33



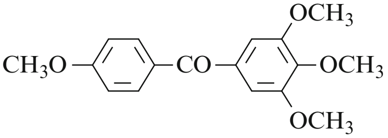
New compound

Synthesis

– Refer to: [1700].

(4-Methoxyphenyl)(3,4,5-trimethoxyphenyl)methanone

[109091-08-9]



$C_{17}H_{18}O_5$ mol.wt. 302.33

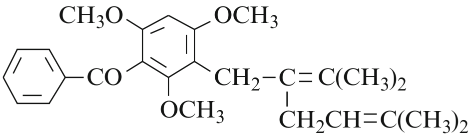
New compound

Synthesis

- Refer to: [1774].

Phenyl[2,4,6-trimethoxy-3-[5-methyl-2-(1-methylethylidene)-4-hexen-1-yl] phenyl]-methanone

[950201-80-6]



$C_{26}H_{32}O_4$ mol.wt. 408.54

New compound

Synthesis

- Obtained by reaction of 2,4,6-trimethoxy-benzophenone with 2-(1-methyl-ethylidene)-4-hexenyl di-n-butyl-sulfonium tetrafluoroborate salt (2.5 equiv) in the presence of Hunig's base (5 equiv) in toluene at 80° for 16 h (30%) [1536].

Part II Diarolyphenols, Polyaroylphenols

Chapter 5. Phenols with One Benzoyl Group and One or Several Acetyl Groups *(Class of ETHANONES)*

5.1 Monohydroxylated Ketones [1476] p. 523

5.2 Dihydroxylated Ketones [1476] p. 525

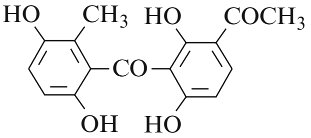
Symmetrical ketones [1476] p. 525
Asymmetric ketones [1476] p. 525

5.3 Trihydroxylated Ketone [1476] p. 528

5.4 Tetrahydroxylated Ketone [1476] p. 528

1-[3-(3,6-Dihydroxy-2-methylbenzoyl)-2,4-dihydroxyphenyl]ethanone
(Baishouwubenzophenone)

[115834-34-9] $C_{16}H_{14}O_6$ mol.wt. 302.28



Described [1476] **p. 528**

Isolation from natural sources

- From the ethyl acetate extract of the rhizome of *Cynanchum otophyllum* (Schneid Asclepiadaceae) [1775].
- From *Cynanchum auriculatum* Royle ex Wight (Asclepiadaceae) [1776].

^{1}H NMR [1776], ^{13}C NMR [1776], IR [1776], UV [1776], MS [1776].

Chapter 6. Phenols with Two or Several Benzoyl Groups *(Class of METHANONES)*

6.1 Monohydroxylated Ketones [1476] p. 529

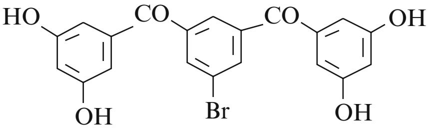
Symmetrical ketones [1476] p. 529
Asymmetric ketones [1476] p. 532

6.2 Di- and Polyhydroxylated Ketones [1476] p. 534

Symmetrical ketones [1476] p. 534

(5-Bromo-1,3-phenylene)bis[(3,5-dihydroxyphenyl)methanone

[873220-62-3] $C_{20}H_{13}BrO_6$ mol.wt. 429.22



New compound

Synthesis

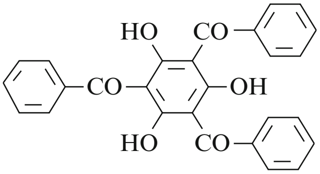
- Obtained by total demethylation of its tetramethyl ether with boron tribromide in methylene chloride at 0° [1563].

Tetramethyl ether [873220-61-2] $C_{24}H_{21}BrO_6$ mol.wt. 485.33

- Obtained by oxidation of 5-bromo-α,α′-bis(3,5-dimethoxyphenyl)benzenedimethanol [873220-60-1] with manganese dioxide in methylene chloride at r.t., followed by demethylation of the obtained product with boron tribromide in methylene chloride at 0° [1563].

(2,4,6-Trihydroxy-1,3,5-benzenetriyl)tris[phenylmethanone

[1818-24-2]



Asymmetric ketones [1476] p. 543

$C_{27}H_{18}O_6$ mol.wt. 438.44

Described [1476] **p. 541**

Synthesis

- Obtained by Fries rearrangement of phloroglucinol tribenzoate [1777].

Part III Miscellaneous Related Compounds *(Class of METHANONES)*

Chapter 7. Miscellaneous Related Compounds

7.1 Diphenyl Derivatives [1476] p. 549

7.2 Diphenylmethane Derivatives [1476] p. 551

7.3 Diphenylethane Derivative [1476] p. 553

7.4 Diphenylpropane Derivatives [1476] p. 554

7.5 Diphenyl Oxide Derivatives [1476] p. 554

7.6 Diphenyl Sulfoxide Derivatives [1476] p. 556

7.7 Diphenyl Sulfone Derivatives [1476] p. 557

7.8 Others Acylated Compounds [1476] p. 559

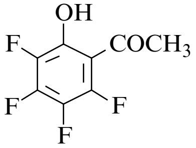
Part V

Monoketones Unsubstituted on the Acetyl Groups

Chapter 9
Compounds Derived from Acetic Acid

1-(2,3,4,5-Tetrafluoro-6-hydroxyphenyl)ethanone

[182951-74-2] $C_8H_4F_4O_2$ mol.wt. 208.11



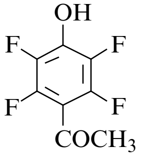
Synthesis

– Obtained by hydrolytic cleavage of 2-amino-3-(2-hydroxy-3,4,5,6-tetrafluorobenzoyl)acrylic acid (**I**) or of 3-(2-hydroxy-3,4,5,6-tetrafluorobenzoylmethylene)-piperazin-2-one (**II**) in boiling aqueous sodium hydroxide for 20 min (53% and 44% yields, respectively) [1778].

m.p. 36° [1778]; ^{1}H NMR [1778], ^{19}F NMR [1778], IR [1778].

1-(2,3,5,6-Tetrafluoro-4-hydroxyphenyl)ethanone

[145797-51-9] $C_8H_4F_4O_2$ mol.wt. 208.11



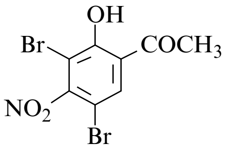
Synthesis

– Preparation by reaction of acetyl chloride on 2,3,5,6-tetrafluorophenol with aluminium chloride in carbon disulfide at 5° (10%) [1779].

m.p. 109–111° [1779].

1-(3,5-Dibromo-2-hydroxy-4-nitrophenyl)ethanone

$C_8H_5Br_2NO_4$ mol.wt. 338.94



Synthesis

– Preparation by reaction of bromine on 2-hydroxy-4-nitro-acetophenone in refluxing acetic acid–sodium acetate mixture (68%) [1780].

m.p. 157° [1780].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
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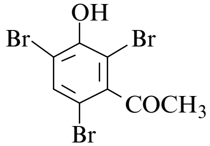
1-(2,4,6-Tribromo-3-hydroxyphenyl)ethanone

[49605-14-3] $C_8H_5Br_3O_2$ mol.wt. 372.84



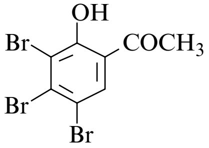
Synthesis

– Preparation by bromination of 3-hydroxyacetophenone in water at 50° (94%) [1781,1782].

m.p. 127°5 [1781,1782].

1-(3,4,5-Tribromo-2-hydroxyphenyl)ethanone

[145666-19-9] $C_8H_5Br_3O_2$ mol.wt. 372.84

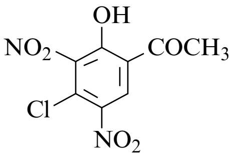


Synthesis not yet described.

– Refer to: [1783] (compound **1h**).

1-(4-Chloro-2-hydroxy-3,5-dinitrophenyl)ethanone

$C_8H_5ClN_2O_6$ mol.wt. 260.59



Syntheses

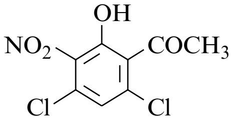
– Preparation by nitration of 4-chloro-2-hydroxy-acetophenone (77%) [1784].

– Also obtained (by-product) by nitration of 2-acetyl-5-chlorophenyl acetate in sulfuric acid solution at −10° (24%) [1784].

m.p. 49–150° [1784].

1-(4,6-Dichloro-2-hydroxy-3-nitrophenyl)ethanone

[81515-01-7] $C_8H_5Cl_2NO_4$ mol.wt. 250.04



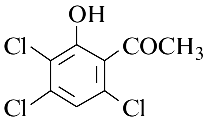
Synthesis

– Preparation by nitration of 2,4-dichloro-6-hydroxy-acetophenone with potassium nitrate in concentrated sulfuric acid, first between −10° and 0°, then at r.t. [1785].

m.p. 62–64° [1785].

1-(3,4,6-Trichloro-2-hydroxyphenyl)ethanone

[126712-08-1] $C_8H_5Cl_3O_2$ mol.wt. 239.48



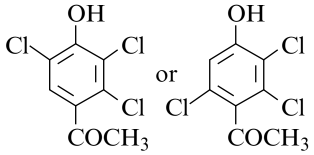
Synthesis

– Preparation by Fries rearrangement of 2,3,5-trichlorophenyl acetate with aluminium chloride without solvent at 130–140° (49%) [1786].

m.p. 103–104° [1786]; ^{1}H NMR [1786], MS [1786].

1-(Trichloro-4-hydroxyphenyl)ethanone

[94650-96-1] $C_8H_5Cl_3O_2$ mol.wt. 239.48

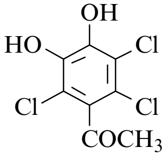


Synthesis not yet described.

- Identified in wheat and rye straw pulp bleaching and combined mill effluents [1787].
- Identified during control of effluent from the manufacturing of bleached pulp and paper from sugarcane bagasse [1788].

1-(2,3,6-Trichloro-4,5-dihydroxyphenyl)ethanone

[154638-87-6] $C_8H_5Cl_3O_3$ mol.wt. 255.48



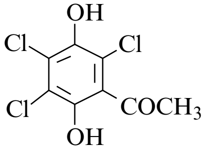
Synthesis

- Obtained (by-product) by chlorination of 4-hydroxy-3-methoxyacetophenone in dioxane–water mixture at 40° (4%) [1789].

MS [1789].

1-(2,4,5-Trichloro-3,6-dihydroxyphenyl)ethanone

[7714-14-9] $C_8H_5Cl_3O_3$ mol.wt. 255.48



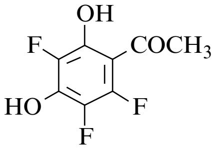
Syntheses

- Preparation by reaction of chlorine on 2,5-dihydroxyacetophenone in chloroform solution containing a drop of triethylamine, under UV light, at 0° (50%) [1790].
- Preparation by reaction of excess of chlorine on acetyl-1,4-benzoquinone in chloroform, followed by treatment of the adduct obtained with hydrochloric acid in ethyl ether (60%) [1791].

m.p. 127°5–129° [1791], 79–81° [1790]. One of the reported melting points is obviously wrong. ^{1}H NMR [1791], IR [1791], MS [1791].

1-(2,3,5-Trifluoro-4,6-dihydroxyphenyl)ethanone

[182951-75-3] $C_8H_5F_3O_3$ mol.wt. 206.12



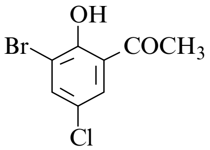
Syntheses

- Obtained by treatment of 2-amino-3-(2-hydroxy-3,4,5,6-tetrafluorobenzoyl)acrylic acid with boiling aqueous sodium hydroxide for 2 h (40%) [1778].
- Also obtained by treatment of 3-(2-hydroxy-3,4,5,6-tetra-fluorobenzoyl-methylene)piperazin-2-one with boiling aqueous sodium hydroxide for 2 h (30%) [1778].

m.p. 146–147° [1778]; ^{1}H NMR [1778], ^{19}F NMR [1778], IR [1778].

1-(3-Bromo-5-chloro-2-hydroxyphenyl)ethanone

[59443-15-1] $C_8H_6BrClO_2$ mol.wt. 249.49



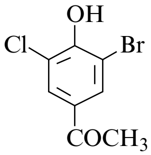
Syntheses

- Preparation by bromination of 5-chloro-2-hydroxyacetophenone in acetic acid (65%) [1792].
- Preparation by Fries rearrangement of 2-bromo-4-chlorophenyl acetate with aluminium chloride without solvent at 140° [1793].
- Also refer to: [1794,1795] (compound **1b**), [1783] (compound **1e**) and [1796] (compound **1c**).

m.p. 104–105° [1792].

1-(3-Bromo-5-chloro-4-hydroxyphenyl)ethanone

$C_8H_6BrClO_2$ mol.wt. 249.49



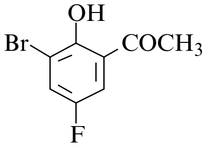
Synthesis

- Preparation by bromination of 3-chloro-4-hydroxyacetophenone [1797,1798].

m.p. 170° [1797,1798].

1-(3-Bromo-5-fluoro-2-hydroxyphenyl)ethanone

[393-62-4] $C_8H_6BrFO_2$ mol.wt. 233.04



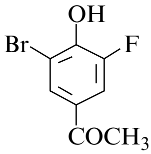
Syntheses

- Preparation by bromination of 5-fluoro-2-hydroxyacetophenone in acetic acid [1799].
- Preparation by Fries rearrangement of 2-bromo-4-fluorophenyl acetate with aluminium chloride without solvent at 130° [1800].

m.p. 97° [1799,1800].

1-(3-Bromo-5-fluoro-4-hydroxyphenyl)ethanone

[402-84-6] $C_8H_6BrFO_2$ mol.wt. 233.04



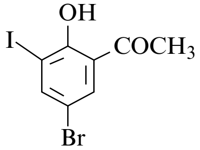
Synthesis

- Preparation by bromination of 3-fluoro-4-hydroxy-acetophenone in acetic acid [1801].

m.p. 173° [1801].

1-(5-Bromo-2-hydroxy-3-iodophenyl)ethanone

[28467-11-0] $C_8H_6BrIO_2$ mol.wt. 340.94



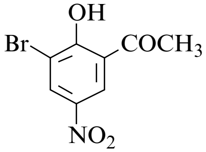
Synthesis

- Preparation by reaction of iodine–iodic acid mixture on 5-bromo-2-hydroxyacetophenone in ethanol (75–85%) [1802], (68%) [1803].

m.p. 116° [1803], 105° [1802]; ^{1}H NMR [1802], IR [1802].

1-(3-Bromo-2-hydroxy-5-nitrophenyl)ethanone

[90004-97-0] $C_8H_6BrNO_4$ mol.wt. 260.04



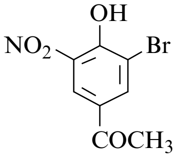
Syntheses

- Preparation by reaction of bromine on 2-hydroxy-5-nitro-acetophenone in refluxing acetic acid–sodium acetate mixture (83%) [1780].
- Also obtained by treatment of 2-hydroxy-5-nitro-acetophenone with NBS in acetonitrile (56%) (compound **31**) [1804].

m.p. 132° [1780]; ^{1}H NMR [1804], MS [1804].

1-(3-Bromo-4-hydroxy-5-nitrophenyl)ethanone

[90004-98-1] $C_8H_6BrNO_4$ mol.wt. 260.04



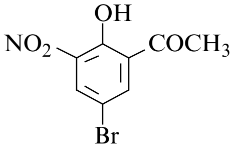
Synthesis

- Preparation by reaction of bromine on 4-hydroxy-3-nitro-acetophenone in refluxing acetic acid–sodium acetate mixture (83%) [1780].

m.p. 136° [1780].

1-(5-Bromo-2-hydroxy-3-nitrophenyl)ethanone

[70978-54-0] $C_8H_6BrNO_4$ mol.wt. 260.04



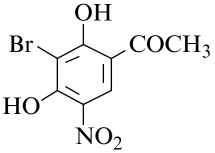
Syntheses

- Preparation by nitration of 5-bromo-2-hydroxy-acetophenone in refluxing carbon tetrachloride (88%) [1805].
- Preparation by reaction of nitric acid on 5-bromo-2-hydroxyacetophenone in concentrated sulfuric acid between −2° and 0° (75%) [1806].

m.p. 99°5 [1806]; IR [1807].

1-(3-Bromo-2,4-dihydroxy-5-nitrophenyl)ethanone

[116465-22-6] $C_8H_6BrNO_5$ mol.wt. 276.04



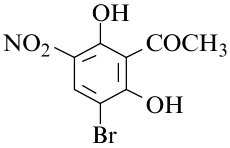
Syntheses

- Preparation by bromination of 5-nitroresacetophenone in acetic acid at 90° [1808].
- Obtained by saponification of 8-bromo-7-hydroxy-2-methyl-6-nitrochromone with 10% aqueous sodium hydroxide [1809].
- Also obtained by reaction of nitric acid on 3,3′-diacetyl-5,5′-dibromo-4,4′,6,6′-tetrahydroxy-diphenyl thioether in acetic acid at r.t. [1810].

m.p. 182–183° [1808,1810], 181–182° [1809].

1-(3-Bromo-2,6-dihydroxy-5-nitrophenyl)ethanone

$C_8H_6BrNO_5$ mol.wt. 276.04



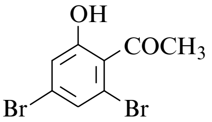
Syntheses

- Preparation by bromination of 2,6-dihydroxy-3-nitroacetophenone in acetic acid [1809].
- Preparation by reaction of nitric acid on 3,5-dibromo-2,6-dihydroxyacetophenone in acetic acid at 0° [1809].
- Obtained by saponification of 6-bromo-5-hydroxy-2-methyl-8-nitrochromone or of 8-bromo-5-hydroxy-2-methyl-6-nitrochromone with 10% aqueous sodium hydroxide at reflux [1809].

m.p. 127–128° [1809].

1-(2,4-Dibromo-6-hydroxyphenyl)ethanone

$C_8H_6Br_2O_2$ mol.wt. 293.94



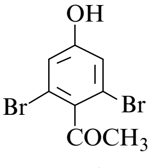
Synthesis

- Preparation by reaction of acetyl chloride on 3,5-dibromo-anisole with aluminium chloride in refluxing carbon disulfide (33%) [1811].

m.p. 96–97° [1811].

1-(2,6-Dibromo-4-hydroxyphenyl)ethanone

$C_8H_6Br_2O_2$ mol.wt. 293.94



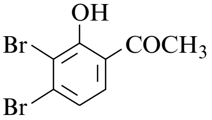
Synthesis

- Obtained by reaction of acetyl chloride on 3,5-dibromo-anisole with aluminium chloride in refluxing carbon disulfide (10%) [1811].

m.p. 141–142° [1811].

1-(3,4-Dibromo-2-hydroxyphenyl)ethanone

[145666-18-8] $C_8H_6Br_2O_2$ mol.wt. 293.94

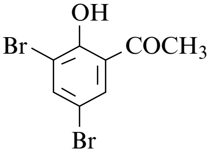


Synthesis

– Refer to: [1783] (compound **1f**).

1-(3,5-Dibromo-2-hydroxyphenyl)ethanone

[22362-66-9] $C_8H_6Br_2O_2$ mol.wt. 293.94



Syntheses

– Preparation by Fries rearrangement of 2,4-dibromophenyl acetate with aluminium chloride without solvent between 150° and 165° [1812–1814], (46%) [1813], (51%) [1812].

– Preparation by reaction of acetic anhydride on 2,4-dibromo-phenol with aluminium chloride in nitrobenzene at 120° (41%) [1813,1814].

– Preparation by bromination of 2-hydroxyacetophenone [1815,1816], (61%) [1815] or 5-bromo-2-hydroxyacetophenone in acetic acid (78%) [1792].

– Preparation by reaction of bromine on 2-hydroxyacetophenone in acetic acid–potassium acetate mixture (55%) [1816].

– Also obtained (by-product) by Fries rearrangement of 2-bromophenyl acetate with aluminium chloride without solvent at 180° (2%) [1817].

– Also obtained by reaction of sodium iodide on 3,5-dibromo-2-hydroxy-α,α,α-tribromo-acetophenone in acetic acid–dioxane–hydrochloric acid mixture (50%) [1818].

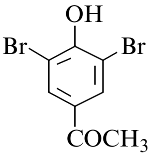
– Also obtained by action of hydriodic acid with 3,5-dibromo-2-hydroxy-α,α,α-tribromo-acetophenone [1818].

m.p. 111° [1815], 110° [1813], 109–110° [1792,1812,1818], 108–109° [1816,1817], 108° [1819];

^{1}H NMR [1817,1820], ^{13}C NMR [1821], UV [1820,1822], fluorescence spectra [1820].

1-(3,5-Dibromo-4-hydroxyphenyl)ethanone

[2887-72-1] $C_8H_6Br_2O_2$ mol.wt. 293.94



Syntheses

– Preparation by bromination of 4-hydroxyacetophenone in dilute acetic acid [1797,1798,1823], (94%) [1824], (80%) [1816], (75%) [1825], (62%) [1801].

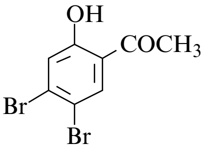
– Preparation by reaction of bromine on 4-hydroxy-acetophenone in acetic acid–potassium acetate mixture (91%) [1825], (80%) [1816].

- Preparation by bromination of 4-hydroxyacetophenone [1826].
- Preparation by Fries rearrangement of 2,6-dibromophenyl acetate with aluminium chloride without solvent at 120° (60%) [1826].
- Also refer to: [1827] (compound **9**) and [1828].

m.p. 187° [1797,1798], 184° [1826], 181° [1801,1816,1824], 180–184° [1825]; UV [1829].

1-(4,5-Dibromo-2-hydroxyphenyl)ethanone

[30186-15-3] $C_8H_6Br_2O_2$ mol.wt. 293.94



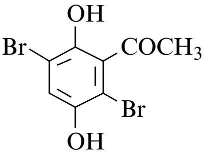
Syntheses

- Obtained (by-product) by Fries rearrangement of 2-bromophenyl acetate with aluminium chloride without solvent at 180° (1%) [1817].
- Also obtained (by-product) by Fries rearrangement of 3-bromophenyl acetate with aluminium chloride without solvent at 175–180° [1830].

m.p. 136° [1830], 130–131° [1817]; ^{1}H NMR [1817,1830].

1-(2,5-Dibromo-3,6-dihydroxyphenyl)ethanone

$C_8H_6Br_2O_3$ mol.wt. 309.94



Synthesis not yet described

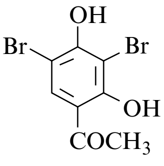
- Obtained by alkaline hydrolysis of 5,8-dibromo-6-hydroxy-2-methylchromone [1831].
- Also obtained by bromination of quinacetophenone [1831].

N.B.: The results of reference [1831] were erroneous [1832].

m.p. 86° [1831].

1-(3,5-Dibromo-2,4-dihydroxyphenyl)ethanone

[36772-98-2] $C_8H_6Br_2O_3$ mol.wt. 309.94



Syntheses

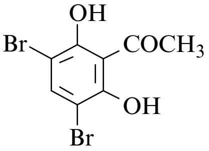
- Preparation by bromination of resacetophenone in acetic acid [1833–1838].
- Preparation by saponification of 6,8-dibromo-7-hydroxy-2-methylchromone with 5% aqueous sodium hydroxide [1809,1839].
- Obtained by reaction of bromine on 3,3′-diacetyl-4,4′,6,6′-tetrahydroxydiphenyl thioether in acetic acid at 50° [1810].
- Preparation by saponification of 6,8-dibromo-7-hydroxyflavone with refluxing 10% aqueous sodium hydroxide [1840].
- Preparation by saponification of 3-benzoyl-6,8-dibromo-7-hydroxyflavone with 10% ethanolic potassium hydroxide in a water bath [1840].

- Also obtained (by-product) by reaction of bromine with resacetophenone in ethanol at −78° [1841].

m.p. 174° [1839], 173–174° [1810,1833,1834,1838], 173° [1835], 172–173° [1809,1840].

1-(3,5-Dibromo-2,6-dihydroxyphenyl)ethanone

[63411-84-7] $C_8H_6Br_2O_3$ mol.wt. 309.94



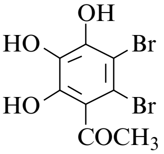
Syntheses

- Preparation by bromination of 2-acetylresorcinol in acetic acid [1809].
- Obtained by saponification of 6,8-dibromo-5-hydroxy-2-methylchromone with 10% aqueous sodium hydroxide at reflux [1809].
- Also refer to: [1840] (compound **IVc**).

m.p. 173° [1809].

1-(2,3-Dibromo-4,5,6-trihydroxyphenyl)ethanone

[65883-24-1] $C_8H_6Br_2O_4$ mol.wt. 325.94



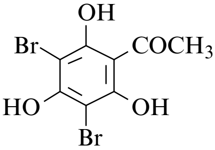
Synthesis

- Preparation by reaction of bromine on gallacetophenone in chloroform at 10° [1842,1843].

Pale yellow crystals [1842,1843].

1-(3,5-Dibromo-2,4,6-trihydroxyphenyl)ethanone

[63990-67-0] $C_8H_6Br_2O_4$ mol.wt. 325.94



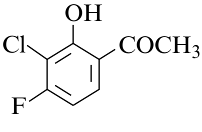
Syntheses

- Preparation by reaction of acetyl chloride or acetic anhydride on 2,4-dibromophloroglucinol with boron trifluoride (72–78%) [1844].
- Preparation by bromination of phloroacetophenone with cupric bromide in refluxing chloroform–ethyl acetate mixture [1845].

m.p. 202–204° [1844], 200–202° [1845]; ^{1}H NMR [1845], MS [1845].

1-(3-Chloro-4-fluoro-2-hydroxyphenyl)ethanone

[703-97-9] $C_8H_6ClFO_2$ mol.wt. 188.59

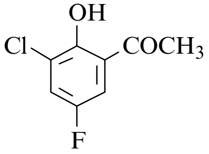


Synthesis not yet described

- There is a single reference, erroneous, concerning the 3-chloro-5-fluoro-2-hydroxyacetophenone [1800].

1-(3-Chloro-5-fluoro-2-hydroxyphenyl)ethanone

[445-38-5] $C_8H_6ClFO_2$ mol.wt. 188.59



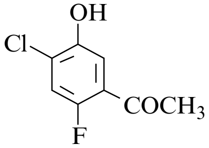
Syntheses

- Preparation by Fries rearrangement of 2-chloro-4-fluoro-phenyl acetate with aluminium chloride without solvent at 130° [1800].
- Preparation by reaction of chlorine on 5-fluoro-2-hydroxyacetophenone in acetic acid solution [1799].

m.p. 84° [1799,1800].

1-(4-Chloro-2-fluoro-5-hydroxyphenyl)ethanone

[148254-30-2] $C_8H_6ClFO_2$ mol.wt. 188.59

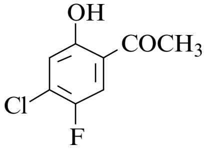


Synthesis

- Refer to: [1846] (Japanese patent).

1-(4-Chloro-5-fluoro-2-hydroxyphenyl)ethanone

[105533-69-5] $C_8H_6ClFO_2$ mol.wt. 188.59



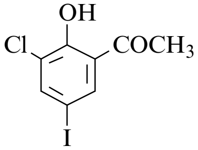
Synthesis

- Preparation by reaction of acetyl chloride on 3-chloro-4-fluorophenol with aluminium chloride at 95–100° (86%) [1847].

m.p. 72° [1847].

1-(3-Chloro-2-hydroxy-5-iodophenyl)ethanone

[292144-84-4] $C_8H_6ClIO_2$ mol.wt. 296.49



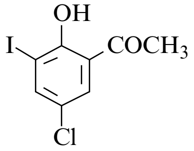
Synthesis

- Preparation by iodination of 3-chloro-2-hydroxy-acetophenone with iodine (1 mol) and iodic acid (1 mol) in ethanol at 35–40° for 1.5 h (75–85%) [1802].

m.p. 112° [1802].

1-(5-Chloro-2-hydroxy-3-iodophenyl)ethanone

[175655-11-5] $C_8H_6ClIO_2$ mol.wt. 296.49



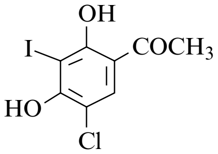
Synthesis

– Preparation by iodination of 5-chloro-2-hydroxy-acetophenone with iodine (1 mol) and iodic acid (1 mol) in ethanol at 35–40° for 1.5 h (75–85%) [1802].

m.p. 89° [1802].

1-(5-Chloro-2,4-dihydroxy-3-iodophenyl)ethanone

[102297-89-2] $C_8H_6ClIO_3$ mol.wt. 312.49



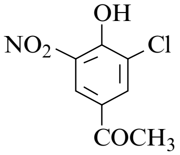
Synthesis

– Preparation by reaction of a iodine–iodic acid mixture on 5-chloro-2,4-dihydroxyacetophenone in water (73%) [1848].

m.p. 173–176° [1848]; ^{1}H NMR [1848].

1-(3-Chloro-4-hydroxy-5-nitrophenyl)ethanone

[52129-62-1] $C_8H_6ClNO_4$ mol.wt. 215.59

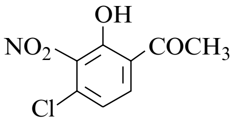


Synthesis

– Preparation by reaction of potassium nitrate on 3-chloro-4-hydroxyacetophenone in concentrated sulfuric acid between 5° and 10° [1849,1850].

1-(4-Chloro-2-hydroxy-3-nitrophenyl)ethanone

$C_8H_6ClNO_4$ mol.wt. 215.59



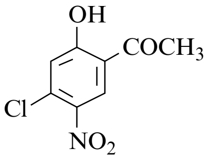
Synthesis

– Obtained (by-product) by nitration of 2-acetyl-5-chlorophenyl acetate in sulfuric acid at −20° [1784].

m.p. 105–106° [1784]; IR [1784].

1-(4-Chloro-2-hydroxy-5-nitrophenyl)ethanone

$C_8H_6ClNO_4$ mol.wt. 215.59



Syntheses

– Obtained by Fries rearrangement of 3-chloro-4-nitrophenyl acetate with aluminium chloride in nitrobenzene at 120° (18%) [1784].

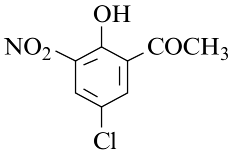
- Preparation by nitration of 2-acetyl-5-chlorophenyl acetate in sulfuric acid solution at −10° (50%) [1784].
- Preparation by hydrolysis of 2-acetyl-5-chloro-4-nitrophenyl 4-methyl-3-nitrobenzenesulfonate with 2 N sodium hydroxide (86%) [1784].

m.p. 104–105° [1784]; IR [1784].

1-(5-Chloro-2-hydroxy-3-nitrophenyl)ethanone

[84942-40-5] $C_8H_6ClNO_4$ mol.wt. 215.59

Syntheses



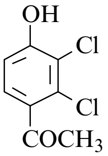
- Preparation by reaction of nitric acid on 5-chloro-2-hydroxyacetophenone in acetic acid at r.t. [1851–1853], (98%) [1852], (63%) [1853].
- Preparation by reaction of nitric acid on 5-chloro-2-hydroxyacetophenone in concentrated sulfuric acid between −2° and 0° (80%) [1806].
- Preparation by reaction of copper nitrate on 5-chloro-2-hydroxyacetophenone in acetic anhydride [1851].
- Also refer to: [1854].

m.p. 135–136° [1852], 132° [1806], 131–132° [1853]; IR [1853].

1-(2,3-Dichloro-4-hydroxyphenyl)ethanone

[2977-53-9] $C_8H_6Cl_2O_2$ mol.wt. 205.04

Syntheses



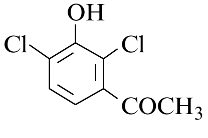
- Preparation by reaction of aluminium chloride on 2,3-dichloro-4-methoxyacetophenone in methylene chloride at 5° [1855].
- Preparation by reaction of aluminium chloride on 2,3-dichloro-4-methoxyacetophenone (or 2,3-dichloro-4-ethoxyacetophenone) in heptane [1856–1858].

m.p. 153–155° [1858].

1-(2,4-Dichloro-3-hydroxyphenyl)ethanone

[92119-05-6] $C_8H_6Cl_2O_2$ mol.wt. 205.04

Syntheses

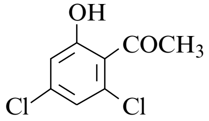


- Preparation from 3-hydroxyacetophenone by chlorination of its ethylene ketal using two equivalents of tert-butyl hypochlorite. Hydrolysis of the obtained dichloroketal with dilute hydrochloric acid in tetrahydrofuran gave essentially 2,4-dichloro-3-hydroxyacetophenone [1859].
- Also obtained (by-product) by reaction of tert-butyl hypochlorite on 3-hydroxyacetophenone in chloroform at 15°, in subdued light (9%) [1859].

Oil [1859].

1-(2,4-Dichloro-6-hydroxyphenyl)ethanone

[57051-50-0] $C_8H_6Cl_2O_2$ mol.wt. 205.04



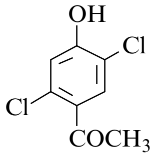
Syntheses

- Preparation by reaction of acetyl chloride on 3,5-di-chloroanisole with aluminium chloride in boiling carbon disulfide [1811,1814], (35–40%) [1811].
- Preparation by Fries rearrangement of 3,5-dichlorophenyl acetate with aluminium chloride without solvent at 120° [1785,1811].

m.p. 49–50° [1811], 48–49° [1785].

1-(2,5-Dichloro-4-hydroxyphenyl)ethanone

[73239-04-0] $C_8H_6Cl_2O_2$ mol.wt. 205.04



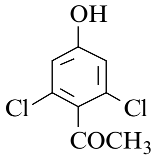
Syntheses

- Preparation by reaction of acetyl chloride on 2,5-di-chloroanisole with aluminium chloride at 110° (36%) [1860].
- Preparation by Fries rearrangement of 2,5-dichlorophenyl acetate with aluminium chloride without solvent at 155–165° (36%) [1861].

m.p. 151–152° [1860], 148–149° [1861]; ^{1}H NMR [1860], IR [1860], UV [1860].

1-(2,6-Dichloro-4-hydroxyphenyl)ethanone

$C_8H_6Cl_2O_2$ mol.wt. 205.04



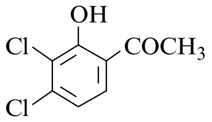
Synthesis

- Obtained by reaction of acetyl chloride on 3,5-dichloroanisole with aluminium chloride in refluxing carbon disulfide (30%) [1811].

m.p. 117–119° [1811]; b.p.$_{14}$ 197° [1811].

1-(3,4-Dichloro-2-hydroxyphenyl)ethanone

[55736-71-5] $C_8H_6Cl_2O_2$ mol.wt. 205.04



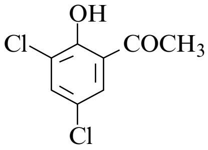
Syntheses

- Preparation by Fries rearrangement of 2,3-dichlorophenyl acetate with aluminium chloride without solvent at 130–140° [1786,1814,1862,1863], (75%) [1786].
- Also obtained by reaction of 40% peracetic acid on 2-(benzyloxy)-3,4-dichloroacetophenone in acetic acid at 50° (40%) [1864].
- Preparation by reaction of aqueous sodium hydroxide solution on 7,8-dichlorochromone-2-carboxylic acid at 95° (38%) [1865].

m.p. 113–114° [1864], 109–111° [1865], 109–110° [1786];
^{1}H NMR [1786], IR [1786], MS [1786].

1-(3,5-Dichloro-2-hydroxyphenyl)ethanone

[3321-92-4] $C_8H_6Cl_2O_2$ mol.wt. 205.04



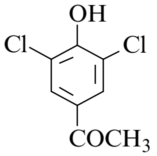
Syntheses

- Preparation by Fries rearrangement of 2,4-dichlorophenyl acetate with aluminium chloride in tetrachloroethane at 150–160° [1866] and without solvent between 115° and 170° (43–75%) [1867–1872].
- Preparation by reaction of chlorine on 2-hydroxy-acetophenone with ferric chloride in dilute acetic acid [1849,1850].
- Preparation by reaction of hydrated sodium sulfide (containing 7–9 mol of water) with 2-piperidino-4-[1873,5-dichloro-2-hydroxyphenyl]-1,3-dithiolane perchlorate in ethanol (63%) [1874].

m.p. 100–101° [1874], 97° [1870], 95–96° [1849,1850,1868,1869], 95°5 [1866], 94–96° [1872]; b.p.$_{18}$ 132–134° [1867,1871];
^{1}H NMR [1820,1869,1874], UV [1820], IR [1874], MS [1869], fluorescence spectra [1820].

1-(3,5-Dichloro-4-hydroxyphenyl)ethanone

[17044-70-1] $C_8H_6Cl_2O_2$ mol.wt. 205.04



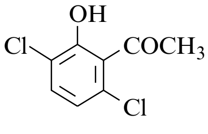
Syntheses

- Preparation by Fries rearrangement of 2,6-dichlorophenyl acetate with aluminium chloride without solvent at 140–150° [1875,1876], (69%) [1876].
- Preparation by chlorination of 4-hydroxyacetophenone in acetic acid [1849,1850,1877].

m.p. 164–165°5 [1876], 164–165° [1849], 160–160°5 [1877];
pK_a [1877].

1-(3,6-Dichloro-2-hydroxyphenyl)ethanone

[55736-72-6] $C_8H_6Cl_2O_2$ mol.wt. 205.04



Syntheses

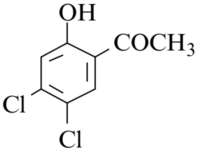
- Preparation by Fries rearrangement of 2,5-dichlorophenyl acetate with aluminium chloride,
  - without solvent, at 155–165° (57%) [1861];
  - in nitrobenzene, at 130° (42%) [1878].
- Preparation by reaction of methyl 3,6-dichloro-2-methoxybenzoate with carbanion reagent (78% yield), followed by ether cleavage (95%) [1879].
- Preparation by reaction of aqueous sodium hydroxide solution on 5,8-dichlorochromone-2-carboxylic acid on a steam bath (92%) [1865].

- Preparation by hydrolysis of 5,8-dichlorochromone with 10% sodium hydroxide at 100° (80%) [1880].
- Also refer to: [1865,1881,1882], and [1883] (compound **III**); [1884] (compound **VIIIk**); [1885] (compound **11**).

m.p. 55–56° [1865].

1-(4,5-Dichloro-2-hydroxyphenyl)ethanone

[22526-30-3] $C_8H_6Cl_2O_2$ mol.wt. 205.04



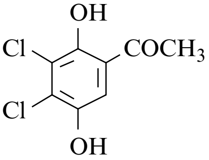
Syntheses

- Preparation by Fries rearrangement of 3,4-dichlorophenyl acetate with aluminium chloride without solvent between 120° and 140° [1786,1814,1862,1886,1887], (72–73%) [1786,1887], (52%) [1886] or at 200° [1888].
- Also obtained by cleavage of 5,6-dichloro-2,3-dimethyl-benzofuran with chromium trioxide in acetic acid at 50°, the keto ester formed was saponified with potassium hydroxide (70%) [1887].

m.p. 104–105° [1786], 102° [1887], 99–105° [1886];
b.p.$_{15}$ 151° [1887]; ^{1}H NMR [1786], IR [1786], MS [1786].

1-(3,4-Dichloro-2,5-dihydroxyphenyl)ethanone

[79755-07-8] $C_8H_6Cl_2O_3$ mol.wt. 221.04



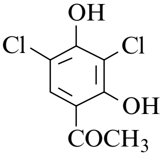
Syntheses

- The preparation from acetylhydroquinone required conversion into its ethylene acetal, oxidation to the quinone with silver oxide, addition of chlorine in acetic acid and enolisation and cleavage of the acetal with, at first hydrogen chloride in ethyl ether, then sulfuric acid in aqueous ethanol (overall yield of 51%) [1791].
- Also obtained by Fries rearrangement of 2,3-dichlorohydroquinone diacetate with aluminium chloride without solvent at 165° (poor yield) [1791].

m.p. 219–220° [1791]; ^{1}H NMR [1791], IR [1791], MS [1791].

1-(3,5-Dichloro-2,4-dihydroxyphenyl)ethanone

$C_8H_6Cl_2O_3$ mol.wt. 221.04



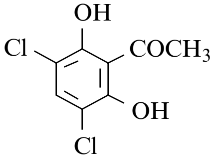
Syntheses

- Preparation by reaction of chlorine on resacetophenone in acetic acid solution [1889,1890].
- Also obtained by reaction of sulfuryl chloride on the 3,3′-di-acetyl-4,4′,6,6′-tetrahydroxydiphenyl thioether with a crystal of bismuth chloride as a catalyst [1810].

m.p. 195–196° [1810,1889,1890].

1-(3,5-Dichloro-2,6-dihydroxyphenyl)ethanone

[87953-95-5] $C_8H_6Cl_2O_3$ mol.wt. 221.04



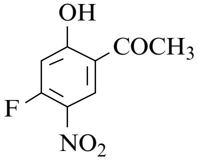
Syntheses

- Preparation by reaction of chlorine on 2,6-dihydroxyacetophenone in ethanol at 0° (84%) [1891].
- Also refer to: [1892] (compound **1d**).

m.p. 174°5–175° [1891]; IR [1891].

1-(4-Fluoro-2-hydroxy-5-nitrophenyl)ethanone

[119994-02-4] $C_8H_6FNO_4$ mol.wt. 199.14



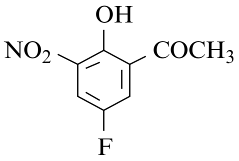
Syntheses

- Preparation by reaction of fuming nitric acid with 4-fluoro-2-hydroxyacetophenone, first between −5° and 0°, then at 0° for 30 min [1893].
- Also refer to: [1894–1896] (Japanese patents).

m.p. 96° [1893].

1-(5-Fluoro-2-hydroxy-3-nitrophenyl)ethanone

[70978-39-1] $C_8H_6FNO_4$ mol.wt. 199.14



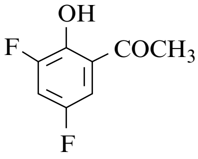
Synthesis

- Preparation by nitration of 5-fluoro-2-hydroxy-acetophenone with nitric acid (d = 1.42) in concentrated sulfuric acid between −15° and −5° (46%) [1897,1898].

m.p. 87–90° [1898], 87–89° [1897].

1-(3,5-Difluoro-2-hydroxyphenyl)ethanone

[140675-42-9] $C_8H_6F_2O_2$ mol.wt. 172.13



Syntheses

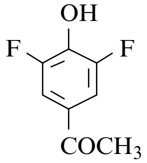
- This compound can be easily obtained by Fries rearrangement of 2,4-difluorophenyl acetate with aluminium chloride without solvent at 150°, and purified by recrystallization from ethanol (80–90%) [1899]; the 2,4-difluorophenyl acetate is prepared by reaction of acetic anhydride on 2,4-difluorophenol [1900,1901].
- Also refer to: [1902].

N.B.: This compound was purchased from Fluorochem Ltd., (Old Glossop, UK) – Catalogue 1993–1994 page 48-reference F-02826 [1821].

m.p. 108–110° [1821]; ^{13}C NMR [1821].

1-(3,5-Difluoro-4-hydroxyphenyl)ethanone

[133186-55-7] $C_8H_6F_2O_2$ mol.wt. 172.13



Syntheses

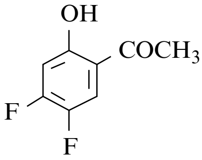
- Preparation by Fries rearrangement of 2,6-difluorophenyl acetate (b.p.$_7$ 62–63°) with aluminium chloride at 140–150° for 5 h under nitrogen atmosphere (56%) [1903].
- Also refer to: [1904].

N.B.: Refer to: [1905] (Japanese patent) and [1906]; there is one erroneous reference. It concerns the 2,6-difluorophenyl acetate (compound **11**) [1906]. This ester, by Fries rearrangement with aluminium chloride, can easily give the aforesaid ketone.

Black solid [1903]; ^{1}H NMR [1903], IR [1903].

1-(4,5-Difluoro-2-hydroxyphenyl)ethanone

[199586-38-4] $C_8H_6F_2O_2$ mol.wt. 172.13

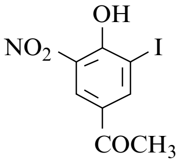


Synthesis

- Refer to: [1907].

1-(4-Hydroxy-3-iodo-5-nitrophenyl)ethanone

[76748-71-5] $C_8H_6INO_4$ mol.wt. 307.04

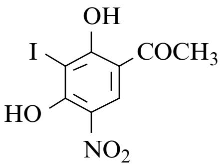


Synthesis

- Refer to: [1908,1909] (compound NIP).

1-(2,4-Dihydroxy-3-iodo-5-nitrophenyl)ethanone

$C_8H_6INO_5$ mol.wt. 323.04



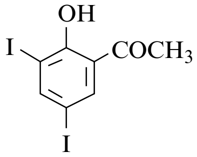
Syntheses

- Preparation by nitration of 2,4-dihydroxy-3-iodoacetophenone [1910].
- Preparation by iodination of 2,4-dihydroxy-5-nitroacetophenone [1910].

m.p. 189° [1910].

1-(2-Hydroxy-3,5-diiodophenyl)ethanone

[7191-46-0] $C_8H_6I_2O_2$ mol.wt. 387.94



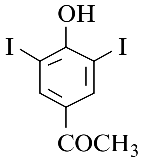
Syntheses

- Preparation by iodination of 2-hydroxyacetophenone,
  - with iodine in aqueous sodium carbonate at r.t. [1911–1915], (25%) [1914], (42%) [1915];
  - with iodine and iodic acid in ethanol at 35–40° for 1.5 h (75–85%) [1802] or at 60° [1837,1916], (75%) [1916].

m.p. 128° [1802], 127° [1916], 126° [1912], 125°5–126° [1913,1914]; UV [1914].

1-(4-Hydroxy-3,5-diiodophenyl)ethanone

[7191-55-1] $C_8H_6I_2O_2$ mol.wt. 387.94



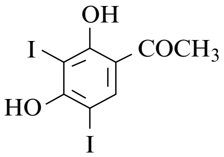
Syntheses

- Preparation by reaction of iodine on 4-hydroxy-acetophenone in aqueous sodium carbonate solution at r.t. [1913,1915,1917–1919], (75%) [1915,1918].
- Preparation by iodination of 4-hydroxyacetophenone with iodine–iodic acid mixture in ethanol at 35–40° for 1.5 h (75–85%) [1802] or in 50% aqueous ethanol (61–69%) [1837,1916,1920].
- Preparation by iodination of 4-hydroxyacetophenone by treatment with iodine monochloride (good yield) [1921,1922].
- Also obtained by Fries rearrangement of 2-iodophenyl acetate with aluminium chloride [1920].

m.p. 174–175° [1918], 172–173° [1921,1922], 171–172° [1920], 162° [1802], 158° [1916]; UV [1918].

1-(2,4-Dihydroxy-3,5-diodophenyl)ethanone

[62069-34-5] $C_8H_6I_2O_3$ mol.wt. 403.94



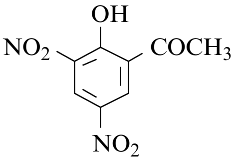
Syntheses

- Preparation by addition of an aqueous iodine and potassium iodide solution on resacetophenone in 30% ammonium hydroxide solution at r.t. (93%) [1923].
- Preparation by addition of an aqueous iodine and iodic acid solution on resacetophenone in ethanol at ambient temperature (86%) [1837,1924].

m.p. 189–190° [1923], 180° [1924]; ^{1}H NMR [1923].

1-(2-Hydroxy-3,5-dinitrophenyl)ethanone

[69027-37-8] $C_8H_6N_2O_6$ mol.wt. 226.15



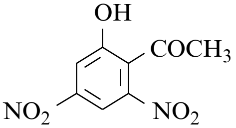
Syntheses

- Preparation by nitration of 2-hydroxyacetophenone,
  - in acetic acid (27–36%) [1925,1926];
  - without solvent (34%) [1927].

m.p. 123°5–124°5 [1926], 123–124° [1925]; IR [1928].

1-(2-Hydroxy-4,6-dinitrophenyl)ethanone

[13684-24-7] $C_8H_6N_2O_6$ mol.wt. 226.15



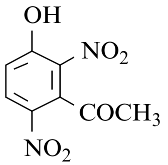
Synthesis not yet described

- Claimed to be prepared by reaction of boiling 50% nitric acid with 2-hydroxyacetophenone (34%) [1927].

N.B.: No physical data is indicated in the patent for this compound. According to the rules dealing with aromatic substitution (Holleman rules) [1929] and electronic induction using the Hammett substituent constants [1930], the obtained product most likely is the 2-hydroxy-3,5-dinitroacetophenone.

1-(3-Hydroxy-2,6-dinitrophenyl)ethanone

[172669-49-7] $C_8H_6N_2O_6$ mol.wt. 226.15



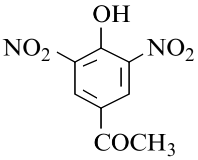
Syntheses

- Obtained by action of concentrated nitric acid (d = 1.42) with 3-hydroxyacetophenone in concentrated sulfuric acid at –20° for 15 min (25%) [1931].
- Also refer to: [1932].

m.p. 182–183° [1931]; ^{1}H NMR [1931]; Crystal data [1931].

1-(4-Hydroxy-3,5-dinitrophenyl)ethanone

[52129-61-0] $C_8H_6N_2O_6$ mol.wt. 226.15



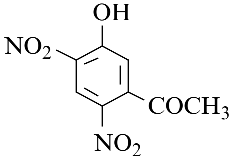
Syntheses

- Preparation by nitration of 4-hydroxyacetophenone with potassium nitrate in concentrated sulfuric acid at 5–10° [1849,1850].
- Preparation by nitration of 4-hydroxy-3-nitroacetophenone with potassium nitrate in sulfuric acid at 5–10° (64–70%) [1933,1934].

m.p. 123°2–123°5 [1933], 119–121° [1849], 119–120°5 [1934].

1-(5-Hydroxy-2,4-dinitrophenyl)ethanone

[22633-36-9] $C_8H_6N_2O_6$ mol.wt. 226.15

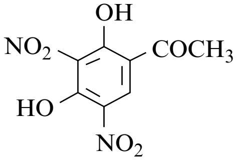


Synthesis not yet described

- There is a single reference, erroneous. It concerns the 2-hydroxy-3,5-dinitroacetophenone or 6′-hydroxy-2′,4′-di-nitroacetophenone [1928].

1-(2,4-Dihydroxy-3,5-dinitrophenyl)ethanone

[54917-82-7] $C_8H_6N_2O_7$ mol.wt. 242.14



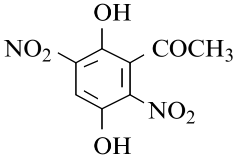
Syntheses

- Preparation by reaction of nitric acid on resacetophenone [1818,1935] or 4-acetoxy-2-hydroxyacetophenone in acetic acid [1818].
- Also obtained by reaction of nitric acid on 3-benzoyl-2,4-dihydroxyacetophenone 4-β-glucopyranoside (35–36%) [1936].
- Also obtained by reaction of nitric acid on 3,3′-diacetyl-4,4′,6,6′-tetrahydroxydiphenyl thioether in a water bath [1810].
- Resacetophenone by treatment with cerium (IV) ammonium nitrate in hot acetic acid yields 2,4-di-hydroxy-3,5-dinitroacetophenone (22%) [1937].
- Preparation by reaction of an ammonia liquor on 7-hydroxy-4-methyl-3,6,8-trinitrocoumarin in a boiling water bath [1938].
- Also obtained by hydrolysis of 7-hydroxy-2-methyl-6,8-dinitrochromone by heating its solution in sodium hydroxide (4%) on a steam bath [1935].
- Also obtained by reaction of concentrated nitric acid (d = 1.42) or fuming nitric acid on the 7-hydroxy-2-methylchromone in acetic acid, heated on a steam bath [1935].
- Also obtained by hydrolysis of 7-hydroxy-8-nitroflavone by refluxing with 10% sodium hydroxide solution [1935].

m.p. 168–169° [1938], 168° [1937], 167° [1935,1939], 166–167° [1810,1818], 165–167° [1936].

1-(2,5-Dihydroxy-3,6-dinitrophenyl)ethanone

$C_8H_6N_2O_7$ mol.wt. 242.14



Syntheses

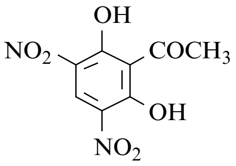
- Obtained by alkaline hydrolysis of 6-hydroxy-2-methyl-5,8-dinitrochromone [1831].
- Also obtained by nitration of quinacetophenone [1831].

N.B.: The results of reference [1831] were erroneous [1832].

m.p. 69–70° [1831].

1-(2,6-Dihydroxy-3,5-dinitrophenyl)ethanone

$C_8H_6N_2O_7$ mol.wt. 242.14



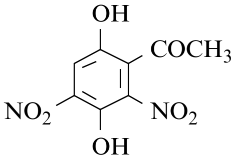
Synthesis

- Preparation by nitration of 2,6-dihydroxyacetophenone (31%) [1940].

m.p. 104° [1940].

1-(3,6-Dihydroxy-2,4-dinitrophenyl)ethanone

$C_8H_6N_2O_7$ mol.wt. 242.14



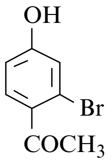
Synthesis

- Preparation by Fries rearrangement of 3,5-dinitro-4-hydroxyphenyl acetate with aluminium chloride in nitrobenzene (61%) [1941].

m.p. 133–134° [1941].

1-(2-Bromo-4-hydroxyphenyl)ethanone

[61791-99-9] $C_8H_7BrO_2$ mol.wt. 215.05



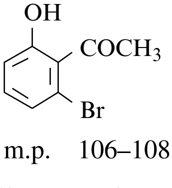
Syntheses

- Obtained (by-product) by Fries rearrangement of 3-bromophenyl acetate with aluminium chloride without solvent at 45° (10%) [1942].
- Also refer to: [1943,1944] and [1945] (Japanese patent).

m.p. 85–89° [1942].

1-(2-Bromo-6-hydroxyphenyl)ethanone

[55736-69-1] $C_8H_7BrO_2$ mol.wt. 215.05



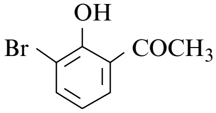
Synthesis

- Preparation by diazotization of 2-amino-6-bromoacetophenone, followed by hydrolysis of the obtained diazonium salt (50%) [1865].

m.p. 106–108° [1865].

1-(3-Bromo-2-hydroxyphenyl)ethanone

[1836-05-1] $C_8H_7BrO_2$ mol.wt. 215.05



Syntheses

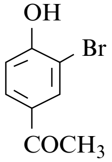
- Preparation by diazotization of 5-amino-3-bromo-2-hydroxyacetophenone, followed by hydrolysis of the obtained diazonium salt (49%) [1817].

- Also obtained (by-product) by Fries rearrangement of 2-bromophenyl acetate with aluminium chloride without solvent at 120–140° (11–13%) [1817,1946,1947].
- Also obtained by reaction of bromine on 2-hydroxyacetophenone with tert-butylamine in methylene chloride at −70° (12%) [1948] or in aqueous acetic acid [1821].

m.p. 33° [1817], 30°5–32°5 [1949,1950]; b.p.$_6$ 140–145° [1946];
^{1}H NMR [1817], ^{13}C NMR [1821], UV [1946,1947].

1-(3-Bromo-4-hydroxyphenyl)ethanone

[1836-06-2] $C_8H_7BrO_2$ mol.wt. 215.05



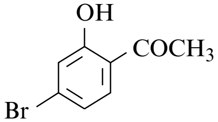
Syntheses

- Preparation by reaction of acetyl chloride on 2-bromo-phenol with aluminium chloride in refluxing carbon disulfide (86%) [1951].
- Preparation by bromination of 4-hydroxyacetophenone (70%) [1952].
- Preparation by diazotization of 3-amino-4-hydroxyacetophenone and replacement of the diazonium group by bromine (Sandmeyer reaction) (51%) [1953].
- Preparation by Fries rearrangement of 2-bromophenyl acetate with aluminium chloride without solvent at 100–120° [1817,1946,1947,1954], (68%) [1817].
- Also obtained by reaction of ammonium tribromide on 4-hydroxyacetophenone in methylene chloride–methanol mixture [1955].
- Also refer to: [1828].

m.p. 129° [1817], 125–128° [1951], 119–121° (anhydrous) [1952], 112° [1953], 97–99° (hydrate) [1952], 95° [1946], 93–94° [1955], 92–95° [1954];
^{1}H NMR [1817], UV [1946,1947].

1-(4-Bromo-2-hydroxyphenyl)ethanone

[30186-18-6] $C_8H_7BrO_2$ mol.wt. 215.05



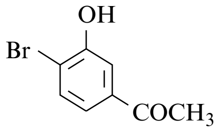
Syntheses

- Preparation by Fries rearrangement of 3-bromophenyl acetate with aluminium chloride [1942,1956–1959],
  - without solvent, at 170–180° (85–88%) [1942,1956,1957];
  - in chlorobenzene, heating in a water bath [1959].
- Also obtained (by-product) by Fries rearrangement of 2-bromophenyl acetate with aluminium chloride without solvent at 180° (6%) [1817] (intermolecular bromine migration).
- Also refer to: [1960–1962].

m.p. 43° [1959], 42–43° [1942,1956,1957]; ^{1}H NMR [1817], IR [1963].

1-(4-Bromo-3-hydroxyphenyl)ethanone

[73898-22-3] $C_8H_7BrO_2$ mol.wt. 215.05



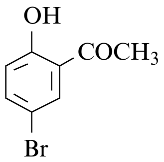
Synthesis

- Preparation by diazotization of 4-amino-3-hydroxyacetophenone and replacement of the diazonium group by bromine (Sandmeyer reaction) (36%) [1951].

m.p. 122–123° [1951].

1-(5-Bromo-2-hydroxyphenyl)ethanone

[1450-75-5] $C_8H_7BrO_2$ mol.wt. 215.05



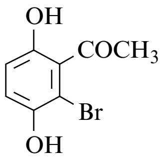
Syntheses

- Preparation by Fries rearrangement of 4-bromophenyl acetate with aluminium chloride without solvent between 110° and 160° [1805,1837,1946,1947,1964–1968], (84–91%) [1805,1946,1964,1968].
- Preparation by reaction of acetic acid on 4-bromophenol with boron trifluoride in a sealed tube at 120° (44%) [1969].
- Preparation by bromination of 2-hydroxyacetophenone in acetic acid [1792,1815,1821,1970], (63%) [1815].
- Also obtained by reaction of bromine on 2-hydroxyacetophenone with tert-butylamine in methylene chloride at −70° (17%) [1948].
- Also obtained by reaction of sodium ethoxide on 6-bromo-4-phenacylideneflavene in refluxing ethanol [1971,1972].
- Also obtained (by-product) by Fries rearrangement of 2-bromophenyl acetate with aluminium chloride without solvent at 180° (6%) [1817].
- Also refer to: [1973,1974].

m.p. 62–63° [1946], 62° [1815], 61–62° [1792,1971,1972], 59–60° [1965,1975], 59° [1967], 57°4–59°2 [1970], 57°3 [1966], 56° [1969], 55–56° [1964], 43–44° [1968];
b.p.$_7$ 110–115° [1968], b.p.$_{20}$ 143° [1964], b.p.$_{20}$ 145–148° [1815];
^{1}H NMR [1817,1976], ^{13}C NMR [1821,1976], IR [1963,1964], UV [1946,1947,1964] ;
pK$_a$ [1977].

1-(2-Bromo-3,6-dihydroxyphenyl)ethanone

[30095-76-2] $C_8H_7BrO_3$ mol.wt. 231.05



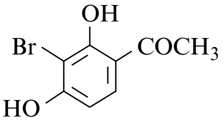
Synthesis

- Obtained from 5-bromo-6-hydroxy-2-methylchromone by alkaline degradation with 10% aqueous sodium hydroxide solution at reflux (22%) [1832].

m.p. 143–144° [1832];
^{1}H NMR [1832], IR [1832], UV [1832].

1-(3-Bromo-2,4-dihydroxyphenyl)ethanone

[60990-39-8] $C_8H_7BrO_3$ mol.wt. 231.05



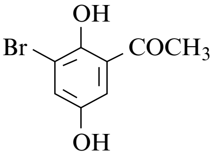
Syntheses

- Obtained by reaction of cupric bromide on resacetophenone in refluxing chloroform–ethyl acetate mixture (6%) [1978].
- Also obtained by reaction of acetic acid on 2-bromo-resorcinol with zinc chloride (Nencki reaction) (26%) [1840].
- Also obtained by saponification of 8-bromo-7-hydroxyflavone with 10% aqueous sodium hydroxide on a steam bath (14%) [1840].
- Preparation by bromination of resacetophenone in acetic acid with bromine in the presence of quinoline sulfate while cooling (90%) [1979] according to the method [1980] or in ethanol at −78° (43%) [1841].

m.p. 139° [1979], 134–135° [1978], 133° [1840], 118–121° [1841]; ^{1}H NMR [1841,1978], IR [1841], MS [1841].

1-(3-Bromo-2,5-dihydroxyphenyl)ethanone

[33857-20-4] $C_8H_7BrO_3$ mol.wt. 231.05



Syntheses

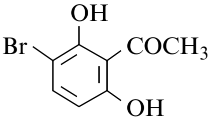
- Preparation from 3-bromo-2-hydroxyacetophenone by persulfate oxidation (Elbs reaction) [1949].
- Also obtained by hydrolysis of 8-bromo-6-hydroxy-2-methylchromone with a 10% aqueous solution of sodium hydroxide, heated on a sand bath [1831].
- Also obtained by reaction of bromine on quinacetophenone in acetic acid at r.t. [1831].
- Also refer to: [1854,1981].

N.B.: All the results of reference [1831] were erroneous [1832].

m.p. 142–144° [1949], 132° [1831].

1-(3-Bromo-2,6-dihydroxyphenyl)ethanone

[82320-47-6] $C_8H_7BrO_3$ mol.wt. 231.05



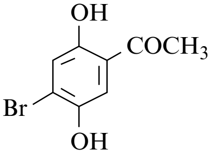
Syntheses

- Obtained by saponification of 8-acetyl-6-bromo-7-hydroxy-4-methylcoumarin with 10% aqueous sodium hydroxide solution at reflux (64%) [1982,1983].
- Also obtained by decarboxylation of 3-acetyl-5-bromo-2,4-dihydroxybenzoic acid with dilute acetic acid containing few drops of concentrated hydrochloric acid, at reflux (40%) [1982,1983].
- Preparation by reaction of 2-carboxyethyltriphenylphosphonium perbromide on 2,6-dihydroxy-acetophenone in tetrahydrofuran at r.t. [1984].

m.p. 143° [1982,1983], 140–142° [1984]; ^{1}H NMR [1984].

1-(4-Bromo-2,5-dihydroxyphenyl)ethanone

[52376-19-9] $C_8H_7BrO_3$ mol.wt. 231.05



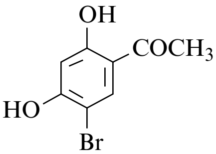
Syntheses

- Preparation by reaction of boron tribromide on 4-bromo-2,5-dimethoxyacetophenone in methylene chloride at −70° (90%) [1985].
- Preparation by Fries rearrangement of 2-bromo-hydroquinone diacetate with aluminium chloride without solvent between 160° and 180° (66%) [1986], (25%) [1987].

m.p. 152° [1985], 148–149° [1986], 132° [1987].

1-(5-Bromo-2,4-dihydroxyphenyl)ethanone

[60965-25-5] $C_8H_7BrO_3$ mol.wt. 231.05



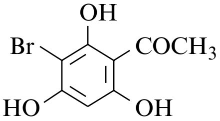
Syntheses

- Preparation by reaction of acetic acid on 4-bromoresorcinol with zinc chloride (Nencki reaction) [1890,1988].
- Preparation by bromination of resacetophenone in acetic acid at r.t. [1834].
- Preparation by bromination of resacetophenone with cupric bromide in refluxing chloroform–ethyl acetate mixture (26%) [1978].
- Preparation by saponification of 4-acetoxy-5-bromo-2-hydroxyacetophenone with sodium hydroxide in dilute methanol at r.t. (76–83%) [1836,1989].
- Also obtained by saponification of 3-benzoyl-6-bromo-7-hydroxyflavone with 10% ethanolic potassium hydroxide in a water bath [1840].
- Preparation by Fries rearrangement of 4-bromoresorcinol diacetate with aluminium chloride in boiling nitrobenzene [1836].

m.p. 171° [1836], 170–171° [1978], 167° [1834,1840,1988], 165–170° [1989]; ^{1}H NMR [1978,1989].

1-(3-Bromo-2,4,6-trihydroxyphenyl)ethanone

[155818-27-2] $C_8H_7BrO_4$ mol.wt. 247.05

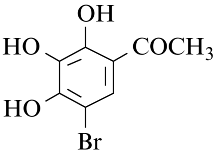


Synthesis

- Preparation by reaction of cupric bromide on phloroacetophenone in refluxing chloroform–ethyl acetate mixture [1845].

^{1}H NMR [1845].

1-(5-Bromo-2,3,4-trihydroxyphenyl)ethanone



$C_8H_7BrO_4$ mol.wt. 247.05

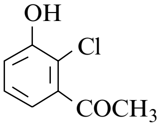
Syntheses

- Preparation by bromination of gallacetophenone in acetic acid with bromine in the presence of quinoline sulfate while cooling [1990], (95%) [1979] according to the method [1980].
- Also obtained by reaction of acetyl chloride with 4-bromo-pyrogallol [1990].

m.p. 186° [1979], 183° [1990].

1-(2-Chloro-3-hydroxyphenyl)ethanone

[69240-96-6] $C_8H_7ClO_2$ mol.wt. 170.60



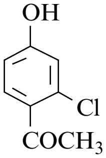
Syntheses

- Preparation by reaction of tert-butyl hypochlorite on 3-hydroxyacetophenone in chloroform at 15°, in subdued light (49%) [1859].
- Also refer to: [1991] (compound **XVIIa**) and to [1992].

m.p. 61–63° [1859]; ^{1}H NMR [1859], IR [1859].

1-(2-Chloro-4-hydroxyphenyl)ethanone

[68301-59-7] $C_8H_7ClO_2$ mol.wt. 170.60



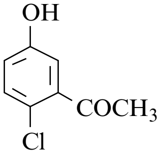
Syntheses

- Obtained (by-product) by Fries rearrangement of 3-chlorophenyl acetate with aluminium chloride [1958,1993,1994].
- Preparation by reaction of acetyl chloride on 3-chloro-phenol with aluminium chloride (Friedel–Crafts reaction) [1958].
- Also refer to: [1991] (compound **XVIIb**) and to [1992].

m.p. 110° [1994].

1-(2-Chloro-5-hydroxyphenyl)ethanone

[58020-38-5] $C_8H_7ClO_2$ mol.wt. 170.60

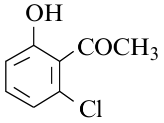


Syntheses

- Obtained by reaction of tert-butyl hypochlorite on 3-hydroxyacetophenone in chloroform at 15°, in subdued light (24%) [1859].
- Also refer to: [1995] (Japanese patent), [1991] (compound **XVIIc**) and to [1992].

1-(2-Chloro-6-hydroxyphenyl)ethanone

[55736-04-4] $C_8H_7ClO_2$ mol.wt. 170.60



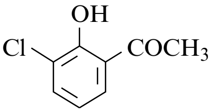
Synthesis

- Preparation by diazotization of 2-amino-6-chloro-acetophenone, followed by hydrolysis of the obtained diazonium salt (55%) [1865].
- The reference [1996] is erroneous. The compound above mentioned is, in reality, the 5-chloro-2-hydroxyacetophenone.

Oil [1865], b.p.$_{0.5}$ 78–80° [1865].

1-(3-Chloro-2-hydroxyphenyl)ethanone

[3226-34-4] $C_8H_7ClO_2$ mol.wt. 170.60



Syntheses

- Preparation by Fries rearrangement of 2-chlorophenyl acetate with aluminium chloride,
  - without solvent, between 110° and 180° (40–21%) [1817,1837,1947,1994,1997];
  - in tetrachloroethane at 70–80° [1866].
- Preparation in two steps: At first, by reaction of acetyl chloride on 2-chlorophenol with aluminium chloride in refluxing petroleum ether, one obtains the 2-acetoxy-3-chloroacetophenone (83%); the saponification of this keto ester leads to the 3-chloro-2-hydroxyacetophenone (93%) [1867,1871].
- Obtained by reaction of acetyl chloride on 2-chlorophenol with ferric chloride at r.t. (22%) [1998].

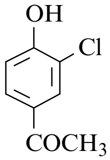
m.p. 84° [1997], 55° [1994], 49–50° [1817], 48°5 [1866]. One of the reported melting points is obviously wrong.

b.p.$_6$ 66–67° [1998], b.p.$_1$ 87–89° [1867,1871], b.p.$_{20}$ 148–153° [1866]; UV [1947,1998].

1-(3-Chloro-4-hydroxyphenyl)ethanone

[2892-29-7] $C_8H_7ClO_2$ mol.wt. 170.60

Syntheses



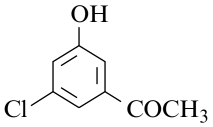
- Preparation by reaction of acetyl chloride on 2-chloro-phenol with aluminium chloride in refluxing carbon disulfide (98%) [1951].
- Preparation by diazotization of 3-amino-4-hydroxy-acetophenone and replacement of diazonium group by chlorine (Sandmeyer reaction) [1953,1999], (84%) [1953].

- Preparation by Fries rearrangement of 2-chlorophenyl acetate with aluminium chloride without solvent [1797,1817,1947,1954,1994,1998], (60–66%) [1817,1998].
- Preparation by Fries rearrangement of 2-chlorophenyl acetate with aluminium chloride in nitrobenzene at 100° (30%) [1994].
- Preparation by Fries rearrangement of 2-chlorophenyl acetate with aluminium chloride in tetrachloroethane at 70–80° [1866].
- Also obtained by reaction of acetyl chloride on 2-chlorophenol with ferric chloride [1823,1998,2000].
- Also obtained (by-product) by reaction of acetyl chloride on 2-chloroanisole or 2-chlorophenetole with aluminium chloride [2001].

m.p. 107–108° [1866], 107° [2001], 100-100°5 [1817], 96° [1823,1994,1999], 95° [1953], 94–95° [1998], 93° [1797], 92–95° [1951,1954];
^{1}H NMR [1817], IR [1866], UV [1947,1998]; pK_a [1977].

1-(3-Chloro-5-hydroxyphenyl)ethanone

[23164-97-8] $C_8H_7ClO_2$ mol.wt. 170.60

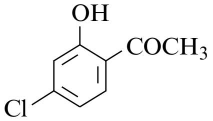


Synthesis not yet described

- The reference [1947] is erroneous. In this one, the compound **4** is, in reality, the 5-chloro-2-hydroxyacetophenone.
- Also refer to: [2002].

1-(4-Chloro-2-hydroxyphenyl)ethanone

[6921-66-0] $C_8H_7ClO_2$ mol.wt. 170.60



Syntheses

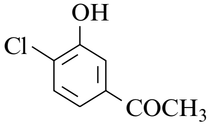
- Preparation by Fries rearrangement of 3-chlorophenyl acetate with aluminium chloride,
  - without solvent at 130–135° (74–75%) [2003,2004], between 135° and 200° [1784,1956,1993,1994,2003–2005], and 175–200° (85–89%) [1784,1956];
  - with solvent, at r.t., in nitrobenzene (88%) [2004], in tetrachloroethane (50%) [1994] or in chlorobenzene [1959].
- Also obtained by treatment of 4-chloroacetophenone with sodium trifluoroacetate in nitromethane–trifluoroacetic acid–trifluoroacetic anhydride mixture in the presence of a platinum electrode followed by treatment of the intermediate trifluoroacetate ester with 10% potassium hydrogen carbonate solution (51%) [2006] (hydroxylation of aromatic compounds).
- Preparation by reaction of methyl magnesium iodide on 4-chloro-2-hydroxybenzoyl chloride in ethyl ether, at –70°, followed by hydrolysis of the complex so obtained [2007].

m.p. 50–51° [1993], 26° [2005]. One of the reported melting points is obviously wrong.

b.p.$_2$ 96° [2004], b.p.$_{14}$ 119–122° [1784], b.p.$_{10}$ 120–122° [2003], b.p.$_{15}$ 121–124° [1956], b.p.$_{16}$ 126° [2005], b.p. 247° [1959]; IR [1784,1963], (Sadtler: standard n° 8980); pK$_a$ [1977].

1-(4-Chloro-3-hydroxyphenyl)ethanone

[61124-56-9] $C_8H_7ClO_2$ mol.wt. 170.60



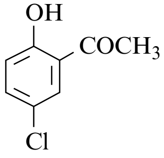
Syntheses

- Preparation by diazotization of 3-amino-4-chloro-acetophenone (quantitative yield) [2007], (16–18%) [1951].
- Obtained by treatment of 4-chloroacetophenone with sodium trifluoroacetate in nitromethane–trifluoroacetic acid–trifluoroacetic anhydride mixture in the presence of a platinum electrode, followed by treatment of the intermediate trifluoroacetate ester with 10% potassium hydrogen carbonate solution (33%) [2006] (hydroxylation of aromatic compound).
- Preparation by reaction of pyridinium chloride on 4-chloro-3-methoxyacetophenone between 170° and 200° (40%) [1951].
- Preparation from 3-hydroxyacetophenone by chlorination of its 2,3-butylene ketal or its ethylene ketal using tert-butyl hypochlorite. The hydrolysis of these chloroketals with concentrated hydrochloric acid in tetrahydrofuran–water mixture gave 3-hydroxy-4-chloroacetophenone (94% and 48% yields, respectively) [1859].
- Also obtained by reaction of acetyl chloride on 2-chlorophenol with ferric chloride [1823].
- Also obtained (by-product) by reaction of tert-butyl hypochlorite on 3-hydroxyacetophenone in chloroform at 15° (6%) [1859].

m.p. 106–107° [1859], 103–104° [1951], 96° [1823]; ^{1}H NMR [1859], IR [1859].

1-(5-Chloro-2-hydroxyphenyl)ethanone

[1450-74-4] $C_8H_7ClO_2$ mol.wt. 170.60



Syntheses

- Preparation by Fries rearrangement of 4-chlorophenyl acetate with aluminium chloride without solvent between 110° and 200° [1837,1851,1867,1871,1947,1965,1967, 1970,1994,1998,2008–2014], (90–100% yield) [1970,1994,1998,2008–2010,2012–2014].

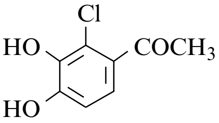
- Preparation by Fries rearrangement of 4-chlorophenyl acetate with aluminium chloride,
  - in tetrachloroethane at 150–160° [1866];
  - in chlorobenzene, in a sealed tube and subjected to high power microwave irradiation for 2 min only (85%) [2013].
- Preparation by Fries rearrangement of 4-chlorophenyl acetate with boron trifluoride in acetic acid at 125°, in a sealed tube (91%) [1969].
- Preparation by photo-Fries rearrangement of 4-chlorophenyl acetate with potassium carbonate in hexane at r.t. (88%) [2015].
- Preparation by reaction of acetyl chloride on 4-chlorophenol with ferric chloride [1823,2000,2008].
- Preparation by reaction of acetyl chloride on 4-chlorophenol with aluminium chloride in refluxing petroleum ether, and saponification of the obtained keto ester, the 2-acetoxy-5-chloroacetophenone (90%) [1871].
- Preparation by reaction of acetic acid on 4-chlorophenol with boron trifluoride at 150°, in a sealed tube (94%) [1969].
- Preparation by diazotization of 5-amino-2-hydroxyacetophenone and replacement of the diazonium group by chlorine (Sandmeyer reaction) [2016].
- Also obtained (by-product) by heating 4-chlorophenyl 3,3-dimethylacrylate at 255–260°, in a sealed tube (4%) [2017].
- Also refer to: [1794] (compound **1b**); [1795] (compound **1a**); and to [1783] (compound **1d**).

m.p. 72° [2010], 57° [2016], 56–57° [1994], 55° [1823,1998], 54° [1969,2011], 53°5–54°5 [2014], 53–54° [1965,2012], 53° [1866,2017], 52°5–53° [1975], 52°2–53°6 [1970], 52°5 [1967], 52–53° [2009], 52° [1851], 50–52° [2015];

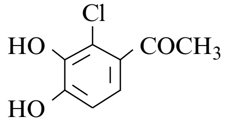
b.p.$_2$ 97–99° [2011], b.p.$_{12}$ 107–109° [1867,1871], b.p.$_{14}$ 125–126° [1969], b.p.$_{28}$ 126–128° [1871], b.p.$_{26-27}$ 134–136° [2009];
^{1}H NMR [1820,1976], ^{13}C NMR [1976], IR [1963,2010],
UV [1820,1947,1970,1998], fluorescence spectra [1820]; pK$_a$ [1977].

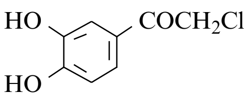
1-(2-Chloro-3,4-dihydroxyphenyl)ethanone

[56961-48-9] $C_8H_7ClO_3$ mol.wt. 186.59



N.B.: The titled compound seems, to our knowledge, to have never been prepared so far [2018]. However, in the various considered publications which mentioned the usage of this compound, for example [2019], it is always another substance that is used as starting material, namely [99-40-1].



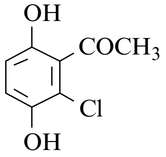


As far as the titled compound is concerned, the same confusion occurs when consulting the *Chemical Abstracts Service*, *Registry Handbook*, Number Section, (1975 supplement, p. 2202RD) and *the Beilstein Institut zur Foerderung der Chemischen Wissenschaften* (Copyright 1988–2001), Beilstein Registry Number 4921697.

Both above mentioned documents use the same Registry Number [56961-48-9], though both compounds are different even if have the same raw formula $C_8H_7ClO_3$.

1-(2-Chloro-3,6-dihydroxyphenyl)ethanone

[52095-12-2] $C_8H_7ClO_3$ mol.wt. 186.59



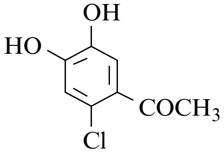
Syntheses

- Preparation by reaction of aluminium chloride on 2-acetylquinone in acetone (87%) [2020].
- Preparation by reaction of hydrochloric acid on 2-acetylquinone in benzene (89%) [2020].

m.p. 94–96° [2020]; ^{1}H NMR [2020], IR [2020].

1-(2-Chloro-4,5-dihydroxyphenyl)ethanone

[69240-97-7] $C_8H_7ClO_3$ mol.wt. 186.59

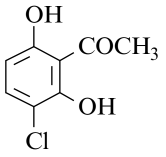


Synthesis

- Refer to: [1991] (compound **XVIId**).

1-(3-Chloro-2,6-dihydroxyphenyl)ethanone

[87953-93-3] $C_8H_7ClO_3$ mol.wt. 186.59



Syntheses

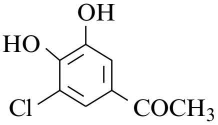
- Preparation by reaction of sulfuryl chloride on 2,6-dihydroxyacetophenone in refluxing ethyl ether (90%) [2021].
- Preparation by hydrolysis of 8-acetyl-6-chloro-7-hydroxy-4-methylcoumarin with 10% aqueous sodium hydroxide solution at reflux [1982,1983].
- Obtained by decarboxylation of 3-acetyl-5-chloro-2,4-dihydroxybenzoic acid [1982,1983].

- Preparation by reaction of thionyl chloride on 2,6-dihydroxyacetophenone in refluxing acetic acid (77%) [2022,2023].

m.p. 135° [2021], 134–135° [1982,1983].

1-(3-Chloro-4,5-dihydroxyphenyl)ethanone

[154638-86-5] $C_8H_7ClO_3$ mol.wt. 186.59



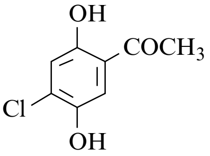
Synthesis

- Obtained (by-product) by chlorination of 4-hydroxy-3-methoxyacetophenone (acetoguaiacone) in dioxane–water mixture at 40° (8%) [1789].

MS [1789].

1-(4-Chloro-2,5-dihydroxyphenyl)ethanone

[90110-31-9] $C_8H_7ClO_3$ mol.wt. 186.59



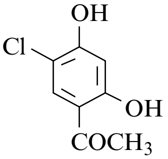
Syntheses

- Preparation by Fries rearrangement of 2-chloro-1,4-di hydroxyphenyl diacetate with aluminium chloride without solvent [1987,2024], at 160° (35%) [1987].
- Also refer to: [2025].

m.p. 145° [1987,2024].

1-(5-Chloro-2,4-dihydroxyphenyl)ethanone

[90110-32-0] $C_8H_7ClO_3$ mol.wt. 186.59



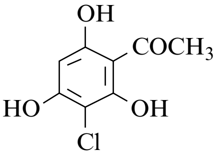
Syntheses

- Preparation by reaction of acetic acid on 4-chlororesorcinol with zinc chloride at 145° (Nencki reaction) (31%) [1988].
- Preparation by reaction of acetic anhydride on 4-chlororesorcinol with polyphosphoric acid in the presence of one drop of concentrated sulfuric acid at reflux (12%) [2026].
- Also obtained by reaction of cuprous cyanide on 5-chloro-2,4-dihydroxy-3-iodoacetophenone in HMPT at 90° (conditions of the Rosenmund-von Braun reaction) (80%) [1848].

m.p. 176–177° [2026], 174° [1848], 171° [1988].

1-(3-Chloro-2,4,6-trihydroxyphenyl)ethanone

[130435-29-9] $C_8H_7ClO_4$ mol.wt. 202.59



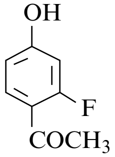
Synthesis

- Preparation by reaction of sulfuryl chloride on phloroacetophenone in ethyl ether at r.t. (86%) [2027].

m.p. 215–218° [2027]; ^1H NMR [2027], MS [2027].

1-(2-Fluoro-4-hydroxyphenyl)ethanone

[98619-07-9] $C_8H_7FO_2$ mol.wt. 154.14



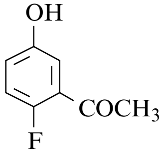
Synthesis

– Preparation by reaction of acetyl chloride on 3-fluorophenol with aluminium chloride in refluxing ethylene dichloride [2028].

^{1}H NMR [2028], IR [2028], MS [2028].

1-(2-Fluoro-5-hydroxyphenyl)ethanone

[145300-04-5] $C_8H_7FO_2$ mol.wt. 154.14



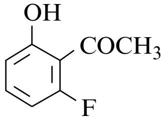
Synthesis

– Preparation by aromatization of 5-acetyl-4-fluoro-3-cyclohexenone promoted by cupric bromide–lithium bromide mixture in refluxing acetonitrile (70%) [2029,2030].

m.p. 97° [2029,2030]; ^{1}H NMR [2030], IR [2030].

1-(2-Fluoro-6-hydroxyphenyl)ethanone

[93339-98-1] $C_8H_7FO_2$ mol.wt. 154.14



Syntheses

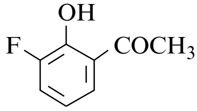
– Preparation by reaction of boron tribromide on 2-fluoro-6-methoxyacetophenone (**I**) in methylene chloride at –65° to –80° [2031,2032], (56%) [2032]. The precursor (**I**) was obtained by reaction of methyl magnesium iodide on 2-fluoro-6-methoxybenzonitrile in refluxing toluene (quantitative yield) [2031].

– Preparation from 2-fluoro-6-methoxybenzonitrile [2033] according to the procedure [2034].

oil [2031,2032]; b.p.$_{0.2}$ 170–172° [2031];
^{1}H NMR [2032,2033], ^{13}C NMR [2033], ^{19}F NMR [2033], MS [2033].

1-(3-Fluoro-2-hydroxyphenyl)ethanone

[699-92-3] $C_8H_7FO_2$ mol.wt. 154.14



Syntheses

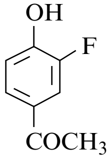
– Obtained by Fries rearrangement of 2-fluorophenyl acetate with aluminium chloride without solvent at 115° [1869], 150° [2031] or at 180–190° (16%) [2035,2036].

– Also obtained by Fries rearrangement of 2-fluorophenyl acetate with aluminium chloride in chlorobenzene at 100° for 24 h (38%) [2037].

m.p. 75–77° [2031], 75–76° [1869], 72–73° [2035,2036];
^{1}H NMR [1869,2037], MS [1869].

1-(3-Fluoro-4-hydroxyphenyl)ethanone

[403-14-5] $C_8H_7FO_2$ mol.wt. 154.14



Syntheses

- Preparation by reaction of pyridinium chloride on 3-fluoro-4-methoxyacetophenone at reflux (78%) [2038].
- Preparation by reaction of acetyl chloride on 2-fluorophenol with aluminium chloride in refluxing carbon disulfide (74%) [1951].
- Preparation by Fries rearrangement of 2-fluorophenyl acetate with aluminium chloride without solvent at 115° [1869] or 140° (51–52%) [2039–2042].

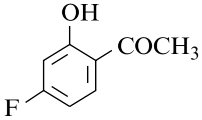
m.p. 128–128°5 [1869], 127–129° [1951], 126–127° [2040], 125–126°6 [2042], 125° [2038];

b.p.$_2$ 125–135° [2042];

^{1}H NMR [1869], IR [2040], MS [1869,2040].

1-(4-Fluoro-2-hydroxyphenyl)ethanone

[1481-27-2] $C_8H_7FO_2$ mol.wt. 154.14



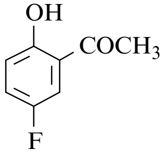
Syntheses

- Preparation by Fries rearrangement of 3-fluorophenyl acetate with aluminium chloride without solvent at 160–180° [2043–2045], (75%) [2045], (88–90%) [2043,2044].
- Preparation by Fries rearrangement of 3-fluorophenyl acetate with alumina in methanesulfonic acid for 3 h at 160° (60%) [2046].
- Preparation by Friedel–Crafts acylation of 3-fluorophenol with acetic acid in the presence of alumina in methanesulfonic acid for 2 h at 120° (63%) [2046].
- Also refer to: [1893,2047,2048].

m.p. 24° [2043,2044].

1-(5-Fluoro-2-hydroxyphenyl)ethanone

[394-32-1] $C_8H_7FO_2$ mol.wt. 154.14



Syntheses

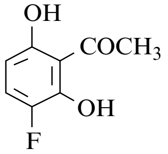
- Preparation by Fries rearrangement of 4-fluorophenyl acetate with aluminium chloride without solvent between 115° and 150° [1800,1869,1965,2044,2049–2051], (88–89%) [1965,2044,2049], (62–63%) [2050,2051].
- Preparation by reaction of acetic acid on 4-fluorophenol with boron trifluoride at 150°, in a sealed tube (89%) [1969].
- Preparation by reaction of pyridinium chloride on 5-fluoro-2-methoxyacetophenone at reflux (74%) [1799], (59%) [1970].

- Preparation by reaction of acetyl chloride on 4-fluoroanisole with aluminium chloride in carbon tetrachloride (44%) [2052,2053].
- Also obtained (by-product) by reaction of acetyl chloride on 4-fluoroanisole with aluminium chloride in carbon disulfide [1799].
- Also refer to: [2025].

m.p. 57–58° [1965,1975], 57° [1799,1969], 56°4–57°6 [1970], 56°5–57° [2049], 56–57° [2052,2053], 56–56°5 [2051], 56° [2044], 55° [1869]; b.p.$_8$ 65–66° [2050], b.p.$_{12}$ 94–99° [2051], b.p.$_{13}$ 103–104° [1799], b.p.$_{16}$ 105° [1969];
^{1}H NMR [1869,2052], ^{13}C NMR [1821], UV [1970], MS [1869,2052]; pK$_a$ [1977].

1-(3-Fluoro-2,6-dihydroxyphenyl)ethanone

[117902-14-4] $C_8H_7FO_3$ mol.wt. 170.14



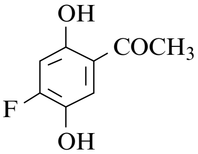
Synthesis

- Obtained (by-product) by reaction of boron tribromide on 2,6-dimethoxy-3-fluoroacetophenone in methylene chloride–methanol mixture (14%) [2054].

m.p. 134–135° [2054]; ^{1}H NMR [2054].

1-(4-Fluoro-2,5-dihydroxyphenyl)ethanone

[88772-48-9] $C_8H_7FO_3$ mol.wt. 170.14



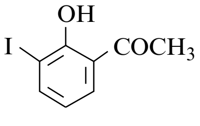
Synthesis

- Preparation by Fries rearrangement of 2-fluoro-hydroquinone diacetate with aluminium chloride in nitrobenzene at 140° (66%) [1884].

m.p. 210° [1884]; ^{1}H NMR [1884], IR [1884].

1-(2-Hydroxy-3-iodophenyl)ethanone

$C_8H_7IO_2$ mol.wt. 262.05



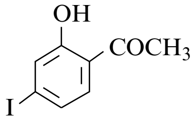
Synthesis

- Preparation by diazotization of 3-amino-2-hydroxyacetophenone with sodium nitrite in dilute sulfuric acid at 0°, and replacement of the diazonium group by iodine with potassium iodide at 65° (Sandmeyer reaction) [1920,1965,2036], (50%) [1965].

m.p. 59°5–60°5 [1920], 58–59° [1965,2036].

1-(2-Hydroxy-4-iodophenyl)ethanone

[39730-66-0] $C_8H_7IO_2$ mol.wt. 262.05



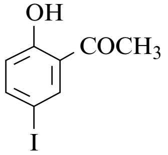
Syntheses

- Preparation by Fries rearrangement of 3-iodophenyl acetate with aluminium chloride [1942,1956–1959,2055],
  - in chlorobenzene, at 125–135° (64%) [1956,1959];
  - in nitrobenzene, at 110–140° (40–45%) [1956,1957,2055].
- Preparation by diazotization of 4-amino-2-hydroxyacetophenone and replacement of the diazonium group by iodine (Sandmeyer reaction) (46%) [1956].
- Preparation by reaction of acetyl chloride on 3-iodoanisole with aluminium chloride in refluxing carbon disulfide (14%) [2055].
- Also refer to: [1848] (compound **6**).

m.p. 54° [1959], 53–54° [1942,1956,1957], 52–54° [2055].

1-(2-Hydroxy-5-iodophenyl)ethanone

[7191-41-5] $C_8H_7IO_2$ mol.wt. 262.05



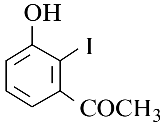
Syntheses

- Preparation by saponification of ethyl 4-hydroxy-6-iodocoumarin-3-carboxylate with boiling 10% aqueous potassium hydroxide solution (92%) [1914,2056].
- Preparation by diazotization of 2-hydroxy-5-nitro-acetophenone and replacement of the diazonium group by iodine (Sandmeyer reaction) [1920,1965,1975,2057], (75–80%) [1965,1975].
- Preparation by diazotization of 4-acetamido-2-methoxyacetophenone and replacement of the diazonium group by iodine (73%) [1975].
- Also obtained by reaction of iodine on 2-hydroxyacetophenone in aqueous sodium carbonate solution (37%) [1915], (14%) [1913,1914].
- Also obtained by Fries rearrangement of 4-iodophenyl acetate with aluminium chloride in nitrobenzene at 25° (13%) [2058].
- Also refer to: [1961].

m.p 91–92° [1965], 90–92° [1920,1975], 90° [2057], 88–89° [1914,2056], 67–69° [2058]. One of the reported melting points is obviously wrong. UV [1914].

1-(3-Hydroxy-2-iodophenyl)ethanone

[348616-32-0] $C_8H_7IO_2$ mol.wt. 262.05



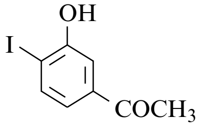
Synthesis

- Obtained by debenzylation of 3-(benzyloxy)-2-iodo-acetophenone (m.p. 53–55°) with boron tribromide in methylene chloride at −70° for 50 min (93%) [2059].

m.p. 93–97° [2059]; ^1H NMR [2059], ^{13}C NMR [2059], IR [2059], MS [2059].

1-(3-Hydroxy-4-iodophenyl)ethanone

[73898-23-4] $C_8H_7IO_2$ mol.wt. 262.05



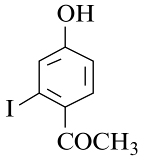
Synthesis

- Obtained by reaction of iodine–potassium iodide mixture in aqueous solution with 3-hydroxyacetophenone in concentrated aqueous ammonia at r.t. (15%) [1951].

m.p. 134–135° [1951].

1-(4-Hydroxy-2-iodophenyl)ethanone

$C_8H_7IO_2$ mol.wt. 262.05



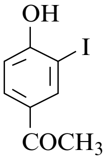
Syntheses

- Preparation by reaction of 48% hydrobromic acid on 2-iodo-4-methoxyacetophenone in acetic acid at 100°, in a sealed tube (45%) [2055].
- Also obtained (by-product) by reaction of acetyl chloride on 3-iodoanisole with aluminium chloride in refluxing carbon disulfide (9%) [2055].
- Also obtained in very small quantities by Fries rearrangement of 3-iodophenyl acetate with aluminium chloride in nitrobenzene at 90–95° [1942].

m.p. 131–132° [2055], 128–136° [1942].

1-(4-Hydroxy-3-iodophenyl)ethanone

[62615-24-1] $C_8H_7IO_2$ mol.wt. 262.05



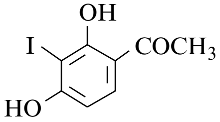
Syntheses

- Preparation by reaction of iodine and potassium iodide on 4-hydroxyacetophenone in aqueous ammonium hydroxide at r.t. [1951,1989,2060], (54–57%) [1951,1989].
- Preparation by adding an aqueous solution of potassium iodide to a solution of 4-hydroxyacetophenone in concentrated ammonium hydroxide at 35° (49%) [2061].
- Preparation by adding a 5% aqueous solution of sodium hypochlorite to a solution of 4-hydroxyacetophenone and sodium iodide in methanol at 15° (60%) [2062].
- Also refer to: [2063].

m.p. 155–156° [2062], 154–156° [1989], 153–155° [2061], 153–154° [1951]; ^{1}H NMR [1989,2061,2062], IR [2061].

1-(2,4-Dihydroxy-3-iodophenyl)ethanone

[71243-12-4] $C_8H_7IO_3$ mol.wt. 278.05



Syntheses

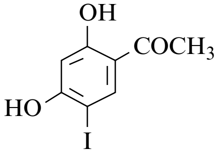
– Preparation by iodination of resacetophenone,

- with iodine and iodic acid in dilute ethanol at r.t. [1837,1910,1924,2064–2066], (82–84%) [1924,2065];
- with iodine in the presence of diisopropylamine in methanol at r.t. (14%) [2065];
- with potassium iodate and potassium iodide in dilute acetic acid [1890].

m.p. 164° [1924], 158–162° [2065], 158–159° [1890]; ^{1}H NMR [2065].

1-(2,4-Dihydroxy-5-iodophenyl)ethanone

[62069-33-4] $C_8H_7IO_3$ mol.wt. 278.05



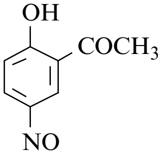
Syntheses

- Preparation by selective deiodination of 2,4-dihydroxy-3,5-diiodoacetophenone with stannous chloride in refluxing acetic acid (40%) [1923].
- Also obtained by reaction of aqueous iodine–potassium iodide solution on resacetophenone in 22% aqueous ammonia at r.t. (15%) [1924].
- Preparation by iodination of resacetophenone using iodine and iodic acid in dilute ethanol at r.t. [1837].
- Preparation by reaction of boron tribromide with 2,4-bis(benzyloxy)-5-iodoacetophenone (SM) in methylene chloride with stirring for 5 min at −70° (97%). SM was obtained from 2,4-bis-(benzyloxy)acetophenone with iodine in the presence of silver trifluoroacetate in chloroform (89%) [2062].

m.p. 185–186° [1923], 184° [1924], 180–181° [2062]; ^{1}H NMR [1923,2062].

1-(2-Hydroxy-5-nitrosophenyl)ethanone

[15516-61-7] $C_8H_7NO_3$ mol.wt. 165.15



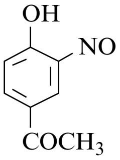
Synthesis

– Refer to: [2067] (compound **XVII**).

UV [2067].

1-(4-Hydroxy-3-nitrosophenyl)ethanone

[97871-70-0] $C_8H_7NO_3$ mol.wt. 165.15



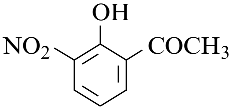
Synthesis

– Preparation by direct nitrosation [2068] of 4-hydroxy-acetophenone with aqueous sodium nitrite solution in dilute hydrochloric acid between 0° and 5° [2069].

UV [2069]; pK_a [2069].

1-(2-Hydroxy-3-nitrophenyl)ethanone

[28177-69-7] $C_8H_7NO_4$ mol.wt. 181.15



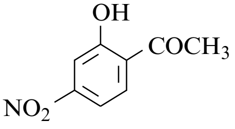
Syntheses

- Preparation by reaction of boiling 5% aqueous potassium hydroxide on 8-nitrochromone (78%) [2070].
- Also obtained by reaction of nitric acid on 2-hydroxy-acetophenone in acetic acid [2071], (30%) [2022,2023], (9–10%) [1926,2072], (2%) [1925,1965,2073].
- Preparation by diazotization of 5-amino-2-hydroxy-3-nitroacetophenone, followed by decomposition of the obtained diazonium salt (90%) [2074], (56%) [2075].
- Also obtained by reaction of boiling 20% aqueous hydrochloric acid on 2-hydroxy-3-nitro-acetophenone oxime [2076].
- Also obtained by nitration and hydroxylation of acetophenone with pernitrous acid (2%) [2077].
- Also refer to: [2078–2081].

m.p. 103–104° [2070], 99–101° [1949], 98°5–99°5 [2072], 97–98° [2071], 90° [2077], 89–90° [2076], 84–85° [1965], 82–83° [1925,1926,2073–2075]; ^{1}H NMR [2074,2078], IR [2074]; pK_a [2078].

1-(2-Hydroxy-4-nitrophenyl)ethanone

[1834-91-9] $C_8H_7NO_4$ mol.wt. 181.15



Syntheses

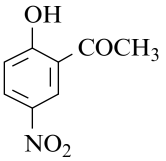
- Obtained by Fries rearrangement of 3-nitrophenyl acetate with aluminium chloride without solvent at 125° [2082–2084], (20%) [2084], (32%) [2082].
- Also obtained by reaction of acetyl chloride on 3-nitro-phenol with aluminium chloride at 125° (16–18%) [2084].
- Claimed to be prepared by reaction of boiling 50% nitric acid on 2-hydroxy acetophenone (59%) [1927].

N.B.: No physical data is indicated in the patent for this compound. According to the rules dealing with aromatic substitution (Holleman rules) [1929] and electronic induction using the Hammett substituent constants [1930], the obtained product most likely is the 2-hydroxy-5-nitro-acetophenone.

m.p. 67–68° [2082,2085,2086], 67° [2084]; ^{1}H NMR [1963], IR [1963].

1-(2-Hydroxy-5-nitrophenyl)ethanone

[1450-76-6] $C_8H_7NO_4$ mol.wt. 181.15



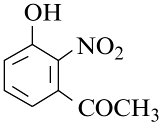
Syntheses

- Obtained by nitration of 2-hydroxyacetophenone oxime, followed by treatment of this oxime with boiling 20% hydrochloric acid (73%) [2016,2076].
- Preparation by reaction of acetyl chloride on 4-nitrophenol with aluminium chloride in nitrobenzene at 130° (44%) [1925].
- Preparation by Fries rearrangement of 4-nitrophenyl acetate with aluminium chloride,
  - without solvent, at 140–150° (28%) [2087];
  - with solvent, in nitrobenzene, at 120–130° (20–35%) [1925,2083,2085,2088,2089].
- Preparation by nitration of 2-hydroxyacetophenone,
  - in acetic acid (20–30%) [1925,1926,1965,2072,2073];
  - without solvent (59%) [1927].
- Also obtained by reaction of acetic anhydride on 4-nitrophenol with aluminium chloride [2088].
- Also obtained from mixture of 2-chloro-5-nitroacetophenone, sodium acetate and acetamide heated at 180–200° (49%) [1806].
- Also refer to: [2090].

m.p. 111–112° [2076], 102–103° [1926,2083,2085,2088], 101–102° [1965,2087], 99°5 [1806,1925], 98°5–99°5 [2072], 98–99° [2073]; ^{1}H NMR [1963,1976], ^{13}C NMR [1976], IR [1963].

1-(3-Hydroxy-2-nitrophenyl)ethanone

[53967-72-9] $C_8H_7NO_4$ mol.wt. 181.15



Syntheses

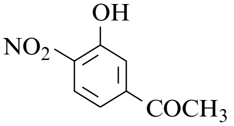
- Preparation by nitration of 3-hydroxyacetophenone,
  - with concentrated nitric acid in concentrated sulfuric acid at –20° (35–45%) [1932];
  - with nitric acid (d = 1.4) in acetic acid at 70° (16%) [2091];
  - with cupric nitrate in acetic acid–acetic anhydride mixture between 12° and 15° (18%) [1932,2092].

- Preparation by demethylation of 3-methoxy-2-nitroacetophenone with pyridinium chloride at 200° [2093].

m.p. 138° [1932], 136° [2093], 135° [2091]; IR [2091], UV [1932,2091]; pK_a [2091].

1-(3-Hydroxy-4-nitrophenyl)ethanone

[89942-63-2] $C_8H_7NO_4$ mol.wt. 181.15



Syntheses

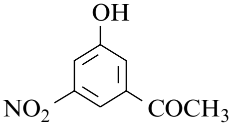
- Obtained (poor yield) by nitration of 3-hydroxyacetophenone,
  - with nitric acid (d = 1.4) in acetic acid, at 70° (10%) [2091];
  - with cupric nitrate in acetic acid–acetic anhydride mixture, between 12° and 15° (<1%) [1932].

m.p. 131–132° [2085], 71°5–72°5 [1932], 69–70° [2091];
IR [2091], UV [1932,2091]; pK_a [2091].

N.B.: The melting point 131–132° [2085] was erroneous. This melting point is the one of an isomer, the 3-nitro-4-hydroxyacetophenone (130–131°2) [2094].

1-(3-Hydroxy-5-nitrophenyl)ethanone

[70284-07-0] $C_8H_7NO_4$ mol.wt. 181.15



Syntheses

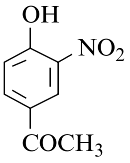
- Obtained in two steps: First, a mixture of 3-hydroxyacetophenone and dysprosium nitrate in ethyl acetate were refluxed (85–105°) for 75 min. Then, the isolated intermediate $(C_8H_6NO_4)_3Dy$ (59%) was dissolved in 6 N hydrochloric acid (50%) [2095].
- Also refer to: [2096] (compound NP), [2097].

N.B.: Dy (III) salt [193693-92-4] [2095].

m.p. 122–124° [2095]; MS [2095].

1-(4-Hydroxy-3-nitrophenyl)ethanone

[6322-56-1] $C_8H_7NO_4$ mol.wt. 181.15



Syntheses

- Preparation by nitration of 4-hydroxyacetophenone [1933,1934,1953,1999,2091,2098–2100], (85%) [1953,1999], (70–75%) [1933,2099,2100], (56%) [2091].
- Also obtained by nitration of 4-hydroxyacetophenone,
  - using properties of dinitrogen tetroxide complexes of iron – $Fe(NO_3)_3\cdot 1.5\ N_2O_4$ – and copper nitrates – $Cu(NO_3)_2\cdot N_2O_4$ – in acetone for 5–10 min at r.t. (97–100%) [2101];

  - by reaction of ferric nitrate nonahydrate – $Fe(NO_3)_3$. 9 H_2O – in ethanol for 24 h at 65° (91%) [2102].

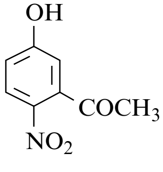
- Also obtained by reaction of peroxynitrite ($ONOO^-$) with 4-hydroxyacetophenone in aqueous phosphate buffer and acetonitrile solution [2103].
- Also obtained from aromatic nucleophilic substitution of 3,4-dichloroacetophenone with sodium nitrite in DMSO (40%) [2104].
- Preparation by reaction of acetyl chloride on 2-nitrophenol with aluminium chloride in nitrobenzene (46–47%) [1925,2094,2105,2106].
- Also obtained by reaction of acetyl chloride on 2-nitroanisole with aluminium chloride (11%) [2107].
- Preparation by Fries rearrangement of 2-nitrophenyl acetate with aluminium chloride in nitrobenzene (40–45%) [1925,2082], (30%) [2086,2094].
- Also obtained by Fries rearrangement of 2-nitrophenyl acetate [2108], which occurs under mild conditions on K 10 montmorillonite using microwave radiations [2109].
- Also obtained on heating 4-bromo-3-nitroacetophenone with acetamide–sodium acetate mixture between 175° and 200° (low yield) [2110].
- Also obtained by mononitration of 4-hydroxyacetophenone with a combination,
  - trichloroisocyanuric acid, sodium nitrite and wet silicone dioxide (50% w/w) in methylene chloride at r.t. for 20 min (97%) [2111];
  - magnesium bisulfate or sodium bisulfate monohydrate, sodium nitrite and wet silicone dioxide (50% w/w) in methylene chloride at r.t. for 3 h (80–82%) [2112].
- Also refer to: [2078,2113–2119].

N.B.: Na salt [42247-95-0] [2120].

m.p. 135–136° [2091], 135° [2100], 133–134° [1933], 133° [2099], 132–132°5 [2094], 132° [1925], 131–132° [2106], 130°5 [2107], 130° [1999,2110], 129°5 [1953], 128–130° [2082], 123° [2102], 122–124° [2111,2112]; pK_a [2078,2091,2106]; GC [2104]; ^1H NMR [2078,2102], ^{17}O NMR [2121], IR [2091,2102], UV [2091,2106], MS [2104].

1-(5-Hydroxy-2-nitrophenyl)ethanone

[30879-49-3] $C_8H_7NO_4$ mol.wt. 181.15



Syntheses

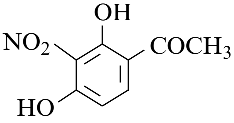
- Preparation by nitration of 3-hydroxyacetophenone,
  - with nitric acid (d = 1.4) in acetic acid, at 70° (31%) [2091], (15%) [2099];

- with cupric nitrate in acetic acid–acetic anhydride mixture, between 12° and 15° (20%) [1932,2092].

m.p. 148–149° [1932,2091,2099]; IR [2091], UV [1932,2091,2122]; pK_a [2091].

1-(2,4-Dihydroxy-3-nitrophenyl)ethanone

[89684-58-2] $C_8H_7NO_5$ mol.wt. 197.15



Syntheses

– Resacetophenone by treatment with cerium (IV) ammonium nitrate in acetic acid at 50–60° yields 2,4-dihydroxy-3-nitro-acetophenone (good yield) [2123], (23%) [1937].

– Preparation by reaction of acetic anhydride on 2-nitro-resorcinol with aluminium chloride in nitrobenzene at 100° [2124,2125], (53%) [2124].

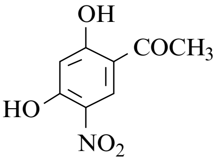
– Also obtained by reaction of aqueous hydrochloric acid–acetic acid mixture on 5-acetyl-2,4-di-hydroxy-3-nitrobenzoic acid in a sealed tube at 140–145° [1938].

– Also refer to: [2126].

m.p. 103° [2125], 102° [2124], 93° [1938,1939,2127], 90–91° [1937,2123]; ^1H NMR [2123], IR [2123].

1-(2,4-Dihydroxy-5-nitrophenyl)ethanone

[3328-77-6] $C_8H_7NO_5$ mol.wt. 197.15



Syntheses

– Preparation by nitration of resacetophenone [2128,2129], (71%) [2130], (54–50%) [1836,2131], (44%) [2074].

– Also obtained by reaction of nitric acid on 3,3′-diacetyl-4,4′,6,6′-tetrahydroxydiphenyl thioether in acetic acid at r.t. [1810].

– Also obtained (by-product) by reaction of acetic anhydride on 4-nitroresorcinol with aluminium chloride in nitrobenzene (6%) [1940].

– Also obtained by reaction of sodium hydroxide on 3-benzoyl-2,4-dihydroxy-5-nitroacetophenone in boiling aqueous ethanol [1936].

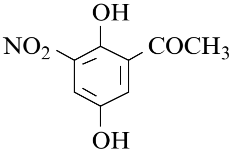
– Resacetophenone by treatment with cerium (IV) ammonium nitrate in hot acetic acid yields 2,4-di-hydroxy-5-nitroacetophenone (31%) [1937].

m.p. 145–147° [2074], 145° [1836], 143° [1937], 142° [1810,1936,1939,1940,2125,2128–2131].

^1H NMR [2074], IR [2074].

1-(2,5-Dihydroxy-3-nitrophenyl)ethanone

[30095-74-0] $C_8H_7NO_5$ mol.wt. 197.15



Syntheses

- Preparation by nitration of 3-acetyl-4-hydroxyphenyl acetate, followed by hydrolysis of the obtained keto ester with hydrochloric acid in refluxing methanol (61%) [1832].
- Also obtained by hydrolysis of 6-hydroxy-2-methyl-8-nitro-chromone with 10% aqueous sodium hydroxide [1831].
- Also obtained by nitration of quinacetophenone in acetic acid [1831].
- Also obtained by demethylation of 2-hydroxy-5-methoxy-3-nitroacetophenone with 48% hydrobromic acid in the presence of red phosphorous at 85–90° for 16 h under argon atmosphere (90%) [2074].

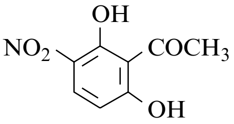
N.B.: All the results of reference [1831] were erroneous [1832].

m.p. 141–142° [1832], 136–138° [2074], 58° [1831]. One of the reported melting points is obviously wrong.

^{1}H NMR [1832,2074], IR [1832], UV [1832].

1-(2,6-Dihydroxy-3-nitrophenyl)ethanone

[25205-34-9] $C_8H_7NO_5$ mol.wt. 197.15



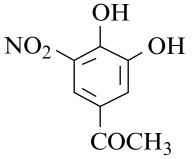
Syntheses

- Preparation by reaction of acetic anhydride on 4-nitro-resorcinol with aluminium chloride in nitrobenzene [1940,2125], (37%) [1940].
- Preparation by Fries rearrangement of 4-nitroresorcinol diacetate with aluminium chloride in nitrobenzene at 95–100° (38%) [2132].
- Also obtained by nitration of 2,6-dihydroxyacetophenone (77%) [2074], (27%) [1940].
- Also obtained by demethylation of 2,6-dimethoxy-3-nitroacetophenone with aluminium chloride or with boiling 10% aqueous sodium hydroxide (quantitative yield) [1940].
- Also obtained by reaction of 4-methoxy-2-methyl-8-nitrochromone or its 3-acetyl derivative with boiling 10% aqueous sodium carbonate (50% and 58% yields, respectively) [2133].
- Also refer to: [2134].

m.p. 119° [1940,2125], 116–117° [2132], 114–115° [2074]; ^{1}H NMR [2074], IR [2074].

1-(3,4-Dihydroxy-5-nitrophenyl)ethanone

[116313-84-9] $C_8H_7NO_5$ mol.wt. 197.15



Syntheses

– Preparation from 4-hydroxy-3-methoxyacetophenone by nitration and subsequent demethylation of 4-hydroxy-3-methoxy-5-nitroacetophenone obtained,

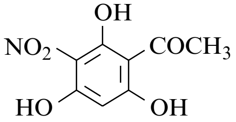
- with boiling concentrated hydrobromic acid [2135];
- with concentrated hydrobromic acid in acetic acid (35%) [2136].

– Preparation by demethylation of 3,4-dimethoxy-5-nitroacetophenone with concentrated hydrobromic acid at 140° [2135].

m.p. 161–169° [2136], 159–160° [2135]; pK_a [2137].

1-(2,4,6-Trihydroxy-3-nitrophenyl)ethanone

[81325-88-4] $C_8H_7NO_6$ mol.wt. 213.15



Syntheses

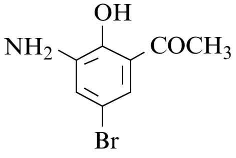
– Preparation by addition of a mixture of concentrated sulfuric acid and fuming nitric acid into a solution of phloroaceto-phenone in concentrated sulfuric acid and hexane mixture under cooling with an ice bath (70–80%) [2138].

– Also obtained by adding acetic anhydride to a mixture of 1,3,5-trihydroxy-2-nitrobenzene and aluminium chloride in nitrobenzene and heating on a steam bath for 7 h (20%) [2139].

m.p. 133–135° [2138], 131–132° [2139]; ^{1}H NMR [2138,2139], IR [2138], MS [2138].

1-(3-Amino-5-bromo-2-hydroxyphenyl)ethanone

[70977-85-4] $C_8H_8BrNO_2$ mol.wt. 230.06



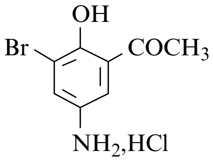
Synthesis

– Preparation by reaction of 20% aqueous titanium trichloride solution on 5-bromo-2-hydroxy-3-nitroacetophenone in toluene, at r.t., in a sealed tube [1897,1898], (31%) [1897].

m.p. 99–102° [1897,1898].

1-(5-Amino-3-bromo-2-hydroxyphenyl)ethanone *(Hydrochloride)*

[30186-22-2] $C_8H_8BrNO_2$, HCl mol.wt. 266.52



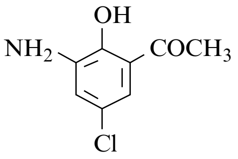
Synthesis

– Preparation by reduction of 3-bromo-2-hydroxy-5-nitro-acetophenone with an excess of tin in concentrated hydrochloric acid at 100° (82%) [1817].

m.p. 200°(d) [1817].

1-(3-Amino-5-chloro-2-hydroxyphenyl)ethanone

[21312-85-6] $C_8H_8ClNO_2$ mol.wt. 185.61



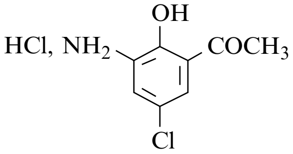
Syntheses

- Preparation by reaction of acetic anhydride on 2-amino-4-chlorophenol with aluminium chloride in 1,2,4-trichloro-benzene at 120° [2140].
- Preparation by reaction of 75% hydrochloric acid solution on 3-acetamido-5-chloro-2-hydroxyacetophenone; the mixture was heated on a steam bath (98%) [2141].

m.p. 110–112° [2141]; UV [2141].

1-(3-Amino-5-chloro-2-hydroxyphenyl)ethanone *(Hydrochloride)*

[153404-65-0] $C_8H_8ClNO_2$, HCl mol.wt. 222.07

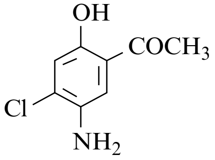


Synthesis

- Preparation by reaction of acetic anhydride on 2-amino-4-chlorophenol hydrochloride with aluminium chloride in 1,2,4-trichlorobenzene at 120° (79%) [2140].

1-(5-Amino-4-chloro-2-hydroxyphenyl)ethanone

$C_8H_8ClNO_2$ mol.wt. 185.61



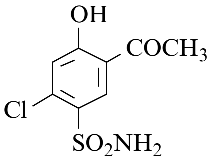
Synthesis

- Preparation by reduction of 4-chloro-2-hydroxy-5-nitro-acetophenone with iron powder in dilute acetic acid at 95° (93%) [1784] (Béchamp reduction).

m.p. 117–118° [1784]; IR [1784].

1-[5-(Aminosulfonyl)-4-chloro-2-hydroxyphenyl]ethanone

$C_8H_8ClNO_4S$ mol.wt. 249.67



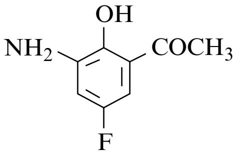
Synthesis

- Preparation by reaction of ammonia gas on 4-chloro-5-chlorosulfonyl-2-hydroxyacetophenone in tetrahydrofuran at r.t. (57%) [1784].

m.p. 181–182° [1784].

1-(3-Amino-5-fluoro-2-hydroxyphenyl)ethanone

[70977-84-3] $C_8H_8FNO_2$ mol.wt. 169.16



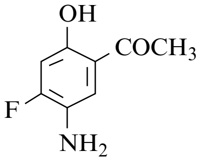
Synthesis

– Preparation by catalytic hydrogenation of 5-fluoro-2-hydroxy-3-nitroacetophenone in the presence of 5% Pd/C in ethanol at 25° [1897,1898], (99%) [1897].

m.p. 113–114° [1897].

1-(5-Amino-4-fluoro-2-hydroxyphenyl)ethanone

$C_8H_8FNO_2$ mol.wt. 169.16

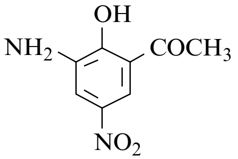


Synthesis

– Refer to: [1893] (Japanese patent).

1-(3-Amino-2-hydroxy-5-nitrophenyl)ethanone

[70977-79-6] $C_8H_8N_2O_4$ mol.wt. 196.16



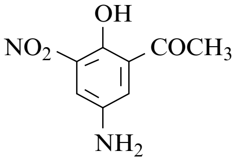
Synthesis

– Preparation by reaction of sodium sulfide on 2-hydroxy-3,5-dinitroacetophenone with ammonium chloride in refluxing methanol [1897,1898], (55%) [1898], (72%) [1897].

m.p. 172–175° [1898], 172–174° [1897].

1-(5-Amino-2-hydroxy-3-nitrophenyl)ethanone

[108129-55-1] $C_8H_8N_2O_4$ mol.wt. 196.16



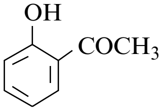
Synthesis

– Preparation by hydrolysis of 5-acetamido-2-hydroxy-3-nitroacetophenone (96%) [2074], (82%) [2075].

m.p. 141–142° [2075], 129–130° [2074]; ^{1}H NMR [2074], IR [2074].

1-(2-Hydroxyphenyl)ethanone

[118-93-4] $C_8H_8O_2$ mol.wt. 136.15



Syntheses

- Preparation by Fries rearrangement of phenyl acetate, **with Lewis acids**
  - aluminium chloride,

with solvent:

- in petroleum ether at 25° (80%) [2142] (result not reproducible);
- in nitrobenzene [2143–2146] at 60° (26%) [2145];
- in nitroethane at 60° (12%) [2147];
- in chlorobenzene at 60–65° (23%) [2148] or in a sealed tube and subjected to high power microwave irradiation for 2 min only (30%) [2013].

without solvent:

- between 130° and 165° (63–82%) [1967,2013,2149–2153];
- between 120° and 180° (35–46%) [2143,2154–2159];
- between 90° and 200° (25–31%) [1797,1888,1914,2150,2160–2165];
- at 60° (7%) [2166];

- boron trifluoride etherate, in boiling benzene (70%) [2167];
- aluminium chloride–sodium chloride mixture at 240–250° (47%) [2168];
- boron trifluoride at 90° (43%) [2164];
- titanium tetrachloride at 110° (22%) [2169];
- scandium tris(trifluoromethanesulfonate), in nitromethane, at 50° (17%) [2170];
- zinc chloride between 125° and 160° (4–7%) [2164,2171];

with Protic acids

- p-Toluenesulfonic acid at 160° (25%) [2164];
- methanesulfonic acid at 160° (22%) [2164];
- polyphosphoric acid at 100° (20%) [2172];
- monohydrated sulfuric acid at 190° (20%) [2164];
- methanetrisulfonic acid at 160° (11%) [2164];
- phosphoric acid at 190° (8%) [2164];

with a cation exchange resin

- sulfonated polystyrene resin;
  - (Dowex 50 X 8) at 150° (8%) [2164];
  - (Dowex 50 WX 8) at 115° (6%) [2164];
- Nafion-XR 500 at 100° [2173];

with Zeolites molecular sieves

- Ga ZSM-5 at 250° (46%) [2174];

- ZSM-5 (Si/Al = 20) in sulfolane, at 180° (34%) [2175];
- H-Nu-2 (unknown structure) at 170° (6%) [2176];
- H-ZSM-5 (MFI structure) at 210° (4%) [2176].
- HY (Si/Al = 3) at 400° (3%) [2177];
- fluorided alumina (Al_2O_3-F; 3 % wt. F) at 400° (3%) [2177];
- H-ZSM-5 (Si/Al = 45) at 400° (1%) [2177].

- Also obtained by Fries rearrangement of 4-trimethylsilylphenyl acetate with aluminium chloride without solvent at 140° (60%) [2178].
- Also obtained by reaction of triethylamine hydrochloride on phenyl acetate at 260°, in a sealed tube [2179].
- Also obtained (by-product) by Fries rearrangement of 2-bromophenyl acetate with aluminium chloride without solvent, at 180° (15%) [1817].
- Also obtained by reaction of acetic acid on phenol,
    - with polyphosphoric acid at 100° (20%) [2172];
    - with zinc chloride (Nencki reaction) at reflux (2–5%) [2165,2171,2180].
- Also obtained by reaction of acetyl chloride on phenol, in nitrobenzene, between 45° and 60°,
    - with aluminium chloride (14–15%) [2144,2181];
    - with titanium tetrachloride (11%) [2181].
- Also obtained by reaction of acetyl chloride on phenyl borate with aluminium chloride in refluxing carbon disulfide (54%) [2182].
- Also obtained by reaction of acetic anhydride on phenol, using a steam bath,
    - with 70% $HClO_4$ (51%) [2183];
    - with aluminium chloride (38%) [2183];
    - with zinc chloride at 145–150° (36%) [2184].
- Also obtained by reaction of acetylacetone on phenyl benzoate with aluminium chloride in nitrobenzene, at 45° (3%) [2144].
- Preparation by diazotization of 2-aminoacetophenone and hydrolysis of the obtained diazonium salt [1819,2185,2186].
- Also obtained by demethylation of 2-methoxyacetophenone with hydrochloric acid, in a sealed tube (low yield) [2185] or at 130° [2187].
- Also obtained by hydroxylation of acetophenone,
    - this reaction was accomplished by oxidation of acetophenone at platinum in methylene chloride–trifluoroacetic acid mixture containing tetraethylammonium fluoborate, at r.t. (85% yield) [2188];
    - this reaction was realized by treatment of acetophenone with sodium trifluoroacetate in nitromethane–trifluoroacetic acid–trifluoroacetic anhydride mixture in the presence of a platinum electrode, followed by treatment of the intermediate trifluoroester with 10% potassium hydrogen carbonate solution (50% yield) [2006];
    - the 2-hydroxyacetophenone was obtained by nitration and hydroxylation of acetophenone with pernitrous acid (2% yield) [2077].

- Also obtained by reaction of boiling dilute potassium hydroxide solution on flavone [2189].
- Also obtained by reaction of sodium ethoxide on chromone in refluxing ethanol [2190].
- Also obtained by reaction of aqueous potassium hydroxide on 4-hydroxycoumarin (benzotetronic acid) at 180° (80%) [2180].
- Also obtained by reaction of sodium ethoxide on 4-phenacylidenflaven in refluxing ethanol [1971].
- Also obtained by reaction of aqueous potassium hydroxide solution on methyl or ethyl 4-hydroxycoumarin-3-carboxylate at 180° (80%) [2191].
- Also obtained by reaction of potassium hydroxide on the 2-hydroxyseneciophenone (2′-hydroxy-3,3-dimethylacrylophenone) in boiling ethanol (60%) [2017].
- Also obtained by UV light irradiation of phenyl acetate (photo-Fries rearrangement),
  - in hexane, at 25° (13%) [2015];
    - in the presence of potassium carbonate (78%) [2015];
  - in benzene (59%) [2192];
  - in cyclohexane, at 25° (43%) [2193];
  - in methanol (28%) [2192];
  - in water, at 25–30° (25–28%) [2194,2195];
    - in the presence of β-cyclodextrin (89%) [2192], (35%) [2195], (11%) [2194];
    - in the presence of methyl α-D-glucopyranoside (32%) [2194];
  - in ethyl ether, at 25° (24%) [2193];
  - in ethanol [2196,2197], at 30° (19%) [2196];
  - in isopropanol, at 25° (9%) [2193].
- Also obtained by UV light irradiation of 2-methoxyphenyl acetate in benzene (14%) [2198], in ethyl ether (4%) [2193].
- Also obtained (by-product) on UV light irradiation of 2-hydroxy-α-chloroacetophenone in ethanol (3%) [2199].

Isolation from natural sources

- From essential oil of *Chione Glabra* (Rubiaceae) [2200].
- From essential oil of *Acacia farnesiana Willd* [2201].

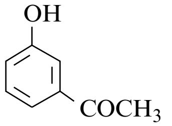
m.p. 28° [2171];

b.p.$_{0.2}$ 56° [1817], b.p.$_{6}$ 73° [2142], b.p.$_{7}$ 86° [2183], b.p.$_{13}$ 91–92° [2155], b.p.$_{10}$ 92–94° [2157], b.p.$_{7}$ 93–94° [2158], b.p.$_{11}$ 95–100° [2017], b.p.$_{10}$ 96–97° [2191], b.p.$_{15}$ 96–98° [2157], b.p.$_{15}$ 97–98° [1797], b.p.$_{6}$ 98° [2162], b.p.$_{15}$ 99–100° [2156], b.p.$_{14}$ 100° [2160], b.p.$_{15}$ 101–101°5 [2168], b.p.$_{17}$ 101–102° [2150], b.p.$_{20}$ 104–105° [1914], b.p.$_{20}$ 106° [2159], b.p.$_{17}$ 106° [2153], b.p.$_{17}$ 106–107° [2160], b.p.$_{17}$ 109° [1967], b.p.$_{32}$ 109–110° [2202], b.p.$_{15}$ 110° [2171], b.p.$_{33}$ 113°5–113°8 [2203], b.p.$_{30}$ 115–120° [2178], b.p.$_{22}$ 130–133°

[2186,2204], b.p.$_{34}$ 160–165° [2200], b.p.$_{717}$ 213° [2015,2187], b.p. 213–214° [2180,2184], b.p. 215–218° [2148], b.p. 216–217° [2165], b.p. 218° [1971,2161], b.p.$_{744}$ 220° [2015];
^{1}H NMR [1817,1963,1976,2205–2209],
^{13}C NMR [1821,1976,2206,2210,2211], IR [1797,1963,2091,2207,2209,2212], UV [1822,2091,2213–2216],[2165,2186]; pK$_a$ [1949,1977,2091,2217].

1-(3-Hydroxyphenyl)ethanone

[121-71-1] $C_8H_8O_2$ mol.wt. 136.15



Syntheses

- Preparation by diazotization of 3-aminoacetophenone, followed by hydrolysis of the obtained diazonium salt [1782,1932,2145,2186,2218–2223], (78–82%) [1782,1932].
- Synthesis of 3-hydroxyacetophenone by means of organocadmium derivatives (77%) [2224].
- Preparation by reductive deamination of 2-amino-5-hydroxyacetophenone [2225].
- Preparation by aromatization of 5-acetyl-2-cyclohexenone [2030] promoted by,
  - cupric bromide and lithium bromide in refluxing acetonitrile (75%);
  - Pd/C in refluxing xylene (40%).
- Also obtained by treatment of acetophenone with sodium trifluoroacetate in nitromethane–trifluoroacetic acid–trifluoroacetic anhydride mixture in the presence of a platinum electrode, followed by treatment of the intermediate trifluoroester with 10% potassium hydrogen carbonate solution (14%) [2006], (hydroxylation of aromatic compounds).
- Also obtained (trace) by UV irradiation of phenyl acetate in ethanol at r.t. [2196].
- Also obtained from 3-(allyloxy)acetophenone by cleavage of allyl group with bis(benzonitrile)-palladium (II) chloride in refluxing benzene (90%) [2226].
- Also refer to: [2227].

Isolation from natural sources

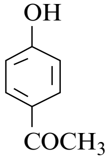
- From *Propolis* and *Populus nigra* bud exudate (compound **36**) [2228].
- From commercial wood vinegar liquor (*Cryptomeria japonica*) [2229].

N.B.: Toxic action of 3-hydroxyacetophenone to the ciliate *Tetrahymena pyriformis* [2230,2231].

m.p. 96° [2145,2223,2224], 95–96° [2221], 95° [2222], 94–96° [1859],94–95° [1782,2219,2225], 94° [2186,2204] , 92–93° [2218], 92° [1932], 86–88° [2226];
b.p.$_{0.01}$ 82° [2226], b.p.$_5$ 153° [2220], b.p.$_{756}$ 296° [2220];
HPLC [2228]; GC-MS [2228]; pK$_a$ [1977,2091,2217];
^{13}C NMR [2219], IR [2091,2232], UV [1822,2091,2186,2223], MS [2229].

1-(4-Hydroxyphenyl)ethanone

[99-93-4] $C_8H_8O_2$ mol.wt. 136.15



Syntheses

– Preparation by Fries rearrangement of phenyl acetate

with Lewis acids

- aluminium chloride,

with solvent:

- in nitrobenzene at 20–25° [1914,2146,2151,2153] or at 50–60° [2013,2143,2145,2146,2151], (75–76%) [2013,2143,2151], (60–64%) [2145,2146];
- in chlorobenzene between 45° and 65° [2144,2148], (69%) [2148] or in a sealed tube and subjected to high power microwave irradiation for 2 min only (36%) [2013];
- in nitroethane at 60° (44%) [2147];
- in carbon disulfide at 45° (40%) [1953];
- in petroleum ether at 50° (20%) [2142];

without solvent:

- [1797,2143,2150,2152,2154,2157,2159,2162–2164,2166,2233,2234], but between 130° and 175° (40–60%) [2143,2150,2154,2159,2164,2233];
- aluminium chloride–sodium chloride mixture at 240–250° (10%) [2168];
- boron trifluoride at 90° (56%) [2164];
- scandium tris(trifluoromethanesulfonate), in nitromethane, at 50° (39%) [2170];
- titanium tetrachloride at 90–100° (34%) [2162];
- ferric chloride at 65° (25%) [2235];
- zinc chloride at 125° (8%) [2171];

with Protic acids

- hydrofluoric acid [2236–2238], between 20° and 100° (94%) [2237], (81%) [2236];
- polyphosphoric acid, between 20° and 100° (69%) [2239], (50–53%) [2172,2240], (44%) [2241];

with a cation exchange resin

- Nafion-XR 500, sulfonic acid type at 100° [2173];

with Zeolites molecular sieves

- ZSM-5, in sulfolane, at 180° (28%) [2175];
- H-ZSM-5 at 400° [2177] or at 210° (6%) [2176];
- H-Nu-2 at 170° (15%) [2176];
- HY (Si/Al = 3) or fluorided alumina (Al_2O_3-F; 3 % wt. F), at 400° [2177].

- Also obtained by Fries rearrangement of 4-trimethylsilylphenyl acetate with aluminium chloride in refluxing carbon disulfide (55%) [2178].
- Also obtained (by-product) by Fries rearrangement of 2-bromophenyl acetate with aluminium chloride without solvent at 180° (8%) [1817].
- Preparation by reaction of acetic acid on phenol,
  - with boron trifluoride, between 30° an 80° (95%) [2242], (85%) [2243], (77%) [2244];
  - with polyphosphoric acid [2239–2241], between 70° and 100° (65–67%) [2239,2240];
  - with hydrofluoric acid, at 100° (61%) [2236];
  - with zinc chloride (Nencki reaction) [2171,2180,2186,2245], (11%) [2180];
  - with Nafion-XR 500, sulfonic acid type, at 100° [2173].
- Preparation by reaction of acetyl chloride on phenol,
  - with aluminium chloride, in nitrobenzene, between 45° and 60° (70–74%) [2181,2233], (50–55%) [2144,2246];
  - with titanium tetrachloride, in nitrobenzene, at 60° (70%) [2181];
  - with butanesulfonic acid, at 85–90° (23%) [2247];
  - with ferric chloride, in carbon disulfide [1823,2221], (21%) [1823];
  - with zinc chloride [2220].
- Also obtained by reaction of acetyl chloride on phenyl borate with aluminium chloride in refluxing carbon disulfide (35%) [2145].
- Preparation by reaction of acetic anhydride on phenol,
  - with 70% perchloric acid (20%) [2183];
  - with aluminium chloride (48%) [2248], (19%) [2183];
  - with zinc chloride at 145–150° (40%) [2184].
- Also obtained by reaction of phenyl 2-acetoxybenzoate on phenol with aluminium chloride at 180° (18%) [2249].
- Preparation by reaction of acetonitrile on phenol with triflic acid (trifluoromethanesulfonic acid) at r.t. (74%) [2250].
- Preparation by dealkylation of 4-ethoxyacetophenone [2251,2252], (70%) [2252] or 4-methoxy-acetophenone (70%) [2252] with aluminium chloride between 140° and 180°.
- Also obtained by UV light irradiation of phenyl acetate (photo-Fries rearrangement),
  - in water, at 25–30° (24–25%) [2194,2195],
    - in the presence of β-cyclodextrin [2192,2194], (69%) [2194];
    - in the presence of methyl α-D-glucopyranoside (32%) [2194];
  - in benzene or methanol, at r.t. (38–39%) [2192],
  - in cyclohexane, ethyl ether, ethanol or isopropanol, at 25–30° (10–15%) [2193,2196,2197],
  - in hexane, at 25° (4%) [2015],
    - in the presence of potassium carbonate (10%) [2015].

- Also obtained by UV light irradiation of 4-methoxyphenyl acetate in ethyl ether, at 25° (7%) [2193].
- Also obtained by UV light irradiation of 4-hydroxy-α-chloroacetophenone in ethanol (26%) [2199].
- Also obtained by reaction of acetylacetone on phenyl benzoate with aluminium chloride in nitrobenzene at 45° (12%) [2144].
- Preparation by diazotization of 4-aminoacetophenone, followed by hydrolysis of the obtained diazonium salt [2253,2254].
- Also obtained by treatment of acetophenone with sodium trifluoroacetate in nitromethane–trifluoroacetic acid–trifluoroacetic anhydride mixture in the presence of a platinum electrode, followed by treatment of the intermediate trifluoroester with 10% potassium hydrogen carbonate solution (16%) [2006], (hydroxylation of aromatic compounds).
- Also obtained by reaction of stannous chloride on 4-hydroxyacetophenone 2,4-dinitro-phenylhydrazone in refluxing aqueous acetic acid–hydrochloric acid mixture (66%) [2255].

Isolation from natural sources:

- From the roots of *Paeonia broteroi* (Paeoniaceae) [2256].
- The Picein or 4-hydroxyacetophenone-D-glucoside has been isolated from leaves of *Picea Glehnii Masters* (Coniferae). This compound, by hydrolysis with dilute mineral acids or with emulsin leads to 4-hydroxyacetophenone [2257,2258].
- Hinokiflavone has been isolated from *Chamaecyparis obtusa* (Coniferae) [2259]. When treated with potassium hydroxide, Hinokiflavone produces 4-hydroxyacetophenone [2260].
- From spruce needles (*Picea abies* L. Karst) [2261].
- Also obtained by saponification of Apiin or Apigenin with boiling 25% aqueous sodium hydroxide (87%) [2262]. Apiin or 4′,5,7-trihydroxyflavone-7-apiosylglucoside was isolated from parsley or from celery [2263,2264].

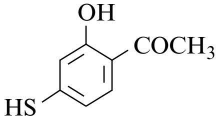
m.p. 115° [2168], 112° [2262], 110–111° [2144], 110° [1953,2142,2221], 109–110° [1797], 109° [1914,2145,2186,2204,2239,2242,2249,2258], 108–110° [2238], 108–109° [2148,2178,2183,2265], 108° [1823,2143,2150,2151,2153,2162,2180,2235,2245,2255], 107–109° [2157,2196,2236,2240], 107°5–108° [2244], 107–108° [1789,2257], 107° [2220,2241,2250,2252,2253,2261,2266], 106°2–107°8 [2162], 106–107° [2154,2171,2246,2267], 105–108° [2243], 104–106° [2184];

b.p.$_3$ 147–148° [2180], b.p.$_{20}$ 170–194° [2240], b.p.$_4$ 175° [1914], b.p.$_{15}$ 190° [2171];

^{1}H NMR [1817,2250,2256,2261], ^{13}C NMR [2219], IR [1797,2091,2261],
UV [1822,1829,2091,2165,2186,2216,2261,2268,2269];
pK$_a$ [1977,2091,2217].

1-(2-Hydroxy-4-mercaptophenyl)ethanone

[35204-52-5] $C_8H_8O_2S$ mol.wt. 168.22



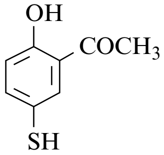
Syntheses

- Preparation by reaction of stannous chloride with 2-acetyl-5-chlorosulfonylphenol in acetic acid previously saturated with gaseous hydrochloric acid (63%) [2270].
- Also refer to: [2271].

m.p. 60–62° [2270].

1-(2-Hydroxy-5-mercaptophenyl)ethanone

[35204-45-6] $C_8H_8O_2S$ mol.wt. 168.22

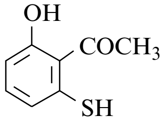


Synthesis

- Refer to: [2272].

1-(2-Hydroxy-6-mercaptophenyl)ethanone

[83080-88-0] $C_8H_8O_2S$ mol.wt. 168.22

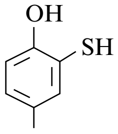


Synthesis

- Preparation by reaction of 2 N aqueous sodium hydroxide on 2-dimethylcarbamylthio-6-hydroxyacetophenone in refluxing methanol (65%) [2273].

1-(4-Hydroxy-3-mercaptophenyl)ethanone

[66264-55-9] $C_8H_8O_2S$ mol.wt. 168.22



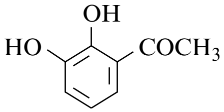
Synthesis

- Preparation by reaction of stannous chloride dihydrate on 3-(chlorosulfonyl)-4-hydroxyacetophenone with gaseous hydrochloric acid in acetic acid at 25–30° (31%) [2274,2275].

m.p. 117–120° [2274,2275].

1-(2,3-Dihydroxyphenyl)ethanone

[13494-10-5] $C_8H_8O_3$ mol.wt. 152.15



Syntheses

- Preparation by reaction of acetic anhydride on pyrocatechol with 70% perchloric acid on a steam bath (58%) [2183], at 100° (4%) [2276].

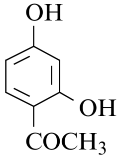
- Preparation by demethylation of 2,3-dimethoxy-acetophenone [2277–2281], (73%) [2278], (50%) [2280], itself obtained by oxidation of 2,3-dimethoxyphenyl methyl carbinol [2277,2279–2281]. This "carbinol" was prepared either from 2,3-dimethoxybenzaldehyde by an organomagnesium synthesis [2277,2279,2280] or from 2,3-dimethoxycinnamic acid by Posner's method [2280,2281].
- Also obtained by acid hydrolysis of 8-hydroxyflavone [2280].
- Also obtained by UV light irradiation of pyrocatechol monoacetate in ethanol at 30° (19–22%) [2196,2197,2282].
- Also obtained (low yield) by degradation of an aqueous solution (pH 4.5) of D-xylose at 96° (0.5%) [2283].
- Also obtained (low yields) by degradation of aqueous solutions (pH 3.5 and 4.5) of D-glucuronic acid at 96° (0.3% and 0.2% yields, respectively) [2283].
- Also obtained from 3-acetyl-1-oxocyclohexane-2,3,6-triol, either on sublimation at 0.5 mm Hg (70% yield) or when heated in an aqueous solution (pH 4.5) and 96° (50% yield). The above triol was obtained in crystalline form (m.p. 149–150°) by reaction of D-glucuronic acid in aqueous solutions (pH 3.5 and 4.5) at 96° [2283].
- Also obtained (by product) by UV light irradiation of 3-methyl-1,2-benzisoxazole in 96% sulfuric acid (10%) [2284,2285].
- Also obtained by hydrolytic rearrangement of 2-acetoacetyl-2,5-dimethoxytetrahydrofuran in refluxing 0.1 N hydrochloric acid (88%) [2286], (52%) [2287].
- Also obtained by hydrolysis of (2,2-dimethyl-1,3-benzodioxol-4-yl)acetone (m.p. 72–73°) with concentrated hydrochloric acid in refluxing ethanol under argon atmosphere (84%) [2288].

m.p. 98–98°5 [2286], 98° [2196,2197,2278], 97–98° [2280,2282], 97° [2183], 96°5–97°5 [2284], 96–98° [2276,2283], 96–97° [2288], 95–96° [2287];

^{1}H NMR [2282,2284,2287,2288], ^{13}C NMR [2288], IR [2284], UV [2284], MS [2284,2288].

1-(2,4-Dihydroxyphenyl)ethanone *(Resacetophenone)*

[89-84-9] $C_8H_8O_3$ mol.wt. 152.15



Syntheses

- Preparation by reaction of acetic acid on resorcinol,
  - with zinc chloride (Nencki reaction) [1833,1835,1838,2129, 2289–2296], (94%) [2294], (57–65%) [2129,2291,2292];
  - with boron trifluoride [2242,2297,2298], (85–94%) [2242, 2298];
  - with Amberlite IR-120 (a cation exchange resin, sulfonic acid type) (87%) [2026];
  - with polyphosphoric acid [2241,2299], (63%) [2299];
  - with 70% perchloric acid (33%) [2297].
- Preparation by reaction of acetonitrile on resorcinol,
  - with triflic acid (trifluoromethanesulfonic acid) (87%) [2250];
  - with zinc chloride (Hoesch reaction) [2300–2304], (77%) [2303], (70%) [2301].

- Preparation by reaction of acetic anhydride on resorcinol,
  - with boron trifluoride (91%) [2305];
  - with 70% perchloric acid (90%) [2183], (70%) [2306];
  - with Amberlite IR-120 (83%) [2026];
  - with zinc chloride at 145–150° (2%) [2184];
  - with a trace of concentrated sulfuric acid at 130° (75%) [2307].
- Preparation by reaction of acetyl chloride on resorcinol,
  - with aluminium chloride in nitrobenzene at r.t. (65%) [2308], (50%) [2309];
  - with Amberlite IR-120 (52%) [2026];
  - with zinc chloride [2310,2311].
- Preparation by Fries rearrangement of resorcinol monoacetate with 70% perchloric acid in acetic anhydride at r.t. (83%) [2183].
- Preparation by Fries rearrangement of resorcinol diacetate,
  - with Amberlite IR-120 (64%) [2026];
  - with aluminium chloride [2024,2312,2313], (63%) [2313], (60%) [2024];
  - with boron trifluoride etherate in benzene at reflux (60%) [2167];
  - with zinc chloride [2129,2314], (52%) [2314].
- Preparation by Fries rearrangement of 3-methoxyphenyl acetate with aluminium chloride without solvent at 180–185° (60%) [2024].
- Also obtained by UV light irradiation of resorcinol diacetate in methanol at 25° [2315].
- Also obtained by demethylation of 2-hydroxy-4-methoxyacetophenone,
  - with potassium hydroxide [2316];
  - with hydriodic acid [2316].
- Also obtained by reaction of concentrated sulfuric acid on resacetophenone diacetate [2312].
- Also obtained by reaction of potassium hydroxide on 4-methylumbelliferone [2317] or on 4-methylene-2-phenyl-4*H*-chromen-7-ol [2318].
- Also obtained by decarboxylation of 2-acetyl-3,5-dihydroxybenzoic acid with copper powder in quinoline at 220–230° [2319].
- Also obtained by decarboxylation of 3-acetyl-2,6-dihydroxybenzoic acid with dilute hydrochloric acid at reflux [2320,2321].
- Also obtained by decarboxylation of 5-acetyl-2,4-dihydroxybenzoic acid with dilute hydrochloric acid at 160–170°, in a sealed tube [2322].

Isolation from natural sources

- From Chinese Moutan Cortex, the root of *Paeonia suffruticosa* Andrews (Paeoniaceae) [2323].
- Also obtained by thermal decomposition of the resin from *Ferula pyramidata* (Kar. et Kir.) eug. kor. [2293].

m.p. 147° [1833,2242,2298], 146° [2306–2308], 145–146° [2293], 145° [2026,2300], 144–145° [2301,2305], 144° [2184], 143° [2297,2322], 142–144° [2291], 142–143° [2320,2324], 142° [2266,2278,2289,2290,2313,2314,2316,2325] [2129,2186,2204,2241,2299,2309], 141–142° [2317,2319], 141° [1835], 140–141° [2292], 138–140° [2250], 133–140° [2294];

b.p. 303–305° (d) [2129];

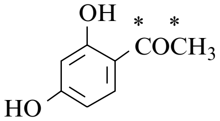
$d^{141^\circ 1}$ = 1.18 [2220]; $n_D^{141^\circ 1}$ = 1.56467 [2220];

^{1}H NMR [2205,2250,2326,2327], ^{13}C NMR [1821,2219,2328,2329],

UV [2213,2296,2326,2330], MS [2331]; pK_a [1977].

1-(2,4-Dihydroxyphenyl)ethanone-$^{13}C_2$

[74291-78-4] $C_8H_8O_3$ mol.wt. 154.13

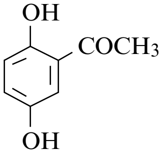


Synthesis

– Preparation by reaction of [1,2-$^{13}C_2$]acetyl chloride with resorcinol in nitrobenzene in the presence of aluminium chloride for 24 h at r.t. (65%) [2332].

1-(2,5-Dihydroxyphenyl)ethanone *(Quinacetophenone)*

[490-78-8] $C_8H_8O_3$ mol.wt. 152.15



Syntheses

- Preparation by Fries rearrangement of hydroquinone diacetate,
  - with aluminium chloride [1970,2024,2072,2333–2341], (91%) [2337], (76%) [2072,2341], (63–77%) [2333,2334,2340], (55–60%) [1970,2024,2336,2338];
  - with zinc chloride in refluxing acetic acid (quantitative yield) [2342];
  - with boron trifluoride etherate in benzene at reflux (65%) [2167].
- Preparation by Fries rearrangement of hydroquinone diacetate with aluminium chloride in the presence of hydroquinone (54%) [2339].
- Also obtained by UV light irradiation of hydroquinone diacetate in methanol (35%) [2343].
- Preparation by Fries rearrangement of 4-methoxyphenyl acetate with aluminium chloride without solvent at 130° (40%) [2344].
- Preparation by Fries rearrangement of 4-(benzoyloxy)phenyl acetate with aluminium chloride without solvent at 125–130° (22%) [2344].
- Preparation by reaction of acetic acid on hydroquinone,
  - with zinc chloride (Nencki reaction) [2000,2186,2289,2336,2342,2345–2347], (25–28%) [2345,2347];
  - with boron trifluoride [2242,2298,2348–2350], (95%) [2350], (66–70%) [2242,2298];
  - with Amberlite IR-120 or Zeokarb 225 (22%) [2026].

- Preparation by reaction of acetyl chloride on hydroquinone with aluminium chloride [2289,2336,2339], (35–40%) [2336,2339].
- Also obtained by reaction of acetic anhydride on hydroquinone,
  - with zinc chloride at 145–150° (76%) [2184];
  - with Amberlite IR-120 (27%) [2026].
- Also obtained (by-product) by reaction of acetic acid on 1,4-dimethoxybenzene with boron trifluoride at 70° [2298].
- Preparation by dealkylation of,
  - 2,5-dimethoxyacetophenone with aluminium bromide in refluxing carbon disulfide (81%) [2351];
  - 2-hydroxy-5-methoxyacetophenone with hydriodic acid [2352];
  - 2,5-diethoxyacetophenone or 5-ethoxy-2-hydroxyacetophenone with aluminium chloride [2289].
- Preparation by diazotization of 5-amino-2-hydroxyacetophenone, connected with hydrolysis of the obtained diazonium salt [2076,2353].
- Preparation by hydrolysis of keto esters further on,
  - 5-(benzoyloxy)-2-hydroxyacetophenone with concentrated sulfuric acid at r.t. [2344];
  - 5-acetoxy-2-hydroxyacetophenone,
    - with 5% aqueous sodium hydroxide (96%) [2338];
    - with a 5% solution of hydrogen chloride in methanol (75%) [2354];
    - with aluminium chloride in refluxing carbon disulfide (55%) [2338].
- Also obtained by reduction of 2-acetyl-1,4-benzoquinone,
  - with aqueous sodium hydrosulfite, in ethyl ether [2351];
  - with allyltrimethylstannane, in benzene (36%) [2355].
- Also obtained by reaction of acetaldehyde on 1,4-benzoquinone with sunlight, in a sealed tube (good yield) [2356].
- Also obtained (by-product) from 5-bromo-6-hydroxy-2-methylchromone by alkaline degradation with 10% aqueous sodium hydroxide solution at reflux (50%) [1832].
- Also obtained (poor yield) by alkaline degradation of a solution of D-xylose or D-glucose in 0.63 M sodium hydroxide at 96° under nitrogen [2276].
- Also obtained by UV light irradiation of 3-methyl-1,2-benzisoxazole in 96–98% sulfuric acid (52–57%) [2284,2285].
- Also obtained by heating 5′-cinnamyloxy-2′-hydroxyacetophenone at 220° (64%) [2357].
- Also obtained from 5-(allyloxy)-2-hydroxyacetophenone by cleavage of allyl group with bis(benzonitrile)palladium (II) chloride in refluxing benzene (89%) [2226].

m.p. 206° [2336], 205–206° [2276,2354], 204°6–205°4 [1970], 204° [2350], 202°6–203°6 [2284], 202–204° [2026], 202–203° [2334,2338,2340], 202° [2076,2242,2278,2289,2298,2337,2339,2346,2352,2356] [2341,2343], 201–203° [2355], 201–202° [2266,2333,2342,2344,2345,2351], 201° [2186,2204], 200–201° [2347], 200° [2184], 198–200° [2358], 197°5–198°5 [2072], 196–198° [2226,2357];

b.p.$_{0.01}$ 86° [2226]; pK$_a$ [1977];

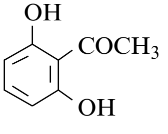
^{1}H NMR [2338,2348], (Sadtler: standard n° 4286); ^{13}C NMR [1821,2328],

IR [2284,2338,2355], (Sadtler: standard n° 10815);

UV [1779,2186,2284,2348], (Sadtler: standard n° 6276); MS [2348,2355].

1-(2,6-Dihydroxyphenyl)ethanone *(γ-Resacetophenone)*

[699-83-2] $C_8H_8O_3$ mol.wt. 152.15



Syntheses

- Preparation by hydrolysis of 8-acetyl-4-methyl-umbelliferone (8-acetyl-7-hydroxy-4-methylcoumarin) with aqueous sodium hydroxide solution at reflux (56–73%) [2213,2359–2361], (77–89%) [2362–2367], (89–100%) [2363,2368–2370].
- Preparation by hydrolysis of 8-acetyl-4-phenylumbelliferone with aqueous sodium hydroxide solution at reflux (33%) [2371].
- Preparation by demethylation of 2,6-dimethoxyacetophenone with aluminium chloride in toluene at 120° (24%) [2366,2367], (59%) [2372]. The 2,6-dimethoxyacetophenone was obtained from 2,6-dimethoxybenzonitrile and methylmagnesium iodide.

From Microorganisms

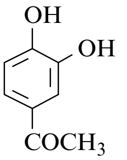
- Isolation from *Daldinia concentrica* [2373].

m.p. 157–158° [2372], 157° [2278,2369,2371], 156–157° [2359,2361,2366,2367], 155–156° [2213], 154–157° [2374], 154–156° [2370], 154–155° [2363–2365], 154° [2360], 152–154° [2368];

^{1}H NMR [2206,2374], ^{13}C NMR [2206,2219], UV [2213,2375].

1-(3,4-Dihydroxyphenyl)ethanone

[1197-09-7] $C_8H_8O_3$ mol.wt. 152.15



Syntheses

- Preparation by Fries rearrangement of pyrocatechol diacetate with aluminium chloride in nitrobenzene between 75° and 95° [2282,2339,2376], (80%) [2339], (64%) [2282], (43%) [2376] or in chlorobenzene at 80° (83%) [2377].
- Also obtained by Fries rearrangement of guaiacol acetate with aluminium chloride in nitrobenzene at r.t. (30%) [2339] or without solvent between 20° and 50° (6%) [2378].

- Preparation by reaction of acetic acid on pyrocatechol,
    - with boron trifluoride in a sealed tube at 150° (43%) [2298];
    - with zinc chloride (Nencki reaction) [2171,2379], (20%) [2171];
    - with polyphosphoric acid (10%) [2299].
- Also obtained by reaction of acetic anhydride on pyrocatechol,
    - with perchloric acid at 100° (6%) [2276];
    - with zinc chloride at 145–150° (51%) [2184].
- Preparation by reaction of acetyl chloride with pyrocatechol with aluminium chloride at 140° [2380].
- Also obtained by UV light irradiation of pyrocatechol monoacetate in ethanol at 30° (18%) [2196,2197].
- Also obtained by reaction of 5% aqueous potassium hydroxide on Luteolin, at reflux [2381].
- Preparation by demethylation of acetovanillone,
    - with dilute hydrochloric acid in a sealed tube at 140–150° [2379];
    - using aluminium chloride and pyridine [2331,2382].
- Also obtained by reaction of zinc powder [2266,2383–2386] or stannous chloride [2384] and hydrochloric acid on 3,4-dihydroxy-α-chloroacetophenone (quantitative yield) [2266,2384], (45–49%) [2385].
- Also obtained by reaction of aluminium bromide on (3,4-methylenedioxy)acetophenone (acetopiperone) in nitrobenzene at r.t. (48%) [2387].
- Also obtained from 3,4-diacetoxyacetophenone [2383],
    - by heating with a concentrated solution of sodium carbonate;
    - by refluxing with 5% sulfuric acid;
    - by treatment with porcine pancreatic lipase in diisopropyl ether and n-butanol at 42–45° (80%) [2388,2389].
- Also obtained (by-product) by chlorination of acetoguaiacone in dioxane–water mixture at 40° (3%) [1789].
- Also obtained (poor yield) by alkaline degradation of a solution of D-xylose or D-glucose in 0.63 M sodium hydroxide at 96° under nitrogen (<1%) [2276].
- Also obtained from neutral glucose and fructose solutions heated at 120° [2390].

Isolation from natural sources

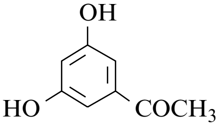
- From the needles of *Picea obovata* Ledeb. and *Picea koraiensis* Nakai (Pinaceae) [2391].
- This ketone was shown to occur in natural humic acids* and fulvic acids by hydrolysis with 2 N sodium hydroxide at 170° [2392]. Allomelanins* found in soils, coals and peat, resulting from the decomposition of organic matter, particularly dead plants [2393].
- By hydrolysis of its 3-O-β-D-glucopyranoside (*poungenoside*) (m.p. 200–202°) [2391].

N.B.: it was found to be an antimicrobial substance in coffee residue [2394].

m.p. 122° [2196,2197], 120° [2298], 119–121° [2385], 119°2–119°7 [2380], 118–120° [2276], 117–118° [2387], 117° [2331], 116–117° [2391], 116° [2171,2184,2299,2339,2376,2382,2384], 115–116° [2266,2282], 114–116° [2381], 114–115° [2383], 114° [2386], 110–112° [2377], 96–98° [2379];
b.p.$_{11}$ 127–133° [2299]; pK_a [2091]; TLC [2391]; HPLC [2394];
^{1}H NMR [2276,2282,2391,2394], ^{13}C NMR [2394], IR [2091,2276,2391,2392], UV [2091,2196,2282,2391], MS [1789,2276,2331,2392,2394].

1-(3,5-Dihydroxyphenyl)ethanone

[51863-60-6] $C_8H_8O_3$ mol.wt. 152.15



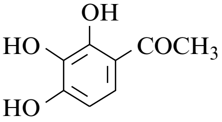
Syntheses

- Preparation from 3,5-dimethoxyacetophenone (SM) by demethylation with aluminium chloride in refluxing chlorobenzene (71%) [2395]. The starting material (SM) was prepared by a three-step procedure from 3,5-dimethoxy-benzoic acid.
- Preparation from 3,5-diacetoxyacetophenone by hydrolysis with 10% sulfuric acid at reflux (41%) [2396]. The starting ketone was prepared by reaction of methyl bromide on 3,5-diacetoxybenzoyl chloride in the presence of dimethyl cadmium.

m.p. 148° [2396], 147–148° [2395].

1-(2,3,4-Trihydroxyphenyl)ethanone *(Gallacetophenone)*

[528-21-2] $C_8H_8O_4$ mol.wt. 168.15



Syntheses

- Preparation by Fries rearrangement of pyrogallol triacetate with aluminium chloride without solvent [2312,2335], (very good yield) [2335].
- Preparation by reaction of acetic anhydride on pyrogallol,
  - with Amberlite IR-120 or Zeokarb 225, at 160° (84%) [2026];
  - with few drops sulfuric acid at 130° (65%) [2307];
  - with zinc chloride at 145–150° (53%) [2184] or in the presence of acetic acid (58–63%) [2397,2398];
  - with sulfuric acid and benzene-1,3-disulfonic acid mixture at 135° (54%) [2399];
  - with 70% perchloric acid on a steam bath (44%) [2183].
- Preparation by reaction of acetyl chloride on pyrogallol with aluminium chloride [2309,2400–2402].
- Preparation by reaction of acetic acid on pyrogallol,

  - with boron trifluoride at 28–30° (90%) [2242,2403] alone or in ethyl ether at 0° (90%) [2268];
  - with Amberlite IR-120 or Zeokarb 225 at 160° (75%) [2026];
  - with zinc chloride at 140–150° (Nencki reaction) (good yield) [2129,2186,2290,2404], (58%) [2405];
  - with 70% perchloric acid, at reflux (30%) [2297].

– Also obtained by dealkylation of 2,3-dihydroxy-4-methoxyacetophenone with aluminium chloride in refluxing chlorobenzene [2406].

– Also obtained by hydrolysis of 3,4-diacetoxy-2-hydroxyacetophenone [2343].

– By other method (90%), also refer to: [2407].

– Preparation by reaction of hydrogen peroxide on 3-formylresacetophenone in 1 N aqueous sodium hydroxide (79%) [2360].

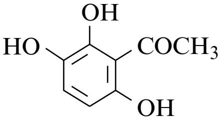
– Also obtained by cleavage of 4-methylene-2-phenyl-4*H*-chromene-7,8-diol with boiling 10% potassium hydroxide [2318].

m.p. 186° [2312], 173° [2183,2242,2297,2401,2403,2404], 172° [2186,2204], 171–173° [2184], 171–172° [2397,2398], 171° [2268,2400], 170° [2405], 169–171° [2026], 169–170° [2307,2343], 168° [2129,2266,2290,2360,2402,2406], 167° [2309];

IR [2403], UV [2186,2268].

1-(2,3,6-Trihydroxyphenyl)ethanone

[85918-30-5] $C_8H_8O_4$ mol.wt. 168.15



Syntheses

– Photolysis of 1,2,4-triacetoxybenzene gave a complex mixture from which 2-acetyl-3,6-diacetoxyphenol could be isolated. The triacetate of this one, by deacetylation afforded 3-acetyl-1,2,4-trihydroxybenzene [2343].

– Preparation by reaction of potassium persulfate on 2,6-dihydroxyacetophenone in aqueous sodium hydroxide solution at 15–20° (29%) [2408].

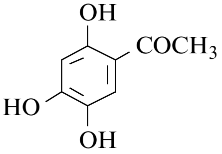
– Preparation by reaction of hydrogen peroxide on 3-formyl-2,6-dihydroxyacetophenone in 1 N aqueous sodium hydroxide (71–74%) [2360,2409].

m.p. 160° [2343], 157° [2360],
230° (d) [2408], 96° [2409]. These reported melting points are obviously wrong.

(The triacetate melted to 155° [2408]).

1-(2,4,5-Trihydroxyphenyl)ethanone

[1818-27-5] $C_8H_8O_4$ mol.wt. 168.15



Syntheses

– Preparation by Fries rearrangement of 1,2,4-triacetoxy-benzene,

  - with aluminium chloride in nitrobenzene [2312,2410–2413], (30–43%) [2411–2413];

- with zinc chloride at 135–140° [2399,2414–2416], (49–53%) [2399,2414];
- with p-toluenesulfonic acid in refluxing tetrachloroethane or benzene or without solvent at 135–140° (60%) [2399].

– Also obtained by reaction of potassium persulfate on resacetophenone (Elbs reaction),

- in aqueous solution of potassium hydroxide at r.t. (24–30%) [2417,2418];
- with ferrous sulfate in aqueous sodium hydroxide at r.t. (18%) [2415].

– Preparation by reaction of acetonitrile on hydroxyquinol with zinc chloride (Hoesch reaction) [2411–2413,2419], (25%) [2411].

– Also obtained by reaction of acetic acid–acetic anhydride mixture on hydroxyquinol with zinc chloride at 140–150° (32%) [2411].

– Also obtained by reaction of acetic anhydride on hydroxyquinol with concentrated sulfuric acid at 135° (61–81%) [2399].

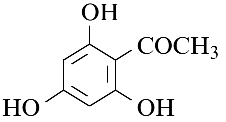
– Preparation by reaction of acetic anhydride on p-quinone with concentrated sulfuric acid alone or with benzenesulfonic acid, p-toluenesulfonic acid, dl-camphorsulfonic acid or benzene-m-disulfonic acid at 135° (53 to 70%) [2399].

– Also refer to: [2420].

m.p. 208° [2416], 206–207° [2312,2411–2413], 206° [2419], 202–204° [2417], 200–202° [2414,2415];
^{13}C NMR [2421], MS [2331].

1-(2,4,6-Trihydroxyphenyl)ethanone *(Phloroacetophenone)*

[480-66-0] $C_8H_8O_4$ mol.wt. 168.15



Syntheses

– Preparation by reaction of acetic anhydride on phloroglucinol,

- with concentrated sulfuric acid at 130° (70%) [2307];
- with boron trifluoride at 10° (62.5–68%) [1844];
- with zinc chloride at 145–150° (50%) [2184];
- with Amberlite IR-120 or Zeokarb 225 (cation exchange resins, sulfonic acid type) at 160° (39%) [2026].

– Preparation by reaction of acetonitrile on phloroglucinol,

- with zinc chloride (Hoesch reaction) [2268,2301,2422–2427], (93%) [2425], (74–87%) [2301,2422,2424,2426];
- with triflic acid, at r.t. (40%) [2250].

– Preparation by reaction of acetyl chloride on phloroglucinol,

- with boron trifluoride, at 10° (62.5–68%) [1844];
- with aluminium chloride, in nitrobenzene [2309,2312,2428], (25–30%) [2312,2428];
- with ferric chloride, in a boiling water bath [2000].

- Preparation by reaction of acetic acid on phloroglucinol with zinc chloride (Nencki reaction), and then saponification of 5,7-dihydroxy-4-methylene-2-(2,4,6-trihydroxyphenyl)-4*H*-benzopyran first formed (76%) [2429].
- Preparation by reaction of phenyl acetate on phloroglucinol with boron trifluoride etherate in refluxing benzene (30%) [2167].
- Also obtained by Fries rearrangement of phloroglucinol triacetate with aluminium chloride in nitro-benzene at 27° (10%) [2312].
- Also obtained by deacetylation of 4,6-diacetoxy-2-hydroxyacetophenone [2343].
- Also obtained (high yields) by hydrolysis of undermentioned compounds with water at 160–170° [2430],
    - ethyl 5,7-dihydroxy-2,4-dioxo-chroman-8-carboxylate;
    - ethyl 3-ethoxycarbonylacetyl-2,4,6-trihydroxybenzoate;
    - diethyl 5-ethoxycarbonylacetyl-2,4,6-trihydroxy-isophthalate.
- Also obtained by cleavage of 4-methylene-2-phenyl-4*H*-chromen-5,7-diol with boiling 10% potassium hydroxide (low yield) [2318,2431].

m.p. 284–285° [2429], 280° [2318], 221–223° [1844], 220–222° [2026], 219° [2423], 218°5 [2425], 218–219° [2424,2426], 218° [2301,2309,2312,2430], 217–218° [2422], 216–218° [2250], 214–216° [2184], 214–215° [2432], 213–214° [2307,2428], 209–210° [2431];

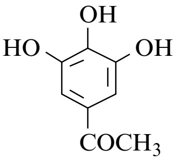
TLC [2433]; tautomerism [2434];

^{1}H NMR [2206,2250,2435], ^{13}C NMR [2206,2219,2328,2436], IR [2435],

UV [2268,2437], MS [2331].

1-(3,4,5-Trihydroxyphenyl)ethanone

[33709-29-4] $C_8H_8O_4$ mol.wt. 168.15



Syntheses

- Preparation by reaction of aluminium chloride on gallacetophenone trimethyl ether in refluxing chlorobenzene (71%) [2438].
- Preparation from diethyl 3,4,5-triacetoxybenzoylmalonate by hydrolysis and decarboxylation with 10% sulfuric acid in acetic acid at 80° (92%) [2439].
- Also refer to: [2440,2441].

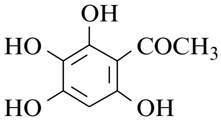
Isolation from natural sources

- This ketone was shown to occur in natural humic acids* and fulvic acids by hydrolysis with 2 N sodium hydroxide at 170° [2392]. Allomelanins* found in soils, coals and peat, resulting from the decomposition of organic matter, particularly dead plants [2393].

m.p. 187–188° [2438]; 178–180° [2439]; IR [2392], MS [2392,2439].

1-(2,3,4,6-Tetrahydroxyphenyl)ethanone

[63635-39-2] $C_8H_8O_5$ mol.wt. 184.15



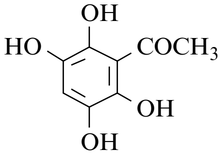
Syntheses

- Preparation by reaction of acetic acid on 1,2,3,5-tetrahydroxybenzene by heating with zinc chloride (Nencki reaction) [2442,2443], (84–85%) [2443].
- Preparation by reaction of acetonitrile on 1,2,3,5-tetra-hydroxybenzene (Hoesch reaction) [2444,2445], (65%) [2445].
- Also obtained by demethylation of 3,6-dihydroxy-2,4-dimethoxyacetophenone with aluminium chloride in boiling chlorobenzene (42%) [2446].
- Also refer to: [2442] (compound **I**); [2447] (compound **9**); [2448] (compound **2**); [2449] (compound **12**).

m.p. 243–244° [2446], 236–238° [2444,2445], 204–205° [2443];
^{1}H NMR [2445], IR [2445], MS [2445].

1-(2,3,5,6-Tetrahydroxyphenyl)ethanone

$C_8H_8O_5$ mol.wt. 184.15



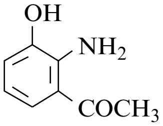
Syntheses

- Preparation by reaction of aluminium bromide on 2,3,5,6-tetramethoxyacetophenone in refluxing benzene (84%) [2450].
- Preparation by reaction of concentrated hydrochloric acid on 2,3,5,6-tetraacetoxyacetophenone in refluxing methanol (77%) [2450].

m.p. 134° [2450].

1-(2-Amino-3-hydroxyphenyl)ethanone

[4502-10-7] $C_8H_9NO_2$ mol.wt. 151.17



Syntheses

- Preparation from 3-hydroxy-2-nitroacetophenone,
  - by catalytic hydrogenation in the presence of Raney nickel in ethanol (good yield) [2451] or in the presence of 5% Pt/C catalyst in THF under normal pressure at r.t. for 5 h [2452], (96%) [2453];
  - by reduction with iron filings in aqueous hydrochloric acid [2093] (Béchamp reduction).
- Preparation by reaction of hydriodic acid with 3-methoxy-2-nitroacetophenone in the presence of phosphorous. Simultaneous demethylation and reduction of the nitro group occur [2093].
- Also obtained as a side product by photolysis of 3-methylanthranil in 98% sulfuric acid (6%) [2285,2454,2455].

- Also obtained from 2-azidoacetophenone, by thermal decomposition in 98% sulfuric acid (9%) [2454,2455] or by photolysis in dioxane/water/sulfuric acid mixture (3%) [2454,2455].
- Also refer to: [2456] (compound **8b**) and [2457,2458].

Isolation from natural sources
By alkaline degradation,

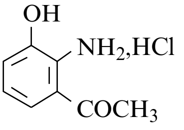
- of *3-hydroxykynurenine* [2459–2461], (13%) [2462], itself obtained by gentle alkaline degradation of *Xanthommatin* [2461];
- of *Ommin* (SM) by heating with 2 N sodium hydroxide in a water bath for 4 h under oxygen atmosphere (53%) [2460]. SM was isolated from the eyes of silkworms (*Bombyx Mori*) and prawns (*Crangon vulgaris*);
- of *Ommatin D* [2459] according to the method [2462];
- of *Ommochromes* (*Xanthommatin*, *Rhodommatin*, *Ommatin D* and *Bombyx Ommin*) with 2 N sodium hydroxide by heating in a water bath for 2 h [2459].

Also refer to: [2463].

m.p. 185–187° [2453], 185° [2093], 184–185° [2455], 183° [2462];
paper chromatography [2459,2460];
^{1}H NMR [2453,2455], ^{13}C NMR [2453], IR [2455,2462], UV [2122,2455,2459,2460],
MS [2455].

1-(2-Amino-3-hydroxyphenyl)ethanone *(Hydrochloride)*

$C_8H_9NO_2$, HCl mol.wt. 187.63



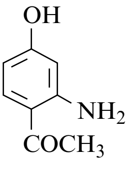
Syntheses

- Obtained by reaction of gaseous hydrochloric acid on 2-amino-3-hydroxyacetophenone in ethanol–ethyl ether mixture (93%) [2451].
- Also refer to: [2458].

m.p. 215° (d) [2451].

1-(2-Amino-4-hydroxyphenyl)ethanone

[90033-64-0] $C_8H_9NO_2$ mol.wt. 151.17



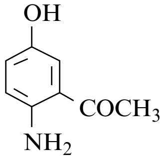
Syntheses

- Preparation by reaction of aluminium chloride with 2-amino-4-methoxyacetophenone in methylene chloride (63%) [2464].
- Preparation from 3-hydroxyaniline using acetonitrile with boron trichloride as a catalyst [2465] or by classical Friedel–Crafts techniques [2466].

^{1}H NMR [2464].

1-(2-Amino-5-hydroxyphenyl)ethanone

[30954-71-3] $C_8H_9NO_2$ mol.wt. 151.17



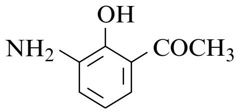
Syntheses

- Preparation by catalytic hydrogenation of 5-hydroxy-2-nitroacetophenone in the presence of platinic oxide in methanol (quantitative yield) [2091].
- Also obtained by UV light irradiation of,
  - 3-methyl-2,1-benzisoxazole (3-methylanthranil), in 98% sulfuric acid (83%) [2455], (87%) [2285,2454] or in 66% sulfuric acid at 80–90° (88–95%) [2225];
  - 3-methyl-1*H*-indazole, at 11–15°, in dilute sulfuric acid (26–28%) [2285,2467] or in methanol, water and sulfuric acid mixture (19%) [2285].
- Also obtained from 2-azidoacetophenone,
  - by thermal decomposition in 98% sulfuric acid (67%) [2454,2455];
  - by UV light irradiation in dioxane, water and sulfuric acid mixture (21%) [2454,2455].

m.p. 178–179° [2455,2467], 176–177° [2091,2225];
^{1}H NMR [2455,2467], IR [2455,2467], UV [2455,2467], MS [2455,2467].

1-(3-Amino-2-hydroxyphenyl)ethanone

[70977-72-9] $C_8H_9NO_2$ mol.wt. 151.17



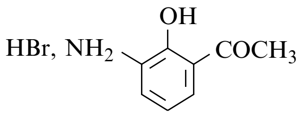
Syntheses

- Preparation by catalytic hydrogenation of 2-hydroxy-3-nitroacetophenone in the presence of Pt/C in ethanol at 25° (90%) [1898].
- Preparation by reaction of stannous chloride on 2-hydroxy-3-nitroacetophenone with hydrochloric acid [1965].
- Preparation by hydrogenolysis of 3-amino-5-chloro-2-hydroxyacetophenone [2140].
- Preparation by reduction of 2-hydroxy-3-nitroacetophenone (65%) [2075].

m.p. 95–97° [1898], 95–96° [2075], 93–94° [1965,2036].

1-(3-Amino-2-hydroxyphenyl)ethanone *(Hydrobromide)*

[136450-03-8] $C_8H_9NO_2$, HBr mol.wt. 232.08

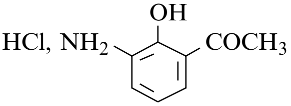


Synthesis

- Preparation by catalytic hydrogenation of 5-bromo-2-hydroxy-3-nitroacetophenone in presence of Pd/C in solution of methanol and methylene chloride mixture at r.t. (99%) [1805].

1-(3-Amino-2-hydroxyphenyl)ethanone *(Hydrochloride)*

[90005-55-3] $C_8H_9NO_2$, HCl mol.wt. 187.63

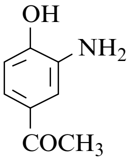


Synthesis

– Preparation by catalytic hydrogenolysis of 3-amino-5-chloro-2-hydroxyacetophenone hydrochloride at 25° in the presence of Pd/C in isopropanol (94%) [2140].

1-(3-Amino-4-hydroxyphenyl)ethanone

[54255-50-4] $C_8H_9NO_2$ mol.wt. 151.17



Syntheses

– Preparation by hydrolysis of 3-acetamido-4-hydroxy-acetophenone with boiling aqueous hydrochloric acid at 50% HCl [2468] or 10 N HCl (78%) [2469].

– Preparation from 4-hydroxy-3-nitroacetophenone,

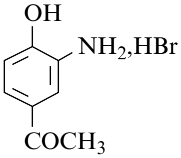
- by catalytic hydrogenation in acetone [1999,2098], (67%) [2098] or in the presence of 10% Pd/C in methanol at r.t. (95%) [2105];
- by reaction of tin in boiling concentrated hydrochloric acid [1953].

m.p. 127–128° [2469], 98–99° [2105], 98° [1999,2098,2468]. One of the reported melting points is obviously wrong.

oil [1953]; ^{1}H NMR [2469], IR [2469].

1-(3-Amino-4-hydroxyphenyl)ethanone *(Hydrobromide)*

$C_8H_9NO_2$, HBr mol.wt. 232.08



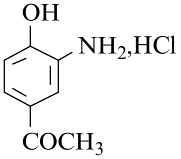
Synthesis

– Refer to: [2105].

m.p. 250° (d) [2105].

1-(3-Amino-4-hydroxyphenyl)ethanone *(Hydrochloride)*

[14347-14-9] $C_8H_9NO_2$, HCl mol.wt. 187.63



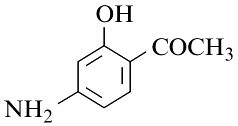
Synthesis

– Obtained on saturating with dry hydrogen chloride gas an acetone-ethyl ether solution of the amine [2098].

m.p. > 250° (d) (anhydrous) [2098],
232° (monohydrate) [2105].

1-(4-Amino-2-hydroxyphenyl)ethanone

[2476-29-1] $C_8H_9NO_2$ mol.wt. 151.17



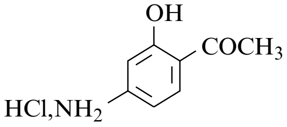
Synthesis

– Preparation by hydrolysis of 4-acetamido-2-hydroxy-acetophenone with boiling aqueous hydrochloric acid at 50% HCl [2468] or at 15% HCl [1956,2470], (86%) [1956].

m.p. 130° [2468], 129–130° [1956], 122–123° [2470].

1-(4-Amino-2-hydroxyphenyl)ethanone *(Hydrochloride)*

[51410-07-2] $C_8H_9NO_2$, HCl mol.wt. 187.63

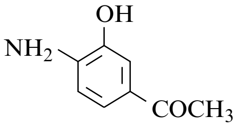


Synthesis

– Refer to: [1961].

1-(4-Amino-3-hydroxyphenyl)ethanone

[54903-54-7] $C_8H_9NO_2$ mol.wt. 151.17



Syntheses

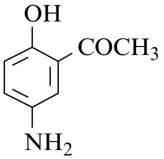
– Preparation from 6-acetyl-2-(3*H*)-benzoxazolinone by alkaline hydrolysis with boiling 10% aqueous sodium hydroxide solution (90–100%) [2469,2471].

– Also refer to: [2472] (compound **VI**) and [2456], (compound **8d**).

m.p. 170° [2469,2471]; ^{1}H NMR [2469], IR [2469], UV [2122].

1-(5-Amino-2-hydroxyphenyl)ethanone

[50-80-6] $C_8H_9NO_2$ mol.wt. 151.17



Syntheses

– Preparation by hydrolysis of 5-acetamido-2-methoxy-acetophenone (80%) [1975].

– Preparation by hydrolysis of 5-acetamido-2-hydroxy-acetophenone with boiling aqueous hydrochloric acid solution [1965,2016,2057,2468,2470,2473,2474], (50%) [2016,2473], (84%) [1965].

– Preparation by reduction of 2-hydroxy-5-nitroacetophenone,

  - with stannous chloride [1965,2016,2076], (53%) [2076];
  - by electrolytic way in concentrated sulfuric acid [2475].

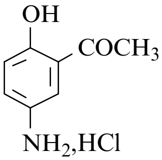
– The 5-amino-2-hydroxyacetophenone hydrochloride, by treatment with ammonia gave the keto-base [2353].

- Also obtained by electrolytic reduction of 3-nitroacetophenone in concentrated sulfuric acid [2107].

m.p. 121–122° [2076], 118° [2057,2353], 112–113°5 [1975], 112–113° [1965], 110° [2016,2107,2468,2475], 105° [2473,2474].

1-(5-Amino-2-hydroxyphenyl)ethanone *(Hydrochloride)*

[57471-32-6] $C_8H_9NO_2$, HCl mol.wt. 187.63

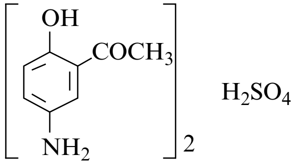


Synthesis

- Obtained by reaction of gaseous hydrochloric acid on 5-amino-2-hydroxyacetophenone in ethanol [1965,2057] or in ethyl ether [2473,2474].

m.p. 230–240° (d) [1965], 231° [2353], 155° (d) [2473,2474].

1-(5-Amino-2-hydroxyphenyl)ethanone *(Sulfate)*



2 $C_8H_9NO_2$, H_2SO_4 mol.wt. 400.41

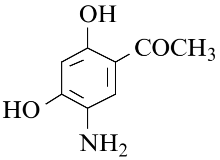
Synthesis

- Easily obtained by reaction of sulfuric acid on 5-amino-2-hydroxyacetophenone in ethyl ether [2474].

m.p. 150° [2474].

1-(5-Amino-2,4-dihydroxyphenyl)ethanone

[5528-13-2] $C_8H_9NO_3$ mol.wt. 167.16



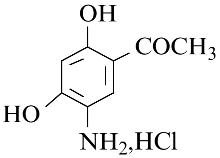
Synthesis

- Preparation from 2,4-dihydroxy-5-nitroacetophenone by reduction in acetone solution using Raney catalyst [2130].

m.p. 137–142° (d) [2130].

1-(5-Amino-2,4-dihydroxyphenyl)ethanone *(Hydrochloride)*

$C_8H_9NO_3$, HCl mol.wt. 203.61



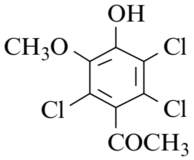
Synthesis

- Prepared by passing dry hydrochloric acid in 2,4-dihydroxy-5-aminoacetophenone in acetone solution, and adding ethyl ether when needed for precipitation [2130].

m.p. >300° [2130].

1-(2,3,6-Trichloro-4-hydroxy-5-methoxyphenyl)ethanone

[94649-69-1] $C_9H_7Cl_3O_3$ mol.wt. 269.51

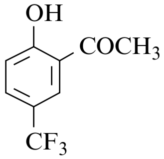


Synthesis not yet described

- Identified in wheat and rye straw pulp bleaching and combined mill effluents [1787].
- Identified on control of effluent from the manufacturing of bleached pulp and paper from sugarcane bagasse [1788].

1-[2-Hydroxy-5-(trifluoromethyl)phenyl]ethanone

[67589-15-5] $C_9H_7F_3O_2$ mol.wt. 204.15



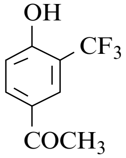
Synthesis

- Preparation by reaction of acetyl chloride with p-(trifluoromethyl)phenol in hydrofluoric acid for 6 h to 100° under 3 atm (88%) [2476].

^{1}H NMR [2476], IR [2476].

1-[4-Hydroxy-3-(trifluoromethyl)phenyl]ethanone

[149105-11-3] $C_9H_7F_3O_2$ mol.wt. 204.15



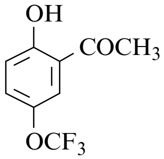
Synthesis

- Obtained by heating to 180° under nitrogen a mixture of 4-methoxy-3-(trifluoromethyl)acetophenone and pyridinium chloride (39%) [2477].

m.p. 168–170° [2477].

1-[2-Hydroxy-5-(trifluoromethoxy)phenyl]ethanone

[146575-64-6] $C_9H_7F_3O_3$ mol.wt. 220.15

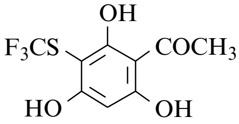


Synthesis

- Preparation by reaction of pyridinium dichromate with 4-trifluoromethoxy-2-(α-hydroxyethyl)phenol in the presence of Celite in methylene chloride at r.t. (85%) [2478,2479].

1-[2,4,6-Trihydroxy-3-[(trifluoromethyl)thio]phenyl]ethanone

[66625-03-4] $C_9H_7F_3O_4S$ mol.wt. 268.21



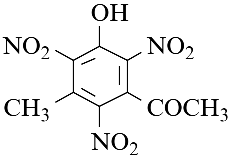
Synthesis

- Preparation by reaction of trifluoromethanesulfonyl chloride with phloroacetophenone in chloroform in the presence of a slight excess of pyridine, first at –40°, then at 60° for 3 h (33%) [2480].

m.p. 139–140° [2480]; ^{1}H NMR [2480], IR [2480].

1-(3-Hydroxy-5-methyl-2,4,6-trinitrophenyl)ethanone

$C_9H_7N_3O_8$ mol.wt. 285.17



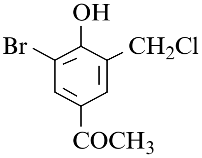
Synthesis

- Obtained by reaction of nitric acid on 3-hydroxy-5-methylacetophenone in acetic anhydride at 10° (23%) [2122].

m.p. 195–196° [2122].

1-[3-Bromo-5-(chloromethyl)-4-hydroxyphenyl]ethanone

[107724-60-7] $C_9H_8BrClO_2$ mol.wt. 263.52



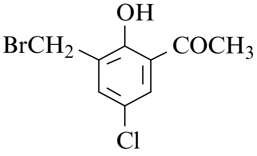
Synthesis

- Preparation by adding a methylene chloride solution of bromine to a methanol/methylene chloride solution of 3-(chloromethyl)-4-hydroxyacetophenone at 0° (85%) [2481].

m.p. 120–121° [2481]; ^1H NMR [2481], IR [2481], MS [2481].

1-[3-(Bromomethyl)-5-chloro-2-hydroxyphenyl]ethanone

[50317-56-1] $C_9H_8BrClO_2$ mol.wt. 263.52



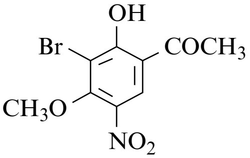
Syntheses

- Preparation by heating 3,3′-diacetyl-5,5′-dichloro-2,2′-di-hydroxydibenzyl ether with an 48% aqueous hydrobromic acid solution during 8 h (61%) [2482].
- Preparation by [Quelet (bromomethylation) reaction] of 5-chloro-2-hydroxyacetophenone [2482] according to [2483].

m.p. 74–76° [2482].

1-(3-Bromo-2-hydroxy-4-methoxy-5-nitrophenyl)ethanone

[134700-74-6] $C_9H_8BrNO_5$ mol.wt. 290.07

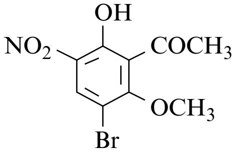


Synthesis

- Refer to: [2484] (compound **1d**).

1-(3-Bromo-6-hydroxy-2-methoxy-5-nitrophenyl)ethanone

[7253-20-5] $C_9H_8BrNO_5$ mol.wt. 290.07



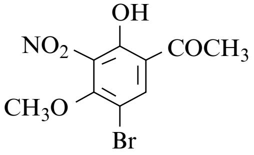
Syntheses

- Preparation by bromination of 2-hydroxy-6-methoxy-3-nitroacetophenone [1807,2485,2486], (44%) [1807].
- Obtained by reaction of nitric acid (d = 1.4) on 2,2′-di-hydroxy-3,3′-diacetyl-4,4′-dimethoxy-5,5′-dibromophenyl thioether, at 0° [2485].

m.p. 160–162° [1807], 156–157° [2485,2486]; IR [1807].

1-(5-Bromo-2-hydroxy-4-methoxy-3-nitrophenyl)ethanone

$C_9H_8BrNO_5$ mol.wt. 290.07



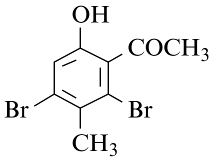
Synthesis

- Preparation by reaction of nitric acid on 5-bromo-2-hydroxy-4-methoxyacetophenone in acetic acid at r.t. [1818].

m.p. 112–114° [1818].

1-(2,4-Dibromo-6-hydroxy-3-methylphenyl)ethanone

[212494-38-7] $C_9H_8Br_2O_2$ mol.wt. 307.97

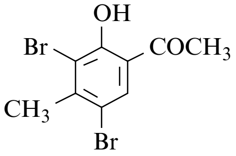


Synthesis

- Refer to: [2487].

1-(3,5-Dibromo-2-hydroxy-4-methylphenyl)ethanone

[145666-17-7] $C_9H_8Br_2O_2$ mol.wt. 307.97



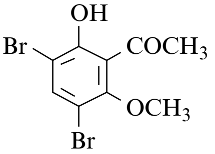
Syntheses

- Preparation by reaction of bromine with 2-hydroxy-4-methylacetophenone in aqueous acetic acid (68%) [2488].
- Also refer to: [1783] (compound **1b**).

m.p. 107–108° [2488]; ^1H NMR [2488], IR [2488].

1-(3,5-Dibromo-2-hydroxy-6-methoxyphenyl)ethanone

[16290-04-3] $C_9H_8Br_2O_3$ mol.wt. 323.97



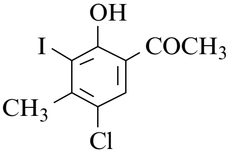
Synthesis

– Preparation by reaction of bromine with 2-hydroxy-6-methoxyacetophenone in acetic acid at 35° (62%) [2489].

m.p. 90–90°5 [2489]; ^{1}H NMR [2489].

1-(5-Chloro-2-hydroxy-3-iodo-4-methylphenyl)ethanone

[292144-86-6] $C_9H_8ClIO_2$ mol.wt. 310.52



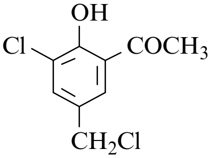
Synthesis

– Preparation by iodination of 5-chloro-2-hydroxy-4-methyl-acetophenone with iodine (1 mol) and iodic acid (1 mol) in ethanol at 35–40° for 1.5 h (75–85%) [1802].

m.p. 76° [1802].

1-[3-Chloro-5-(chloromethyl)-2-hydroxyphenyl]ethanone

[66883-87-2] $C_9H_8Cl_2O_2$ mol.wt. 219.07



Syntheses

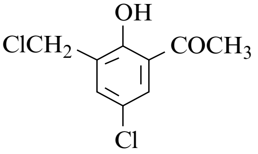
– Preparation from 3-chloro-2-hydroxyacetophenone in acetic acid solution by introduction of the chloromethyl group into aromatic ring by treatment with formaldehyde and hydrogen chloride in the presence of zinc chloride (44%) [2490] (Blanc [2490] reaction).

– Also refer to: [2491,2492].

m.p. 145° [2490].

1-[5-Chloro-3-(chloromethyl)-2-hydroxyphenyl]ethanone

[34987-36-5] $C_9H_8Cl_2O_2$ mol.wt. 219.07



Syntheses

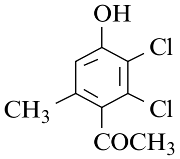
– Preparation from 5-chloro-2-hydroxyacetophenone in ethylene dichloride solution by introduction of the chloromethyl group into aromatic ring by treatment with 37% formalin and hydrogen chloride in the presence of zinc chloride at 60–65° (76–68%) [2009,2493] [Blanc (Chloromethylation) reaction].

- Preparation by reaction of concentrated hydrochloric acid with 5-chloro-2-hydroxy-3-hydroxy-methylacetophenone for 5 days at 20° (95%) [2482].
- Also refer to: [2491,2492,2494].

m.p. 68°5–69° [2009], 67–68° [2482];
b.p.$_2$ 130–135° [2009], b.p.$_3$ 140–142° [2482]; IR [2493].

1-(2,3-Dichloro-4-hydroxy-6-methylphenyl)ethanone

[21472-87-7] $C_9H_8Cl_2O_2$ mol.wt. 219.07



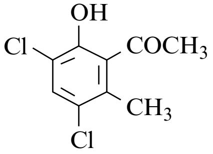
Synthesis

- Obtained as a trace during the rearrangement of 1,2,3,3-tetrachlorocyclopropane-cis-1,2-diacetone by heating with a saturated solution of sodium bisulfate in acetic acid at 100° (<1%) [2495].

m.p. 107–108° [2495]; ^{1}H NMR [2495], IR [2495].

1-(3,5-Dichloro-2-hydroxy-6-methylphenyl)ethanone

$C_9H_8Cl_2O_2$ mol.wt. 219.07



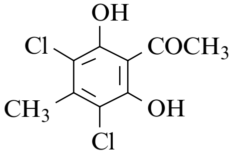
Syntheses

- Obtained by Fries rearrangement of 2,4-dichloro-5-methyl-phenyl acetate with aluminium chloride at 135° (21%) [2496].
- Obtained by reaction of pyridinium chloride on 3,5-di-chloro-2-methoxy-6-methylacetophenone at 210° (21%) [2496].
- Also obtained by reaction of levulinic acid on 3,5-dichloro-2-hydroxy-6-methylacetophenone hydrazone in 1 N hydrochloric acid using a steam bath (15%) [2496].
- Also obtained by basic hydrolysis of 6,8-dichloro-2,5-dimethylchromone with 5% aqueous sodium hydroxide in refluxing methanol (4%) [2496].

m.p. 104–105°5 [2496].

1-(3,5-Dichloro-2,6-dihydroxy-4-methylphenyl)ethanone

[3361-23-7] $C_9H_8Cl_2O_3$ mol.wt. 235.07



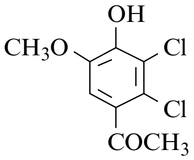
Synthesis

- Preparation by reaction of sulfuryl chloride with 2,6-di-hydroxy-4-methylacetophenone in ethyl ether (71%) [2497].

m.p. 164–165° [2497].

1-(2,3-Dichloro-4-hydroxy-5-methoxyphenyl)ethanone

[154638-85-4] $C_9H_8Cl_2O_3$ mol.wt. 235.07



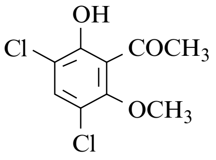
Synthesis

- Obtained by chlorination of acetoguaiacone in dioxane–water mixture at 40° (29%) [1789].

MS [1789].

1-(3,5-Dichloro-2-hydroxy-6-methoxyphenyl)ethanone

[87953-94-4] $C_9H_8Cl_2O_3$ mol.wt. 235.07



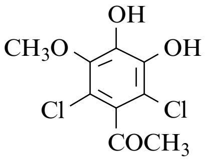
Syntheses

- Preparation by reaction of chlorine on 2-hydroxy-6-methoxyacetophenone in chloroform at r.t. (50%) [1884].
- Preparation by reaction of sulfuryl chloride on 2-hydroxy-6-methoxyacetophenone in refluxing acetic acid [2021].
- Also obtained by reaction of dimethyl sulfate on 3,5-dichloro-2,6-dihydroxyacetophenone with potassium carbonate in refluxing benzene (very low yield) [2021].

m.p. 99° [1884], 97–98° [2021].

1-(2,6-Dichloro-3,4-dihydroxy-5-methoxyphenyl)ethanone

[75322-34-8] $C_9H_8Cl_2O_4$ mol.wt. 251.07

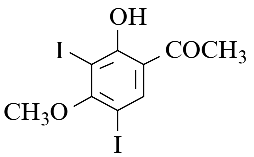


Synthesis

- Preparation by chlorination of acetosyringone (main product) [2498].

Visible light absorption spectra [2498].

1-(2-Hydroxy-3,5-diiodo-4-methoxyphenyl)ethanone



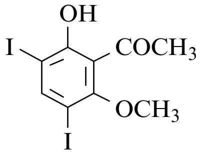
$C_9H_8I_2O_3$ mol.wt. 417.97

Syntheses

- Obtained by reaction of an aqueous iodine–iodic acid solution on paeonol in ethanol at r.t. (8%) [1924].
- Also obtained by hydrolysis of 6,8-diiodo-7-methoxy-2-methylchromone by boiling 10% aqueous sodium hydroxide solution [1924].

m.p. 98–99° [1924].

1-(2-Hydroxy-3,5-diiodo-6-methoxyphenyl)ethanone



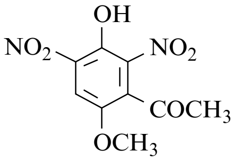
$C_9H_8I_2O_3$ mol.wt. 417.97

Syntheses

- Preparation by reaction of an aqueous iodine–iodic acid solution on 2-hydroxy-6-methoxyacetophenone in ethanol at r.t. (76%) [1924].
- Also obtained by hydrolysis of 6,8-diiodo-5-methoxy-2-methylchromone by boiling 10% aqueous sodium hydroxide solution [1924].

m.p. 111° [1924].

1-(3-Hydroxy-6-methoxy-2,4-dinitrophenyl)ethanone



$C_9H_8N_2O_7$ mol.wt. 256.17

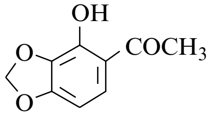
Synthesis

- Preparation by reaction of dimethyl sulfate on 3,6-di-hydroxy-2,4-dinitroacetophenone with sodium hydroxide in water at 60–65° (53%) [1941].

m.p. 94–96° [1941]; IR [1941], UV [1941].

1-(4-Hydroxy-1,3-benzodioxol-5-yl)ethanone

[23780-60-1] $C_9H_8O_4$ mol.wt. 180.16



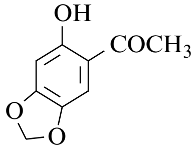
Synthesis

- Obtained (poor yield) by reaction of methylene iodide with gallacetophenone in the presence of potassium carbonate in refluxing acetone for 48 h (9%) [2499,2500].

m.p. 85–86° [2499,2500]; TLC [2500];
column chromatography [2499];
^{1}H NMR [2499,2500], IR [2499,2500], UV [2499,2500].

1-(6-Hydroxy-1,3-benzodioxol-5-yl)ethanone

[66003-50-7] $C_9H_8O_4$ mol.wt. 180.16



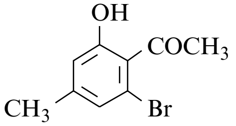
Syntheses

- Obtained by refluxing methylene iodide and 2,4,5-trihydroxyacetophenone in acetone in the presence of potassium carbonate (56%) [2501], (51%) [2420].
- Also obtained by Fries rearrangement of 5-acetoxy-1,3-benzodioxole with aluminium chloride in nitrobenzene at −10 to 7°, then 3 days at r.t. (5%) [2502].

- Also obtained by Friedel–Crafts acylation of 5-hydroxy-1,3-benzodioxole *(Sesamol)* with acetic anhydride in the presence of boron trifluoride etherate for 1 h at 80–90° (75%) [2503].
- Also obtained by reaction of acetonitrile with 5-hydroxy-1,3-benzodioxole (Hoesch reaction) [2501], (35%) [2504], (31%) [2505].
- Also obtained by reaction of acetyl chloride with sesamol methyl ether (b.p.$_{18}$ 110–114°) in the presence of aluminium chloride in ethyl ether at r.t. overnight (36%) [2504].
- Also obtained (poor yield) by reaction of acetic acid with sesamol in the presence of boron trifluoride for 3 h, then heating at 80–90° for 1.5 h (5%) [2504].
- Also refer to: [2504,2505].

m.p. 114° [2502], 113–114° [2504], 112° [2501], 111–112° [2503,2505]; ^{1}H NMR [2501,2505], IR [2501,2504,2505], UV [2505], MS [2505].

1-(2-Bromo-6-hydroxy-4-methylphenyl)ethanone



$C_9H_9BrO_2$ mol.wt. 229.07

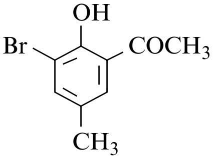
Synthesis

- Obtained by reaction of acetyl chloride with 3-bromo-5-methylanisole in the presence of aluminium chloride in refluxing carbon disulfide [2506].

m.p. 50–52° [2506].

1-(3-Bromo-2-hydroxy-5-methylphenyl)ethanone

[56609-15-5] $C_9H_9BrO_2$ mol.wt. 229.07



Syntheses

- Preparation by Fries rearrangement of 2-bromo-4-methyl-phenyl acetate,
  - with aluminium chloride without solvent at 110° or in refluxing nitrobenzene (90%) [2507];
  - with zinc chloride or boron trifluoride in nitrobenzene at 100° (80–85%) [2507];
  - with ferric chloride or stannic chloride in nitrobenzene at 100° (55–70%) [2507].
- Preparation by reaction of bromine on 2-hydroxy-5-methylacetophenone in acetic acid at r.t. (quantitative yield) [2508], (65–70%) [1792,1815,2509,2510].
- Also refer to: [1783,1795,2511,2512].
- Preparation by reaction of N-bromosuccinimide with 2-hydroxy-5-methylacetophenone in DMF at r.t. (93–96%) [2513,2514].

N.B.: It is mentioned in the patent [2515] (page 26) that this hydroxyketone, the 1-(3-bromo-2-hydroxy-5-methylphenyl)ethanone (**I**) has been prepared by Fries rearrangement ($AlCl_3$/165°) of 4-bromo-3-methylphenyl acetate, itself obtained from the 4-bromo-3-methylphenol. This is impossible. The Fries rearrangement of the above ester always leads to 1-(5-bromo-2-hydroxy-4-methylphenyl)ethanone (**II**). There are two possibilities:

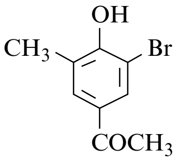
- Either the ester used for Fries rearrangement was the 2-bromo-4-methylphenyl acetate and provides (**I**).
- Or the obtained hydroxyketone should be (**II**). This appears confirmed later on by obtaining 6-bromo-3,4-dihydro-2,2,7-trimethyl-2*H*-1-benzopyran-4-one with this hydroxyketone [2515].

m.p. 95° [2509], 94–95° [1792], 89° [1815], 88°5–89°5 [2508], 88–89° [2513,2514].

b.p.$_2$ 126–127° [2507]; pK$_a$ [2516]; ^{1}H NMR [2513], MS [2513].

1-(3-Bromo-4-hydroxy-5-methylphenyl)ethanone

[127923-55-1] $C_9H_9BrO_2$ mol.wt. 229.07



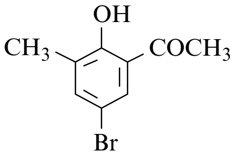
Synthesis

– Preparation by reaction of bromine with 4-hydroxy-3-methyl-acetophenone in aqueous acetic acid, first at 5°, then at r.t. (89%) [1825].

m.p. 145–146° [1825].

1-(5-Bromo-2-hydroxy-3-methylphenyl)ethanone

[72422-80-1] $C_9H_9BrO_2$ mol.wt. 229.07



Syntheses

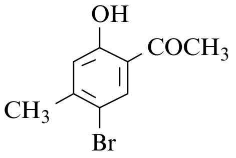
– Preparation by reaction of bromine with 2-hydroxy-3-methylacetophenone in acetic acid at r.t. (80%) [2517] (66%) [1792].

– Also refer to: [1795] (compound **1e**) and [1783] (compound **1a**).

m.p. 78–79° [2517], 77–78° [1792]; ^{1}H NMR [2517], IR [2517].

1-(5-Bromo-2-hydroxy-4-methylphenyl)ethanone

[50342-17-1] $C_9H_9BrO_2$ mol.wt. 229.07



Syntheses

– Preparation by reaction of bromine on 2-hydroxy-4-methyl-acetophenone in chloroform at <–10° (74%) [2518] or in acetic acid at r.t. (38%) [1792].

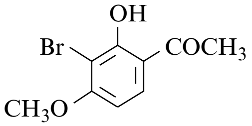
– Preparation by Fries rearrangement of 4-bromo-3-methyl-phenyl acetate with aluminium chloride without solvent at 165–180° [2515,2518], (80%) [2518].

– Also refer to: [1783].

m.p. 86° [1792], 81–82° [2515], 80° [2518]; ^{1}H NMR [2518].

1-(3-Bromo-2-hydroxy-4-methoxyphenyl)ethanone

[39503-62-3] $C_9H_9BrO_3$ mol.wt. 245.07



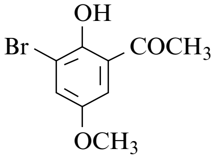
Syntheses

- Preparation by reaction of bromine on 2-hydroxy-4-methoxyacetophenone with titanium tetrachloride in methylene chloride at r.t. (65%) [2519].
- Also obtained (by-product) by reaction of cupric bromide with 2-hydroxy-4-methoxyacetophenone in refluxing dioxane (3%) [1978].

m.p. 130–131° [2519], 120–122° [1978]; ^{1}H NMR [1978,2519].

1-(3-Bromo-2-hydroxy-5-methoxyphenyl)ethanone

[37113-61-4] $C_9H_9BrO_3$ mol.wt. 245.07



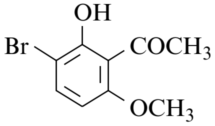
Syntheses

- Preparation by reaction of bromine on 2-hydroxy-5-methoxyacetophenone with aluminium chloride,
  - in chloroform at 10° (80%) [2520];
  - in carbon disulfide at r.t. (65%) [2521].

m.p. 78–79° [2520], 76–76°5 [2521]; ^{1}H NMR [2520], IR [2520].

1-(3-Bromo-2-hydroxy-6-methoxyphenyl)ethanone

[37113-62-5] $C_9H_9BrO_3$ mol.wt. 245.07



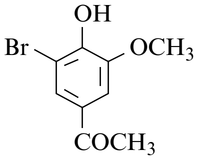
Syntheses

- Preparation by diazotization of 3-amino-2-hydroxy-6-methoxyacetophenone and replacement of the diazonium group by bromine (Sandmeyer reaction) (28%) [2522], (19%) [2523].
- Preparation by bromination of 2-hydroxy-6-methoxyacetophenone, with bromine [2522], in methylene chloride (76%) [2524], in chloroform [2520,2523,2525], (68%) [2523] or in acetic anhydride (44%) [2526] or with N-bromosuccinimide (41%) [2522].

m.p. 102–103° [2525], 101–102° [2526], 101° [2524], 100–101° [2520,2522], 99–100° [2523]; ^{1}H NMR [2520,2525,2526], IR [2520].

1-(3-Bromo-4-hydroxy-5-methoxyphenyl)ethanone

[103653-14-1] $C_9H_9BrO_3$ mol.wt. 245.07



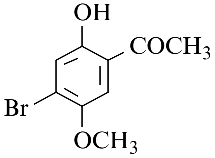
Syntheses

- Preparation by reaction of bromine with acetovanillone in aqueous acetic acid at 0°, then oil r.t. (54%) [2527].
- Preparation by adding bromine to a solution of acetovanillone, sodium acetate and potassium bromide in aqueous methanol cooled to −60° [2528].

m.p. 156–157° [2527], 155–157° [2528].

1-(4-Bromo-2-hydroxy-5-methoxyphenyl)ethanone

[90971-91-8] $C_9H_9BrO_3$ mol.wt. 245.07



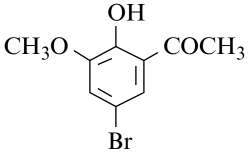
Synthesis

- Preparation by reaction of methyl iodide on 4-bromo-2,5-dihydroxyacetophenone with potassium carbonate in refluxing methyl ethyl ketone [1987].

m.p. 115° [1987].

1-(5-Bromo-2-hydroxy-3-methoxyphenyl)ethanone

$C_9H_9BrO_3$ mol.wt. 245.07



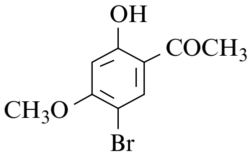
Syntheses

- Preparation by reaction of bromine on 2-hydroxy-3-methoxyacetophenone in the presence of sodium bromide in 1:1 aqueous ethanol at 25° (48%) [2215].
- Preparation by reaction of hydrobromic acid on 5-bromo-2,3-dimethoxyacetophenone in acetic acid (36%) [2215].

m.p. 108–109° [2215]; UV [2215].

1-(5-Bromo-2-hydroxy-4-methoxyphenyl)ethanone

[39503-61-2] $C_9H_9BrO_3$ mol.wt. 245.07

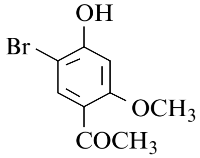


Syntheses

- Preparation by reaction of bromine on paeonol (2-hydroxy-4-methoxyacetophenone),
  - in methylene chloride at r.t. (quantitative yield) [2519];
  - in acetic acid at r.t. [1818,1836,2293,2529,2530], (60%) [1836];
  - in acetic anhydride (48%) [2526].
- Preparation by diazotization of 5-amino-2-hydroxy-4-methoxyacetophenone and replacement of the diazonium group by bromine (Sandmeyer reaction) [1836].
- Also obtained by reaction of dimethyl sulfate on 5-bromoresacetophenone in sodium hydroxide [1836].
- Also obtained as one of the products of bromination of paeonol acetate in carbon disulfide [2531].
- Also obtained as one of the products of reaction of cupric bromide on paeonol in refluxing dioxane (17%) [1978].
- Also obtained (by-product) by reaction of acetic anhydride on 4-bromoresorcinol dimethyl ether with aluminium chloride in refluxing carbon disulfide [2532].
- Also obtained (by-product) by reaction of N-bromosuccinimide on 2,4-dimethoxyacetophenone with benzoyl peroxide in refluxing carbon tetrachloride [2532].
- Also refer to: [2533].

m.p. 172–174° [2530], 172–172°5 [2519], 172° [1836], 171–172° [2532], 171° [2531], 170–172° [2526], 169–170° [1978], 169° [1818], 168–170° [2293]; ^{1}H NMR [1978,2519,2526].

1-(5-Bromo-4-hydroxy-2-methoxyphenyl)ethanone



$C_9H_9BrO_3$ mol.wt. 245.07

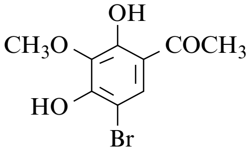
Synthesis

- Preparation by bromination of isopaeonol (4-hydroxy-2-methoxyacetophenone) in chloroform [1836].

m.p. 198° [1836].

1-(5-Bromo-2,4-dihydroxy-3-methoxyphenyl)ethanone

[62615-25-2]



$C_9H_9BrO_4$ mol.wt. 261.07

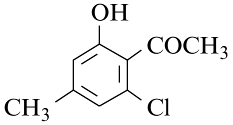
Synthesis

- Preparation by bromination of 2,4-dihydroxy-3-methoxy-acetophenone with bromine in 80% acetic acid at r.t. (64%) [1989].

m.p. 110°5–112°5 [1989]; ^{1}H NMR [1989].

1-(2-Chloro-6-hydroxy-4-methylphenyl)ethanone

[24490-25-3]



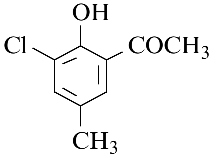
$C_9H_9ClO_2$ mol.wt. 184.62

Synthesis not yet described

- Refer to: [2534]; this sole reference mentioned for this compound (Chem. Abstr., **71**, 86368b (1969)) is erroneous. Actually, the ketone described as being the 2′-chloro-6′-hydroxy-4′-methylacetophenone is in fact the 5′-chloro-2′-hydroxy-4′-methylacetophenone in original publication.

1-(3-Chloro-2-hydroxy-5-methylphenyl)ethanone

[7507-88-2]



$C_9H_9ClO_2$ mol.wt. 184.62

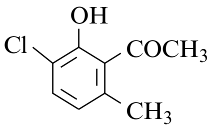
Syntheses

- Preparation by Fries rearrangement of 2-chloro-4-methyl-phenyl acetate with aluminium chloride without solvent at 120° (quantitative yield) [2151].
- Obtained by reaction of aluminium chloride on a mixture of 2-chloro-4-methylphenyl acetate and 4-methylphenyl benzoate without solvent at 150° (33–37%) [2535].
- Obtained by reaction of aluminium chloride on a mixture of 2,4,6-trimethylphenyl acetate or 4-methylphenyl acetate and 2-chloro-4-methylphenyl benzoate without solvent at 150° (67% yield and small amounts, respectively) [2535].

m.p. 91° [2151,2535]; pK_a [2516].

1-(3-Chloro-2-hydroxy-6-methylphenyl)ethanone

[85128-50-3] $C_9H_9ClO_2$ mol.wt. 184.62

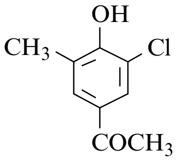


Synthesis

- Refer to: [1881,1885] (ketone **13**).

1-(3-Chloro-4-hydroxy-5-methylphenyl)ethanone

[54556-95-5] $C_9H_9ClO_2$ mol.wt. 184.62



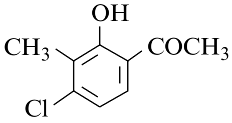
Syntheses

- Preparation by Fries rearrangement of 2-chloro-6-methylphenyl acetate in the presence of aluminium chloride without solvent at 140° (74%) [2536].
- Preparation by chlorination of 4-hydroxy-3-methylacetophenone [2536].
- Also refer to: [2537].

m.p. 123°5–124° [2536]; IR [2536].

1-(4-Chloro-2-hydroxy-3-methylphenyl)ethanone

[198344-86-4] $C_9H_9ClO_2$ mol.wt. 184.62

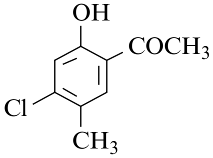


Synthesis

- Refer to: [1873].

1-(4-Chloro-2-hydroxy-5-methylphenyl)ethanone

[57051-51-1] $C_9H_9ClO_2$ mol.wt. 184.62

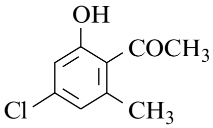


Synthesis

- Preparation by Fries rearrangement of 3-chloro-4-methyl-phenyl acetate with aluminium chloride without solvent at 125° [1814].

1-(4-Chloro-2-hydroxy-6-methylphenyl)ethanone

[87239-37-0] $C_9H_9ClO_2$ mol.wt. 184.62

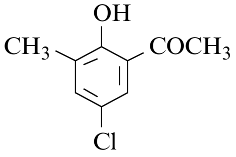


Synthesis

- Refer to: [2538].

1-(5-Chloro-2-hydroxy-3-methylphenyl)ethanone

[50343-12-9] $C_9H_9ClO_2$ mol.wt. 184.62



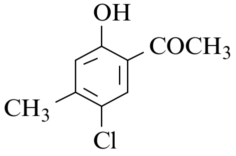
Syntheses

- Preparation by Fries rearrangement of 4-chloro-2-methyl-phenyl acetate with aluminium chloride without solvent at 120° (97%) [2482], (75%) [2539].
- Preparation by adding zinc dust to an acetic acid solution of 5-chloro-3-(chloromethyl)-2-hydroxyacetophenone at 100° (82%) [2482].
- Also obtained (by-product) by chloromethylation of 5-chloro-2-hydroxyacetophenone (5%) [2493].
- Also obtained (poor yield) by treatment of 5-chloro-3-(chloromethyl)-2-hydroxyacetophenone with zinc chloride in methylene chloride in the presence of water for 45 h at 60° (5%) [2493].

m.p. 70° [2539], 65–66° [2482], 63–64° [2493]; b.p.$_{16}$ 141° [2482].

1-(5-Chloro-2-hydroxy-4-methylphenyl)ethanone

[28480-70-8] $C_9H_9ClO_2$ mol.wt. 184.62



Syntheses

- Preparation by reaction of acetic acid on 4-chloro-3-methylphenol with boron trifluoride at 70–100° (80–85%) [1969].
- Preparation by Fries rearrangement of 4-chloro-3-methyl-phenyl acetate with aluminium chloride without solvent between 110° and 155° [1867,1871,2011,2014,2151,2540,2541], (quantitative yield) [2014,2541], (64%) [1867,1871].
- Also obtained by reaction of acetyl chloride on 4-chlorothymol methyl ether with aluminium chloride in carbon disulfide at r.t. (8%) [2542].
- Also refer to: [2540] (compound CHMA).

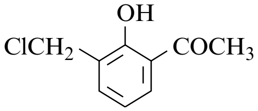
m.p 75°5 [2541], 71–72° [2151], 71° [2011], 70°5 [1969], 70° [2542], 69–70° [2014];

b.p.$_{15}$ 137° [2151], b.p.$_{21}$ 140–142° [1867,1871];

^{1}H NMR [1820], UV [1820], fluorescence spectra [1820,2534].

1-[3-(Chloromethyl)-2-hydroxyphenyl]ethanone

[87165-49-9] $C_9H_9ClO_2$ mol.wt. 184.62



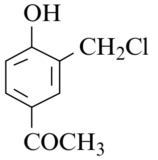
Synthesis

- Preparation by reaction of polyoxymethylene with 2-hydroxyacetophenone in the presence of concentrated hydrochloric acid at 50–60° [2494].

m.p. 45° [2494]; ^{1}H NMR [2494], IR [2494].

1-[3-(Chloromethyl)-4-hydroxyphenyl]ethanone

[24085-05-0] $C_9H_9ClO_2$ mol.wt. 184.62



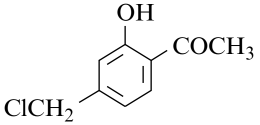
Syntheses

- Preparation by introduction of the chloromethyl group into 4-hydroxyacetophenone by treatment with formaldehyde and hydrochloric acid [2543–2546], in the presence of zinc chloride [2547Blanc (Chloromethylation) reaction], (85–92%) [2543,2545,2546].
- Also refer to: [2548,2549].

m.p. 160° (d) [2543,2546].

1-[4-(Chloromethyl)-2-hydroxyphenyl]ethanone

[107223-42-7] $C_9H_9ClO_2$ mol.wt. 184.62



Synthesis

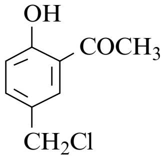
- Preparation by reaction of ethyl chloroformate with 4-(dimethylaminomethyl)-2-hydroxyacetophenone in toluene (22%) [2550].

m.p. 34–37° [2550].

1-[5-(Chloromethyl)-2-hydroxyphenyl]ethanone

[30787-43-0] $C_9H_9ClO_2$ mol.wt. 184.62

Syntheses

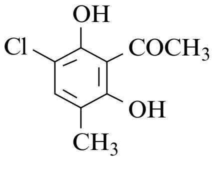


- Preparation by reaction of formaldehyde solution with hydrochloric acid on 2-hydroxyacetophenone at 25–30° (57–61%) [2551].
- Preparation by reaction of polyoxymethylene with 2-hydroxyacetophenone in the presence of hydrochloric acid at 50–60° [2494].

m.p. 94–95° [2551].

1-(3-Chloro-2,6-dihydroxy-5-methylphenyl)ethanone

[56581-46-5] $C_9H_9ClO_3$ mol.wt. 200.62



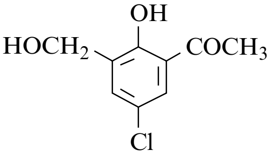
Synthesis

- Preparation by reaction of chlorine on 2,6-dihydroxy-3-methylacetophenone in acetic acid at r.t. [2552].

m.p. 152° [2552];
^{1}H NMR [2552], IR [2552], MS [2552].

1-[5-Chloro-2-hydroxy-3-(hydroxymethyl)phenyl]ethanone

[50317-52-7] $C_9H_9ClO_3$ mol.wt. 200.62



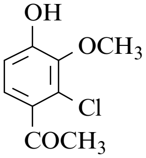
Syntheses

- Preparation by boiling an aqueous solution of 5-chloro-3-(chloromethyl)-2-hydroxyacetophenone for 6 h (73%) [2482].
- Also obtained as a labile intermediate product during the chloromethylation of 5-chloro-2-hydroxyacetophenone (<5%) [2493].

m.p. 82°5–84° [2482]; b.p.$_1$ 155° [2482].

1-(2-Chloro-4-hydroxy-3-methoxyphenyl)ethanone

[151340-06-6] $C_9H_9ClO_3$ mol.wt. 200.62



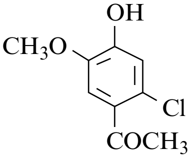
Synthesis

- Preparation from the acetate of 2-chlorovanillin by reaction with diazomethane and subsequent hydrolysis [2553,2554]

m.p. 97–98° [2553,2554];
^{1}H NMR [2553], ^{13}C NMR [2553], MS [2553].

1-(2-Chloro-4-hydroxy-5-methoxyphenyl)ethanone

[69240-98-8] $C_9H_9ClO_3$ mol.wt. 200.62



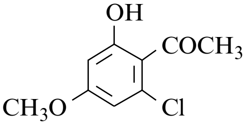
Syntheses

- Obtained from the acetate of 6-chlorovanillin by reaction with diazomethane and subsequent hydrolysis [2553,2554], (11–17%) [2553,2555].
- Also refer to: [1991] (compound **XVIIe**).

m.p. 109–110° [2555], 107–108° [2553];
^{1}H NMR [2553], ^{13}C NMR [2553], MS [2553].

1-(2-Chloro-6-hydroxy-4-methoxyphenyl)ethanone

[112954-19-5] $C_9H_9ClO_3$ mol.wt. 200.62



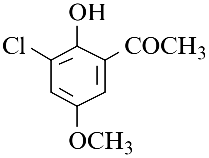
Syntheses

- Preparation by reaction of acetyl chloride with 3,5-di-methoxy-1-chlorobenzene in the presence of aluminium chloride (Friedel–Crafts reaction) (38%) [2556].
- Also refer to: [2557,2558].

^{1}H NMR [2556].

1-(3-Chloro-2-hydroxy-5-methoxyphenyl)ethanone

[286931-53-1] $C_9H_9ClO_3$ mol.wt. 200.62



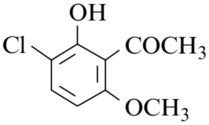
Synthesis

- Preparation by chlorination of 2-hydroxy-5-methoxyacetophenone with N-chlorosuccinimide in acetic acid containing magnesium acetate at r.t. for 24 h under nitrogen atmosphere (80%) [2559,2560].

m.p. 78–79° [2560]; ^{1}H NMR [2560], MS [2560].

1-(3-Chloro-2-hydroxy-6-methoxyphenyl)ethanone

[87953-91-1] $C_9H_9ClO_3$ mol.wt. 200.62



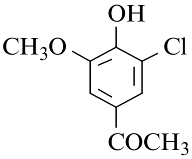
Syntheses

- Preparation by reaction of dimethyl sulfate on 3-chloro-2,6-dihydroxyacetophenone with potassium carbonate in refluxing benzene (38%) [2021].
- Preparation by diazotization of 3-amino-2-hydroxy-6-methoxyacetophenone with sodium nitrite in dilute sulfuric acid at 0°, and replacement of the diazonium group by chlorine with a solution of cuprous chloride in 2 N hydrochloric acid at 100° (Sandmeyer reaction) (37%) [1884].
- Also obtained (by-product) by reaction of sulfuryl chloride on 2-hydroxy-6-methoxy-acetophenone in refluxing acetic acid [2021].
- Also obtained (crude product) by a one-pot acylation-deprotection of 4-methoxy-2-methoxy-methoxychlorobenzene (about 93%). No physical data available [2561].

m.p. 88°5–89° [2021], 82° [1884].

1-(3-Chloro-4-hydroxy-5-methoxyphenyl)ethanone

[116296-35-6] $C_9H_9ClO_3$ mol.wt. 200.62



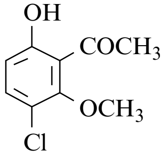
Syntheses

- Preparation from the acetate of 5-chlorovanillin by reaction with diazomethane and subsequent hydrolysis [2553–2555], (35%) [2553], (23%) [2555].
- Also obtained by chlorination of acetoguaiacone in dioxane–water mixture at 40° (21%) [1789].
- Preparation by adding sodium hypochlorite solution to a solution of acetovanillone and sodium acetate in methanol cooled to −60° [2528].
- Also obtained (by-product) by chlorination of apocynol in dioxane–ethyl ether mixture at 40° (3%) [1789].

m.p. 124–126° [2528], 124–125° [2555], 123–124° [2553]; MS [1789,2553].

1-(3-Chloro-6-hydroxy-2-methoxyphenyl)ethanone

[87953-86-4] $C_9H_9ClO_3$ mol.wt. 200.62



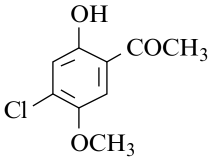
Syntheses

- Preparation by reaction of sulfuryl chloride on 2-hydroxy-6-methoxyacetophenone in refluxing ethyl ether (93%) or in refluxing acetic acid (66%) [2021].
- Preparation by reaction of chlorine on 2-hydroxy-6-methoxyacetophenone in carbon tetrachloride at −20° (62%) [1884].

m.p. 31° [2021], 30–35° [1884]; b.p.$_{0.001}$ 40° [2021], b.p.$_2$ 120° [1884].

1-(4-Chloro-2-hydroxy-5-methoxyphenyl)ethanone

$C_9H_9ClO_3$ mol.wt. 200.62



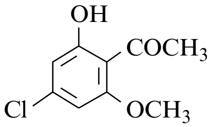
Synthesis

- Preparation by reaction of methyl iodide on 4-chloro-2,5-dihydroxyacetophenone with potassium carbonate in refluxing acetone [1987].

m.p. 130° [1987].

1-(4-Chloro-2-hydroxy-6-methoxyphenyl)ethanone

[140155-06-2] $C_9H_9ClO_3$ mol.wt. 200.62



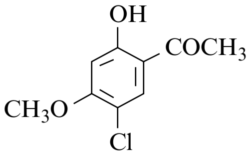
Syntheses

- Preparation by reaction of acetyl chloride with 3,5-dimethoxy-1-chlorobenzene in the presence of aluminium chloride (Friedel–Crafts reaction) (38%) [2556].
- Also refer to: [2557,2558].

^{1}H NMR [2556].

1-(5-Chloro-2-hydroxy-4-methoxyphenyl)ethanone

[116265-99-7] $C_9H_9ClO_3$ mol.wt. 200.62



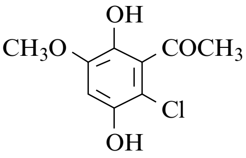
Syntheses

- Preparation by diazotization of 5-amino-2-hydroxy-4-methoxyacetophenone and replacement of diazonium group by chlorine (Sandmeyer reaction) [2530].
- Preparation by reaction of acetyl chloride with 4-chloro-resorcinol dimethyl ether in the presence of aluminium chloride in ethylene dichloride (79%) [2562].
- Also refer to: [2563].

m.p. 154–155° [2562], 153–155° [2530].

1-(2-Chloro-3,6-dihydroxy-5-methoxyphenyl)ethanone

[34603-08-2] $C_9H_9ClO_4$ mol.wt. 216.62



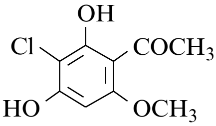
Synthesis

- Preparation by metallation of 2-chloro-5-methoxy-hydro-quinone-bis-[tetrahydropyranyl-(2)-ether], followed by treatment of the intermediate aryllithium compound with acetic anhydride in tetrahydrofuran at r.t. (56%) [2564].

m.p. 100° [2564].

1-(3-Chloro-2,4-dihydroxy-6-methoxyphenyl)ethanone

[200878-65-5] $C_9H_9ClO_4$ mol.wt. 216.62

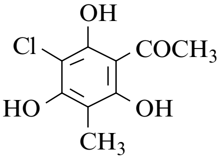


Synthesis

- Refer to: [2565].

1-(3-Chloro-2,4,6-trihydroxy-5-methylphenyl)ethanone

[23053-47-6] $C_9H_9ClO_4$ mol.wt. 216.62



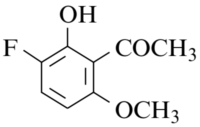
Synthesis

- Preparation by reaction of sulfuryl chloride with methyl-phloroacetophenone in ethyl ether, first at 0°, then at r.t. (58%) [2566].

m.p. 223–224° [2566].

1-(3-Fluoro-2-hydroxy-6-methoxyphenyl)ethanone

[117902-13-3] $C_9H_9FO_3$ mol.wt. 184.17



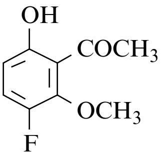
Synthesis

- Preparation by demethylation of 2,6-dimethoxy-3-fluoro-acetophenone with boron tribromide (62%) [2054].

m.p. 87–88° [2054]; ^{1}H NMR [2054].

1-(3-Fluoro-6-hydroxy-2-methoxyphenyl)ethanone

[117902-12-2] $C_9H_9FO_3$ mol.wt. 184.17



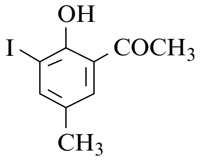
Synthesis

- Obtained (by-product) by demethylation of 2,6-dimethoxy-3-fluoroacetophenone with boron tribromide (6%) [2054].

^{1}H NMR [2054].

1-(2-Hydroxy-3-iodo-5-methylphenyl)ethanone

[175655-10-4] $C_9H_9IO_2$ mol.wt. 276.07



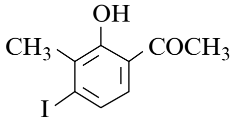
Syntheses

- Preparation by iodination of 2-hydroxy-5-methyl-acetophenone with iodine (1 mol) and iodic acid (1 mol) in ethanol at 35–40° for 1.5 h (75–85%) [1802].
- Also refer to: [2567,2568].

m.p. 80° [1802]; ^{1}H NMR [1802].

1-(2-Hydroxy-4-iodo-3-methylphenyl)ethanone

[40591-02-4] $C_9H_9IO_2$ mol.wt. 276.07



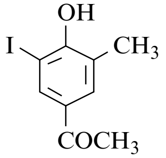
Synthesis

- Obtained (by-product) by Fries rearrangement of 3-iodo-4-methylphenyl acetate with aluminium chloride without solvent at 120° [1830].

m.p. 80° [1830]; ^{1}H NMR [1830].

1-(4-Hydroxy-3-iodo-5-methylphenyl)ethanone

[292144-89-9] $C_9H_9IO_2$ mol.wt. 276.07



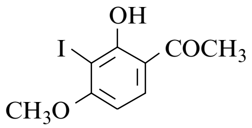
Synthesis

- Preparation by iodination of 4-hydroxy-3-methyl-acetophenone with iodine (1 mol) and iodic acid (1 mol) in ethanol at 35–40° for 1.5 h (75–85%) [1802].

m.p. 149° [1802]; ^{1}H NMR [1802].

1-(2-Hydroxy-3-iodo-4-methoxyphenyl)ethanone

$C_9H_9IO_3$ mol.wt. 292.07



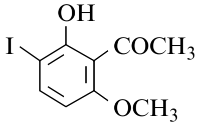
Syntheses

- Obtained from 8-iodo-7-methoxy-2-methylchromone by boiling 10% aqueous sodium hydroxide solution [1924].
- Preparation by adding an aqueous solution of iodine and iodic acid to an ethanolic solution of 2-hydroxy-4-methoxy-acetophenone (15%) [1924].

m.p. 152° [1924].

1-(2-Hydroxy-3-iodo-6-methoxyphenyl)ethanone

[103440-57-9] $C_9H_9IO_3$ mol.wt. 292.07



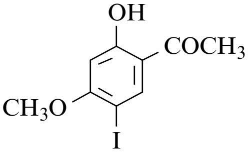
Syntheses

- Preparation by reaction of iodine monochloride with 2-hydroxy-6-methoxyacetophenone in acetic acid at 17° (88%) [2524].

- Also obtained from 8-iodo-5-methoxy-2-methylchromone by boiling 10% aqueous sodium hydroxide solution [1924].
- Preparation by adding an aqueous solution of iodic acid and iodine to an ethanolic solution of 2-hydroxy-6-methoxyacetophenone (85%) [2569].

m.p. 115° [2524,2569], 57° [1924]; ^{13}C NMR [2524], IR [2524].

1-(2-Hydroxy-5-iodo-4-methoxyphenyl)ethanone



$C_9H_9IO_3$ mol.wt. 292.07

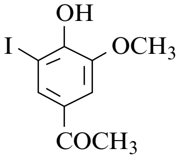
Syntheses

- Obtained by reaction of an aqueous iodine–potassium iodide solution on paeonol in 22% aqueous ammonia; then, the mixture was poured into excess of dilute ice-cold sulfuric acid (13%) [1924].
- Also obtained by hydrolysis of 6-iodo-7-methoxy-2-methyl-chromone by boiling 10% aqueous sodium hydroxide solution [1924].

m.p. 161° [1924].

1-(4-Hydroxy-3-iodo-5-methoxyphenyl)ethanone

[103440-59-1] $C_9H_9IO_3$ mol.wt. 292.07



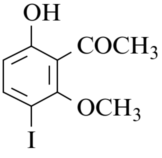
Syntheses

- Preparation by reaction of iodine with acetovanillone in 0.8 N aqueous sodium hydroxide (94%) [2527].
- Preparation by adding an aqueous solution of iodine and potassium iodide to and aqueous solution of acetovanillone and sodium bicarbonate at 80° (82%) [2570].
- Preparation by adding potassium iodide and iodine to an aqueous solution of acetovanillone and sodium acetate at 90° [2528].
- Preparation by adding 0.1 M hydrogen peroxide to a solution of acetovanillone and potassium iodide in 0.025 M phosphate buffer (pH 3) (75%) [2571].

m.p. 179° [2527], 178–179° [2570,2572], 174–176° [2528];
^{1}H NMR [2571], MS [2571].

1-(6-Hydroxy-3-iodo-2-methoxyphenyl)ethanone

$C_9H_9IO_3$ mol.wt. 292.07



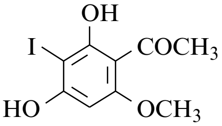
Synthesis

- Preparation by iodination of 2-hydroxy-6-methoxy-acetophenone with an aqueous iodine-iodic acid solution in ethanol at r.t. (68%) [1924].

m.p. 116° [1924].

1-(2,4-Dihydroxy-3-iodo-6-methoxyphenyl)ethanone

[74047-32-8] $C_9H_9IO_4$ mol.wt. 308.07



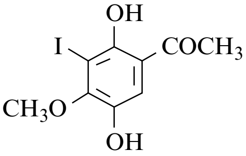
Synthesis

- Preparation by adding an aqueous solution of iodine and periodic acid to an ethanolic solution of 2,4-dihydroxy-6-methoxyacetophenone at 60–70° [2064,2573, 2574], (86%) [2574], (74%) [2573].

m.p. 198–200° [2574], 193–195° [2573];
^{1}H NMR [2574], ^{13}C NMR [2574], IR [2574], MS [2574].

1-(2,5-Dihydroxy-3-iodo-4-methoxyphenyl)ethanone

$C_9H_9IO_4$ mol.wt. 308.07



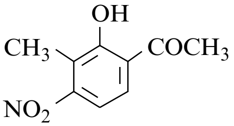
Synthesis

- Obtained from 2-hydroxy-3-iodo-4-methoxyacetophenone oxidised with potassium persulfate in aqueous sodium hydroxide solution (21%) [1924] (Elbs persulfate oxidation).

m.p. 174° (d) [1924].

1-(2-Hydroxy-3-methyl-4-nitrophenyl)ethanone

[190730-40-6] $C_9H_9NO_4$ mol.wt. 195.17



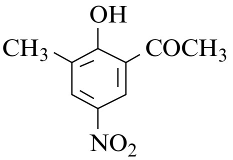
Syntheses

- Preparation by reaction of acetyl chloride (1.3 mol) with 2-methyl-3-nitrophenol (1 mol) in nitrobenzene under nitrogen. The reaction mixture was warmed at 45°, a small amount of aluminium chloride was added and the reaction mixture was stirred at 45° for 1 h. After the addition of another portion of aluminium chloride (1 mol), the temperature rose to 60° and the reaction mixture was slowly heated at 120° and allowed to stirring for another 16 h (72%) [2575].
- Also refer to: [2576,2577].

m.p. 40–41° [2575]; ^{1}H NMR [2575], IR [2575], MS [2575].

1-(2-Hydroxy-3-methyl-5-nitrophenyl)ethanone

$C_9H_9NO_4$ mol.wt. 195.17

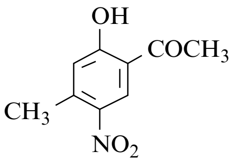


Synthesis

- Preparation by reaction of nitric acid on 2-hydroxy-3-methylacetophenone in acetic acid at 0° (58%) [2578]; first at 0°, then 30° (72%) [1925].

m.p. 115–116° [2578], 114–114°5 [1925].

1-(2-Hydroxy-4-methyl-5-nitrophenyl)ethanone



$C_9H_9NO_4$ mol.wt. 195.17

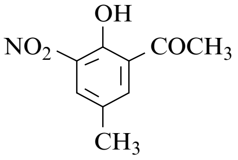
Synthesis

- Preparation by reaction of nitric acid on 2-hydroxy-4-methylacetophenone in acetic acid first at 0°, then 30° (72%) [1925].

m.p. 99°5 [1925].

1-(2-Hydroxy-5-methyl-3-nitrophenyl)ethanone

[66108-30-3] $C_9H_9NO_4$ mol.wt. 195.17



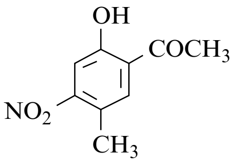
Syntheses

- Preparation by reaction of nitric acid on 2-hydroxy-5-methylacetophenone in acetic acid [1851,1852, 1925,2579], (90%) [1852], (64%) [1925].
- Preparation by reaction of potassium nitrate on 2-hydroxy-5-methylacetophenone in dilute sulfuric acid (77%) [2072].
- Also obtained by reaction of copper nitrate on 2-hydroxy-5-methylacetophenone in ice-cooled acetic anhydride solution (80%) [1851].

m.p. 135–136° [1852], 135° [1851], 132° [1925,2579], 130–130°5 [2072]; pK_a [2516].

1-(2-Hydroxy-5-methyl-4-nitrophenyl)ethanone

[56609-14-4] $C_9H_9NO_4$ mol.wt. 195.17

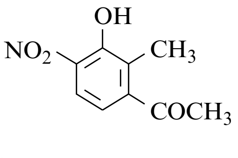


Synthesis not yet described

- There is a single reference, erroneous. It concerns the 2-hydroxy-5-methyl-3-nitroacetophenone [2516] (see above).

1-(3-Hydroxy-2-methyl-4-nitrophenyl)ethanone

[89877-53-2] $C_9H_9NO_4$ mol.wt. 195.17

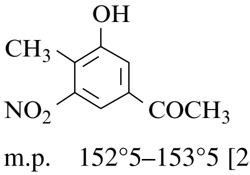


Synthesis

- Refer to: [2580].

1-(3-Hydroxy-4-methyl-5-nitrophenyl)ethanone

$C_9H_9NO_4$ mol.wt. 195.17

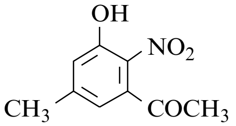


Synthesis

- Preparation by diazotization of 3-amino-4-methyl-5-nitro-acetophenone (m.p. 158–159°), followed by hydrolysis of the diazonium salt so obtained (62%) [2581].

m.p. 152°5–153°5 [2581].

1-(3-Hydroxy-5-methyl-2-nitrophenyl)ethanone



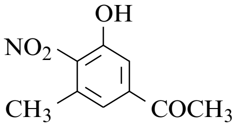
$C_9H_9NO_4$ mol.wt. 195.17

Synthesis

- Obtained (by-product) by reaction of fuming nitric acid on 3-hydroxy-5-methylacetophenone in ethyl ether between −20° and −10° (4%) [2122].

m.p. 66–67° [2122].

1-(3-Hydroxy-5-methyl-4-nitrophenyl)ethanone



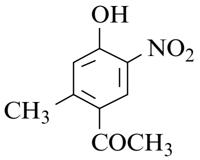
$C_9H_9NO_4$ mol.wt. 195.17

Synthesis

- Preparation by reaction of fuming nitric acid on 3-hydroxy-5-methylacetophenone in ethyl ether between −20° and −10° (23%) [2122].

m.p. 148–150° [2122].

1-(4-Hydroxy-2-methyl-5-nitrophenyl)ethanone



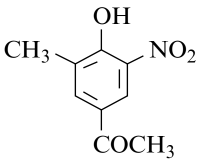
$C_9H_9NO_4$ mol.wt. 195.17

Synthesis

- Preparation by reaction of nitric acid on 4-hydroxy-2-methylacetophenone in acetic acid first at 0°, then 30° (61%) [1925].

m.p. 125–126° [1925].

1-(4-Hydroxy-3-methyl-5-nitrophenyl)ethanone



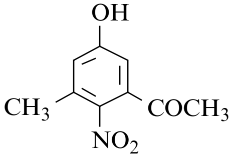
$C_9H_9NO_4$ mol.wt. 195.17

Syntheses

- Preparation by reaction of nitric acid on 4-hydroxy-3-methylacetophenone in acetic acid first at 0°, then 30° (67%) [1925].
- Preparation by reaction of acetyl chloride on 2-methyl-6-nitrophenol with aluminium chloride in nitrobenzene at 130° (50%) [1925].

m.p. 131°5 [1925].

1-(5-Hydroxy-3-methyl-2-nitrophenyl)ethanone



$C_9H_9NO_4$ mol.wt. 195.17

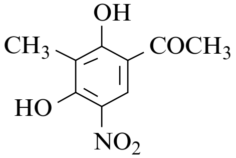
Synthesis

- Preparation by reaction of fuming nitric acid on 3-hydroxy-5-methylacetophenone in ethyl ether between −20° and −10° (29%) [2122].

m.p. 148–150° [2122]; UV [2122].

1-(2,4-Dihydroxy-3-methyl-5-nitrophenyl)ethanone

[118824-94-5] $C_9H_9NO_5$ mol.wt. 211.17



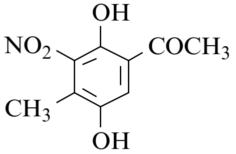
Synthesis

- Preparation by reaction of acetic anhydride on 2-methyl-4-nitroresorcinol with aluminium chloride in nitrobenzene at 80° (73%) [2582].

m.p. 178–179° [2582]; ^{1}H NMR [2582], IR [2582].

1-(2,5-Dihydroxy-4-methyl-3-nitrophenyl)ethanone

[43140-83-6] $C_9H_9NO_5$ mol.wt. 211.17

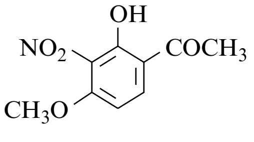


Synthesis

- Preparation by demethylation of 2-hydroxy-5-methoxy-4-methyl-3-nitroacetophenone with aluminium bromide in carbon disulfide at r.t. (96%) [2583].

m.p. 179° [2583]; ^{1}H NMR [2583], IR [2583].

1-(2-Hydroxy-4-methoxy-3-nitrophenyl)ethanone



$C_9H_9NO_5$ mol.wt. 211.17

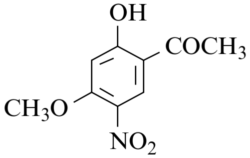
Syntheses

- Preparation by reaction of dimethyl sulfate on 2,4-di-hydroxy-3-nitroacetophenone with potassium carbonate in refluxing acetone [2125].
- Also refer to: [2126].

m.p. 211–212° [2125].

1-(2-Hydroxy-4-methoxy-5-nitrophenyl)ethanone

[102877-53-2] $C_9H_9NO_5$ mol.wt. 211.17



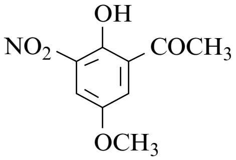
Syntheses

- Preparation by nitration of 2-hydroxy-4-methoxy-aceto-phenone (paeonol) with nitric acid (d = 1.42) in acetic acid [1818,1836,2530,2584], (47%) [1836].
- Also obtained by reaction of concentrated nitric acid with 3,3′-diacetyl-4,4′-dihydroxy-6,6′-dimethoxydiphenyl thioether at r.t. overnight [2585].
- Also refer to: [2484] (compound **1b**).

m.p. 157–158° [2530], 155° [1818,2585], 154° [1836], 153° [2584].

1-(2-Hydroxy-5-methoxy-3-nitrophenyl)ethanone

[90564-25-3] $C_9H_9NO_5$ mol.wt. 211.17



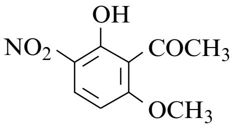
Syntheses

- Preparation from 2-hydroxy-5-methoxyacetophenone by reaction,
  - with nitric acid (d = 1.2) in acetic acid at <15° (83%) [2586];
  - with nitric acid (d = 1.5) in ice-cooled acetic acid and acetic anhydride mixture [1832,2587–2589], (57%) [2588].

m.p. 113–114° [2586], 112° [2588], 110–112° [2587,2589]; IR [1807,1853].

1-(2-Hydroxy-6-methoxy-3-nitrophenyl)ethanone

[38226-01-6] $C_9H_9NO_5$ mol.wt. 211.17



Syntheses

- Obtained by reaction of acetic anhydride on 5-methoxy-2-nitrophenol with aluminium chloride in nitrobenzene at 135° [1940].
- Obtained by reaction of nitric acid (d = 1.42) on 2-hydroxy-6-methoxyacetophenone in acetic acid at r.t. [1807,1940,2522,2523], (43%) [2522], (26%) [1807].
- Also obtained by reaction of fuming nitric acid with 2-hydroxy-6-methoxyacetophenone in acetic acid, first at r.t. for 40 min, then at 45–50° for 16 h (30%) [2074].
- Also obtained (small quantity) by partial methylation of 2,6-dihydroxy-3-nitroacetophenone with dimethyl sulfate in the presence of potassium carbonate in boiling acetone [1940].

- Also obtained by partial demethylation of 2,6-dimethoxy-3-nitroacetophenone,
  - with aluminium chloride in nitrobenzene, heated on a steam bath (by-product) [1940];
  - with concentrated hydrochloric acid in boiling acetic acid [1940];
  - with boiling concentrated hydrochloric acid [1940];
  - with concentrated sulfuric acid at 30° [1940];
  - with potassium hydroxide in boiling ethanol [1940].
- Also obtained by reaction of nitric acid (d = 1.4) on 2,2′-dihydroxy-3,3′-diacetyl-4,4′-di-methoxyphenyl thioether at 0° [2485].

m.p. 104–105° [2522], 102–103° [1940,2485], 100°5–101°5 [1807], 98–100° [2074];

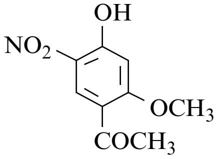
^{1}H NMR [2074], ^{13}C NMR [2590], IR [1807,2074].

1-(4-Hydroxy-2-methoxy-5-nitrophenyl)ethanone

$C_9H_9NO_5$ mol.wt. 211.17

Synthesis

- Obtained by reaction of fuming nitric acid on 4-hydroxy-2-methoxyacetophenone (isopaeonol) at 10° (24%) [1836].



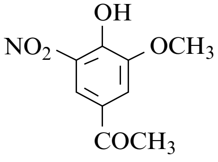
m.p. 95° [1836].

1-(4-Hydroxy-3-methoxy-5-nitrophenyl)ethanone

[20716-41-0] $C_9H_9NO_5$ mol.wt. 211.17

Synthesis

- Preparation by nitration of 4-hydroxy-3-methoxyaceto-phenone with 70% nitric acid in acetic acid, first at 10°, then at 25° (95%) [2215].



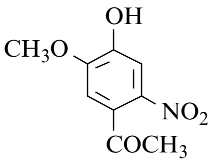
m.p. 148°1–149°5 [2215].

1-(4-Hydroxy-5-methoxy-2-nitrophenyl)ethanone

[418759-58-7] $C_9H_9NO_5$ mol.wt. 211.17

Syntheses

- Preparation by treatment of 4-benzyloxy-5-methoxy-2-nitro-acetophenone (m.p. 142–143°) with trifluoroacetic acid at r.t. for 14 h (89%) [2591].
- Also refer to: [2592].

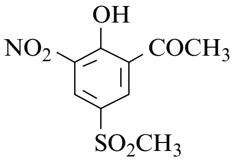


m.p. 151°5–152°5 [2591]; Crystal data [2591];

^{1}H NMR [2591], ^{13}C NMR [2591].

1-[2-Hydroxy-5-(methylsulfonyl)-3-nitrophenyl]ethanone

[70978-46-0] $C_9H_9NO_6S$ mol.wt. 259.24



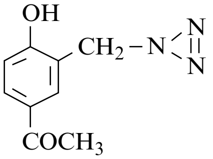
Syntheses

- Preparation by nitration of 2-hydroxy-5-(methylsulfonyl)-acetophenone in concentrated sulfuric acid,
  - with 100% nitric acid at r.t. (89%) [1852];
  - with nitric acid (d = 1.42) between −15° and −5° [1897,1898], (50%) [1897].

m.p. 192–193° [1852], 189–191° [1897,1898].

1-[3-(Azidomethyl)-4-hydroxyphenyl]ethanone

[154603-69-7] $C_9H_9N_3O_2$ mol.wt. 191.19

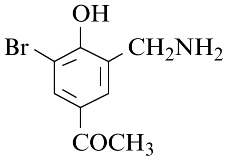


Synthesis

- Preparation by action of sodium azide with 3-chloromethyl-4-hydroxyacetophenone in DMF at 30° for 4 h (60%) [2593].

1-[3-(Aminomethyl)-5-bromo-4-hydroxyphenyl]ethanone

[109314-52-5] $C_9H_{10}BrNO_2$ mol.wt. 244.09

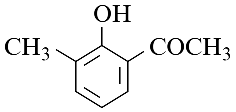


Synthesis

- Refer to: [2594] (Japanese patent).

1-(2-Hydroxy-3-methylphenyl)ethanone

[699-91-2] $C_9H_{10}O_2$ mol.wt. 150.18



Syntheses

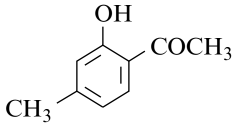
- Preparation by reaction of acetic acid on o-cresol with zinc chloride at reflux (Nencki reaction) (20%) [2595].
- Preparation by Fries rearrangement of o-tolyl acetate with aluminium chloride without solvent between 130° and 180° [2022,2023,2143,2151,2161,2203,2596,2597], (45–55%) [2151,2596,2597], (18–26%) [2022,2023,2143,2161].

- Also obtained (by-product) by Fries rearrangement of o-tolyl acetate with aluminium chloride, ferric chloride or titanium tetrachloride in nitrobenzene as solvent between 20° and 60° (3–5%) [2143,2598–2600].
- Preparation by reaction of acetic anhydride on o-cresol without solvent, at 120°,
  - with 70% perchloric acid (56%) [2183];
  - with aluminium chloride (27%) [2578].
- Also obtained (by-product) by reaction of acetyl chloride on o-cresol with aluminium chloride or titanium tetrachloride in nitrobenzene at 30–60° (3–6%) [2181,2600].
- Preparation by treatment of methyl 4-hydroxy-8-methylcoumarin-3-carboxylate with potassium hydroxide (69%) [2191].
- Also obtained by reaction of stannous chloride on 2-hydroxy-3-methylacetophenone 2,4-dinitro-phenylhydrazone in refluxing aqueous acetone-hydrochloric acid mixture (81%) [2255].
- Also obtained by UV light irradiation of o-tolyl acetate at 25° (photo-Fries rearrangement), in the presence of potassium carbonate in hexane (74%) [2015] or without potassium carbonate in hexane (32%) [2015] or in ethyl ether (16%) [2193].
- Also refer to: [2494].

b.p.$_1$ 62–63° [2578], b.p.$_5$ 91°7–91°9 [2203], b.p.$_9$ 103–104° [2161], b.p.$_9$ 103–105° [2597], b.p.$_{10}$ 105° [2183], b.p.$_{10-10.5}$ 106–107° [2191,2595], b.p.$_{12}$ 107° [2255], b.p.$_{11}$ 108° [2015], b.p.$_{15}$ 111–112° [2215], b.p. 235–237° [2161]; ^{1}H NMR [2203], UV [2203,2215].

1-(2-Hydroxy-4-methylphenyl)ethanone

[6921-64-8] $C_9H_{10}O_2$ mol.wt. 150.18



Syntheses

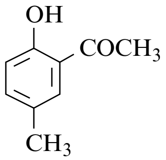
- Preparation by treatment of methyl 4-hydroxy-7-methyl-coumarin-3-carboxylate with potassium hydroxide at 200° (87%) [2191].
- Preparation by Fries rearrangement of m-tolyl acetate [2596,2601–2603],
- *with solvent:*
  - with aluminium chloride,
    - in tetrachloroethane at 130–135° (99%) [2604];
    - in nitrobenzene at 25–30° or at 165° (44–45%) [2143,2605];
  - with titanium tetrachloride in nitrobenzene at 165° (73%) [2143];
- *without solvent:*
  - with aluminium chloride,

    - at 165–170° (88–95%) [2143,2151,2606];
    - between 120° and 165° (70–90%) [2022,2023,2152,2161,2607,2608];
    - between 60° and 165° (38–65%) [2151,2597,2609];
  - with titanium tetrachloride at 95° (83%) [2606];
  - with hydrofluoric acid at 100° (80%) [2236];
  - with zinc chloride at 140–160° (20%) [2191,2610].
- Preparation by reaction of acetic acid on m-cresol,
  - with boron trifluoride at 70° (65%) [2611];
  - with zinc chloride (Nencki reaction) (25%) [2595].
- Preparation by reaction of acetic anhydride on m-cresol with 70% perchloric acid,
  - at r.t. (63%) [2183];
  - at 125–135° (30%) [2306].
- Preparation by reaction of acetyl chloride on m-cresol,
  - with titanium tetrachloride,
    - in nitrobenzene at 60° (75%) [2181];
    - in ethylene dichloride at 25° (40%) [2181];
  - with aluminium chloride,
    - in nitrobenzene at 60° (48%) [2181];
    - in ethylene dichloride at 25° (42%) [2181];
  - with zinc chloride at r.t. (11%) [2610] or at 140–160° [2311].
- Preparation by dehydrogenation of 6-acetyl-3-methyl-2-cyclohexen-1-one,
  - with a 5% palladium-barium sulfate catalyst at reflux (47%) [2612];
  - with refluxing 16% solution of bromine in acetic acid [2612].
- Also obtained by treatment of 4-methylacetophenone with sodium trifluoroacetate in nitromethane–trifluoroacetic acid–trifluoroacetic anhydride mixture in the presence of a platinum electrode, followed by treatment of the intermediate trifluoroester with 10% aqueous potassium carbonate solution (33%) [2006], (hydroxylation of aromatic compound).
- Preparation by UV light irradiation of m-tolyl acetate, at r.t. (photo-Fries rearrangement) [2192], in ethanol (37%), with aqueous β-cyclodextrin solution (54%) or with β-cyclodextrin (solid) (95%).

Colourless oil [2607]; m.p. 21° [2151,2191,2306,2311], 20–21° [2236];
b.p.$_{0.6}$ 82–84° [2608], b.p.$_{0.22}$ 87° [2607], b.p.$_{8}$ 101° [2161,2597],
b.p.$_{7.5}$ 102–104° [2601], b.p.$_{7}$ 103° [2183,2191,2311], b.p.$_{9}$ 105–106° [2191],
b.p.$_{7}$ 107° [2306], b.p.$_{15}$ 115° [2236], b.p.$_{14}$ 116–119° [2611],
b.p.$_{17}$ 123–125° [2609], b.p.$_{20}$ 126° [2191,2311,2595], b.p.$_{20}$ 126–127° [2603],
b.p.$_{760}$ 245° [2191,2311,2595,2606,2610];
^{1}H NMR [1963,2607], IR [1963,2607], MS [2607].

1-(2-Hydroxy-5-methylphenyl)ethanone

[1450-72-2] $C_9H_{10}O_2$ mol.wt. 150.18



Syntheses

- Obtained by treatment of methyl 4-hydroxy-6-methyl-coumarin-3-carboxylate with potassium hydroxide at 200° (82%) [2191].
- Preparation by Fries rearrangement of p-tolyl acetate [1851,2613],

- *with solvent:*
  - with titanium tetrachloride in nitrobenzene at 50° (92%) [2614,2615];
  - with alumina in methanesulfonic acid for 5 min at 160° (92%) [2046];
  - with zirconium chloride in nitrobenzene at 60° (86%) [2615];
  - with zirconium chloride in methylene chloride at r.t. for 24 h (83%). The same reaction performed in a simple ultrasound cleaning bath at r.t. for 24 h also leads to 83% yield [2616];
  - with stannic chloride in nitrobenzene at 60° (78%) [2615];
  - with aluminium chloride, (90%) [2617], (86%) [2071], at 130° (90%) [2618] or at 160° (70%) [2619];
    - in nitrobenzene at 60° (92%) [2598], (68%) [2615], at 25° (84%) [2143];
    - in diphenyl ether at 160° (30%) [2620];
    - in chlorobenzene, in a sealed tube and subjected to high power microwave irradiation for 2 min only (85%) [2013];
    - in the presence of 4-ethyl-2,6-dimethylphenyl chloroacetate at 150° (72%) [2535];
    - in the presence of 2-chloro-4-methylphenyl benzoate at 150° (50%) [2535];
  - on K 10 montmorillonite using microwave radiations (640 w, 5 min) (86%) or in refluxing DMF during 4 h (75%) [2109];
- *without solvent:*
  - with aluminium chloride,
    - between 110° and 170° (85–99%) [1852,2012,2014,2143,2151,2606,2621];
    - between 120° and 150° (60–74%) [1967,2150,2622];
    - at 120° (36–45%) [2082,2597], (90%) [2013];
  - with titanium tetrachloride at 120° (70%) [2606];
  - with hydrofluoric acid at 120–125° (63%) [2236];
  - with beryllium chloride at 150° (63%) [2579,2623].
- Preparation by reaction of acetyl chloride on p-cresol,
  - with titanium tetrachloride in nitrobenzene at 60° (93%) [2181];
  - with aluminium chloride,

  - in nitrobenzene at 60° (75–80%) [2181,2624];
  - in ethylene dichloride at 110–120° (56%) [2625];
  - without solvent at 180° (64–72%) [2626].

- Preparation by reaction of acetyl chloride with p-cresol in the presence of aluminium chloride during 30 min at 180°, via a Fries rearrangement (98%) [2627].
- Preparation by acylation of p-cresol with acetic acid in the presence of alumina in methanesulfonic acid for 5 min at 120° (90%) [2046].
- Preparation by reaction of acetyl chloride on 4-methylanisole with aluminium chloride [2160,2161,2628].
- Also obtained by reaction of acetyl chloride on p-tolyl borate with aluminium chloride in refluxing carbon disulfide (15%) [2182].
- Preparation by reaction of acetic anhydride on p-cresol with 70% perchloric acid at 100° (53%) [2183], at 125–135° (30%) [2306].
- Preparation by reaction of acetic acid on p-cresol,
  - with boron trifluoride at 70° (95%) [2611];
  - with zinc chloride at reflux (14%) [2595] (Nencki reaction).
- Preparation by dealkylation of,
  - 2-methoxy-5-methylacetophenone,
    - with pyridinium chloride at reflux (56%) [2609];
    - with hydrobromic acid in acetic acid (7%) [2215];
  - 2-ethoxy-5-methylacetophenone with aluminium chloride [2251].
- Also obtained from 2-hydroxy-5-methyl-α-chloroacetophenone by treatment with zinc powder in acetic acid [2628].
- Also obtained by treatment of 2-hydroxy-5-methyl-α,α,α-trifluoroacetoacetophenone with 2 N sodium hydroxide at r.t. (100%) [2629].
- Also obtained by reaction of stannous chloride on 2-hydroxy-5-methylacetophenone 2,4-dinitro-phenylhydrazone in refluxing aqueous acetic acid–hydrochloric acid mixture (80%) [2255].
- Also obtained by reaction of mesityl acetate on p-cresol with aluminium chloride at 150° (72%) [2535].
- Also obtained by reaction of various aryl acetates on p-tolyl benzoate with aluminium chloride at 150°, which produce the acetyl cation [2535],
  - with mesityl acetate (77%);
  - with 2-chloro-4-methylphenyl acetate (57%);
  - with 2,6-dimethylphenyl acetate (33%).
- Also obtained by degradation of 3-bromo-6-methylchromanone with alkali in boiling water [2630].
- Also obtained by hydrogenation of 2,4-dimethylindoxazen in acetic acid in the presence of Pd/$BaSO_4$ during 4–6 h [2631].
- Also obtained by hydrolysis of 2-(2-hydroxy-5-methylphenyl)-2-methyl-1,3-dioxolane by a catalytic amount of carbon tetrabromide (20%) in acetonitrile/water

solvent mixture under sonication in a commercial ultrasonic cleaning bath for 2 h at 45° (98%) [2632].

- Also obtained by UV light irradiation of p-tolyl acetate at 25°,
  - in hexane,
    - in the presence of potassium carbonate (86%) [2015];
    - without potassium carbonate (35%) [2015], (6%) [2198];
  - in ethyl ether (32%) [2193], in ethanol [2198,2633], (8%) [2198], in benzene (6%) [2198] or in methanol [2633].
- Also refer to: [1794] (compound **1a**), [1795,2634,2635] (compound **1c**), [2567].

Isolation from natural sources

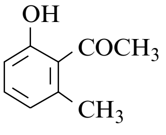
- From the coriander seed essential oil [2636].
- Detected in the sorghum malt beverage [2637].
- Identified in powdered turmeric (curcuminoids) [2638].

N.B.: Na salt [52166-70-8] [2617,2639].

m.p. 50° [1851,2012,2143,2151,2160,2161,2191,2236,2255,2306,2595,2597, 2598,2606,2614,2619,2624,2628,2634], 49°5 [2082], 49–50° [2109,2611], 48–49° [2620,2625], 48° [2631], 47–48° [1852,2579], 47° [2622], 46°5 [1967], 46–48° [2535], 46–47°5 [2215], 45–46° [2014,2015], 45° [2071];
b.p.$_{0.2}$ 60–62° [2622], b.p.$_7$ 101–103° [2183], b.p.$_{15}$ 112° [2161], b.p.$_{13}$ 118° [2611],
b.p.$_{15}$ 118–121° [2609], b.p.$_7$ 120–130° [2620], b.p.$_{16}$ 124° [2236], b.p.$_{740}$ 209° [2071];
GC-MS [2636–2638]; pK$_a$ [1977,2516];
^{1}H NMR [1963,1976,2109,2622,2627,2633,2640], ^{13}C NMR [1976],
IR [1963,2109,2622], UV [2215,2622,2640].

1-(2-Hydroxy-6-methylphenyl)ethanone

[41085-27-2] $C_9H_{10}O_2$ mol.wt. 150.18



Syntheses

- Preparation from 2-hydroxy-6-methylbenzonitrile and methylmagnesium bromide in ethyl ether–tetrahydrofuran mixture at 50° (67%) [2641].
- Preparation by adding a solution of methyl lithium in ethyl ether to 6-methylsalicylic acid in tetrahydrofuran and maintain a gentle reflux for 8 h. Then, the solution was cooled and acidified with hydrochloric acid (62%) [2642].

- Preparation by demethylation of 2-methoxy-6-methylacetophenone,
  - with pyridinium chloride at 200–215° (44%) [2496], (31%) [2643];
  - with boron tribromide in methylene chloride at r.t. [2644,2645], (70%) [2644].
- Preparation by diazotization of 2-amino-6-methylacetophenone, connected with hydrolysis of the diazonium salt obtained [2646].
- Also obtained by a potassium fluoride catalyzed self-condensation of 2,4-pentanedione in DMF solution [2647,2648].
- Also obtained by UV light irradiation of 3-methylphenyl acetate, in ethanol (36%) [2192] or in ethanol in the presence of β-cyclodextrin (42%) [2192].
- Also obtained (poor yield) by irradiation of dehydroacetic acid and vinyl acetate in an ethyl acetate solution [2648,2649], (4%) [2649].

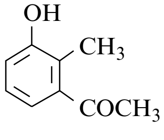
Isolation from natural sources

- The 2-hydroxy-6-methylacetophenone was isolated as exocrine compound in several neotropical species of ants in the dolichoderine genus Hypoclinea (*Hypoclinea analis*, *Hypoclinea abrupta*, *Hypoclinea bidens* A). The ants were collected in Belem, Para, Brazil [2641,2650]. It was also isolated from the bodies of *Rhytidoponera aciculata* (Australian ponerine ant) [2651,2652].
- The hydrolytic cleavage of Peripentadenine gave the 2-hydroxy-6-methylacetophenone. The Peripentadenine was obtained from dried milled bark of *Peripentadenia mearsii* (Elaeocarpaceae), collected at Boonjie (North Queensland) [2653].
- From the essential oil of *Cistus ladanifer* L. (Cistaceae) [2654].
- From the aerial parts of *Gerbera ambigua* (Compositae), collected in Transvaal [2655].

Oil [2641–2644,2646,2649,2652,2655];
After two recrystallizations from water and two sublimations (130°/0.09 mm) gave a white powder, m.p. 93–98° [2643];
b.p.$_7$ 100° [2642], b.p.$_{18}$ 138–142° [2644];
n = 1.5612 [2646], n = 1.5600 [2642];
^{1}H NMR [2641,2642,2644,2648–2650,2654,2655], ^{13}C NMR [2653],
IR [2496,2641,2642,2644,2653,2655], MS [2641,2650,2651,2654,2655].

1-(3-Hydroxy-2-methylphenyl)ethanone

[69976-81-4] $C_9H_{10}O_2$ mol.wt. 150.18



Syntheses

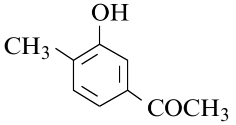
- Preparation by diazotization of 6-amino-3-hydroxy-2-methylacetophenone, followed by hydrolysis of the obtained diazonium salt in the presence of hypophosphorous acid at 0° (42%) [2455].

- Preparation by passing a solution of 3-hydroxy-2-methylbenzoic acid in hot acetic acid over a pelleted thoria catalyst at 470–480° during 4 h (32%) [2656].

m.p. 121° [2656], 120°8–121°7 [2455]; ^{1}H NMR [2455].

1-(3-Hydroxy-4-methylphenyl)ethanone

[33414-49-2] $C_9H_{10}O_2$ mol.wt. 150.18



Syntheses

- Preparation by diazotization of 3-amino-4-methylaceto-phenone, followed by hydrolysis of the obtained diazonium salt [1951,2294,2657], (75%) [2294], (40%) [1951].
- Obtained by treatment of 4-methylacetophenone with sodium trifluoroacetate in nitromethane–trifluoroacetic acid–trifluoroacetic anhydride mixture in the presence of a platinum electrode followed by treatment of the intermediate trifluoroacetate ester with 10% potassium hydrogen carbonate solution (40%) [2006] (of aromatic compounds).

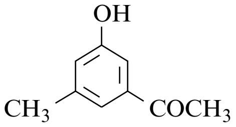
Isolation from natural sources

- From *Laurencia chilensis* De Toni, Forte and Howe (Rhodomelaceae) [2658].

m.p. 119–120° [1951,2294], 105–107° [2658];
^{1}H NMR [2658], UV [2658], IR [2658] (Sadtler: standard n° 8331), MS [2658].

1-(3-Hydroxy-5-methylphenyl)ethanone

[43113-93-5] $C_9H_{10}O_2$ mol.wt. 150.18



Syntheses

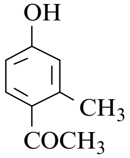
- Preparation by diazotization of 3-amino-5-methylaceto-phenone, followed by hydrolysis of the obtained diazonium salt (70%) [2122].
- Preparation by hydrolysis of diethyl 3-acetoxy-5-methyl-benzoylmalonate with concentrated sulfuric acid in boiling aqueous acetic acid (70%) [2122].
- Also obtained by solvolysis of 3-acetyl-5-methylphenol tetrahydropyranyl ether with p-toluene-sulfonic acid in methanol at r.t. (73%) [2659].
- Preparation by aromatization of 5-acetyl-3-methyl-2-cyclohexen-1-one with cupric bromide and lithium bromide in boiling acetonitrile (46%) [2122].
- Also obtained by alkaline transformation of methyl, tert-butyl or benzyl esters of 4-acetonyl-5-hydroxy-2,5-dimethyl-3-furoic acid with 2 N sodium hydroxide in ethanol at r.t. (35–50%) [2660].
- Preparation by oxidation of 5-acetyl-3-methyl-2-cyclohexen-1-one with air in alkaline medium or by catalytic hydrogenation of the latter in the presence of Pd/C in p-cymene [2660].

– Also obtained (poor yield) by alkaline degradation of a solution of D-xylose or D-glucose in 0.63 M sodium hydroxide at 96° under nitrogen [2276].

m.p. 122–123° [2122,2276], 118–121° [2660], 118–120°5 [2659];
^{1}H NMR [2659,2660], IR [2659,2660], UV [2660], MS [2659,2660].

1-(4-Hydroxy-2-methylphenyl)ethanone

[875-59-2] $C_9H_{10}O_2$ mol.wt. 150.18



Syntheses

– Preparation by dealkylation of 4-hydroxy-2-methyl-5-iso-propylacetophenone with aluminium chloride in chlorobenzene at 50° (80%) [2661].
– Also obtained by reaction of aluminium chloride on 4-acetoxy-2-methylacetophenone at 130° (51%) or on 4-(benzoyloxy)-2-methylacetophenone at 170° [2151].
– Preparation by Fries rearrangement of m-tolyl acetate,

- with aluminium chloride in nitrobenzene at r.t. (80–85%) [2151], (60–66%) [2143,2535,2605,2662,2663], (54%) [2664];
- with aluminium chloride without solvent [2143,2152,2161,2606,2609], at 65° (88%) [2606], at 60–70° (28%) [2609], at 130° (16%) [2152] and at 165° (7%) [2143];
- with zinc chloride and hydrochloric acid at r.t. (30%) [2610];
- with hydrofluoric acid at 20° (17%) [2236];
- with titanium tetrachloride at 95° (7%) [2606].

– Also obtained by reaction of acetyl chloride on m-cresol,

- with aluminium chloride in nitrobenzene at 25° (63%) [2181];
- with titanium tetrachloride in nitrobenzene at 25° (25%) [2181];
- with zinc chloride [2310,2311,2610], (14%) [2610];
- with ferric chloride in carbon disulfide [1823].

– Also obtained by reaction of acetic acid on m-cresol,

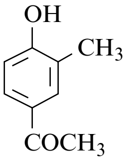
- with boron trifluoride at 70° (16%) [2611];
- by heating the mixture with phosphorous oxychloride (by-product) [2596].

– Also obtained by reaction of acetic anhydride on m-cresol with 70% perchloric acid at r.t. (6%) [2183].
– Also obtained by UV light irradiation on p-tolyl acetate in methanol at 25° (26%) [2315]. (There is a 1,2-migration of the methyl group).
– Also obtained by UV light irradiation of m-tolyl acetate in ethanol (13%) [2192].
– Also obtained by reduction of 4-hydroxy-2-methyl-α-chloroacetophenone with zinc dust in aqueous ethanolic acetic acid [2665].

m.p. 131° [2611], 129° [2661], 128–130° [2315], 128–129° [2663], 128° [2161,2183,2311,2606,2609,2664,2665], 127° [2143,2151,2610,2662], 126° [1823], 125° [2236];
b.p.$_{17}$ 158–160° [2609], b.p. 313° [2311];
^{1}H NMR [2315,2663], IR [2315], UV [2605].

1-(4-Hydroxy-3-methylphenyl)ethanone

[876-02-8] $C_9H_{10}O_2$ mol.wt. 150.18



Syntheses

- Preparation by dealkylation of 4-hydroxy-5-methyl-2-iso-propylacetophenone with aluminium chloride in chloro-benzene at 50° (53%) [2661].
- Preparation by Fries rearrangement of o-tolyl acetate [2161,2171],

with solvent:

- with aluminium chloride in nitrobenzene at r.t. (83–85%) [2151,2598], (41–49%) [1825,2600,2666,2667] or at 60° (82%) [2143];
- with aluminium chloride in diphenyl ether at 175° (54%) [2620];
- with titanium tetrachloride in nitrobenzene at 30° (78%) [2599,2600];
- with ferric chloride in nitrobenzene at 50° (60%) [2600].

without solvent:

- with aluminium chloride at 160–180° (61%) [2143], (27%) [2597];
- with ferric chloride (42%) [2600].

- Preparation by reaction of acetyl chloride on o-cresol,
  - with hydrofluoric acid at 50° (98%) [2668];
  - with aluminium chloride,
    - in refluxing carbon disulfide (96%) [1951];
    - in nitrobenzene at 60° (86%) [2181];
  - with titanium tetrachloride in nitrobenzene at 30° (66%) [2600] or at 60° (78%) [2181];
  - with ferric chloride in carbon disulfide [1823].
- Preparation by reaction of acetonitrile on o-cresol with triflic acid at r.t. (67%) [2250].
- Also obtained by reaction of acetic anhydride with o-cresol in anhydrous hydrofluoric acid at 50° (quantitative yield) [1825] or in 70% perchloric acid at 120° (31%) [2183].
- Also obtained by reaction of acetic acid on o-cresol with zinc chloride (Nencki reaction) (10%) [2595].
- Preparation by diazotization of 4-amino-3-methylacetophenone with sodium nitrite in dilute hydrochloric acid and replacement of the diazonium group by hydroxyl group [2253].

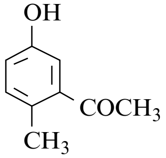
- Also obtained by UV light irradiation of o-tolyl acetate, at 25°,
  - in ethyl ether (10%) [2193];
  - in hexane, in the presence of potassium carbonate (12%) [2015] or without potassium carbonate (3%) [2015].
- Also refer to: [1828].

m.p. 110° [2620,2661], 108–109° [1825], 108° [2598], 107–109° [1951], 106–108° [2666],105°5–106°5 [2250], 104–105° [2266], 104° [1823,2143,2161,2253,2595,2599,2600];

b.p.$_{7.5}$ 166–167° [2597], b.p.$_5$ 170–175° [2620]; pK$_a$ [1977];
^{1}H NMR [2250], IR [2250], MS [2250].

1-(5-Hydroxy-2-methylphenyl)ethanone

[40180-70-9] $C_9H_{10}O_2$ mol.wt. 150.18



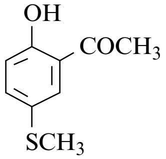
Syntheses

- Preparation from 5-acetyl-4-methyl-3-cyclohexenone by aromatization promoted,
  - by cupric bromide with lithium bromide in refluxing acetonitrile (80%) [2030];
  - by 10% Pd/C in refluxing xylene (50%) [2030].

m.p. 28° [2030]; ^{1}H NMR [2030], IR [2030].

1-[2-Hydroxy-5-(methylthio)phenyl]ethanone

[135936-88-8] $C_9H_{10}O_2S$ mol.wt. 182.24

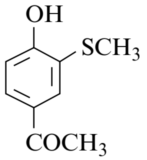


Synthesis

- Refer to: [2272].

1-[4-Hydroxy-3-(methylthio)phenyl]ethanone

[66264-56-0] $C_9H_{10}O_2S$ mol.wt. 182.24



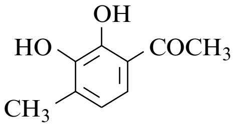
Syntheses

- Preparation by reaction of methyl iodide on 4-hydroxy-3-mercaptoacetophenone with potassium carbonate in acetone at r.t. (83%) [2274,2275].
- Preparation by reaction of acetyl chloride on 2-(methyl-thio) phenol with aluminium chloride in nitrobenzene at 65° (34%) [2274,2275].
- Also refer to: [1828].

m.p. 117–120° [2274,2275].

1-(2,3-Dihydroxy-4-methylphenyl)ethanone

[69751-81-1] $C_9H_{10}O_3$ mol.wt. 166.18



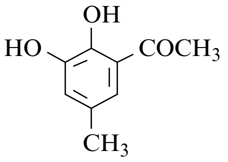
Syntheses

- Preparation by Fries rearrangement of 3-methylpyrocatechol diacetate with a molten mixture of aluminium chloride and sodium chloride at 200° (15%) [2669].
- Also obtained by UV light irradiation of 3,6-dimethyl-1,2-benzisoxazole in 96% sulfuric acid (5%) [2284,2285].

m.p. 70° [2669], 67–68° [2284]; ^{1}H NMR [2284], IR [2284,2669], UV [2284], MS [2284].

1-(2,3-Dihydroxy-5-methylphenyl)ethanone

[69751-80-0] $C_9H_{10}O_3$ mol.wt. 166.18



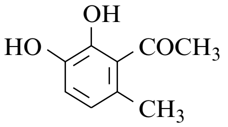
Syntheses

- Preparation by reaction of acetic acid on creosol with boron trifluoride at 160° (78%) [2670].
- Also obtained by UV light irradiation of 3,5-dimethyl-1,2-benzisoxazole in 96% sulfuric acid (6%) [2284].

m.p. 87°5–88° [2284], 86–88° [2670];
^{1}H NMR [2284], IR [2284], UV [2284], MS [2284].

1-(2,3-Dihydroxy-6-methylphenyl)ethanone

[59862-07-6] $C_9H_{10}O_3$ mol.wt. 166.18



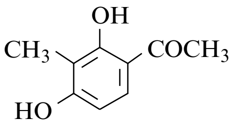
Synthesis

- Obtained (poor yield) by alkaline degradation of a solution of D-xylose or D-glucose in 0.63 M sodium hydroxide at 96° under nitrogen [2276].

m.p. 82–83° [2276]; ^{1}H NMR [2276], IR [2276], MS [2276].

1-(2,4-Dihydroxy-3-methylphenyl)ethanone

[10139-84-1] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

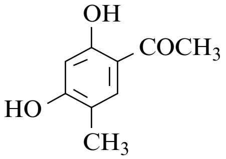
- Preparation by reaction of acetic anhydride on 2-methyl-resorcinol with boron trifluoride-ethyl ether complex at 70–80° (78%) [2582].
- Preparation by reaction of acetic acid on 2-methyl-resorcinol with zinc chloride (Nencki reaction) (59%) [2671].

- Preparation by reaction of acetonitrile on 2-methylresorcinol (Hoesch reaction) [2213,2672].
- Preparation by demethylation of 2-hydroxy-4-methoxy-3-methylacetophenone with hydriodic acid in a boiling mixture of phenol and acetic anhydride [2213,2672].
- Also obtained by catalytic reduction of 2′,4′-dihydroxy-3′-(1-piperidylmethyl) acetophenone in the presence of Pd/C [2673,2674], (65%) [2674]. The starting material was obtained by treatment of resacetophenone in ethanol with methylene-bis-piperidine.
- Also refer to: [2675].

m.p. 157–158° [2582,2673,2674], 156–157° [2672], 155–156° [2213];
^{1}H NMR [2582,2674], ^{13}C NMR [1821], IR [2582,2674], UV [2213], MS [2582].

1-(2,4-Dihydroxy-5-methylphenyl)ethanone

[93578-16-6] $C_9H_{10}O_3$ mol.wt. 166.18



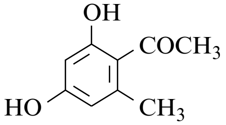
Syntheses

- Preparation by reaction of acetonitrile on 4-methylresorcinol (Hoesch reaction) (75%) [2676], (50–59%) [2302,2677].
- Preparation from 2,4-dimethoxy-5-methylacetophenone by reaction with boron tribromide in methylene chloride at r.t. (48%) [2678,2679].
- Preparation by reaction of acetic acid on 4-methylresorcinol with zinc chloride (Nencki reaction) (46–60%) [2330,2680–2682].
- Preparation by Fries rearrangement of 4-methylresorcinol diacetate with aluminium chloride in nitrobenzene (63%) [2680].

m.p. 170–171° [2330], 170° [2676,2682], 169° [2677], 168–169° [2680];
^{1}H NMR [2680], IR [2680], UV [2213].

1-(2,4-Dihydroxy-6-methylphenyl)ethanone
(Orcacetophenone; Orsacetophenone; β-Orcacetophenone)

[703-29-7] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

- Preparation by reaction of acetonitrile on orcinol (Hoesch reaction) [2301,2307,2683,2684], (75%) [2330], (62%) [2301].
- Preparation by reaction of acetyl chloride on orcinol with aluminium chloride in nitrobenzene (56%) [2308].
- Preparation by reaction of acetic acid with orcinol in the presence of boron trifluoride etherate at 102–115°, then hydrolysis of complex obtained with boiling dilute ethanol (65%) [2685].

- Preparation from 3,3′-dimethyl-5,5′-diisoxazolyl-methane by performing hydrogenolysis and subsequent hydrolysis with hydrochloric acid (80%) [2686] or with 50% sulfuric acid [2687].
- Preparation by reaction of 50% sulfuric acid with nonan-2,4,6,8-tetraone diethylene ketal for 10 min (77%) [2688].
- Preparation by hydrolysis of 2,8-di-(1-pyrrolidinyl)-2,7-nonanediene-4,6-dione (SM) $C_{17}H_{26}N_2O_2$ **4** (m.p. 196–199°) with refluxing 2 N sulfuric acid for 30 min (69%) [2689]. SM was obtained by reaction of 3-acetoacetyl-4-hydroxy-6-methyl-2-pyrone with an excess of pyrrolidine.
- Preparation by hydrolysis of an ionic complex $C_{22}H_{27}NO_5$ **10** (m.p. 161–162°) with refluxing 2 N sulfuric acid for 1 h (87%) [2689].
- Also obtained from 7-hydroxy-4,5-dimethylcoumarin by heating in a boiling water bath with 10% aqueous sodium hydroxide [2683].
- Also obtained by degradation of 3,8-diacetyl-4,5-dihydroxy-7-methylcoumarin by heating with aqueous sodium hydroxide solution [2690].
- Also obtained by decarboxylation of 3-acetyl-p-orsellinic acid (3-acetyl-2,6-dihydroxy-4-methyl-benzoic acid) with copper in boiling quinoline [2691].
- Also obtained (by-product) by reaction of trifluoroacetic anhydride on 2-acetoxy-4-methoxy-6-methylbenzoic in the presence of orcinol at 25° (8%) [2692].
- Also obtained (by-product) by Fries rearrangement of orcinol diacetate in the presence of aluminium chloride in nitrobenzene at 75–80° (5%) [2693].
- Also obtained (by-product) by reaction of acetyl chloride on orcinol dimethyl ether with aluminium chloride in carbon disulfide (3%) [2694].

Isolation from natural sources

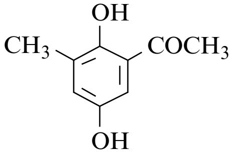
- From *Scolecotrichum graminis* Fuckel [2695].

m.p. 160–161° [2694], 159–160° [2308], 159° [2301,2307,2688,2690], 158–160° [2692], 158–159° [2330,2689], 158° [2686,2691], 157–159° [2683], 140° [2685];

^{1}H NMR [2686,2695], IR [2685,2695], UV [2213,2685,2695], MS [2686,2695].

1-(2,5-Dihydroxy-3-methylphenyl)ethanone

[274259-41-5] $C_9H_{10}O_3$ mol.wt. 166.18



Synthesis

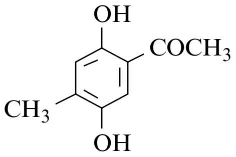
- Preparation by demethylation of 2-hydroxy-5-methoxy-3-methylacetophenone with boron tribromide in methylene chloride at r.t. for 5 h (71%) [2696].

m.p. 111–113° [2696];

^{1}H NMR [2696], IR [2696], MS [2696].

1-(2,5-Dihydroxy-4-methylphenyl)ethanone

[54698-17-8] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

- Preparation by Fries rearrangement of 2-methyl-hydro-quinone diacetate with aluminium chloride without solvent [2024,2697–2699], between 120° and 160° (53–54%) [2698,2699].
- Preparation by reaction of acetic acid on 2-methylhydro-quinone with boron trifluoride [2349,2350,2700], (85%) [2350].
- Also obtained by saponification of 2,5-diacetoxy-4-methylacetophenone with aqueous sodium hydroxide [2349].
- Also obtained by demethylation of 2-hydroxy-5-methoxy-4-methylacetophenone and 2,5-di-methoxy-4-methylacetophenone with boiling pyridinium chloride [2542].
- Also obtained by UV light irradiation of 3,6-dimethyl-1,2-benzisoxazole in 96–98% sulfuric acid (44%) [2284,2285].
- Also refer to: [2701–2703].

Isolation from natural sources

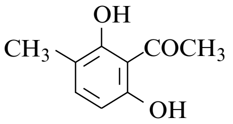
- From Chinese Moutan Cortex, the root of *Paeonia suffruticosa* Andrews (Paeoniaceae) [2323].

m.p. 148–149° [2699], 147–147°5 [2284], 147° [2542], 145–146° [2698], 145° [2349], 141° [2024,2350];

^{1}H NMR [2284], IR [2284,2699], UV [2284], MS [2284].

1-(2,6-Dihydroxy-3-methylphenyl)ethanone

[29183-78-6] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

- Obtained from 8-acetyl-4,6-dimethyl-7-hydroxycoumarin by alkaline degradation with 20% aqueous sodium hydroxide solution at reflux (42%) [2213,2682].
- Obtained from 4-acetoxy-3-acetyl-2-hydroxy-5-methyl-benzoic acid by hydrolysis with 10% ethanolic potassium hydroxide, followed by decarboxylation [2704].

Isolation from natural sources

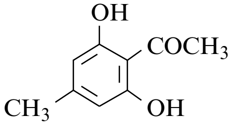
- From dihydrousnic acid [2552,2705] or from tetrahydrodeoxyusnic acid [2705] (from Lichens substances) by potassium permanganate oxidation in 10% aqueous potassium hydroxide at r.t., followed by distillation (5%) [2552].
- From methyldihydrousnic acid [2704], either by potassium permanganate oxidation, followed by vacuum distillation [2706], or by vacuum distillation, followed by ozone oxidation [2706].

m.p. 138–139° [2213], 138° [2552,2682,2705], 136–137° [2706], 134–136° [2704];

^{1}H NMR [2704], UV [2213,2704,2706].

1-(2,6-Dihydroxy-4-methylphenyl)ethanone *(γ- or p-orcacetophenone)*

[1634-34-0] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

- Preparation by reaction of acetic anhydride on orcinol,
  - with concentrated sulfuric acid at 130° (65%) [2307];
  - with Amberlite IR-120 or Zeokarb-225, cation exchange resins (sulfonic acid type) at 160° (20%) [2026].
- Preparation by reaction of acetyl chloride on orcinol with aluminium chloride in boiling nitrobenzene (50%) [2213,2707].
- Preparation by reaction of acetic acid on orcinol,
  - with zinc chloride (Nencki reaction) [2708];
  - with phosphorous oxychloride at 100–110° [2301,2709,2710].
- Also obtained by reaction of acetyl chloride on 2,5-dimethoxytoluene (?) with aluminium chloride in carbon disulfide (26%) [2348].
- Also obtained by partial deacylation of 2,4-diacetylorcinol with 85% sulfuric acid at r.t. [2335,2337,2691], (39%) [2337] or with boiling solution of 1 N sodium hydroxide [2691].
- Also obtained by decarboxylation of methyl 3-acetyl-2,4-dihydroxy-6-methylbenzoate,
  - by reaction of boiling solution of 1 N sodium hydroxide [2691];
  - by heating at 180°, followed by distillation [2711].
- Also obtained by reaction of boiling solution of 1 N sodium hydroxide on methyl 3,5-diacetyl-2,4-dihydroxy-6-methylbenzoate, decarbonylation and decarboxylation occurring simultaneously [2691].
- Also obtained by reaction of 2-acetoxy-4-methoxy-6-methylbenzoic acid on orcinol with trifluoroacetic anhydride at 25° (5%) [2692].
- Also obtained by degradation of 3,8-diacetyl-4,7-dihydroxy-5-methylcoumarin by heating with aqueous sodium hydroxide solution [2690].
- Also obtained by reaction of potassium hydroxide with 2-acetyl-3-dimethylamino-5-hydroxy-5-methyl-2-cyclohexenone in ethanol at 40° (48%) [2712].
- Also refer to: [2713].

Isolation from natural sources

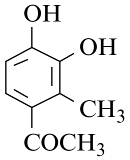
- From *Rumex patientia* (Polygonaceae) [2714].
- The occurrence of 2-acetylorcinol and its monoglucoside was established in Tissue Cultures from *Rumex alpinus* (Polygonaceae) [2715].

m.p. 147–149° [2026], 146–147° [2213,2348], 146° [2301,2307,2337,2691,2708,2710,2711], 144–146° [2712], 142–144° [2707], 142–143° [2690,2692];

^{1}H NMR [2206,2348,2704,2712], ^{13}C NMR [2206], IR [2348,2690,2711,2712]; UV [2213,2711], MS [2348,2712].

1-(3,4-Dihydroxy-2-methylphenyl)ethanone

[66296-84-2] $C_9H_{10}O_3$ mol.wt. 166.18



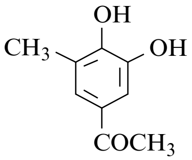
Syntheses

- Obtained (poor yield) by treatment of dihydroxyacetone in aqueous solution (pH 4.5) at 96° (2%) [2716].
- Also obtained (poor yield) by reaction of acetic anhydride on 3-methyl-1,2-benzenediol with perchloric acid [2717].
- Also obtained (poor yield) by Fries rearrangement of 3-methylpyrocatechol diacetate with aluminium chloride in nitrobenzene at 75–80° (< 19%) [2376].

m.p. 149–152° [2716]; ^{1}H NMR [2716], MS [2716].

1-(3,4-Dihydroxy-5-methylphenyl)ethanone

[80547-86-0] $C_9H_{10}O_3$ mol.wt. 166.18



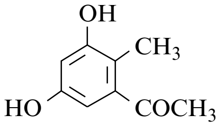
Syntheses

- Preparation by heating 3-methylpyrocatechol in boron trifluoride-acetic acid at 60–70° (62%) [2669].
- Preparation by Fries rearrangement of 3-methylpyrocatechol diacetate,
  - with aluminium chloride in chlorobenzene at 110° (71%) [2718] or in nitrobenzene at 75–80° (<19%) [2376];
  - with a molten mixture of aluminium chloride and sodium chloride at 200° (40%) [2669].
- Also obtained from neutral glucose and fructose solutions heated at 120° [2390].

m.p. 197–199° [2669], 139–140° [2718]; ^{1}H NMR [2718], IR [2669,2718].

1-(3,5-Dihydroxy-2-methylphenyl)ethanone

$C_9H_{10}O_3$ mol.wt. 166.18



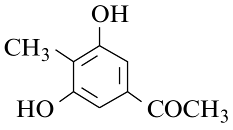
Synthesis

- Preparation by diazotization of (3 or 5)-amino-(5 or 3)-hydroxy-2-methylacetophenone hydrochloride (m.p. 110°5–111°), followed by hydrolysis of the diazonium salt so obtained (51%) [2581].

m.p. 160°5–161°2 [2581].

1-(3,5-Dihydroxy-4-methylphenyl)ethanone

$C_9H_{10}O_3$ mol.wt. 166.18



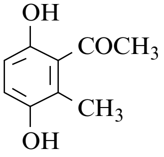
Synthesis

- Preparation by diazotization of 3-amino-5-hydroxy-4-methylacetophenone hydrochloride, followed by hydrolysis of the diazonium salt so obtained (43%) [2581].

m.p. 190–191° [2581].

1-(3,6-Dihydroxy-2-methylphenyl)ethanone

[176177-16-5] $C_9H_{10}O_3$ mol.wt. 166.18



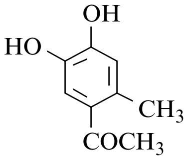
Synthesis

– Preparation by total demethylation of 2,5-dimethoxy-6-methylacetophenone (SM) with aluminium chloride in refluxing chlorobenzene for 4 h (74%) [2719]. SM was obtained in three steps from 2,5-dimethoxy-6-methylaniline *via* the sequence: first, 6-bromo-2,5-dimethoxytoluene (m.p. 97–98°5), then 6-(1-hydroxyethyl)-2,5-dimethoxy-toluene (m.p. 40°5–42°5) and finally SM (m.p 59–59°5).

m.p. 122°5–123°5 [2719]; sublimation at 86–90°/0.1 mm [2719];
^{1}H NMR [2719], IR [2719], MS [2719].

1-(4,5-Dihydroxy-2-methylphenyl)ethanone

[18087-17-7] $C_9H_{10}O_3$ mol.wt. 166.18



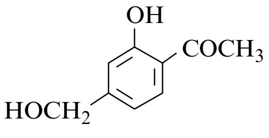
Syntheses

- Preparation by reaction of zinc powder on 4,5-dihydroxy-2-methyl-α-chloroacetophenone in dilute acetic acid between 40° and 70° [2280,2386], (good yield) [2280].
- Preparation by Fries rearrangement of 4-methylpyrocatechol diacetate with aluminium chloride in nitrobenzene at 75–85° (87–97%) [2376,2720], (34%) [2721].
- Also obtained (poor yield) by reaction of acetic anhydride with 3,4-dihydroxytoluene in the presence of 70% perchloric acid on a steam bath for 3 h (10%) [2722].
- Also obtained (by-product) by Fries rearrangement of creosol acetate with aluminium chloride in nitrobenzene at 80° for 1 h (6%) [2723].
- Also refer to: [2724].

m.p. 170°5–171° [2722], 169° [2723], 168–170° [2721], 168–169° [2280,2720], 164° [2386], 160° [2376];
^{1}H NMR [2722], MS [2722].

1-[2-Hydroxy-4-(hydroxymethyl)phenyl]ethanone

[22518-00-9] $C_9H_{10}O_3$ mol.wt. 166.18



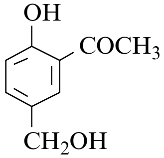
Isolation from natural sources

- By acidic hydrolysis of two esters isolated from *Gaillardia aristata*, the 7-isobutyryloxy-8,9-epoxythymol and 7-(2-methylbutyryloxy)-8,9-epoxythymol isobutyrates [2725].
- From the aerial parts of *Calea nelsonii* Robinson and Greenman (Asteraceae) [2726].

m.p. 61°5–62° [2725]; ^{1}H NMR [2725], IR [2725], UV [2725].

1-[2-Hydroxy-5-(hydroxymethyl)phenyl]ethanone

[31611-90-2] $C_9H_{10}O_3$ mol.wt. 166.18

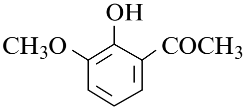


Synthesis

- Obtained by hydrolysis of 2-hydroxy-5-chloromethyl-acetophenone [2727].

1-(2-Hydroxy-3-methoxyphenyl)ethanone *(ortho-Acetovanillone)*

[703-98-0] $C_9H_{10}O_3$ mol.wt. 166.18



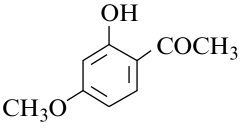
Syntheses

- Preparation from 2,3-dimethoxybenzonitrile and methyl-magnesium iodide in refluxing ethyl ether (Grignard reaction) [1821,2728], (75%) [2728].
- Preparation by reaction of aluminium chloride on the 2,3-dimethoxyacetophenone, in boiling ethyl ether (96%) [2408] or in boiling toluene (67%) [2729].
- Also obtained by reaction of hydrobromic acid on 2,3-dimethoxyacetophenone in acetic acid at 25° [2215].
- The usual method of synthesis involves the successive conversion of o-veratraldehyde to the 2,3-dimethoxyphenyl methyl carbinol, 2,3-dimethoxyacetophenone and demethylation [2728].
- Preparation by UV light irradiation of guaiacol acetate, in benzene (48%) [2198] or in ethyl ether (20%) [2193].
- Also obtained (by-product) by Fries rearrangement of guaiacol acetate with zinc chloride at reflux [2729] or with aluminium chloride without solvent between 20° and 50° (9–11%) [2378].

m.p. 54° [2408], 53–54° [2729], 52–53° [2215], 50–53°1 [2728];
b.p.$_{0.5}$ 110–120° [2729];
^{1}H NMR [2198], ^{13}C NMR [1821], IR [2198], UV [2215].

1-(2-Hydroxy-4-methoxyphenyl)ethanone *(Paeonol)*

[552-41-0] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

- Preparation by reaction of methyl iodide [2220,2730,2731], or dimethyl sulfate [1818,2227,2301,2529,2732–2737] on resacetophenone in sodium hydroxide or potassium hydroxide solution (quantitative yield) [2301], (70–75%) [1818], (good yield) [2731], (60%) [2735], (52%) [2732].
- Preparation by reaction of methyl iodide on resacetophenone,
  - with potassium carbonate in boiling acetone (92%) [2738], (73%) [2368], (55–64%) [2739];
  - with lithium carbonate in DMF at 55° (81%) [2217].

- Preparation by reaction of dimethyl sulfate with resacetophenone in the presence of potassium carbonate in refluxing acetone (81%) [2740].
- Also obtained by reaction of methyl iodide on resacetophenone silver salt in boiling acetone [2293].
- Preparation by reaction of diazomethane with resacetophenone in ethyl ether/ methanol solution (90%) [2302].
- Preparation by reaction of acetic acid,
  - on the resorcinol monomethyl ether, in the presence of polyphosphoric acid (25%) [2299] or zinc chloride at reflux (Nencki reaction) (29%) [2171];
  - on the resorcinol dimethyl ether with boron trifluoride at 125° (42%) [2298].
- Preparation by reaction of acetonitrile on resorcinol monomethyl ether (Hoesch reaction) (27%) [2301].
- Preparation by reaction of bromoacetyl bromide on resorcinol dimethyl ether with aluminium chloride [2741].
- Preparation by Fries rearrangement of 3-methoxyphenyl acetate with zirconium chloride in methylene chloride for 48 h at r.t. (85%) [2616].
- Also obtained by Fries rearrangement of 3-methoxyphenyl acetate with aluminium chloride in nitrobenzene at r.t. (13%) [2742,2743].
- Also obtained by reaction of zinc powder on 2,4-dimethoxy-α-bromoacetophenone in acetic acid in a water bath [2744].
- Also obtained (by-product) by reaction of zinc powder and silver chloride on 2-hydroxy-4-methoxy-α-chloroacetophenone in benzene and crotonaldehyde mixture at 60–70° [2745].
- Also obtained by reaction of 2 N sodium hydroxide on 2-hydroxy-4-methoxy-α,α,α-trifluoro-acetoacetophenone at r.t. (quantitative yield) [2629].
- Also obtained by acetylation of resorcinol monomethyl ether by treatment with complex mixture (acetyl chloride/acetic anhydride/acetic acid/anisole/sodium perchlorate) at 60° (8%) [2746].
- Also obtained by reaction of aluminium chloride on 2,4-dimethoxyacetophenone in benzene at 100–110° [2530] or in acetonitrile for 6 h at 45° (40%) [2747].
- Also obtained on treatment of ethyl 2-acetyl-5-methoxyphenoxyacetate with boron trichloride in methylene chloride, first at –70°, then at r.t. for 5 min (84%). The ethoxycarbonylmethyl group was selectively removed without difficulty [2748].

Isolation from natural sources

- From the root bark of *Paeonia Moutan* (Ranunculaceae) [2316,2331,2731,2749], of *Paeonia broteroi* [2256] and of *Paeonia suffruticosa* Andrews (mudanpi) (Paeoniaceae) [2323] or *Paeania suffruticosa* Anhr. (mudanpi) (Ranunculaceae) (major component) [2750].
- From *paeonia radix* (SM) [2751–2753]. SM is the dried roots of *paeonia veitchii* Lynch or *paeonia lactiflora* Pall, yet named *paeonia albiflora* Pall.

- From macadamia nuts and shells [2754].
- From the roots of *Pentecost rose* (*paeonia arborea*, also named *paeonia Moutan* in China or *paeonia Botan* in Japan) (Renonculaceae) by hydrolysis of its glucoside [2736,2749,2755].
- From the roots of *cynanchum paniculatum* [2756–2758].
- From the volatile oil of *dioscorea japonica* [2759].
- From the leaves of *ficus krishnae* [2760].
- As a major component in the steam distillates of the resins from various species of *Xanthorrhoea* (X.); *X. tateana* F. Muell. and *X. preissi* [2584], but also from *X. arborea* R. Br. and *X. reflexa* [2761].
- By thermal decomposition of the resin from *Ferula pyramidata* (Kar. et Kir.) eug. kor. [2293].

m.p. 51°3 [2762], 51° [2299,2745], 50–51° [2737], 50° [1818,2171,2220,2733273423312301276327352742274323162298253027 31], 49–50° [2368,2738], 49° [2293,2584,2730,2761], 48–50° [2739,2755], 48° [2740,2764], 47–49° [2217], 45° [2749];

b.p.$_{0.001}$ 90–100° [2745], b.p.$_{3}$ 135–137° [2293], b.p.$_{20}$ 154° [2732], b.p.$_{18}$ 155–165° [2761];

b.p.$_{20}$ 158° [2584], b.p.$_{5}$ 180° [2737], b.p.$_{30}$ 210° [2737];

n_D^{15} = 1.5745 [2293]; d_{20}^{40} = 1.1604 [2293];

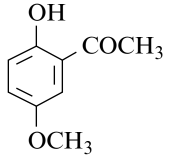
TLC [2754,2765]; HPLC [2752,2766]; GC [2756,2759]; GC-MS [2759];

^{1}H NMR [1869,2256,2326,2327,2738], ^{13}C NMR [1821,2210,2328,2329,2368,2767];

IR [1963,2256], UV [2256,2326], MS [2331,2765].

1-(2-Hydroxy-5-methoxyphenyl)ethanone

[705-15-7] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

- Preparation by reaction of dimethyl sulfate on 2,5-dihydroxyacetophenone [2345,2348,2529,2768],
  - with potassium carbonate in acetone at r.t. (74%) [2348];
  - with aqueous sodium hydroxide solution at reflux (35%) [2345,2768].
- Preparation by reaction of aluminium chloride on 2,5-dimethoxyacetophenone in refluxing ethyl ether (53%) [2408].
- Preparation by reaction of methyl iodide on 2,5-dihydroxyacetophenone with potassium carbonate in refluxing acetone [1984,2769], (55–64%) [2769].
- Also obtained by reaction of acetic acid on hydroquinone dimethyl ether with boron trifluoride at 70° (26%) [2298].
- Also obtained by Fries rearrangement of 4-methoxyphenyl acetate with aluminium chloride without solvent at 60–65° (24%) [2344].
- Also obtained by reaction of acetyl chloride on hydroquinone dimethyl ether with aluminium chloride,

  - in boiling ethyl ether (57%) [2770];
  - in nitrobenzene, at r.t. (45%) [2771];
  - in methylene chloride, at r.t. (16%) [2772];
  - in carbon disulfide, at r.t. (7%) [2773] or by heating at 95°, after elimination of the solvent (72%) [2521].

- Preparation by UV light irradiation of 4-methoxyphenyl acetate at 25°,
  - in hexane (75%) [2015];
  - in hexane with potassium carbonate, (89%) [2015];
  - in ethyl ether (38%) [2193];
  - in ethanol [2774,2775], (40–42%) [2775].
- Also obtained by UV light irradiation of 4-methoxyphenyl 3-(ethylenedioxy)-butanoate in hexane at r.t. (34%) [2776].
- Also obtained by UV light irradiation of 2,4-dimethoxyphenyl acetate in benzene or ethanol (11–13%) [2198].
- Also refer to: [2227,2494,2777].

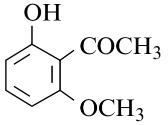
Isolation from natural sources

- From the essential oil of the rhizomes of *Primula acaulis*, an European variety of stemless primrose [2352].

m.p. 52° [2012,2298,2768,2771], 51° [2770], 50–51° [2344,2345,2408,2773], 50° [2015,2775], 49–49°5 [2772], 49° [2352,2521], 48–50° [2769,2778], 48–49° [1984,2215], 47–49° [2348], 46° [2774];
b.p.$_{12}$ 138–142° [2408], b.p.$_{15}$ 146–150° [2298];
^{1}H NMR [1976,2348], ^{13}C NMR [1821,1976,2328,2779], IR [1963,2348];
UV [2215,2348]; pK$_a$ [1977].

1-(2-Hydroxy-6-methoxyphenyl)ethanone

[703-23-1] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

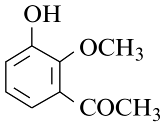
- Preparation by reaction of methyl iodide on 2,6-dihydroxyacetophenone with potassium carbonate in boiling acetone [1821,1984,2780], (56%) [2780].
- Preparation by reaction of dimethyl sulfate on 2,6-di-hydroxyacetophenone,
  - with potassium carbonate in boiling benzene (71–73%) [1891,2781];
  - with potassium carbonate in boiling acetone [2219,2524] (quantitative yield) [2524];
  - with potassium hydroxide in benzene, in a water bath (54%) [2364];
  - with 20% solution of sodium hydroxide (68%) [2361];
  - with 30% solution of sodium hydroxide (20%) [2213,2369].
- Also obtained by partial demethylation of 2,6-dimethoxyacetophenone with aluminium chloride in acetonitrile for 6 h at 45° (30%) [2747].

– Also obtained from 3-acetyl-5-methoxy-2-methylchromone by refluxing with 2% sodium carbonate solution [2131].

m.p. 60° [2131,2361,2369,2781], 59–60° [2364], 58°5 [2780], 58–59° [2213], 57–59° [1891], 57–58° [2219], 55–56° [1984], 55° [2524];
^{1}H NMR [2374], ^{13}C NMR [1821,2219], UV [2213].

1-(3-Hydroxy-2-methoxyphenyl)ethanone

[204781-71-5] $C_9H_{10}O_3$ mol.wt. 166.18



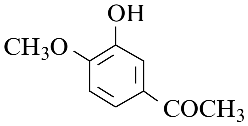
Isolation from natural sources

– Detected in liquid smoke flavouring preparations (compound **61**) [2782].

GC [2782]; GC/MS [2782].

1-(3-Hydroxy-4-methoxyphenyl)ethanone *(Isoacetovanillone)*

[6100-74-9] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

– Preparation by saponification of 3-acetoxy-4-methoxy-acetophenone [2783–2785], (90%) [2785], (59%) [2783].

– Preparation by hydrolysis of 3,4-dimethoxyacetophenone with concentrated sulfuric acid at 65° (58%) [2786].

– Preparation by reaction of methyl iodide on 3,4-dihydroxyacetophenone with lithium carbonate in DMF at 55° (90%) [2217].

– Preparation by reaction of activated zinc dust on 3-hydroxy-4-methoxy-α-chloroacetophenone with 10% acetic acid in refluxing ethanol (48%) [2787].

– Preparation by Fries rearrangement of guaiacol acetate,

- with hydrofluoric acid at 0° (28%) [2236];
- with zinc chloride at reflux (6%) [2729].

– Preparation by reaction of acetic acid on guaiacol,

- with hydrofluoric acid at 0° (25%) [2236];
- with phosphorous oxychloride on a steam bath (20%) [2171].

– Preparation by reaction of acetic anhydride on guaiacol,

- with concentrated sulfuric acid at 80° (20–24%) [2784] (The in situ formed sulfoacetic acid during the reaction was the actual acylating agent);
- with zinc chloride [2331].

– Obtained by reaction of sodium acetate on 3-chloroacetoxy-4-methoxyacetophenone in refluxing methanol [2788].

– Also refer to: [2789].

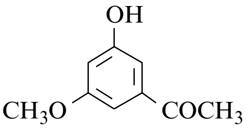
Isolation from natural sources

- From Chinese Moutan Cortex, the root of *Paeonia suffruticosa* Andrews or Anhr. *(mudanpi)* (minor component) (Paeoniaceae) [2323], (Ranunculaceae) [2750].

m.p. (anhydrous): 93° [2236], 92–93° [2750,2786], 92° [2783], 91–92° [2171,2785], 91° [2784], 89° [2217], 87–88° [2787];
m.p. (hydrate): 67–68° [2729,2785], 66–69° [2784], 65–68° [2788], 64–65° [2331], 59–60° [2171];
b.p.$_{15}$ 180–200° [2171], b.p.$_{18}$ 195–200° [2784];
^{1}H NMR (Sadtler: standard n° 28211 M);
IR [2783] and (Sadtler: standard n° 55283); UV [2783], MS [2331].

1-(3-Hydroxy-5-methoxyphenyl)ethanone

[35999-23-6] $C_9H_{10}O_3$ mol.wt. 166.18

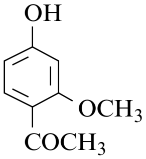


Synthesis

- Refer to: [2790].

1-(4-Hydroxy-2-methoxyphenyl)ethanone *(Isopaeonol)*

[493-33-4] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

- Preparation by reaction of acetonitrile on resorcinol monomethyl ether (Hoesch reaction) (27%) [2301], (10%) [2128].
- Preparation by reaction of acetic acid on resorcinol monomethyl ether,
  - with polyphosphoric acid (27%) [2299];
  - using perchloric acid liberated in situ, at 60° (6%) [2746].
- Preparation by hydrolysis of 4-acetyl-3-methoxyphenyl acetate with boiling 10% sodium hydroxide solution (58%) [2791] or 4-acetyl-3-methoxyphenyl benzoate with potassium hydroxide in refluxing methanol (85%) [2792].
- Also obtained (by-product) by Fries rearrangement of 3-methoxyphenyl acetate with aluminium chloride in nitrobenzene at r.t. (11%) [2742,2743] or at 90° (4%) [2128].
- Also refer to: [2777] and [2793] (compound **8**).

Isolation from natural sources

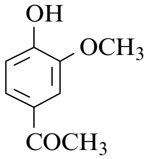
- This ketone was identified in the extract from “Redgold” apple flowers [2794].

m.p. 139–140° [2791,2792], 138° [2128,2301,2742,2743], 137–138° [2746], 134° [2299]; ^{13}C NMR [2328].

1-(4-Hydroxy-3-methoxyphenyl)ethanone

(Apocynin; Acetovanillone; Acetoguaiacone)

[498-02-2] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

- Preparation by reaction of acetyl chloride on guaiacol [2784].
- Preparation by reaction of acetic anhydride on guaiacol with polyphosphoric acid in a water bath (29%) [2788].
- Preparation by Fries rearrangement of guaiacol acetate,
  - with aluminium chloride in nitrobenzene at r.t. [2378,2795–2798], (49–51%) [2378,2797] or at 80° (40%) [2796];
  - with hydrofluoric acid at 0° (50%) [2236];
  - with zinc chloride at 200° (25–26%) [2171,2729].
- Preparation by reaction of acetic acid on guaiacol,
  - with hydrofluoric acid at 0° (38%) [2236];
  - with polyphosphoric acid (36%) [2299];
  - with zinc chloride at reflux (4%) [2171];
  - with aluminium chloride–zinc chloride mixture at 140–150° [2784,2799], (low yield) [2799].
- Also obtained by hydrolysis of 4-acetyl-2-methoxyphenyl acetate with boiling water [2784].
- Also obtained by saponification of 4-acetyl-2-methoxyphenyl benzoate with boiling aqueous sodium hydroxide (56%) [2800].
- Also obtained (low yield) by treatment of 4-hydroxy-3-methoxybenzoic acid (vanillic acid) with calcium carbonate in boiling dilute acetic acid [2379].
- Also obtained from 4-hydroxy-3-methoxy-α-bromoacetophenone [2801] or 4-hydroxy-3-methoxy-α-chloroacetophenone [2802] by reductive removal of the halogen atom with iron filings and 10% sulfuric acid in aqueous ethanol at 60°.
- Also obtained by UV light irradiation,
  - of guaiacol acetate in benzene (12–14%) [2193,2198];
  - of 2,4-dimethoxyphenyl acetate in benzene (8%) [2198] or in ethanol (4%) [2198].
- Also refer to: [1828,2227].

Isolation from natural sources

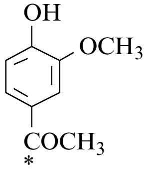
- From rhizomes of Canadian hemp, *Apocynum cannabinum*, of *Apocynum androsaemifolium* (Apocynaceae) [2803,2804], of several species of *Apocynum* [2331].

- From the essential oil of the rhizomes of Iris (Iridaceae) [2805].
- From the roots of *Paeonia broteroi* (Paeoniaceae) [2256].
- From the root of *Paeonia suffruticosa* Andrews (Paeoniaceae) [2323].
- From the bulbs of *Buphane disticha*, Herb. (Amaryllidaceae) [2806].
- From spruce lignin sulfonic acid by treatment with hot aqueous alkali (0.2–0.3%) [2807].
- Described by Shimamoto as one of the fragrant components of Soy [2798].

m.p. 116° [2236], 115–116° [2729,2796], 115–115°5 [2805], 115° [2331,2379,2797,2799,2800,2802–2804,2806], 114–115° [2788], 114° [2299], 113°5–114°5 [2807], 113–114° [2171,2801], 112–114° [1789];
b.p.$_{13}$ 160–170° [2797], b.p.$_{15-20}$ 233–235° [2379], b.p.$_{760}$ 280–300° [2799], b.p. 300° [2803];
^{1}H NMR [2256], IR [2256], UV [2256,2269], MS [1789,2331].

1-(4-Hydroxy-3-methoxyphenyl)ethanone-*1*-^{13}*C*

[199793-91-4] $C_9H_{10}O_3$ mol.wt. 167.17

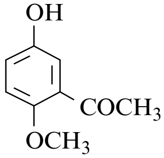


Synthesis

- Obtained by condenof [1-^{13}C]acetic acid with guaiacol [2808].

1-(5-Hydroxy-2-methoxyphenyl)ethanone

[31405-60-4] $C_9H_{10}O_3$ mol.wt. 166.18



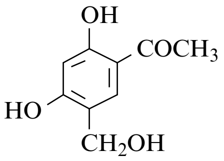
Syntheses

- Preparation by reaction of concentrated hydrochloric acid on 5-(benzyloxy)-2-methoxyacetophenone in acetic acid at 65–70° (31%) [2358].
- Also obtained (by-product) by reaction of dimethyl sulfate on 5-acetoxy-2-hydroxyacetophenone with potassium carbonate in acetone at r.t. (4%) [2348].
- Preparation by reaction of methyl iodide on quinacetophenone with lithium carbonate in DMF at 60° (54%) [2217].
- Preparation by partial demethylation of 2,5-dimethoxyacetophenone with sulfuric acid at 45–55° (42%) [2809].

m.p. 83° [2358], 82–83° [2809], 81–83° [2217], 64–65° [2810], 62–63° [2348];
^{1}H NMR [2348], IR [2348], UV [2348,2810].

1-[2,4-Dihydroxy-5-(hydroxymethyl)phenyl]ethanone

[117705-66-5] $C_9H_{10}O_4$ mol.wt. 182.18



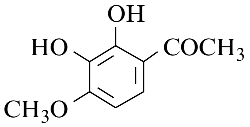
Synthesis

- Preparation by hydrogenation of 5-formyl-2,4-dihydroxy-acetophenone in the presence of 10% Pd/C in ethanol (74%) [2671].

m.p. 51–52° [2671].

1-(2,3-Dihydroxy-4-methoxyphenyl)ethanone

[708-53-2] $C_9H_{10}O_4$ mol.wt. 182.18



Syntheses

- Preparation by reaction of acetic acid on pyrogallol 1-methyl ether,
  - with zinc chloride at reflux (155–160°) (Nencki reaction), (62%) [2781], (46%) [2811];
  - with boron trifluoride on a steam bath (77%) [2403].
- Preparation by reaction of methyl iodide on gallacetophenone monopotassium salt in boiling methanol [2812].
- Preparation by hydrolysis of 3-acetoxy-2-hydroxy-4-methoxyacetophenone with concentrated hydrochloric acid in refluxing aqueous ethanol (84%) [2813].
- Also obtained by reaction of acetyl chloride on 2,6-dimethoxyphenol with zinc chloride at r.t. (8%) [2406].
- Also obtained by Fries rearrangement of 2,6-dimethoxyphenyl acetate with zinc chloride at 120° (6%) [2406].
- Also obtained by partial dealkylation of 2-hydroxy-3,4-dimethoxyacetophenone [2406,2814] with hydrobromic acid in acetic acid at r.t. (46%) [2814] or by partial dealkylation of 3-ethoxy-2-hydroxy-4-methoxyacetophenone in the same conditions (22%) [2814].

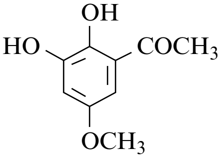
Isolation from natural sources

- From the roots of *Paeonia broteroi* Boiis & Reuter (Paeoniaceae) [2256] or from the root cortex of *Paeonia suffruticosa* Anhr. [2256] (Ranunculaceae) (minor component) [2750].

m.p. 132–133° [2403,2406,2812,2815], 132° [2811], 131–132° [2750], 130–134°5 [2813], 130–132° [2814];

^{1}H NMR [2256,2403], IR [2256,2403], UV [2256], MS [2256,2331].

1-(2,3-Dihydroxy-5-methoxyphenyl)ethanone



$C_9H_{10}O_4$ mol.wt. 182.18

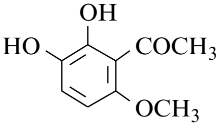
Synthesis

- Obtained by reaction of potassium persulfate on 2-hydroxy-5-methoxyacetophenone in aqueous sodium hydroxide solution (1%) [2408].

m.p. 120° [2408].

1-(2,3-Dihydroxy-6-methoxyphenyl)ethanone

[56358-73-7]



$C_9H_{10}O_4$ mol.wt. 182.18

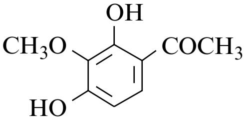
Syntheses

- Preparation by reaction of 3% hydrogen peroxide on 3-acetyl-2-hydroxy-4-methoxybenzaldehyde into solution of 1 N sodium hydroxide (Dakin reaction) (62–65%) [2358,2816].
- Preparation by reaction of 3% hydrogen peroxide on the 3-acetyl-2-hydroxy-6-methoxyacetophenone into solution of 1 N sodium hydroxide at r.t. (33%) [2358].
- Also obtained by reaction of concentrated hydrochloric acid on 2,3-bis(benzyloxy)-6-methoxy-acetophenone in acetic acid at 60° [2358].

m.p. 148–149° [2358], 147° [2816]; ^{1}H NMR [2374], MS [2374].

1-(2,4-Dihydroxy-3-methoxyphenyl)ethanone

[62615-26-3]



$C_9H_{10}O_4$ mol.wt. 182.18

Syntheses

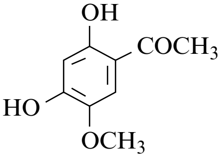
- Preparation by catalytic hydrogenolysis of 4-(benzyloxy)-2-hydroxy-3-methoxyacetophenone in the presence of 5% Pd/C in ethyl acetate [1989,2817], (90%) [2817].
- Preparation by reaction of boron trifluoride–acetic acid complex with the pyrogallol 2-methyl ether at 100° (79–85%) [2403,2818].
- Preparation by reaction of acetonitrile on pyrogallol 2-methyl ether with triflic acid at r.t. (60%) [2819].
- Also obtained by reaction of aluminium chloride on gallacetophenone trimethyl ether in boiling ethyl ether (15%) [2820].
- Also obtained by reaction of methyl iodide on gallacetophenone monopotassium salt in boiling methanol (10%) [2812,2821].
- Also obtained by hydrolysis of 2,4-diacetoxy-3-methoxyacetophenone with boiling aqueous sodium carbonate solution [2822].

N.B.: The melting points of the 1-(2,4-dihydroxy-3-methoxyphenyl)ethanone are conflicting [2819]. One shall thus mention that the melting points of this product, that are in the range 130–135°, can be compared to those of the 1-(2,3-dihydroxy-4-methoxyphenyl)ethanone (132–133°). However, ^{1}H NMR spectral data of this ketone of m.p. 68° [2819] is identical with those reported for the compound of m.p. 141–144° [1989].

m.p. 141–144° [1989], 134–135° [2822], 132–133° [2812,2821], 130–131° [2820], 76° [2817,2818], 75–76° [2403], 68° [2819];
^{1}H NMR [1989,2819], IR [2403,2819], MS [2819].

1-(2,4-Dihydroxy-5-methoxyphenyl)ethanone

[7298-21-7] $C_9H_{10}O_4$ mol.wt. 182.18



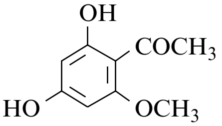
Syntheses

- Preparation by debenzylation of 4-(benzyloxy)-2-hydroxy-5-methoxyacetophenone,
  - by catalytic hydrogenolysis with 5–10% Pd/C in ethyl acetate at r.t. (quantitative yield) [2823,2824];
  - with concentrated hydrochloric acid in boiling acetic acid [2417].
- Preparation by partial demethylation of 2,4,5-trimethoxyacetophenone with hydrobromic acid in acetic acid [2733,2734].
- Preparation by Fries rearrangement of 2,4-diacetoxyanisole with aluminium chloride [2410].

m.p. 174° [2417], 172–173° [2824], 171–172° [2410], 170–171° [2823], 166° [2733,2734].

1-(2,4-Dihydroxy-6-methoxyphenyl)ethanone

[3602-54-8] $C_9H_{10}O_4$ mol.wt. 182.18



Syntheses

- Preparation by reaction of acetonitrile on phloroglucinol monomethyl ether (Hoesch reaction) [1821,2825–2828], (87%) [2828], (73%) [2825], (62%) [2827].
- Preparation by catalytic hydrogenolysis of 2,4-bis-(benzyloxy)-6-methoxyacetophenone in the presence of 10% Pd/C (89%) [2829] in ethyl acetate (64%) [2830].
- Preparation by reaction of aluminium chloride,
  - on the 2,4,6-trimethoxyacetophenone [2325,2826,2831–2833] in boiling chlorobenzene (29–35%) [2831,2832];
  - on the 2-hydroxy-4,6-dimethoxyacetophenone in boiling chlorobenzene [2432,2834–2836], (58%) [2835] according to the method [2836], (58%) [2432];
  - on the 4-hydroxy-2,6-dimethoxyacetophenone in boiling benzene [2837].

- Preparation by partial demethylation of 4-hydroxy-2,6-dimethoxyacetophenone with boron trichloride in cooled methylene chloride (74%) [2748].
- Also obtained by hydrolysis of 2,4-dihydroxy-6-methoxyacetophenone diacetate [2791].
- Also obtained by reaction of concentrated hydrochloric acid on 4-(benzyloxy)-2-hydroxy-6-methoxyacetophenone in acetic acid [2838].

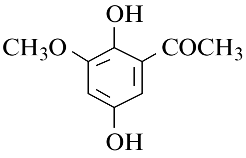
Isolation from natural sources

- From the stem of *Kniphofia fioliosa* (Asphodelaceae) [2839].
- From the aerial parts of *Tanacetum densum* subsp. *eginense* [2840].
- From the roots of *Sanguisorba officinalis* [2841].
- From phloroacetophenone 2,4-di-O-(2,3,4,6-tetra-O-acetyl)-β-D-galactopyranoside. This one was methylated with methyl iodide in the presence of silver carbonate in refluxing acetone for 8 h. The obtained methyl ether was deacetylated, then hydrolyzed with 5% sulfuric acid [2842].
- By reductive cleavage of *isofoliosone*, itself isolated from *Kniphofia foliosa* Hochst [2839].

N.B.: Mg (II) salt [2843].

m.p. 207–209° [2837], 207° [2833], 205–207° [2827,2842,2844,2845], 205–206° [2835], 205° [2325], 204–205° [2432], 203–204° [2791,2838], 203° [2828,2831,2834,2836], 201–202° [2829,2830], 195–198° [2839];
^{1}H NMR [2435,2830,2839], ^{13}C NMR [1821,2839,2846], IR [2435,2830],
UV [2847], MS [2830,2839].

1-(2,5-Dihydroxy-3-methoxyphenyl)ethanone



$C_9H_{10}O_4$ mol.wt. 182.18

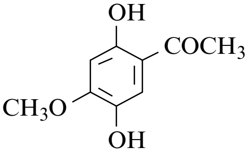
Synthesis

- Obtained by reaction of potassium persulfate on 2-hydroxy-3-methoxyacetophenone in aqueous sodium hydroxide solution (26%) [2848], (2%) [2408].

m.p. 172–174° [2848], 172° [2408].

1-(2,5-Dihydroxy-4-methoxyphenyl)ethanone

[22089-12-9]



$C_9H_{10}O_4$ mol.wt. 182.18

Syntheses

- Preparation by Fries rearrangement of 2-methoxyhydroquinone diacetate,
  - with boron trifluoride in acetic acid (97%) [2849], (62%) [2850];
  - with aluminium chloride in nitrobenzene at r.t. [2410,2742,2743], (38%) [2743], (31%) [2742].

- Preparation by reaction of dimethyl sulfate with 2,4,5-trihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (65%) [2418].
- Preparation from paeonol by persulfate oxidation [2410,2733,2734,2740], (Elbs reaction) (18–22%) [2733,2734,2740].
- Preparation by partial demethylation of 2-hydroxy-4,5-dimethoxyacetophenone with hydrobromic acid in refluxing acetic acid [2417].
- Preparation by diazotization of 5-amino-2-hydroxy-4-methoxyacetophenone, followed by decomposition of the diazonium salt obtained [2530].

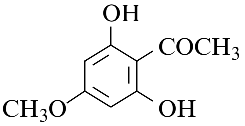
Isolation from natural sources

- From Chinese Moutan Cortex, the root of *Paeonia suffructicosa* Andrews (Paeoniaceae) [2530] in Japanese [2323]. The root cortex of *Paeonia suffruticosa* Anhr. (Ranunculaceae) is also known as [2323] in Chinese. In this, the ketone is a minor component [2750].

m.p. 166–167° [2750], 166° [2849,2850], 165–166° [2742,2743], 164–166° [2418], 164–165° [2417,2530], 164° [2733,2734,2740];
^{1}H NMR [2850], IR [2850], MS [2331].

1-(2,6-Dihydroxy-4-methoxyphenyl)ethanone

[7507-89-3] $C_9H_{10}O_4$ mol.wt. 182.18



Syntheses

- Obtained by reaction of boiling aqueous potassium hydroxide,
  - on the Methyl 3-acetyl-2,4-dihydroxy-6-methoxybenzoate (quantitative yield) [2851,2852];
  - on the 3-trichloroacetyl-2,6-dihydroxy-4-methoxyacetophenone [2368,2851,2852], (quantitative yield) [2851,2852].
- Preparation by adding a solution of 2,6-diacetoxy-4-hydroxyacetophenone in dioxane at r.t.. The residual pale yellow oil obtained after elimination of solvent was hydrolyzed with dilute hydrochloric acid in refluxing methanol (83%) [2853].
- Preparation by selective deacetylation of 2,4-diacetyl-5-methoxyresorcinol with sodium methoxide in refluxing methanol [2826,2854,2855], (76%) [2826] or with boiling 1 N sodium hydroxide (70%) [2856].
- Preparation by reaction of diazomethane on phloroacetophenone in ethyl ether at r.t. [2434,2827,2857,2858], (37%) [2857].
- Preparation by reaction of dimethyl sulfate on phloroacetophenone with potassium carbonate in refluxing acetone (62%) [2859].

Isolation from natural sources

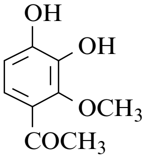
- From the root tissue of *Sanguisorba minor* (Rosaceae) [2859].
- As a decomposition product of sakuranin, a glucoside isolated from the bark of *Prunus pseudocerasus Lindl.* (Var. Sieboldi Maxim.), also called *Prunus yedoensis Matsumura* [2844].

- By alkaline hydrolysis of artocarpetin, a flavonoid pigment isolated from the heartwood of *Artocarpus integrifolia* [2860].
- Claimed to be obtained as a minor component in the steam distillates of the resins from various species of *Xanthorrhoea* (X.); *X. tateana* F. Muell. and *X. preissi* (m.p. 79°) [2584], but also from *X. arborea* R. Br. and *X. reflexa*. (m.p. 79°) [2761]. Thus, this compound, the 2,6-dihydroxy-4-methoxyacetophenone has a melting point of 136–137° (see below). Two possibilities may then occur:
  - either this compound is a monomethyl ether derived from phloroacetophenone, as the authors do pretend, and in this case, it would be at the time the 2,4-dihydroxy-6-methoxyacetophenone (m.p. 205–207°) [2827,2844,2845]. It is not possible;
  - or, it is a dimethyl ether [2845], and in this case, it would be the 2-hydroxy-4,6-dimethoxy-acetophenone *(Xanthoxylin)* (m.p. 78–79°) [2861], (80°) [2845], (82°) [2862], what is later confirmed [2862].

m.p. 139–140° [2844], 139–139°5 [2853], 139° [2860], 138–140° [2857], 137–139° [2856], 136–137° [2826,2827,2833,2837,2845,2852], 134–136° [2368];
b.p.$_{0.5}$ 145–150° [2860];
^{1}H NMR [2206,2368,2826,2853,2856], ^{13}C NMR [2206,2368];
IR [2826], UV [2859];
MS [2859].

1-(3,4-Dihydroxy-2-methoxyphenyl)ethanone

[27829-93-2] $C_9H_{10}O_4$ mol.wt. 182.18



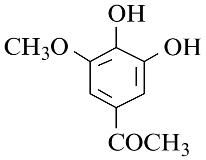
Syntheses

- Obtained by debenzylation of 3,4-bis(benzyloxy)-2-methoxyacetophenone with 15% ethanolic hydrochloric acid, on a steam bath (22%) [2403].
- Also refer to: [2863] (compound **5**).

m.p. 84–85° [2403]; ^{1}H NMR [2403], IR [2403].

1-(3,4-Dihydroxy-5-methoxyphenyl)ethanone

[3934-89-2] $C_9H_{10}O_4$ mol.wt. 182.18

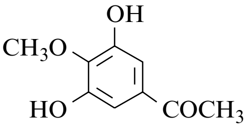


Syntheses

- Cyclic voltammetry showed the electrochemical oxidation of 3,5-dimethoxy-4-hydroxyacetophenone gave 3,4-di-hydroxy-5-methoxyacetophenone as the major product by a one electron oxidation process (20% isolated yield), but 30–40% estimated by thin layer chromatography [2864].
- Also refer to: [1787,2865–2867].

1-(3,5-Dihydroxy-4-methoxyphenyl)ethanone

[148204-58-4] $C_9H_{10}O_4$ mol.wt. 182.18

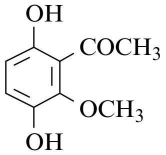


Isolation from natural sources

- Characterization in liquid wastes from eucalyptus wood and kraft lignin charring by flame-ionization gas-chromatography and gas-chromatography/mass-spectrometry [2868].

1-(3,6-Dihydroxy-2-methoxyphenyl)ethanone

[33539-20-7] $C_9H_{10}O_4$ mol.wt. 182.18



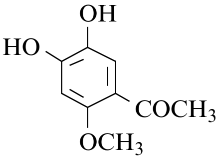
Syntheses

- Preparation from 2-hydroxy-6-methoxyacetophenone by persulfate oxidation (Elbs reaction) (26–33%) [2361,2780, 2781].
- Preparation by reduction of 2-acetyl-3-methoxy-1,4-benzoquinone using conventional methods [2869].
- Also obtained (low yield) by reaction of 2-acetyl-1,4-benzoquinone with an excess of methanol at r.t., with exclusion of light [2869].

m.p. 91° [2780], 90° [2361,2781,2869]; ^{1}H NMR [2374,2869], IR [2869], MS [2374].

1-(4,5-Dihydroxy-2-methoxyphenyl)ethanone

$C_9H_{10}O_4$ mol.wt. 182.18



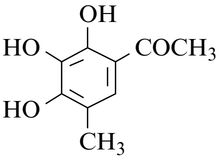
Synthesis

- Obtained by reaction of the Dakin solution on 2,4-diacetyl-5-methoxyphenol [2410].

m.p. 173–175° [2410].

1-(2,3,4-Trihydroxy-5-methylphenyl)ethanone

[59862-06-5] $C_9H_{10}O_4$ mol.wt. 182.18



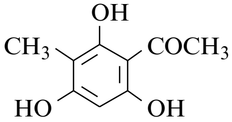
Syntheses

- Preparation by reaction of acetic anhydride and acetic acid on 1,2,3-trihydroxy-4-methylbenzene with zinc chloride at 140–145°, then deacylation of keto esters mixture obtained with boron trifluoride etherate in methanol (25%) [2276].
- Also obtained (poor yield) by alkaline degradation of a solution of D-xylose or D-glucose in 0.63 M sodium hydroxide at 96° under nitrogen [2276].

m.p. 167–168° [2276]; ^{1}H NMR [2276], IR [2276], MS [2276].

1-(2,4,6-Trihydroxy-3-methylphenyl)ethanone

[2657-28-5] $C_9H_{10}O_4$ mol.wt. 182.18



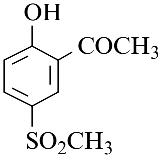
Syntheses

- Preparation by reaction of acetonitrile on 2-methyl-phloroglucinol (Hoesch reaction) [2027,2861,2870,2871], (83%) [2027], (70%) [2871], (33%) [2861].
- Preparation by reduction of 3-formylphloroacetophenone with hydrochloric acid and amalgamated zinc in gently heated aqueous methanol (53%) [2872].
- Preparation by demethylation of 2,4-dimethoxy-6-hydroxy-3-methylacetophenone,
  - with boron tribromide in methylene chloride at 0° (77%) [2873];
  - with aluminium chloride in refluxing benzene (87%) [2874].
- Preparation by reaction of 2-methylphloroglucinol with boron trifluoride-acetic acid complex at 28–30° (50%) [2242].
- Also obtained from phloroacetophenone using methyl iodide and alkali in methanolic solution [2276,2875–2878], (50%) [2876], (31%) [2877].

m.p. 211–212° [2861,2870,2873,2874], 211° [2871], 210–211° [2027,2872,2876], 210° [2242], 209–210° [2879], 205–206° [2877];
^1H NMR [2027,2873,2879], IR [2873], UV [2878], MS [2027].

1-[2-Hydroxy-5-(methylsulfonyl)phenyl]ethanone

[20951-24-0] $C_9H_{10}O_4S$ mol.wt. 214.24



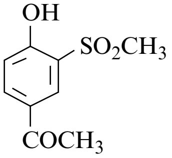
Synthesis

- Preparation by Fries rearrangement of 4-(methylsulfonyl)-phenyl acetate with aluminium chloride without solvent at 120° (42%) [1852].

m.p. 139–140° [1852].

1-[4-Hydroxy-3-(methylsulfonyl)phenyl]ethanone

[56490-43-8] $C_9H_{10}O_4S$ mol.wt. 214.24

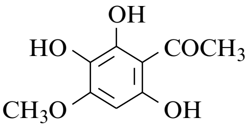


Synthesis

- Preparation by Fries rearrangement of 2-(methylsulfonyl)-phenyl acetate in the presence of aluminium chloride in nitrobenzene at 50–60° (75%) [2544].

m.p. 168–169° [2544].

1-(2,3,6-Trihydroxy-4-methoxyphenyl)ethanone



$C_9H_{10}O_5$ mol.wt. 198.18

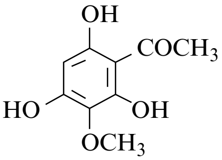
Syntheses

- Preparation by reduction of 2-acetyl-3-hydroxy-5-methoxy-quinone,
  - with sulfur dioxide in water [2880];
  - with sodium hydrosulfite in boiling water (59%) [2881].

m.p. 170–171° [2880,2881].

1-(2,4,6-Trihydroxy-3-methoxyphenyl)ethanone

[16297-01-1] $C_9H_{10}O_5$ mol.wt. 198.18



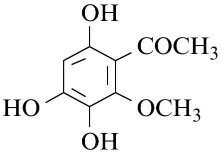
Syntheses

- Preparation by reaction of acetonitrile on 2-methoxy-1,3,5-trihydroxybenzene (2-methoxyphloroglucinol or iretol), (Hoesch reaction) [2882,2883], (71%) [2883].
- Also obtained (poor yield) by partial demethylation of 2,4-di-hydroxy-3,6-dimethoxyacetophenone with aluminium chloride in nitrobenzene at 100° [2884].

m.p. 188° [2884], 169–170° [2882], 168° [2883].

1-(3,4,6-Trihydroxy-2-methoxyphenyl)ethanone

[73239-52-8] $C_9H_{10}O_5$ mol.wt. 198.18



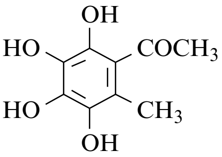
Synthesis

- Refer to: [2885].

TLC [2885].

1-(2,3,4,5-Tetrahydroxy-6-methylphenyl)ethanone

[66296-85-3] $C_9H_{10}O_5$ mol.wt. 198.18

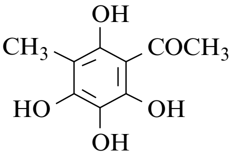


Synthesis

- Obtained (poor yield) by treatment of dihydroxyacetone in aqueous solution (pH 4.5) at 96° (2%) [2716].

m.p. 178–181° [2716]; ^{1}H NMR [2716], MS [2716].

1-(2,3,4,6-Tetrahydroxy-5-methylphenyl)ethanone



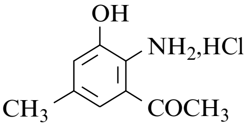
$C_9H_{10}O_5$ mol.wt. 198.18

Syntheses

- Preparation by reaction of sulfur dioxide on 2-acetyl-3,5-dihydroxy-6-methyl-2,5-cyclohexadiene-1,4-dione in dilute methanol (92%) [2886].
- Preparation by hydrolysis of 3-amino-2,4,6-trihydroxy-5-methylacetophenone hydrochloride (89%) [2886].
- Preparation by reaction of aluminium bromide on 2,5-dihydroxy-4,6-dimethoxy-3-methyl-acetophenone in chlorobenzene at 80–85° (61%) [2886].

m.p. 191–192° [2886].

1-(2-Amino-3-hydroxy-5-methylphenyl)ethanone *(Hydrochloride)*



$C_9H_{11}NO_2$, HCl mol.wt. 201.65

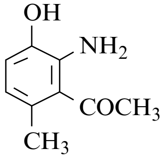
Synthesis

- Preparation by reaction of hydrogen on 3-hydroxy-5-methyl-2-nitroacetophenone with Raney nickel in methanol (80%) [2122].

m.p. 194–196° (d) [2122]; UV [2122].

1-(2-Amino-3-hydroxy-6-methylphenyl)ethanone

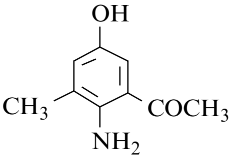
[38968-45-5] $C_9H_{11}NO_2$ mol.wt. 165.19



Syntheses

- Obtained (trace) by photolysis of 3,4-dimethylanthranil in 98% sulfuric acid [2454].
- Also obtained (trace) by thermal decomposition of 2-azido-6-methylacetophenone in 98% sulfuric acid [2454].

1-(2-Amino-5-hydroxy-3-methylphenyl)ethanone



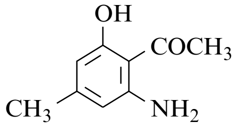
$C_9H_{11}NO_2$ mol.wt. 165.19

Synthesis

- Refer to: [2455] (compound **8e**).

1-(2-Amino-6-hydroxy-4-methylphenyl)ethanone

[97066-15-4] $C_9H_{11}NO_2$ mol.wt. 165.19



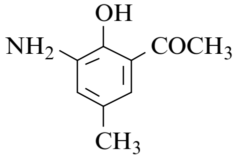
Synthesis

– Obtained by reaction of potassium hydroxide with 2-acetyl-3-amino-5-hydroxy-5-methyl-2-cyclohexenone in ethanol at 40° (44%) [2712].

m.p. 91–92° [2712]; IR [2712], UV [2712], MS [2712].

1-(3-Amino-2-hydroxy-5-methylphenyl)ethanone

[70977-71-8] $C_9H_{11}NO_2$ mol.wt. 165.19



Syntheses

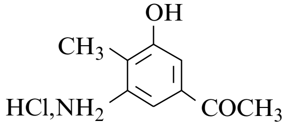
– Preparation by hydrogenation of 2-hydroxy-5-methyl-3-nitroacetophenone using 5% Pd/C catalyst in ethanol (68%) [1898], (48%) [1897].

– Preparation by reduction of 2-hydroxy-5-methyl-3-nitro-acetophenone with stannous chloride dihydrate in hydro-chloric acid (50%) [2586].

m.p. 71–72° [2586], 56–58° [1898], 55–57° [1897].

1-(3-Amino-5-hydroxy-4-methylphenyl)ethanone *(Hydrochloride)*

$C_9H_{11}NO_2$, HCl mol.wt. 201.65

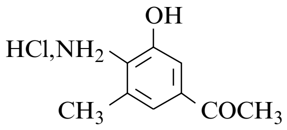


Synthesis

– Preparation by reduction of 3-hydroxy-4-methyl-5-nitro-acetophenone with tin and 25% hydrochloric acid heated under reflux on a steam bath (87%) [2581].

1-(4-Amino-3-hydroxy-5-methylphenyl)ethanone *(Hydrochloride)*

$C_9H_{11}NO_2$, HCl mol.wt. 201.65



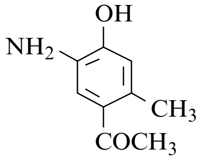
Synthesis

– Preparation by reaction of hydrogen on 3-hydroxy-5-methyl-4-nitroacetophenone with Raney nickel in methanol (92%) [2122].

m.p. 195–198° [2122]; UV [2122].

1-(5-Amino-4-hydroxy-2-methylphenyl)ethanone

$C_9H_{11}NO_2$ mol.wt. 165.19



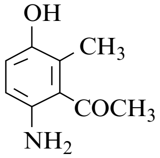
Synthesis

– Obtained by reduction of 4-hydroxy-2-methyl-5-nitro-acetophenone with sodium hydrosulfite in boiling alkaline solution (66%) [2887].

m.p. 116° [2887].

1-(6-Amino-3-hydroxy-2-methylphenyl)ethanone

[69976-76-7] $C_9H_{11}NO_2$ mol.wt. 165.19

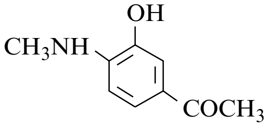


Synthesis

– Preparation by irradiation of 3,4-dimethyl-2,1-benzisoxazole in 96% sulfuric acid (91%) [2455].

1-[3-Hydroxy-4-(methylamino)phenyl]ethanone

[54903-57-0] $C_9H_{11}NO_2$ mol.wt. 165.19



Syntheses

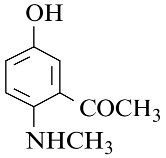
– Preparation from 6-acetyl-3-methylbenzoxazolinone by alkaline hydrolysis with boiling 10% aqueous sodium hydroxide solution (90–100%) [2471], (60%) [2888].

– Also refer to: [2472] (compound **VII**) and [2889].

m.p. 169–170° [2471,2888].

1-[5-Hydroxy-2-(methylamino)phenyl]ethanone

[63609-52-9] $C_9H_{11}NO_2$ mol.wt. 165.19



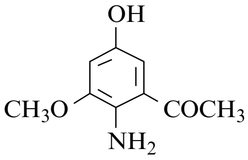
Synthesis

– Preparation by UV light irradiation of 1,3-dimethylindazole in aqueous sulfuric acid at 11–15° (38–44%) [2285,2467].

m.p. 142–143° [2467];
^{1}H NMR [2467], IR [2467], UV [2467], MS [2467].

1-(2-Amino-5-hydroxy-3-methoxyphenyl)ethanone

[126893-27-4] $C_9H_{11}NO_3$ mol.wt. 181.19



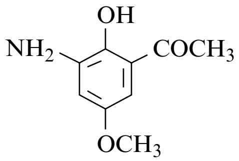
Synthesis

– Obtained (by-product) by reaction of stannous chloride on 3,5-dimethoxy-2-nitroacetophenone in concentrated hydrochloric acid at r.t. [2890].

m.p. 136–138° [2890];
^{1}H NMR [2890], ^{13}C NMR [2890], IR [2890];
UV [2890], MS [2890].

1-(3-Amino-2-hydroxy-5-methoxyphenyl)ethanone

[55008-15-6] $C_9H_{11}NO_3$ mol.wt. 181.19



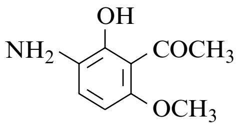
Syntheses

- Preparation by reduction of 2-hydroxy-5-methoxy-3-nitro-acetophenone with stannous chloride dihydrate in hydrochloric acid (47%) [2586].
- Preparation according to [2075] by [2778].

m.p. 107–108° [2586].

1-(3-Amino-2-hydroxy-6-methoxyphenyl)ethanone

[75452-86-7] $C_9H_{11}NO_3$ mol.wt. 181.19



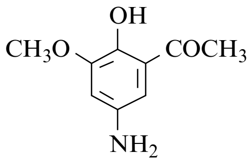
Synthesis

- Preparation by reaction of stannous chloride on 2-hydroxy-6-methoxy-3-nitroacetophenone in refluxing concentrated hydrochloric acid [2522,2523], (51%) [2523].

m.p. 66° [2523].

1-(5-Amino-2-hydroxy-3-methoxyphenyl)ethanone

$C_9H_{11}NO_3$ mol.wt. 181.19



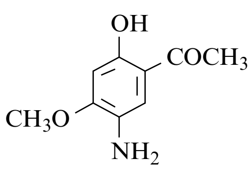
Synthesis

- Preparation from 2-hydroxy-3-methoxyacetophenone that was coupled with diazotized sulfanilic acid and the resulting azo dye reduced with sodium hydrosulfite to 5-amino-2-hydroxy-3-methoxyacetophenone (41%) [2848].

m.p. 145°5–147° [2848].

1-(5-Amino-2-hydroxy-4-methoxyphenyl)ethanone

$C_9H_{11}NO_3$ mol.wt. 181.19



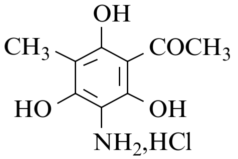
Synthesis

- Preparation by reduction of 2-hydroxy-4-methoxy-5-nitro-acetophenone [2530] with tin in concentrated hydrochloric acid heated in a water bath [1818,1836].

m.p. 113–114° [2530], 113° [1836], 112–113° [1818].

1-(3-Amino-2,4,6-trihydroxy-5-methylphenyl)ethanone *(Hydrochloride)*

$C_9H_{11}NO_4$, HCl mol.wt. 233.65



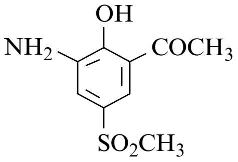
Synthesis

– Preparation by reaction of stannous chloride on 2,4,6-trihydroxy-3-methyl-5-phenylazoacetophenone in acetic acid at 85–90°, in the presence of concentrated hydrochloric acid [2886].

m.p. 210–211° [2886].

1-[3-Amino-2-hydroxy-5-(methylsulfonyl)phenyl]ethanone

[70977-88-7] $C_9H_{11}NO_4S$ mol.wt. 229.26

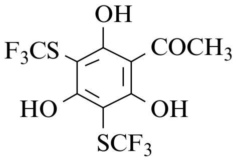


Synthesis

– Preparation by catalytic hydrogenation of 2-hydroxy-5-(methylsulfonyl)-3-nitroacetophenone in the presence of 5% Pd/C in ethanol at 25° [1898].

1-[2,4,6-Trihydroxy-3,5-bis[(trifluoromethyl)thio]phenyl]ethanone

[66625-04-5] $C_{10}H_6F_6O_4S_2$ mol.wt. 368.28



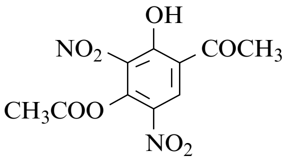
Synthesis

– Preparation by reaction of trifluoromethanesulfenyl chloride with phloroacetophenone in chloroform in the presence of a slight excess of pyridine and a small quantity of iron powder, first at –40°, then at 60° for 3 h (40%) [2480].

m.p. 113–115° [2480]; ^{1}H NMR [2480], IR [2480].

1-[4-(Acetyloxy)-2-hydroxy-3,5-dinitrophenyl]ethanone

$C_{10}H_8N_2O_8$ mol.wt. 284.18

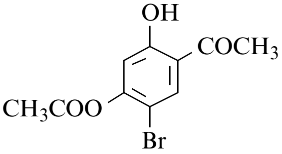


Synthesis

– Preparation by reaction of nitric acid on 4-acetoxy-2-hydroxyacetophenone in acetic acid or in acetic acid–ethanol mixture [1818].

m.p. 121–122° [1818].

1-[4-(Acetyloxy)-5-bromo-2-hydroxyphenyl]ethanone



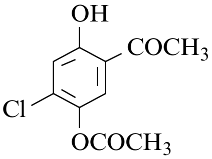
$C_{10}H_9BrO_4$ mol.wt. 273.08

Synthesis

- Preparation by reaction of bromine on 4-acetoxy-2-hydroxyacetophenone in 80% acetic acid at r.t. (34%) [1836,1989].

m.p. 85° [1989], 84° [1836]; ^{1}H NMR [1989].

1-[5-(Acetyloxy)-4-chloro-2-hydroxyphenyl]ethanone



$C_{10}H_9ClO_4$ mol.wt. 228.63

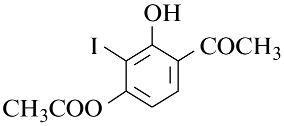
Syntheses

- Preparation by Fries rearrangement of 2-chlorohydroquinone diacetate with aluminium chloride [2024].
- Also obtained by partial acetylation of 4-chloro-2,5-di-hydroxyacetophenone [2024].

m.p. 129° [2024].

1-[4-(Acetyloxy)-2-hydroxy-3-iodophenyl]ethanone

[149810-09-3]



$C_{10}H_9IO_4$ mol.wt. 320.08

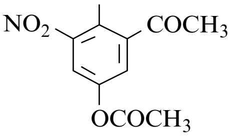
Synthesis

- Preparation by reaction of acetyl chloride with 2,4-di-hydroxy-3-iodoacetophenone in the presence of triethyl-amine in methylene chloride for 1 h at 0° and for 2.5 h at r.t. (77%) [2891].

m.p. 101–102° [2891]; ^{1}H NMR [2891], IR [2891], MS [2891].

1-[5-(Acetyloxy)-2-hydroxy-3-nitrophenyl]ethanone

[30095-73-9]



$C_{10}H_9NO_6$ mol.wt. 239.18

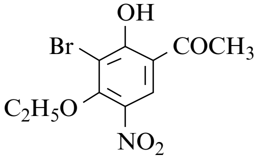
Synthesis

- Preparation by reaction of nitric acid (d = 1.5) on 5-acetoxy-2-hydroxyacetophenone in acetic acid at 10° (41%) [1832].

m.p. 112–113° [1832];
^{1}H NMR [1832], IR [1832], UV [1832].

1-(3-Bromo-4-ethoxy-2-hydroxy-5-nitrophenyl)ethanone

[134716-11-3] $C_{10}H_{10}BrNO_5$ mol.wt. 290.09

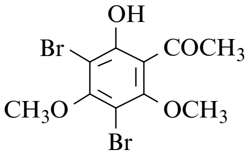


Synthesis

– Refer to: [2484] (compound **1h**).

1-(3,5-Dibromo-2-hydroxy-4,6-dimethoxyphenyl)ethanone

[3410-83-1] $C_{10}H_{10}Br_2O_4$ mol.wt. 354.00



Syntheses

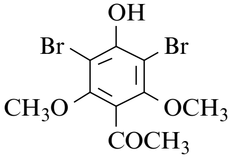
– Preparation by bromination of 2-hydroxy-4,6-dimethoxy-acetophenone (Xanthoxylin) in chloroform containing 6% of pyridine (96%) [2892].

– Also refer to: [2893].

m.p. 111° [2892]; IR [2892].

1-(3,5-Dibromo-4-hydroxy-2,6-dimethoxyphenyl)ethanone

[57393-65-4] $C_{10}H_{10}Br_2O_4$ mol.wt. 354.00



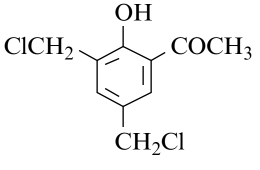
Synthesis

– Obtained (by-product) by reaction of bromine on 4-hydroxy-2,6-dimethoxyacetophenone in chloroform in the presence of aqueous sodium acetate solution (5%) [2894].

m.p. 117–118° [2894]; ^{1}H NMR [2894].

1-[3,5-Bis(chloromethyl)-2-hydroxyphenyl]ethanone

$C_{10}H_{10}Cl_2O_2$ mol.wt. 233.09



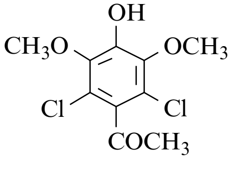
Synthesis

– Preparation by reaction of 37% formaldehyde solution on 2-hydroxyacetophenone with hydrochloric acid in a boiling water bath (81–87%) [2551].

m.p. 83° [2551].

1-(2,6-Dichloro-4-hydroxy-3,5-dimethoxyphenyl)ethanone

[94649-71-5] $C_{10}H_{10}Cl_2O_4$ mol.wt. 265.09



Syntheses

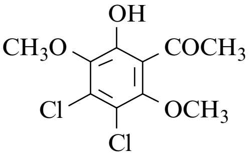
– Preparation by chlorination of acetosyringone (4-hydroxy-3,5-dimethoxyacetophenone) in dioxane with 2.0–2.5 mol. equiv. chlorine in acetic acid [2553,2554], (37%) [2553].

- Identified in wheat and rye straw pulp bleaching and combined mill effluents [1787].
- Identified on control of effluent from the manufacturing of bleached pulp and paper from sugarcane bagasse [1788].
- Also refer to: [2895].

m.p. 114–115° [2553,2554]; ^{1}H NMR [2553], ^{13}C NMR [2553], MS [2553].

1-(3,4-Dichloro-6-hydroxy-2,5-dimethoxyphenyl)ethanone

[88771-63-5] $C_{10}H_{10}Cl_2O_4$ mol.wt. 265.09



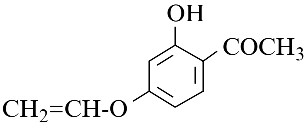
Synthesis

- Preparation by reaction of chlorine on 4-chloro-3,6-di-methoxy-2-hydroxyacetophenone in chloroform at r.t. (57%) [1884].

m.p. 96° [1884].

1-[4-(Ethenyloxy)-2-hydroxyphenyl]ethanone

[109661-95-2] $C_{10}H_{10}O_3$ mol.wt. 178.19



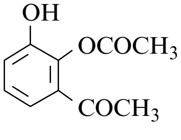
Synthesis

- Preparation by reaction of potassium tert-butoxide with 2-hydroxy-4-(2-chloroethoxy) acetophenone in refluxing tert-butanol (70%) [2295].

^{1}H NMR [2295], IR [2295].

1-[2-(Acetyloxy)-3-hydroxyphenyl]ethanone

[145723-28-0] $C_{10}H_{10}O_4$ mol.wt. 194.19



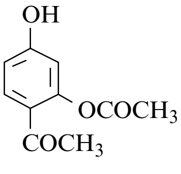
Synthesis

- Obtained by photooxygenation of 2,3-dimethyl-7-hydroxy-benzofuran in methylene chloride at −5° (13%) [2896].

m.p. 53–55° [2896];
^{1}H NMR [2896], ^{13}C NMR [2896], IR [2896], UV [2896].

1-[2-(Acetyloxy)-4-hydroxyphenyl]ethanone

[52751-42-5] $C_{10}H_{10}O_4$ mol.wt. 194.19



Syntheses

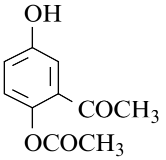
- Preparation by Fries rearrangement of resorcinol diacetate with ferric chloride in boiling acetic acid (46%) [2314].

- Also obtained by enzymatic deacylation of 2,4-diacetoxy-acetophenone with porcine pancreas lipase in tetrahydrofuran at 42–45° (80%) [2388,2897].

m.p. 119–120° [2314], 87–88° [2897]; ^{1}H NMR [2897].

1-[2-(Acetyloxy)-5-hydroxyphenyl]ethanone

[144152-29-4] $C_{10}H_{10}O_4$ mol.wt. 194.19



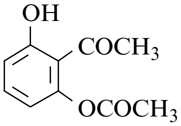
Syntheses

- Obtained by photooxygenation of 2,3-dimethyl-5-hydroxybenzofuran in methylene chloride at −5° (10%) [2896].
- Also obtained by alcoholysis of 2,5-diacetoxyacetophenone with n-butanol mediated by *Pseudomonas cepacia* lipase in cyclohexane/tert-amyl alcohol at 40° (65%) [2898].
- Also obtained by enzymatic deacylation of 2,5-diacetoxy-acetophenone with porcine pancreas lipase in tetrahydrofuran at 42–45° (60%) [2388,2897].

m.p. 105–106° [2896], 93–95° [2897];
^{1}H NMR [2896,2897], ^{13}C NMR [2896], IR [2896], UV [2896].

1-[2-(Acetyloxy)-6-hydroxyphenyl]ethanone

[26674-05-5] $C_{10}H_{10}O_4$ mol.wt. 194.19

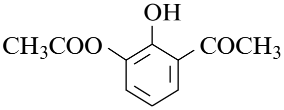


Syntheses

- Preparation by acylation of 2,6-dihydroxyacetophenone (compound **49**) [2899].
- Refer to: [2900]; this reference indicated in Chem. Abstr., **89**, 179792x (1978) for the monoacetate mentioned actually concerns the diacetate, that is to say the 1,3-diacetoxy-2-acetylbenzene.

1-[3-(Acetyloxy)-2-hydroxyphenyl]ethanone

[144224-87-3] $C_{10}H_{10}O_4$ mol.wt. 194.19

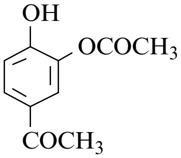


Synthesis

- Refer to: [2675].

1-[3-(Acetyloxy)-4-hydroxyphenyl]ethanone

[115436-75-4] $C_{10}H_{10}O_4$ mol.wt. 194.19



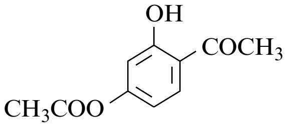
Synthesis

- Obtained (by-product) by reaction of acetic anhydride on pyrocatechol with zinc chloride at 145–150° (8%) [2184].

m.p. 74–76° [2184].

1-[4-(Acetyloxy)-2-hydroxyphenyl]ethanone

[42059-48-3] $C_{10}H_{10}O_4$ mol.wt. 194.19



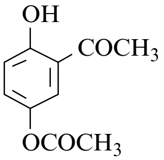
Syntheses

- Preparation by reaction of acetic anhydride on resaceto-phenone,
  - at reflux [1838,2129,2245,2901];
  - with sodium acetate [1833,2290,2613,2902], (78%) [2613];
  - with pyridine (95%) [2358], (54–59%) [1989,2901].
- Preparation by reaction of acetic acid on resacetophenone with polyphosphoric acid (39%) [2241].
- Also obtained by reaction of acetyl chloride on resorcinol at reflux [1838,2903].
- Also obtained (by-product) by reaction of acetic acid on resorcinol with polyphosphoric acid (3%) [2241].
- Also obtained by reaction of vinyl acetate on resacetophenone mediated by *Pseudomonas cepacia* lipase in cyclohexane/tert-amyl alcohol at 40° (55–65%) [2898].
- Also refer to: [2675].

m.p. 76° [2241], 75–76° [2358,2613], 75° [2290], 74° [2245,2314], 72–73° [1833,2901], 72° [2129,2902], 69–71° [1989];
b.p. 303° [2245,2901];
^{1}H NMR [1989,2898]; ^{13}C NMR [2898], IR [2898].

1-[5-(Acetyloxy)-2-hydroxyphenyl]ethanone

[21222-04-8] $C_{10}H_{10}O_4$ mol.wt. 194.19



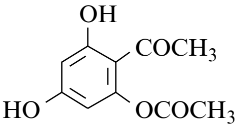
Syntheses

- Preparation by reaction of boiling acetic anhydride on quinacetophenone [2356].
- Preparation by reaction of acetic anhydride and sodium acetate mixture on quinacetophenone at 50° [2359].
- Preparation by Fries rearrangement of hydroquinone diacetate,
  - with boron trifluoride etherate at 120° (90%) [2338];
  - with aluminium chloride at 115–120° [2338,2348,2354] (predominantly) [2354], (31%) [2338].
- Also obtained by UV light irradiation of hydroquinone diacetate in methanol (15%) [2343].
- Also obtained by reaction of vinyl acetate on quinacetophenone mediated by *Pseudomonas cepacia* lipase in cyclohexane/tert-amyl alcohol at 40° (97%) [2898].
- Also obtained by alcoholysis of quinacetophenone diacetate with n-butanol mediated by *Pseudomonas cepacia* lipase in cyclohexane/tert-amyl alcohol at 40° (28%) [2898].

m.p. 91° [2356,2359], 83–84° [2343], 81–82° [2338], 80–85° [2354];
^{1}H NMR [2338,2343,2348,2898], ^{13}C NMR [2898];
IR [2338,2343,2348,2898], UV [2348], MS [2348]; pK_a [1977].

1-[2-(Acetyloxy)-4,6-dihydroxyphenyl]ethanone

[52751-41-4] $C_{10}H_{10}O_5$ mol.wt. 210.19

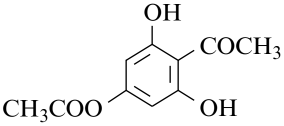


Synthesis

– Refer to: [2904].

1-[4-(Acetyloxy)-2,6-dihydroxyphenyl]ethanone

[29376-65-6] $C_{10}H_{10}O_5$ mol.wt. 210.19



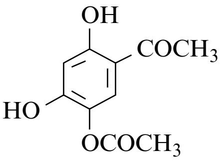
Synthesis

– Preparation by reaction of acetic anhydride on phloro-acetophenone in pyridine at r.t. (17–20%) [2368,2905].

m.p. 165–166° [2905]; ^{13}C NMR [2368].

1-[5-(Acetyloxy)-2,4-dihydroxyphenyl]ethanone

[55168-29-1] $C_{10}H_{10}O_5$ mol.wt. 210.19



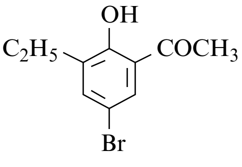
Syntheses

– Obtained by partial Fries rearrangement of 1,2,4-triacetoxy-benzene,
  - with boron trifluoride-acetic acid complex at 100° (78%) [2906];
  - with zinc chloride in acetic acid at 120° (21%) [2416] or at 140° (7%) [2899].

m.p. 171° [2416], 169–170° [2899], 163° [2906];
^{1}H NMR [2416,2899,2906], IR [2416,2899,2906].

1-(5-Bromo-3-ethyl-2-hydroxyphenyl)ethanone

[81591-17-5] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



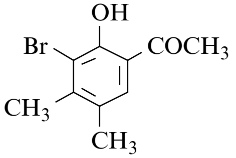
Synthesis

– Preparation by Fries rearrangement of 4-bromo-2-ethyl-phenyl acetate with aluminium chloride without solvent in an oil bath (40%) [2907].

m.p. 42° [2907].

1-(3-Bromo-2-hydroxy-4,5-dimethylphenyl)ethanone

[112747-62-3] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



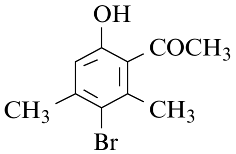
Syntheses

- Preparation by reaction of hydrated sodium sulfide (containing 7–9 mol of water) with 2-dimethylamino-4-[3-bromo-2-hydroxy-4,5-dimethylphenyl]-1,3-dithiole bisulfate in refluxing ethanol (85%) [1874].
- Preparation by reaction of bromine with 2-hydroxy-4,5-di-methylacetophenone in chloroform (69%) [2908].

m.p. 105–106° [1874], 100–101° [2908]; ^{1}H NMR [1874,2908], IR [1874,2908].

1-(3-Bromo-6-hydroxy-2,4-dimethylphenyl)ethanone

[71582-57-5] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



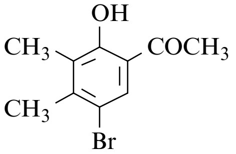
Syntheses

- Preparation by reaction of acetic acid on 4-bromo-3,5-di-methylphenol with boron trifluoride [2909].
- Preparation by Fries rearrangement of 4-bromo-3,5-di-methylphenyl acetate (b.p.$_{15}$ 110°) with aluminium chloride at 120° [2910].

m.p. 99–103° [2909], 95–97° [2910]; ^{1}H NMR [2910], IR [2910].

1-(5-Bromo-2-hydroxy-3,4-dimethylphenyl)ethanone

[319923-51-8] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



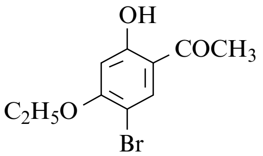
Synthesis

- Preparation by reaction of bromine with 2-hydroxy-3,4-di-methylacetophenone in methylene chloride (61%) [2908].

m.p. 65–66° [2908]; ^{1}H NMR [2908], IR [2908].

1-(5-Bromo-4-ethoxy-2-hydroxyphenyl)ethanone

[131359-44-9] $C_{10}H_{11}BrO_3$ mol.wt. 259.10

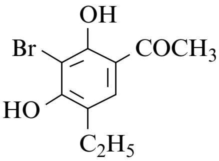


Syntheses

- Preparation by reaction of bromine on 4-ethoxy-2-hydroxyacetophenone in acetic acid [2293].
- Also refer to: [2484]–[2911] (compound **1g**).

m.p. 109–110° [2293].

1-(3-Bromo-5-ethyl-2,4-dihydroxyphenyl)ethanone



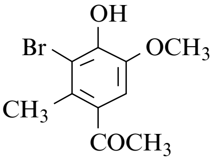
$C_{10}H_{11}BrO_3$ mol.wt. 259.10

Synthesis

- Preparation by bromination of 2,4-dihydroxy-5-ethylaceto-phenone with bromine in acetic acid [1839,2912] or in chloroform [2913].

m.p. 131° [2912], 123–125° [2913], 121° [1839].

1-(3-Bromo-4-hydroxy-5-methoxy-2-methylphenyl)ethanone



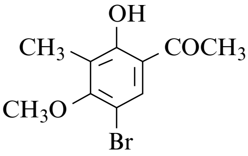
$C_{10}H_{11}BrO_3$ mol.wt. 259.10

Synthesis

- Preparation by reaction of acetyl chloride on 2-bromo-6-methoxy-3-methylphenol with aluminium chloride in carbon disulfide at 50° (50%) [2914].

m.p. 82° [2914].

1-(5-Bromo-2-hydroxy-4-methoxy-3-methylphenyl)ethanone



$C_{10}H_{11}BrO_3$ mol.wt. 259.10

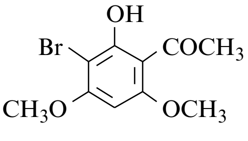
Synthesis

- Preparation by reaction of bromine on 2-hydroxy-4-methoxy-3-methylacetophenone in carbon disulfide [2915].

m.p. 63–64° [2915].

1-(3-Bromo-2-hydroxy-4,6-dimethoxyphenyl)ethanone

[18064-89-6]



$C_{10}H_{11}BrO_4$ mol.wt. 275.10

Syntheses

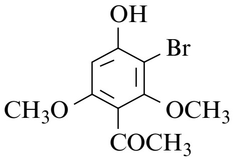
- Preparation by reaction of bromine on 2,4-dimethoxy-6-hydroxyacetophenone [2526,2916–2921],
  - in acetic acid at r.t. (98%) [2526], (43%) [2916];
  - in carbon tetrachloride (85%) [2526];
  - in acetic anhydride (50%) [2526];
  - in chloroform [2526,2918], (33%) [2526].
- Preparation by bromination of 2,4-dimethoxy-6-hydroxyacetophenone with cupric bromide in refluxing chloroform–ethyl acetate mixture [1845,2922], (62%) [2922].

- Preparation by reaction of cupric bromide on 2-acetoxy-4,6-dimethoxyacetophenone in refluxing chloroform–ethyl acetate mixture (76%) [2922].

m.p. 188–189° [2919], 187–189° [2922], 187° [2918,2920,2921], 186–187° [2526,2916]; ^{1}H NMR [1845,2526,2922], MS [1845].

1-(3-Bromo-4-hydroxy-2,6-dimethoxyphenyl)ethanone

[57517-42-7] $C_{10}H_{11}BrO_4$ mol.wt. 275.10



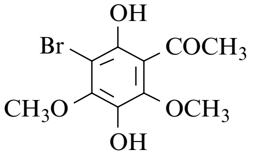
Synthesis

- Preparation by reaction of bromine on 4-hydroxy-2,6-di-methoxyacetophenone in chloroform in the presence of aqueous sodium acetate solution (88%) [2894].

m.p. 156–157° [2894]; ^{1}H NMR [2894].

1-(3-Bromo-2,5-dihydroxy-4,6-dimethoxyphenyl)ethanone

$C_{10}H_{11}BrO_5$ mol.wt. 291.10



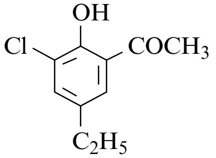
Synthesis

- This compound is obtained by reaction of alkaline potassium persulfate on 3-bromo-2-hydroxy-4,6-dimethoxy-acetophenone [2917].

m.p. 147–149° [2917].

1-(3-Chloro-5-ethyl-2-hydroxyphenyl)ethanone

[58483-48-0] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



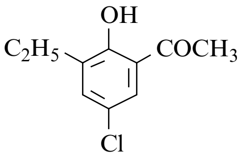
Synthesis

- Preparation by Fries rearrangement of 2-chloro-4-ethyl-phenyl acetate with aluminium chloride without solvent at 150° (75%) [2923].

m.p. 74° [2923].

1-(5-Chloro-3-ethyl-2-hydroxyphenyl)ethanone

[53347-06-1] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



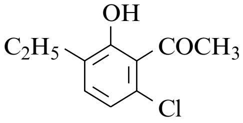
Synthesis

- Preparation by Fries rearrangement of 4-chloro-2-ethyl-phenyl acetate with aluminium chloride without solvent at 120° [2008] or by heating in an oil bath (50%) [2907].

oil [2008]; b.p.$_{12}$ 145–146° [2008], b.p. 265° [2907].

1-(6-Chloro-3-ethyl-2-hydroxyphenyl)ethanone

[81591-14-2] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



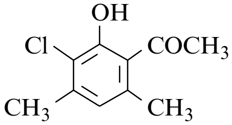
Synthesis

- Preparation by Fries rearrangement of 5-chloro-2-ethyl-phenyl acetate with aluminium chloride without solvent by heating in an oil bath (40%) [2907].

b.p. 258° [2907].

1-(3-Chloro-2-hydroxy-4,6-dimethylphenyl)ethanone

[71582-56-4] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



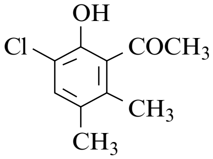
Synthesis

- Preparation by reaction of acetic acid on 2-chloro-3,5-di-methylphenol with boron trifluoride [2909].

m.p. 74–75° [2909].

1-(3-Chloro-2-hydroxy-5,6-dimethylphenyl)ethanone

[90743-03-6] $C_{10}H_{11}ClO_2$ mol.wt. 198.65

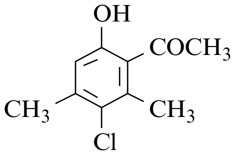


Synthesis

- Refer to: [1885] (ketone 16).

1-(3-Chloro-6-hydroxy-2,4-dimethylphenyl)ethanone

[50343-13-0] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



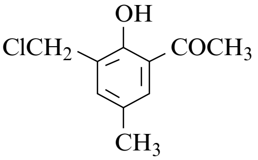
Syntheses

- Preparation by Fries rearrangement of 4-chloro-3,5-di-methylphenyl acetate with aluminium chloride without solvent between 120° and 155° [2011,2909, 2924], (90%) [2924].
- Preparation by reaction of aluminium chloride on 3-chloro-6-methoxy-2,4-dimethylacetophenone without solvent at 140–150° [1811].
- Also obtained by ozonization of 6-chloro-3,4,5,7-tetramethylcoumarin in ethyl acetate, followed first by hydrolysis of the ozonide so formed, then saponification of the resulting oil by sodium hydroxide in refluxing aqueous methanol for 1 h (59%) [2925].

m.p. 110–112° [2925], 106–109° [1811], 94° [2924], 89°7 [2011].

1-[3-(Chloromethyl)-2-hydroxy-5-methylphenyl]ethanone

[87165-62-6] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



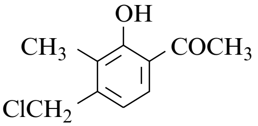
Syntheses

- Preparation from 2-hydroxy-5-methylacetophenone by introduction of the chloromethyl group into aromatic ring by treatment with formaldehyde and hydrogen chloride at 70° [2926] according to [2546].
- Also refer to: [2481] (compound **11**).

m.p. 59–60° [2926]; b.p.$_{0.4}$ 110–116° [2494]; ^{1}H NMR [2926].

1-[4-(Chloromethyl)-2-hydroxy-3-methylphenyl]ethanone

[97582-37-1] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



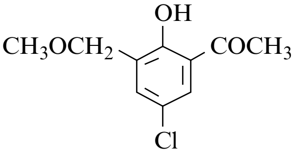
Synthesis

- Preparation by reaction of ethyl chloroformate with 4-(dimethylaminomethyl)-2-hydroxy-3-methylacetophenone in toluene [2550,2927], (72%) [2550].

m.p. 57–59° [2550]; b.p.$_3$ 190–200° [2927]; ^{1}H NMR [2927], IR [2927].

1-[5-Chloro-2-hydroxy-3-(methoxymethyl)phenyl]ethanone

[87165-59-1] $C_{10}H_{11}ClO_3$ mol.wt. 214.65



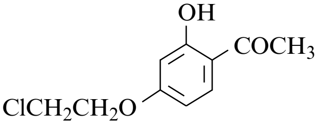
Synthesis

- Obtained by reaction of sodium methoxide with 5-chloro-3-chloromethyl-2-hydroxyacetophenone in refluxing methanol for 2 h (28%) [2494].

b.p.$_{0.6}$ 121–131° [2494]; ^{1}H NMR [2494], IR [2494].

1-[4-(2-Chloroethoxy)-2-hydroxyphenyl]ethanone

[109661-96-3] $C_{10}H_{11}ClO_3$ mol.wt. 214.65



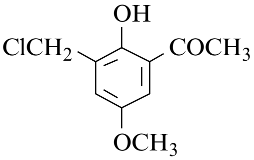
Synthesis

- Preparation by reaction of 1,2-ethylene dichloride with resacetophenone in the presence of n-tetrabutylammonium bromide and potassium hydroxide in water at 60° (41%) [2295].

m.p. 98–100° [2295]; ^{1}H NMR [2295].

1-[3-(Chloromethyl)-2-hydroxy-5-methoxyphenyl]ethanone

[87165-70-6] $C_{10}H_{11}ClO_3$ mol.wt. 214.65



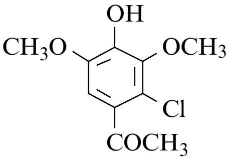
Synthesis

- Preparation by reaction of polyoxymethylene with 2-hydroxy-5-methoxyacetophenone in the presence of concentrated hydrochloric acid at 50° (61%) [2494].

m.p. 71° [2494]; ^{1}H NMR [2494], IR [2494].

1-(2-Chloro-4-hydroxy-3,5-dimethoxyphenyl)ethanone

[94649-70-4] $C_{10}H_{11}ClO_4$ mol.wt. 230.65



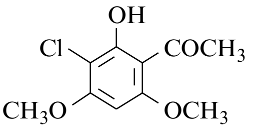
Syntheses

- Preparation by chlorination of acetosyringone (4-hydroxy-3,5-dimethoxyacetophenone) in dioxane with 1.0–1.1 mol. equiv. chlorine in acetic acid [2553,2554], (24%) [2553].
- Identified in wheat and rye straw pulp bleaching and combined mill effluents [1787].
- Identified on control of effluent from the manufacturing of bleached pulp and paper from sugarcane bagasse [1788].
- Isolated from the pyrolysis products of beech wood [2928].

m.p. 93–94° [2553,2554]; GC-MS [2928];
^{1}H NMR [2553], ^{13}C NMR [2553], MS [2553].

1-(3-Chloro-2-hydroxy-4,6-dimethoxyphenyl)ethanone

[81325-85-1] $C_{10}H_{11}ClO_4$ mol.wt. 230.65



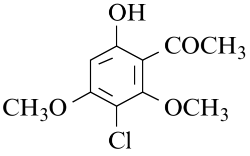
Syntheses

- Preparation by Fries rearrangement of 2-chloro-3,5-di-methoxyphenyl acetate with aluminium chloride in chloro-benzene at reflux (94%) [2929] or in nitrobenzene, from 90° to 130° (24%) [2930].
- Preparation by reaction of acetyl chloride with 2-chloro-3,5-dimethoxyphenol in the presence of aluminium chloride in nitrobenzene at r.t. (87%) [2858].
- Also obtained by reaction of chlorine with 2-hydroxy-4,6-dimethoxyacetophenone in carbon tetrachloride (15%) [2139].
- Preparation by diazotization of 3-amino-2-hydroxy-4,6-dimethoxyacetophenone and treating with cuprous chloride (Sandmeyer reaction) [2139].
- Preparation by partial demethylation of 3-chloro-2,4,6-trimethoxyacetophenone with aluminium chloride in acetonitrile for 4 h at 30° (95%) [2747].
- Also refer to: [2931].

m.p. 191–192° [2139,2858], 188° [2930]; ^{1}H NMR [2139,2930], IR [2858].

1-(3-Chloro-6-hydroxy-2,4-dimethoxyphenyl)ethanone

[81325-86-2] $C_{10}H_{11}ClO_4$ mol.wt. 230.65



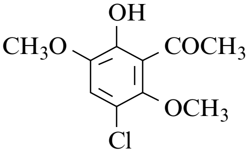
Syntheses

- Preparation by reaction of chlorine with 2-hydroxy-4,6-di-methoxyacetophenone in carbon tetrachloride (57%) [2139].
- Also obtained (poor yield) by Fries rearrangement of 4-chloro-3,5-dimethoxyphenyl acetate with aluminium chloride in nitrobenzene at 90° and then 130° (11%) [2932].
- Also refer to: [2931,2933].

m.p. 193–194° [2932], 91° [2139]; ^{1}H NMR [2139,2932].

1-(3-Chloro-6-hydroxy-2,5-dimethoxyphenyl)ethanone

[88771-58-8] $C_{10}H_{11}ClO_4$ mol.wt. 230.65



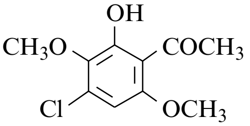
Synthesis

- Preparation by reaction of N-chlorosuccinimide on 3,6-dimethoxy-2-hydroxyacetophenone in refluxing carbon tetrachloride (59%) [1884].

m.p. 108° [1884]; ^{1}H NMR [1884].

1-(4-Chloro-2-hydroxy-3,6-dimethoxyphenyl)ethanone

[88771-46-4] $C_{10}H_{11}ClO_4$ mol.wt. 230.65

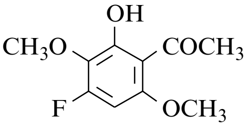


Synthesis

- Refer to: [1884].

1-(4-Fluoro-2-hydroxy-3,6-dimethoxyphenyl)ethanone

[88771-57-7] $C_{10}H_{11}FO_4$ mol.wt. 214.19

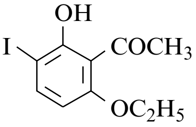


Synthesis

- Refer to: [1884].

1-(6-Ethoxy-2-hydroxy-3-iodophenyl)ethanone

[35292-36-5] $C_{10}H_{11}IO_3$ mol.wt. 306.10



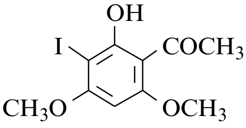
Synthesis

- Preparation by adding an aqueous solution of iodic acid and iodine to an ethanolic solution of 2-ethoxy-6-hydroxyaceto-phenone (88%) [2569].

m.p. 106° [2569].

1-(2-Hydroxy-3-iodo-4,6-dimethoxyphenyl)ethanone

[59656-68-7] $C_{10}H_{11}IO_4$ mol.wt. 322.10



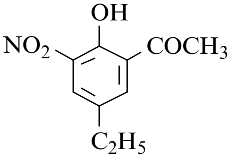
Syntheses

- Preparation by adding an ethanolic solution of iodine and phloroacetophenone dimethyl ether to an aqueous solution of iodic acid at r.t. (81%) [2934].
- Preparation by adding an acetic acid solution of nitric acid to an acetonic solution of iodine and phloroacetophenone dimethyl ether at 0° [2066,2935,2936], (75%) [2936].
- Preparation by reaction of iodine with phloroacetophenone dimethyl ether in methanol in the presence of potassium hydroxide [2937].
- Also refer to: [2931] (compound **23**).

m.p. 201° [2936], 199°5–200°5 [2934].

1-(5-Ethyl-2-hydroxy-3-nitrophenyl)ethanone

[71002-71-6] $C_{10}H_{11}NO_4$ mol.wt. 209.20



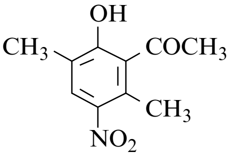
Syntheses

- Preparation by nitration of 5-ethyl-2-hydroxyacetophenone,
  - with 100% nitric acid in acetic acid at r.t. (85%) [1852];
  - with nitric acid (d = 1.42) in concentrated sulfuric acid between −15° and −5° [1897,1898], (44%) [1897].

m.p. 127–128° [1852], 120–122° [1898], 118–120° [1897].

1-(2-Hydroxy-3,6-dimethyl-5-nitrophenyl)ethanone

[207281-53-6] $C_{10}H_{11}NO_4$ mol.wt. 209.20

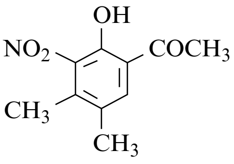


Synthesis

- Refer to: [2938].

1-(2-Hydroxy-4,5-dimethyl-3-nitrophenyl)ethanone

$C_{10}H_{11}NO_4$ mol.wt. 209.20



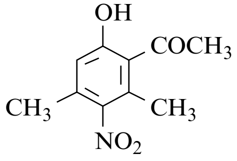
Synthesis

- Preparation by reaction of nitric acid (d = 1.42) on 2-hydroxy-4,5-dimethylacetophenone in acetic acid at r.t. (71%) [2939].

m.p. 143–144° [2939].

1-(6-Hydroxy-2,4-dimethyl-3-nitrophenyl)ethanone

[125249-30-1] $C_{10}H_{11}NO_4$ mol.wt. 209.20



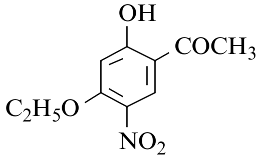
Synthesis

- Preparation is made by adding successively nitromethane, then 3-acetyl-2,6-dimethyl-4*H*-pyran-4-one in a suspension of potassium tert-butoxide in tert-butyl alcohol at 40° under nitrogen (40%) [2940].

m.p. 112–114° [2940]; ^{1}H NMR [2940], IR [2940], MS [2940].

1-(4-Ethoxy-2-hydroxy-5-nitrophenyl)ethanone

[76951-07-0] $C_{10}H_{11}NO_5$ mol.wt. 225.20



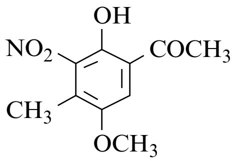
Syntheses

- Prepared by nitration of 2-hydroxy-4-ethoxyacetophenone in glacial acetic acid [2941].
- Also refer to: [2484,2911] (compound **1f**).

m.p. 132° [2941].

1-(2-Hydroxy-5-methoxy-4-methyl-3-nitrophenyl)ethanone

[43140-82-5] $C_{10}H_{11}NO_5$ mol.wt. 225.20



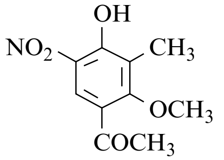
Synthesis

- Preparation by reaction of 10% nitric acid with 2-hydroxy-5-methoxy-4-methylacetophenone at 17–20° (40%) [2583].

m.p. 132° [2583]; ^{1}H NMR [2583], IR [2583].

1-(4-Hydroxy-2-methoxy-3-methyl-5-nitrophenyl)ethanone

[118824-98-9] $C_{10}H_{11}NO_5$ mol.wt. 225.20

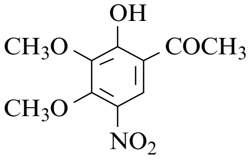


Synthesis

- Preparation by reaction of concentrated nitric acid on 4-hydroxy-2-methoxy-3-methylacetophenone in acetic acid [2582,2942], (64%) [2582].

m.p. 70° [2942], 69° [2582]; ^{1}H NMR [2582], IR [2582], MS [2582].

1-(2-Hydroxy-3,4-dimethoxy-5-nitrophenyl)ethanone



$C_{10}H_{11}NO_6$ mol.wt. 241.20

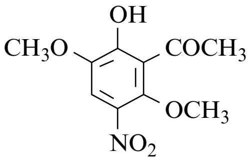
Synthesis

- Preparation by reaction of nitric acid with 3,4-dimethoxy-2-hydroxyacetophenone in ethanol (33%) [2814].

m.p. 83–83°8 [2814].

1-(2-Hydroxy-3,6-dimethoxy-5-nitrophenyl)ethanone

[88771-59-9]



$C_{10}H_{11}NO_6$ mol.wt. 241.20

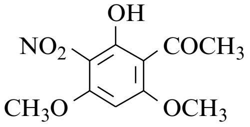
Synthesis

- Preparation by reaction of nitric acid on 3,6-dimethoxy-2-hydroxyacetophenone in acetic acid at 10° (52%) [1884].

m.p. 120° [1884]; ^{1}H NMR [1884].

1-(2-Hydroxy-4,6-dimethoxy-3-nitrophenyl)ethanone

[81325-87-3]



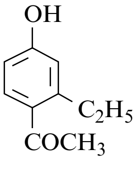
$C_{10}H_{11}NO_6$ mol.wt. 241.20

Synthesis

- Obtained (poor yield) by adding nitric acid (d = 1.42) in acetic acid to an ice-cold solution of 2-hydroxy-4,6-dimethoxyacetophenone in acetic anhydride (2%) [2139].

m.p. 104–105° [2139]; ^{1}H NMR [2139].

1-(2-Ethyl-4-hydroxyphenyl)ethanone



$C_{10}H_{12}O_2$ mol.wt. 164.20

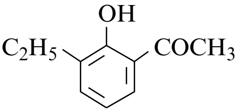
Syntheses

- Obtained by Fries rearrangement of 3-ethylphenyl acetate with aluminium chloride,
  - in nitrobenzene at 0° (varying yield, maximum 50%) [2943];
  - without solvent between 130° and 165° (4–7%) [2152,2944].

m.p. 102° [2943,2944]; b.p.$_{0.45}$ 150–152° [2943], b.p.$_{20}$ 195–200° [2944].

1-(3-Ethyl-2-hydroxyphenyl)ethanone

[103323-22-4] $C_{10}H_{12}O_2$ mol.wt. 164.20



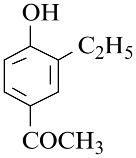
Syntheses

- Obtained by Fries rearrangement of 2-ethylphenyl acetate with aluminium chloride at 130–140° [2233].
- Also refer to: [2080,2945].

b.p.$_{3}$ 90–95° [2945], b.p. 213° [2233].

1-(3-Ethyl-4-hydroxyphenyl)ethanone

[22934-47-0] $C_{10}H_{12}O_2$ mol.wt. 164.20



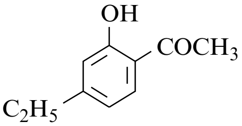
Syntheses

- Preparation by reaction of acetyl chloride on 2-ethylphenol with aluminium chloride,
  - in refluxing carbon disulfide (60%) [1951];
  - in nitrobenzene at 60° (39%) [1964].
- Preparation by acetylation of 2-ethylphenol (60%) [2946].

m.p. 95° [2946], 92–93° [1964], 89–91° [1951];
b.p.$_{1}$ 170–180° [2946], b.p.$_{20}$ 190–195° [1964]; UV [1964].

1-(4-Ethyl-2-hydroxyphenyl)ethanone

[5896-50-4] $C_{10}H_{12}O_2$ mol.wt. 164.20



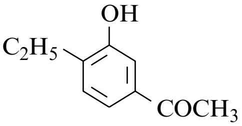
Syntheses

- Preparation by Fries rearrangement of 3-ethylphenyl acetate,
  - with aluminium chloride, without solvent between 120° and 165° (80–88%) [2152,2601,2944,2947–2949], in nitrobenzene at 25° (84–89%) [2948,2949] or at 60° (65%) [2947], in toluene or in xylene at 100° (56–57%) [2947];
  - with titanium tetrachloride, without solvent at 120° (88%) [2948] or in nitrobenzene at 25° (66%) [2948];
  - with stannic chloride, without solvent at 120° (84%) [2948] or in nitrobenzene at 25° (86%) [2948];
  - with zinc chloride, without solvent at 120° (56%) [2948] or in nitrobenzene at 25° (58%) [2948].

b.p.$_{2.5}$ 97° [2601], b.p.$_{4}$ 140° [2949], b.p.$_{20}$ 142° [2944].

1-(4-Ethyl-3-hydroxyphenyl)ethanone

[73898-20-1] $C_{10}H_{12}O_2$ mol.wt. 164.20



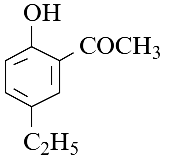
Synthesis

– Preparation by diazotization of 3-amino-4-ethylacetophenone followed by hydrolysis of the diazonium salt obtained (46%) [1951].

m.p. 94–95° [1951].

1-(5-Ethyl-2-hydroxyphenyl)ethanone

[24539-92-2] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

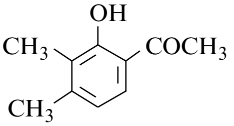
– Preparation by Fries rearrangement of 4-ethylphenyl acetate with aluminium chloride without solvent at 115–120° [1852,1869,2233], (96%) [1852], (70%) [2233].

– Preparation by reaction of acetyl chloride on 4-ethylphenol with aluminium chloride in ethylene dichloride at 110–120° (71%) [2625].

b.p.$_{2.3}$ 94–96° [1869], b.p.$_4$ 102–104° [2625], b.p.$_{10}$ 114–116° [1852], b.p.$_{12}$ 119–121° [2233]; ^{1}H NMR [1869], MS [1869].

1-(2-Hydroxy-3,4-dimethylphenyl)ethanone

[5384-55-4] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

– Preparation by reaction of acetic anhydride on 2,3-di-methylphenol with 70% perchloric acid at 125–135° (35%) [2306].

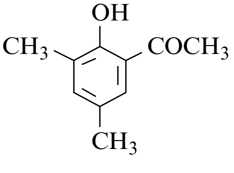
– Preparation by Fries rearrangement of 2,3-dimethylphenyl acetate,

- with aluminium chloride, without solvent, between 100° and 165° (85%) [2950], (70–80%) [1814,2951,2952], (54–69%) [2233,2953–2955] or in refluxing carbon disulfide (20%) [2952];
- with titanium tetrachloride at 100° (60%) [1814,2161,2951].

colourless oil [2950]; m.p. 6–8° [2951];
b.p.$_7$ 105–110° [2306], b.p.$_8$ 120–124° [2955], b.p.$_{12}$ 122–124° [2233];
b.p.$_{10.5}$ 127–129° [2955], b.p.$_{15}$ 131–132° [2951], b.p.$_{12}$ 140° [2953];
^{1}H NMR [2950], ^{13}C NMR [2950], IR [2950,2951], MS [2950].

1-(2-Hydroxy-3,5-dimethylphenyl)ethanone

[1198-66-9] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

– Preparation by reaction of acetyl chloride on 2,4-dimethylphenol with aluminium chloride in nitrobenzene at 50° (80%) [2181].

- Preparation by Fries rearrangement of 2,4-dimethylphenyl acetate with aluminium chloride, in nitrobenzene at 50° (80%) [2181] or in the presence of 2,4-dimethylanisole in refluxing carbon disulfide (50%) [2535] or without solvent [1814,2161,2535,2596,2697] at 130–140° (good yield) [2535], (42%) [2697].
- Also obtained by Fries rearrangement of 2,5-dimethylphenyl acetate with aluminium chloride without solvent [2596,2956], at 80–90° (25%) [2956].
- Also obtained (by-product) by Fries rearrangement of 2,6-dimethyl-4-ethylphenyl acetate with aluminium chloride without solvent (4%) [2957].
- Also obtained by reaction of acetyl chloride on 2,4-dimethylanisole with aluminium chloride in refluxing carbon disulfide [2956].
- Also obtained by reaction of zinc powder on 3,5-bis(chloromethyl)-2-hydroxyacetophenone in aqueous acetic acid (8%) [2551].
- Preparation by UV light irradiation of 2,4-dimethylphenyl acetate at 25°, in benzene (54%) [2198] or in hexane, with potassium carbonate (90%) [2015] or without potassium carbonate (34%) [2015].
- Preparation by reaction of ethyl acetoacetate with 2-methyl-2-pentenal in the presence of pyridine and piperidine as catalysts, in refluxing benzene (49%). The 2-methyl-2-pentenal was first obtained by self-condensation of propionaldehyde in the presence of 15% potassium hydroxide solution [2958–2960].
- Also refer to: [2961,2962].

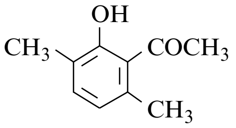
m.p. 55° [2015], 54° [2181,2535,2956], 53–54° [2161,2551,2596], 53–53°5 [2958–2960];

b.p.$_{12}$ 106–135° [2956], b.p.$_{16}$ 124°5–126° [2161], b.p.$_{33}$ 126–140° [2958,2959,2960];

^{1}H NMR [2198,2958–2960], IR [2198,2958–2960], UV [2958–2960].

1-(2-Hydroxy-3,6-dimethylphenyl)ethanone

[90743-02-5] $C_{10}H_{12}O_2$ mol.wt. 164.20



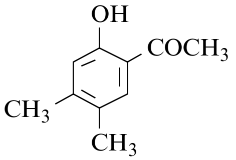
Syntheses

- Obtained by UV irradiation of 2,5-dimethylphenyl acetate in methanol at 254 nm under nitrogen at r.t. (39%) [2963].
- Also refer to: [2964] and [1885] (ketone **14**).

viscous oil [2963]; ^{1}H NMR [2963], ^{13}C NMR [2963], IR [2963], MS [2963].

1-(2-Hydroxy-4,5-dimethylphenyl)ethanone

[36436-65-4] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

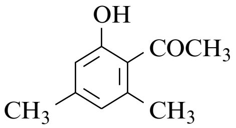
- Preparation by Fries rearrangement of 3,4-dimethylphenyl acetate with aluminium chloride without solvent between 110° and 150° [1814,2212,2596,2939, 2952,2965–2967], (86–100%) [2965–2967], (70–72%) [2939,2952], (23%) [2212].

- Preparation by isomerisation of 2-hydroxy-4,6-dimethylacetophenone with an excess of aluminium chloride without solvent at 140–180° (quantitative yield) [2965].
- Preparation by reaction of acetic acid on 3,4-dimethylphenol,
    - with boron trifluoride at 70° (80%) [2611];
    - with polyphosphoric acid (75%) [2966].
- Preparation by demethylation of 2-methoxy-4,5-dimethylacetophenone with pyridinium chloride at reflux (81%) [2542].
- Preparation by dehydrogenation of 6-acetyl-3,4-dimethyl-2-cyclohexen-1-one,
    - with a 5% palladium-barium sulfate catalyst at reflux [2612];
    - with refluxing 16% solution of bromine in acetic acid [2612].
- Also obtained by reaction of sodium methoxide on 2-acetyl-4,5-dimethyl-4-nitro-1,4-dihydro-phenyl acetate in methanol [2968].
- Also obtained (by-product) by reaction of aluminium chloride on 2,4,5-trimethylphenyl acetate (pseudocumenol acetate) without solvent at 130–140° [2596].

m.p. 74° [2965], 71°5–72°5 [2612], 71–72°5 [2968], 71° [2596,2611,2952,2967], 70°9–71°7 [2212], 70–71° [2939], 70° [2542];
b.p.$_{18}$ 143–144° [2611];
^{1}H NMR [1820,2968], ^{13}C NMR [1821], IR [2212,2968], UV [1820], fluorescence spectra [1820], MS [2968].

1-(2-Hydroxy-4,6-dimethylphenyl)ethanone

[16108-50-2] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

- Preparation by reaction of acetic acid on 3,5-dimethylphenol with boron trifluoride at 60–70° (93%) [2611].
- Preparation by Fries rearrangement of 3,5-dimethylphenyl acetate,
    - with aluminium chloride,
        - without solvent, between 100° and 150° [1814,2947,2948,2955,2965,2969–2973], (81–100%) [2947,2948,2965], (67–75%) [2955,2972];
        - in nitrobenzene at 25° (78%) [2948] or at 60° (67%) [2947];
        - in toluene or xylene at 100° (61–62%) [2947];
        - in refluxing carbon disulfide (60%) [2969].
    - with titanium tetrachloride,
        - without solvent at 120° (82%) [2948];
        - in nitrobenzene at 25° (86%) [2948].

- with stannic chloride,
  - without solvent at 120° (78%) [2948];
  - in nitrobenzene at 25° (76%) [2948].
- with zinc chloride,
  - without solvent at 120° (54%) [2948];
  - in nitrobenzene at 25° (52%) [2948].

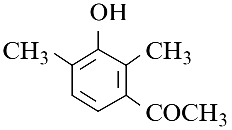
- Also obtained by reaction of 2 N sodium hydroxide on 4,6-dimethyl-2-hydroxy-α,α,α-trifluoro-acetoacetophenone at r.t. (quantitative yield) [2629].
- Also obtained by reaction of acetyl chloride,
  - with 3,5-dimethylanisole with aluminium chloride (60–70%) [2974];
  - with 3,5-dimethylphenol in refluxing carbon disulfide (60%) [2969].
- Also obtained by heating on a steam bath a mixture of 2-acetoxy-4,6-dimethylacetophenone and aluminium chloride (33%) [2973].
- Also obtained by self-condensation of acetylacetone,
- with refluxing 2 N sodium hydroxide, then by heating the residue at 145° [2975];
- catalyzed with potassium fluoride in DMF solution [2648,2976], (64%) [2976]; also refer to "erratum" [2647].
- Also obtained by reaction of acetic anhydride on 3,5-dimethylanisole with aluminium chloride in refluxing carbon disulfide (46%) [2977].
- Preparation by dehydrogenation of 6-acetyl-3,5-dimethyl-2-cyclohexen-1-one with a 5% palladium-barium sulfate catalyst at reflux [2612].
- Also obtained by photoreaction of dehydroacetic acid followed by hydrolysis of the obtained dimer (46%) [2973].
- Also obtained by UV light irradiation of 3,5-dimethylphenyl acetate at 25°, in isopropanol or cyclohexane (32%) [2193] or in ethyl ether (9%) [2193].
- Also refer to: [2025].

m.p. 62° [2611], 60° [2965], 59–60° [2971], 58–59° [2612], 58° [2970,2975], 57–58°5 [2955], 57–58° [2969,2974], 56–58° [2648,2973], 55–56° [2647,2976];

b.p.$_{18}$ 140–141° [2969,2974], b.p.$_{15}$ 140°5–141°5 [2611], b.p.$_{13}$ 144° [2955];
^{1}H NMR [2647,2648,2976], ^{13}C NMR [2647,2976,2978], IR [2647,2970,2976];
MS [2647,2976]; pK$_a$ [2516].

1-(3-Hydroxy-2,4-dimethylphenyl)ethanone

[99892-63-4] $C_{10}H_{12}O_2$ mol.wt. 164.20



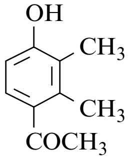
Synthesis

- The 1α,6α,8α-trimethyl-2α-*H*, 4α-*H*, 5α-*H* -3,9-dioxa-tricyclo[3.3.1.0 ^{2,4}]nonan-7-one was rearranged by treatment with sodium ethoxide in ethanol to form 3-hydroxy-2,4-dimethylacetophenone in 11% yield [2979].

m.p. 70° [2979]; ^{1}H NMR [2123], IR [2123], MS [2123].

1-(4-Hydroxy-2,3-dimethylphenyl)ethanone

[5384-57-6] $C_{10}H_{12}O_2$ mol.wt. 164.20



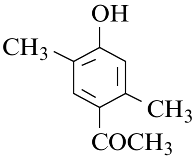
Syntheses

– Obtained by Fries rearrangement of 2,3-dimethylphenyl acetate,
  - with aluminium chloride, in nitrobenzene at r.t. (50–60%) [2952,2953], in carbon disulfide at r.t. (10%) [2952] or without solvent at 100° (17%) [2951,2954];
  - with titanium tetrachloride without solvent at 100° (6%) [2951,2954].

m.p. 148° [2952], 145° [2953], 144° [2951].

1-(4-Hydroxy-2,5-dimethylphenyl)ethanone

[26216-10-4] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

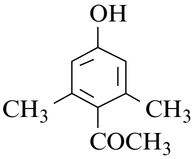
– Preparation by reaction of acetic acid on 2,5-dimethylphenol with boron trifluoride at 70° (95%) [2611].

– Preparation by Fries rearrangement of 2,5-dimethylphenyl acetate with aluminium chloride without solvent at 80–90° (65–70%) [2596,2628], (49%) [2980].

m.p. 131–132° [2611,2980], 130–131° [2596,2628]; ^{1}H NMR [2980], MS [2980].

1-(4-Hydroxy-2,6-dimethylphenyl)ethanone

[91060-92-3] $C_{10}H_{12}O_2$ mol.wt. 164.20



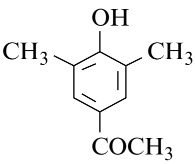
Synthesis

– Preparation by diazotization of 4-amino-2,6-dimethylaceto-phenone, followed by hydrolysis of the obtained diazonium salt (68%) [2981,2982].

m.p. 119–120° [2978,2981–2983];
^{1}H NMR [2981], ^{13}C NMR [2978], IR [2981], MS [2981].

1-(4-Hydroxy-3,5-dimethylphenyl)ethanone

[5325-04-2] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

– Preparation by Fries rearrangement of 2,6-dimethylphenyl acetate,
  - with aluminium chloride [2984], without solvent at 120–140° [2233,2265,2985], (81–100%) [2233], in nitrobenzene at r.t. (75%) [2535] or in nitromethane, first at 0°, then at 50° for 48 h under argon atmosphere (61%) [2986];

- with aluminium bromide without solvent at 130° (71%) [2605].

– Preparation by reaction of 46% hydrobromic acid solution with 4-(benzyloxy)-3,5-dimethyl-acetophenone in the presence of tetrabutylammonium bromide in refluxing methylene chloride (53%) [2987].

– Also obtained by heating various 2,6-dimethyl-4-alkylphenyl acetates** with aluminium chloride without solvent, the reaction being accompanied by an alkyl group elimination,

- **alkyl = benzyl (quantitative yield) [2957], ethyl (50%) [2233], (39%) [2957], dodecyl (38%) [2957], propyl (31%) [2957], butyl (27%) [2957] and heptyl (12%) [2957].

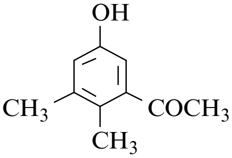
– Also refer to: [2988].

m.p. 162° [2987], 156–157° [2984], 151–152° [2605,2985,2986], 150–151° [2233], 150° [2535];

^{1}H NMR [2984,2986,2987], ^{13}C NMR [2986], IR [2986,2987], MS [2986,2987].

1-(5-Hydroxy-2,3-dimethylphenyl)ethanone

[127701-70-6] $C_{10}H_{12}O_2$ mol.wt. 164.20



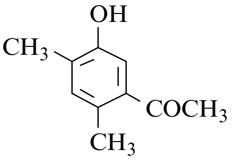
Synthesis

– Preparation by rearrangement of 3-acetyl-4,4-dimethyl-cyclohexa-2,5-dienone with 49% sulfuric acid at 20° (quantitative yield) [2989].

^{1}H NMR [2989].

1-(5-Hydroxy-2,4-dimethylphenyl)ethanone

$C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

– Preparation by demethylation of 5-methoxy-2,4-dimethyl-acetophenone with aluminium chloride (50%) [2956].

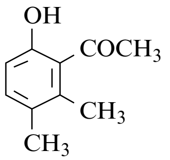
– Also obtained by reaction of acetyl chloride on 2,4-di-methylphenol with aluminium chloride in nitrobenzene at 50° (9%) [2181].

– Also obtained (by-product) by Fries rearrangement of 2,4-dimethylphenyl acetate with aluminium chloride, without solvent [2697] or in nitrobenzene at 50° (9%) [2181].

– Also obtained by reaction of zinc powder on 5-hydroxy-2,4-dimethyl-α-chloroacetophenone in acetic acid [2956].

m.p. 135° [2181], 130–131°5 [2956].

1-(6-Hydroxy-2,3-dimethylphenyl)ethanone



$C_{10}H_{12}O_2$ mol.wt. 164.20

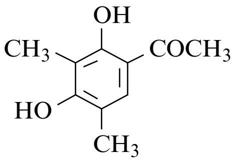
Synthesis

- Preparation by demethylation of 6-methoxy-2,3-dimethylacetophenone with aluminium chloride in boiling benzene (30%) [2212]. The above keto anisole itself was obtained by reaction of dimethylcadmium on 6-methoxy-2,3-di-methylbenzoyl chloride in boiling benzene.

m.p. 73°8–75° [2212]; b.p.$_{4.5}$ 126° [2212]; IR [2212].

1-(2,4-Dihydroxy-3,5-dimethylphenyl)ethanone *(Clavatol)*

[577-45-7]



$C_{10}H_{12}O_3$ mol.wt. 180.20

Syntheses

- Preparation by reaction of acetonitrile on 2,4-dimethylresorcinol (Hoesch reaction) (56–68%) [2302,2990].
- Preparation by reaction of acetic acid on 2,4-dimethyl-resorcinol with zinc chloride (Nencki reaction) [2373,2991].
- Also obtained by reaction of methyl iodide with 2,4-di-hydroxy-5-methylacetophenone in the presence of potassium hydroxide in methanol, in an ice-chest overnight (19%) [2330].

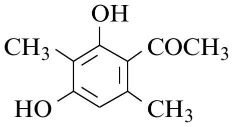
From Microorganisms

- Also obtained by hydrolysis of Sorbicillin (a pigment produced by the mold *Penicillium notatum*) with refluxing 2 N sodium hydroxide solution (16%) [2330].
- Also obtained by direct methylation of resacetophenone or 3-methylresacetophenone using washed cells of *Streptomyces risomus* [2373].
- Isolated in small quantities from cultures of *Aspergillus clavatus* grown in Czapek-Doz medium with molasses as an additional substrate [2373,2992].

m.p. 184–186° [2992], 183° [2990], 181–182° [2330]; UV [2213,2330].

1-(2,4-Dihydroxy-3,6-dimethylphenyl)ethanone

[69082-35-5]



$C_{10}H_{12}O_3$ mol.wt. 180.20

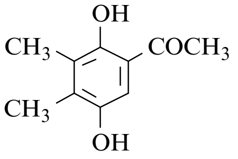
Syntheses

- Obtained by reaction of methyl iodide with 2,4-dihydroxy-6-methylacetophenone in the presence of potassium hydroxide in methanol in an ice-chest overnight (27%) [2330].
- Preparation by reaction of acetonitrile on β-orcinol (1,3-dihydroxy-2,5-dimethylbenzene) (Hoesch reaction) [2693,2993], (70%) [2993].

m.p. 153° [2693], 149–150° [2330]; ^{1}H NMR [2993], IR [2993], UV [2213], MS [2993].

1-(2,5-Dihydroxy-3,4-dimethylphenyl)ethanone

[71582-59-7] $C_{10}H_{12}O_3$ mol.wt. 180.20



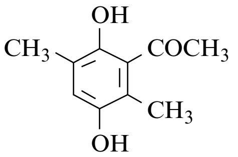
Synthesis

- Preparation by reaction of acetic acid on 2,3-dimethylhydroquinone with boron trifluoride, followed by saponification of the monoacetate [2909,2994] or diacetate [2349] obtained (84–91%) [2349,2994].

m.p. 151° [2349], 150° [2994].

1-(2,5-Dihydroxy-3,6-dimethylphenyl)ethanone

$C_{10}H_{12}O_3$ mol.wt. 180.20



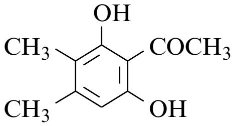
Synthesis

- Obtained (poor yield) by reaction of acetic acid on 2,5-dimethylhydroquinone with boron trifluoride (1%) [2700].

m.p. 88–90° [2700].

1-(2,6-Dihydroxy-3,4-dimethylphenyl)ethanone

[7743-16-0] $C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

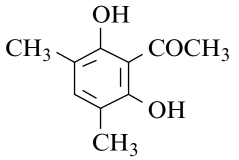
- Obtained from 4,5-dimethylresorcinol by reaction,
  - with acetyl chloride in the presence of aluminium chloride in nitrobenzene, first at r.t., then at 60° (9%) [2213];
  - with acetic acid in the presence of boron trifluoride etherate at reflux [2995].
- Also obtained (by-product) by Fries rearrangement of 4,5-dimethylresorcinol diacetate in the presence of aluminium chloride at 115–120° (10%) [2996].
- Preparation by heating a mixture of 2-acetyl-4,5-dihydroxy-4,5-dimethylcyclohexane-1,3-dione and zinc dust in 50% acetic acid at 65° (71%) [2996]. The same reaction carried out with 2-acetyl-6-hydroxy-5,6-dimethylcyclohex-4-ene-1,3-dione leads to 82% yield [2996].

m.p. 125–127° [2996], 122–124° [2995] and 82–83° [2213]. One of the reported melting points is obviously wrong.

^{1}H NMR [2996], ^{13}C NMR [2996], IR [2996], UV [2213,2996].

1-(2,6-Dihydroxy-3,5-dimethylphenyl)ethanone

[37467-68-8] $C_{10}H_{12}O_3$ mol.wt. 180.20



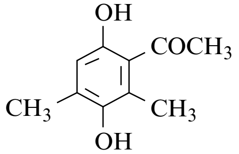
Synthesis

- Obtained by reaction of acetic anhydride with 4,6-dimethyl-resorcinol in the presence of 45% solution of boron trifluoride etherate at r.t. (15%) [2213] or boron trifluoride-acetic acid complex for 2 h at 100° [2997].

m.p. 144–146°5 [2997], 139–140° [2213]; UV [2213].

1-(3,6-Dihydroxy-2,4-dimethylphenyl)ethanone

[71582-58-6] $C_{10}H_{12}O_3$ mol.wt. 180.20



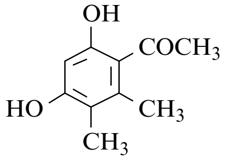
Syntheses

- Preparation by reaction of acetic acid on 2,6-dimethyl-hydroquinone with boron trifluoride [2700,2909], (76%) [2700].
- Preparation by reaction of ammonium persulfate on 2-hydroxy-4,6-dimethylacetophenone (Elbs reaction) (34%) [2998].

m.p. 135–136° [2998], 133–134° [2700]; IR [2998].

1-(4,6-Dihydroxy-2,3-dimethylphenyl)ethanone

$C_{10}H_{12}O_3$ mol.wt. 180.20



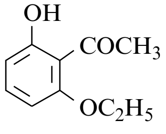
Synthesis

- Preparation by reaction of acetonitrile on 3,5-dihydroxy-o-xylene (Hoesch reaction) (69%) [2330].

m.p. 155–156° [2330]; UV [2213].

1-(2-Ethoxy-6-hydroxyphenyl)ethanone

[2750-25-6] $C_{10}H_{12}O_3$ mol.wt. 180.20



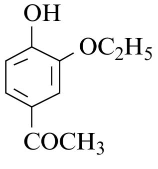
Synthesis

- Preparation by reaction of ethyl iodide on 2,6-dihydroxy acetophenone with potassium carbonate in refluxing acetone (64%) [2999].

m.p. 84–85° [2999].

1-(3-Ethoxy-4-hydroxyphenyl)ethanone

[78268-45-8] $C_{10}H_{12}O_3$ mol.wt. 180.20



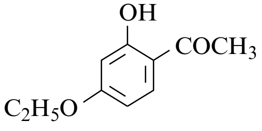
Syntheses

- Preparation by Fries rearrangement of 2-ethoxyphenyl acetate with aluminium chloride in nitrobenzene at r.t. (50%) [3000].
- Preparation by refluxing 4-(benzyloxy)-3-ethoxy-acetophenone in mixture of acetic acid and hydrochloric acid [2528] according to [3001].

m.p. 66° [3000], 59–60° [2528]; ^{1}H NMR [3000].

1-(4-Ethoxy-2-hydroxyphenyl)ethanone

[37470-42-1] $C_{10}H_{12}O_3$ mol.wt. 180.20



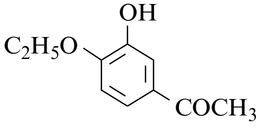
Syntheses

- Preparation by reaction of ethyl iodide on resacetophenone,
  - with potassium hydroxide in refluxing ethanol [2186,2220,2289,2296,2730,3002,3003], (25%) [3002] or boiling acetone [2293];
  - with potassium carbonate in boiling acetone [3004,3005], (88%) [3005].
- Also obtained by reaction of aluminium chloride on 2,4-diethoxyacetophenone [2289].
- Also refer to: [2484,2911] (compound **1e**).

m.p. 50°[2186,2204],49–50°[2293,3005],49°[3002,3003],48°[2220,2289,2730], 45–46° [3004]; UV [2186,2204].

1-(4-Ethoxy-3-hydroxyphenyl)ethanone

[78269-19-9] $C_{10}H_{12}O_3$ mol.wt. 180.20



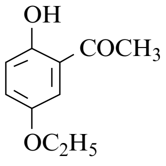
Synthesis

- Preparation by Fries rearrangement of 2-ethoxyphenyl acetate with aluminium chloride in nitrobenzene at r.t. (50%) [3000].

m.p. 100° [3000]; ^{1}H NMR [3000].

1-(5-Ethoxy-2-hydroxyphenyl)ethanone

[56414-14-3] $C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

- Preparation by reaction of ethyl bromide with quinacetophenone in the presence of potassium hydroxide in boiling ethanol [3006].
- Also obtained by condensation of hydroquinone diethyl ether with acetyl chloride according to the Friedel–Crafts method [3007,3008].

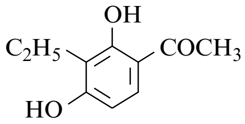
- Also obtained by reaction of aluminium chloride on quinacetophenone diethyl ether [3007,3008].
- Also obtained by alkaline degradation of 6,3′-diethoxyflavone* with sodium ethoxide in refluxing ethanol for some hours [3009].

  N.B.: Former nomenclature (2,3′-diethoxyflavone*).
- Refer to: [2227,3010,3011].

  m.p. 57° [3006], 55° [3007,3008].

1-(3-Ethyl-2,4-dihydroxyphenyl)ethanone

[111224-13-6] $C_{10}H_{12}O_3$ mol.wt. 180.20



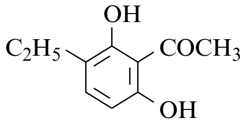
Syntheses

- Preparation by reaction of acetic acid on 2-ethylresorcinol with zinc chloride (Nencki reaction) at 140° [2681,3012,3013], (73%) [3012].
- Preparation from 2-ethylresorcinol (SM) by reaction with acetyl chloride in the presence of aluminium chloride. The starting material (SM) was prepared by a three-step procedure from resorcinol dimethyl ether [3014].
- Also obtained by alkaline degradation of 8-acetyl-6-ethyl-4-methylumbelliferone (m.p. 137°) in refluxing N sodium hydroxide for 1 h (96%) [3015].

  m.p. 137° [3012], 130° [3015]; ^{1}H NMR [3014], MS [3014].

1-(3-Ethyl-2,6-dihydroxyphenyl)ethanone

[54337-59-6] $C_{10}H_{12}O_3$ mol.wt. 180.20



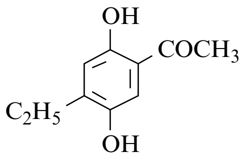
Syntheses

- Preparation by degradation of 8-acetyl-6-ethyl-7-hydroxy-4-methylcoumarin with refluxing 2 N sodium hydroxide [2308].
- Preparation by decarboxylation of 3-acetyl-5-ethyl-2,4-dihydroxybenzoic acid,
  - with refluxing aqueous hydrochloric acid (62%) [3016];
  - with 10% aqueous sodium hydroxide heated in a sand bath [2308].
- Also refer to: [2997].

  m.p. 135° [2308], 130° [3016].

1-(4-Ethyl-2,5-dihydroxyphenyl)ethanone

$C_{10}H_{12}O_3$ mol.wt. 180.20



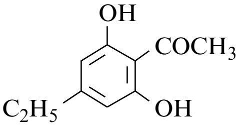
Synthesis

- Obtained (poor yield) by reaction of aqueous ammonium persulfate solution on 4-ethyl-2-hydroxyacetophenone with 10% potassium hydroxide, in aqueous pyridine solution at r.t. (3%) [2944].

m.p. 100° [2944].

1-(4-Ethyl-2,6-dihydroxyphenyl)ethanone

[209746-96-3] $C_{10}H_{12}O_3$ mol.wt. 180.20

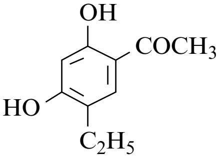


Synthesis

- Refer to: [3017].

1-(5-Ethyl-2,4-dihydroxyphenyl)ethanone

[4460-42-8] $C_{10}H_{12}O_3$ mol.wt. 180.20



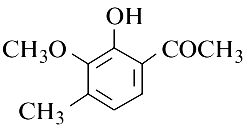
Syntheses

- Preparation by reaction of acetic acid on 4-ethylresorcinol with zinc chloride (Nencki reaction) [2671,2912, 2913,3018,3019], (67–77%) [2671,2913,3019].
- Preparation by Fries rearrangement of 4-ethylresorcinol diacetate with aluminium chloride in nitrobenzene at 50–60° (quantitative yield) [3020] or without solvent at 40–50° (47%) [3020].
- Preparation by reaction of acetonitrile on 4-ethylresorcinol (Hoesch reaction) [2912,2913].
- Preparation from 5-ethyl-2-hydroxy-4-methoxyacetophenone by demethylation with boiling pyridinium chloride (40%) [3013].
- Preparation from 5-ethyl-2,4-dimethoxyacetophenone by demethylation with boron tribromide in methylene chloride at r.t. (36%) [2678,2679].
- Also obtained by hydrolysis of 6-ethyl-7-hydroxy-2-methylchromone with refluxing aqueous 1 N sodium hydroxide solution [3021].

m.p. 118–119° [2913], 118° [2912,3021], 117–118° [3020], 116° [3013], 115–116° [2671], 115° [2676,3019].

1-(2-Hydroxy-3-methoxy-4-methylphenyl)ethanone

[77869-43-3] $C_{10}H_{12}O_3$ mol.wt. 180.20



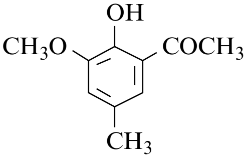
Synthesis

- Isolated as a co-product from the preparation of 2,3-di-methoxy-4-methylacetophenone, obtained by conversion of 2,3-dimethoxy-4-methylbenzoyl chloride with methyl cadmium (10%) [3022].

Crystalline compound [3022]; ^{1}H NMR [3022], MS [3022].

1-(2-Hydroxy-3-methoxy-5-methylphenyl)ethanone

[7452-85-9] $C_{10}H_{12}O_3$ mol.wt. 180.20



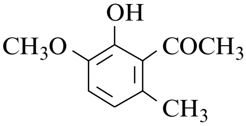
Syntheses

- Preparation by reaction of methylmagnesium iodide on 2-acetoxy-3-methoxy-5-methylbenzonitrile in refluxing ethyl ether (72%) [3023].
- Preparation by Fries rearrangement of 2-methoxy-4-methyl-phenyl acetate with aluminium chloride without solvent (24%) [2697].
- Also obtained by reaction of dimethyl sulfate on 2,3-dihydroxy-5-methylacetophenone with potassium carbonate in acetone (18%) [2670].
- Preparation by UV light irradiation (photo-Fries rearrangement) of 2-methoxy-4-methylphenyl acetate (creosol acetate) in ethanol (68%), in benzene or in hexane (56–55%) [2198].

m.p. 84–85° [2670], 82–84° [3023]; ^{1}H NMR [2198], IR [2198].

1-(2-Hydroxy-3-methoxy-6-methylphenyl)ethanone

[4223-86-3] $C_{10}H_{12}O_3$ mol.wt. 180.20



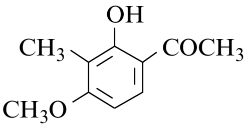
Syntheses

- Preparation by partial demethylation of 2,3-dimethoxy-6-methylacetophenone with aluminium chloride in refluxing methylene chloride (77%) [2524].
- Preparation by chromic acid degradation of 7-methoxy-2,3,4-trimethylbenzofuran (46%) [3013].
- Also obtained (poor yield) by photo-Fries rearrangement of 2-methoxy-5-methylphenyl acetate in ethanol under nitrogen (2%) [3024].

Pale yellow oil [2524,3013,3024]; b.p.$_{12}$ 156–158° [3013];
n_D^{23} = 1.5527 [3013]; ^{1}H NMR [3024], ^{13}C NMR [2524], IR [2524,3024].

1-(2-Hydroxy-4-methoxy-3-methylphenyl)ethanone

[69469-91-6] $C_{10}H_{12}O_3$ mol.wt. 180.20



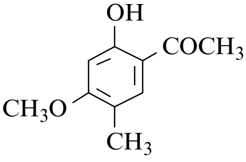
Syntheses

- Preparation by reaction of acetonitrile on 3-methoxy-2-methylphenol (Hoesch reaction) (46%) [3025].
- Preparation by reaction of methyl iodide on resacetophenone,
  - with potassium hydroxide in refluxing methanol [2294,2730,3026], (19–25%) [2294,2672,3026];
  - with sodium methoxide in boiling methanol [2915].

m.p. 83–84° [2915,3026], 83° [3025], 82–83° [2672], 80–82° [2294], 80–81° [2730].

1-(2-Hydroxy-4-methoxy-5-methylphenyl)ethanone

[81511-52-6] $C_{10}H_{12}O_3$ mol.wt. 180.20



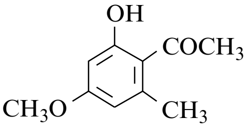
Syntheses

- Preparation by partial methylation of 2,4-dihydroxy-5-methylacetophenone,
  - with methyl iodide in the presence of potassium carbonate in refluxing acetone (78%) [2871];
  - with diazomethane in solution of ethyl ether–methanol mixture (92%) [2302].

m.p. 94° [2871].

1-(2-Hydroxy-4-methoxy-6-methylphenyl)ethanone *(Acetoevernone)*

[6540-66-5] $C_{10}H_{12}O_3$ mol.wt. 180.20



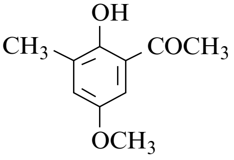
Syntheses

- Preparation by partial methylation of 2,4-dihydroxy-6-methylacetophenone (orcacetophenone or β-orcacetophenone), with diazomethane (78%) [2694] or dimethyl sulfate [2301,2684].
- Preparation by Fries rearrangement of 3-methoxy-5-methylphenyl acetate with aluminium chloride in nitrobenzene at r.t. (52%) [2742,2743].
- Preparation by reaction of acetonitrile on 3-methoxy-5-methylphenol with zinc chloride and hydrochloric acid in ethyl ether at r.t. (Hoesch reaction) [2301,2307], (28%) [2301].
- Also obtained (by-product) by reaction of acetyl chloride on orcinol dimethyl ether with aluminium chloride in carbon disulfide at r.t. [2348,2694,3027,3028], (5–8%) [2348,2694].

m.p. 79–80° [2348], 79° [2301,2307,2742,2743,3028], 78–78°5 [2694];
^{1}H NMR [2348,2694], IR [2348,2694], UV [2348,2694], MS [2348,2694].

1-(2-Hydroxy-5-methoxy-3-methylphenyl)ethanone

[77936-48-2] $C_{10}H_{12}O_3$ mol.wt. 180.20



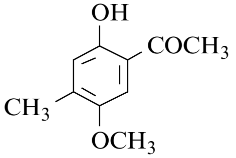
Syntheses

- Preparation by Fries rearrangement of 4-methoxy-2-methyl-phenyl acetate,
  - with boron trifluoride in ethylene dichloride for 3 h (97%) [2696];
  - with aluminium chloride (11%) [2697].

m.p. 52–53° [2696]; sublimation 54–58°/0.2 mm [2696];
^{1}H NMR [2696], IR [2696], MS [2696,3029].

1-(2-Hydroxy-5-methoxy-4-methylphenyl)ethanone

[4223-84-1] $C_{10}H_{12}O_3$ mol.wt. 180.20



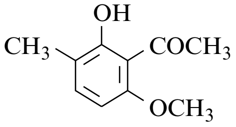
Syntheses

- Obtained by reaction of acetyl chloride on 2,5-dimethoxy-4-isopropyltoluene with aluminium chloride in boiling carbon disulfide (14%) [2542].
- Also obtained by chromic acid degradation of 5-met\ hoxy-2,3,6-trimethylbenzofuran (10%) [3013].
- Also obtained (by-product) by reaction of acetyl chloride with 2,5-dimethoxytoluene in the presence of aluminium chloride in carbon disulfide at 20–25° (4%) [2583].
- Also obtained by photo-Fries rearrangement of 4-methoxy-3-methylphenyl acetate in ethanol under nitrogen (19%) [3024].

m.p. 114° [2583], 112° [2542], 111–112° [3013]; ^{1}H NMR [2583], IR [2583].

1-(2-Hydroxy-6-methoxy-3-methylphenyl)ethanone

[56504-43-9] $C_{10}H_{12}O_3$ mol.wt. 180.20



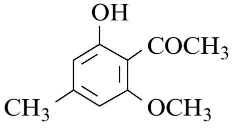
Syntheses

- Preparation by partial methylation of 2,6-dihydroxy-3-methylacetophenone,
  - with methyl iodide in the presence of potassium carbonate in refluxing acetone (55%) [2871];
  - with diazomethane in ethyl ether at r.t. (16%) [2552].
- Also obtained by reduction of 3-formyl-2-hydroxy-6-methoxyacetophenone with hydrochloric acid and amalgamated zinc in methanol at 50° (11%) [2871].

m.p. 59° [2871], 58° [2552]; ^{1}H NMR [2552], IR [2552].

1-(2-Hydroxy-6-methoxy-4-methylphenyl)ethanone

[31405-63-7] $C_{10}H_{12}O_3$ mol.wt. 180.20



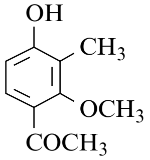
Syntheses

- Preparation by reaction of acetic anhydride on orcinol monomethyl ether with concentrated sulfuric acid, at reflux (45%) [2307].
- Also obtained by reaction of dimethyl sulfate on 2,6-dihydroxy-4-methylacetophenone (p-orcacetophenone) with sodium hydroxide [2307] or potassium hydroxide [2708].
- Also obtained by reaction of acetyl chloride on orcinol dimethyl ether with aluminium chloride in carbon disulfide at r.t. [2348,2694,3028], (11%) [2348], (4%) [2694].

m.p. 81° [2307], 80–81° [2694], 76–77° [2713], 74–75° [2348];
^{1}H NMR [2348,2694], IR [2348,2694], UV [2348,2694], MS [2348,2694].

1-(4-Hydroxy-2-methoxy-3-methylphenyl)ethanone

[118824-97-8] $C_{10}H_{12}O_3$ mol.wt. 180.20



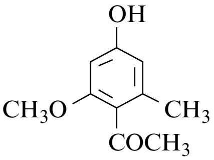
Synthesis

– Preparation by catalytic hydrogenolysis of 4-(benzyloxy)-2-methoxy-3-methylacetophenone at r.t. under pressure in the presence of 10% Pd/C in ethanol (92%) [2582], (84%) [2942].

m.p. 130–132° [2582], 130° [2942]; ^{1}H NMR [2582], IR [2582], MS [2582].

1-(4-Hydroxy-2-methoxy-6-methylphenyl)ethanone *(Isoacetoevernone)*

$C_{10}H_{12}O_3$ mol.wt. 180.20



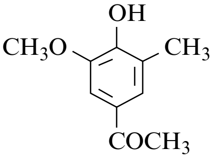
Synthesis

– Preparation by reaction of acetonitrile on orcinol monomethyl ether (Hoesch reaction) (32%) [2301].

m.p. 150° [2301].

1-(4-Hydroxy-3-methoxy-5-methylphenyl)ethanone

$C_{10}H_{12}O_3$ mol.wt. 180.20



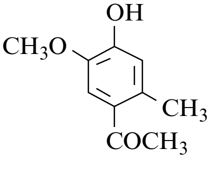
Synthesis

– Preparation by Fries rearrangement of 2-methoxy-6-methyl-phenyl acetate with aluminium chloride in refluxing carbon disulfide (58%) [3030].

m.p. 94° [3030].

1-(4-Hydroxy-5-methoxy-2-methylphenyl)ethanone

[162853-20-5] $C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

– Preparation by reaction of acetyl chloride on isocreosol (2-methoxy-5-methylphenol) with aluminium chloride in nitrobenzene at r.t. (87%) [3031].

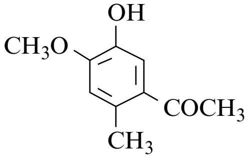
– Preparation by Fries rearrangement of isocreosol acetate with aluminium chloride in nitrobenzene at r.t. (75%) [2914].

– Also obtained by photo-Fries rearrangement of 2-methoxy-5-methylphenyl acetate under nitrogen in ethanol (20%) [3024] or in methanol at 254 nm at r.t. (21%) [2963].

m.p. 167–168° [2963], 124° [2914], 123° [3031];
^{1}H NMR [2963], ^{13}C NMR [2963], IR [2963], MS [2963].

1-(5-Hydroxy-4-methoxy-2-methylphenyl)ethanone

[6948-37-4] $C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

- Preparation by reaction of acetic acid on creosol (2-methoxy-4-methylphenol) with boron trifluoride at 25° [2198,2670], (88%) [2670].
- Preparation by Fries rearrangement of creosol acetate with aluminium chloride, in methylene chloride at 0° [3032] or at 20° (82%) [3033], in nitrobenzene at 80° (70%) [2723] or without solvent (8%) [2697].
- Also obtained by reaction of acetyl chloride on homoveratrole (3,4-dimethoxytoluene) with aluminium chloride in carbon disulfide [3034].

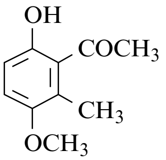
m.p. 129–130° [2670], 129° [2723,3033], 128–129° [3032], 124–126° [2198], 123° [3034];

^{1}H NMR [2198,3033] (Sadtler: standard n°49332 M);

IR [2198,3033] (Sadtler: standard n° 76405 K); UV [3033].

1-(6-Hydroxy-3-methoxy-2-methylphenyl)ethanone

[71452-36-3] $C_{10}H_{12}O_3$ mol.wt. 180.20



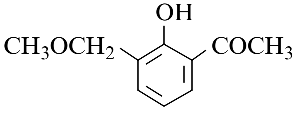
Synthesis

- Obtained by photo-Fries rearrangement of 4-methoxy-3-methylphenyl acetate in ethanol under nitrogen (18%) [3024].

oil [3024]; ^{1}H NMR [3024], IR [3024], MS [3024].

1-[2-Hydroxy-3-(methoxymethyl)phenyl]ethanone

[87165-50-2] $C_{10}H_{12}O_3$ mol.wt. 180.20



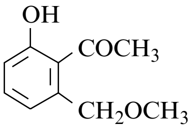
Synthesis

- Preparation from 3-chloromethyl-2-hydroxyacetophenone (m.p. 45°) by reaction with methanol in the presence of concentrated hydrochloric acid and iron powder at reflux for 3.5–4 h (88%) [2494].

b.p.$_{0.3}$ 89–91° [2494]; ^{1}H NMR [2494], IR [2494].

1-[2-Hydroxy-6-(methoxymethyl)phenyl]ethanone

[161358-64-1] $C_{10}H_{12}O_3$ mol.wt. 180.20



Synthesis

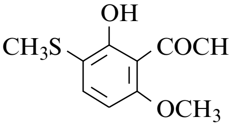
- Obtained by treatment of 2-(methoxymethoxy)-6-(methoxy-methyl)acetophenone with aqueous trifluoroacetic acid at r.t. for 16 h (93%) [2645].

pale yellow oil [2645]; b.p.$_1$ 165–170° [2645];

^{1}H NMR [2645], IR [2645], MS [2645].

1-[2-Hydroxy-6-methoxy-3-(methylthio)phenyl]ethanone

[126405-81-0] $C_{10}H_{12}O_3S$ mol.wt. 212.27



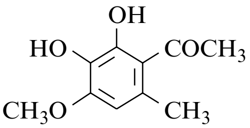
Synthesis

– Preparation by adding 2-hydroxy-3-iodo-6-methoxy-acetophenone and cuprous oxide to a solution of sodium methyl sulfhydrate, first prepared from methanethiol and sodium hydride in DMF [2524].

m.p. 83° [2524]; ^{1}H NMR [2524], IR [2524].

1-(2,3-Dihydroxy-4-methoxy-6-methylphenyl)ethanone

[127940-12-9] $C_{10}H_{12}O_4$ mol.wt. 196.20



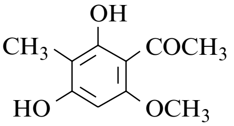
Synthesis

– Preparation by reaction of acetyl chloride on 1,2,3-tri-methoxy-5-methylbenzene with aluminium chloride in refluxing methylene chloride (45%) [3035].

m.p. 132° [3035]; ^{1}H NMR [3035], ^{13}C NMR [3035], IR [3035], MS [3035].

1-(2,4-Dihydroxy-6-methoxy-3-methylphenyl)ethanone

[83459-37-4] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

– Preparation by reduction of 3-formyl-2,4-dihydroxy-6-methoxyacetophenone with hydrochloric acid and amalgamated zinc in gently heated aqueous methanol (64%) [3036].
– Also obtained by reaction of acetonitrile on 3,5-dihydroxy-4-methylanisole (Hoesch reaction) [3037].

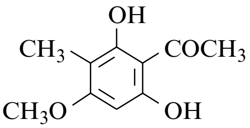
Isolation from natural sources

– From Yuexiandaji *(Euphorbia ebracteolata)* [3038].
– From the roots of *Euphorbia ebracteolata* Hayata [3039,3040].
– From *Pancratium maritimum* (Amaryllidaceae) [3041].

m.p. 225° [3036], 224° [3037], 202–203° (d) [3041]; HPLC [3038];
^{1}H NMR [3041], ^{13}C NMR [3041], IR [3041], EIMS [3041], HRMS [3041].

1-(2,6-Dihydroxy-4-methoxy-3-methylphenyl)ethanone

[69480-06-4] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

– Preparation by reaction of acetonitrile on 3,5-dihydroxy-2-methylanisole (Hoesch reaction) (73%) [3037].

- Also obtained by reaction of 2 N aqueous sodium hydroxide on 2,4-diacetyl-3, 5-dihydoxy-6-methylanisole at 80° (decarbonylation reaction) [3042].
- Preparation by catalytic hydrogenolysis of 6-(benzyloxy)-2-hydroxy-4-methoxy-3-methyl-acetophenone in the presence of Pd/C in acetic acid (75%) [3043].
- Also obtained (by-product) by reaction of acetic anhydride on 3,5-dihydroxy-2-methylanisole with boron trifluoride in ethyl ether at 20° [3042].

Isolation from natural sources

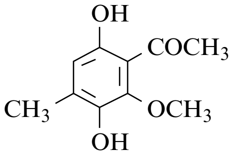
- From the pericarps of *Mallotus japonicus* Muell. Arg. (Euphorbiaceae) [3044–3046].
- Also obtained by reductive alkaline cleavage of 3-(3,3-dimethylallyl)-5-(3-acetyl-2,4-dihydroxy-5-methyl-6-methoxybenzyl)phloroacetophenone (**I**) [3047], according to [3048]. The ketone (**I**) was isolated from *Mallotus japonicus* (Euphorbiaceae) [3047].

m.p. 200–201° [3046], 198° [3042], 197–200° [3047], 197–198° [3037], 196–198° [3045], 196° [3043];

^{1}H NMR [3045–3047], UV [3045], MS [3045–3047].

1-(3,6-Dihydroxy-2-methoxy-4-methylphenyl)ethanone

[90377-24-5] $C_{10}H_{12}O_4$ mol.wt. 196.20

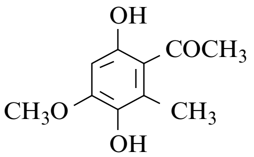


Isolation from natural sources

- From *Trocholejeunea sandvicensis* (Lejeuneaceae) [3049].

1-(3,6-Dihydroxy-4-methoxy-2-methylphenyl)ethanone

[68531-86-2] $C_{10}H_{12}O_4$ mol.wt. 196.20



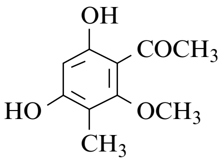
Synthesis

- Preparation by nuclear oxidation of 2-hydroxy-4-methoxy-6-methylacetophenone with alkaline persulfate (Elbs reaction) (45%) [3050].

m.p. 164–165° [3050].

1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)ethanone *(Pseudoaspidinol-A)*

[52200-61-0] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

- Preparation by catalytic hydrogenolysis of 4,6-bis-(benzyloxy)-2-methoxy-3-methylacetophenone in the presence of Pd/C in acetic acid (quantitative yield) [3051], (86%) [3043].

- Preparation by reaction of dimethyl sulfate on 4,6-bis-(benzoyloxy)-2-hydroxy-3-methylacetophenone with potassium carbonate in boiling acetone (46%) [3051].
- Also obtained by heating methyl 5-acetyl-2,6-dihydroxy-4-methoxy-3-methylbenzoate in aqueous glycerol at 180–200° for 30 min (29%) [3052].
- Also refer to: [3053,3054].

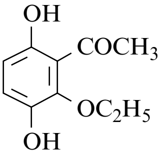
Isolation from natural sources

- From *stereocaulon vesuvianum*, a foliose lichen, abundantly growing over volcanic rocks [3055].

  m.p. 143–144° [3055], 142° [3043,3051], 138–140° [3052]; TLC [3055]; ^{1}H NMR [3052,3055], IR [3052,3055], UV [3055], MS [3055].

1-(2-Ethoxy-3,6-dihydroxyphenyl)ethanone

[33539-21-8] $C_{10}H_{12}O_4$ mol.wt. 196.20



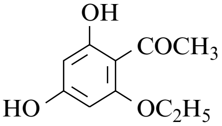
Syntheses

- Easy preparation by reduction of 2-acetyl-3-ethoxy-1,4-benzoquinone using conventional methods [2869].
- Also obtained (low yield) by reaction of 2-acetyl-1,4-benzoquinone with an excess of ethanol at r.t., with exclusion of light [2869].

m.p. 102–103°5 [2869]; ^{1}H NMR [2869], IR [2869].

1-(2-Ethoxy-4,6-dihydroxyphenyl)ethanone

$C_{10}H_{12}O_4$ mol.wt. 196.20



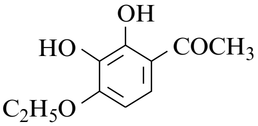
Synthesis

- Preparation by reaction of acetonitrile on phloroglucinol monoethyl ether (Hoesch reaction) (63%) [2851].

m.p. 178° [2851].

1-(4-Ethoxy-2,3-dihydroxyphenyl)ethanone

$C_{10}H_{12}O_4$ mol.wt. 196.20



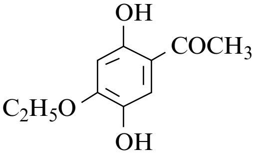
Syntheses

- Preparation by reaction of ethyl iodide on gallacetophenone monopotassium salt in boiling methanol [2812].
- Preparation by reaction of hydrobromic acid with 2,4-diethoxy-3-methoxyacetophenone (43%) or 3,4-diethoxy-2-hydroxyacetophenone (20%) in acetic acid at r.t. [2814].

m.p. 102°2–103°2 [2814], 102° [2812,2815].

1-(4-Ethoxy-2,5-dihydroxyphenyl)ethanone

[58084-93-8] $C_{10}H_{12}O_4$ mol.wt. 196.20



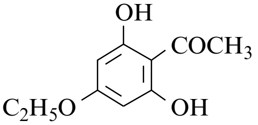
Synthesis

- Preparation from 4-ethoxy-2-hydroxyacetophenone by persulfate oxidation (Elbs reaction) (quantitative yield) [3005], (24%) [3056].

m.p. 129–130° [3056], 125–126° [3005]; ^{1}H NMR [3005].

1-(4-Ethoxy-2,6-dihydroxyphenyl)ethanone

[35028-01-4] $C_{10}H_{12}O_4$ mol.wt. 196.20



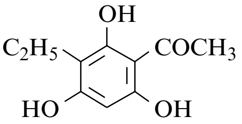
Synthesis

- Preparation from 2,4-diacetyl-5-ethoxyresorcinol by selective deacetylation by refluxing in 1 N sodium hydroxide for 1 h [2856].

m.p. 164–165° [2856]; ^{1}H NMR [2856].

1-(3-Ethyl-2,4,6-trihydroxyphenyl)ethanone

[84653-58-7] $C_{10}H_{12}O_4$ mol.wt. 196.20



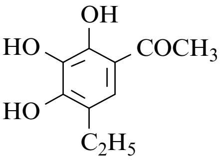
Synthesis

- Refer to: [2879] (compound **VII**).

m.p. 187–189° [2879]; ^{1}H NMR [2879].

1-(5-Ethyl-2,3,4-trihydroxyphenyl)ethanone

[86989-84-6] $C_{10}H_{12}O_4$ mol.wt. 196.20



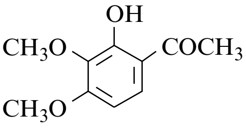
Syntheses

- Preparation by reaction of acetyl chloride on 4-ethylpyrogallol with aluminium chloride [2266].
- Preparation by reaction of acetic acid with 4-ethylpyrogallol in the presence of boron trifluoride in ethyl ether at 0° (78%) [2268].

m.p. 141° [2266,2268]; UV [2268].

1-(2-Hydroxy-3,4-dimethoxyphenyl)ethanone

[5396-18–9] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

- Preparation by reaction of acetyl chloride on pyrogallol trimethyl ether,

  - with aluminium chloride in carbon disulfide [3057–3061], (50%) [3061], in boiling ethyl ether (77%) [2770] or in benzene at 45–50° (77%) [3062];
  - with mercuric chloride without solvent at 100° (40%) [3063,3064].

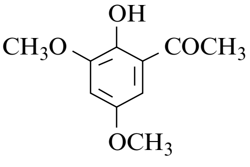
- Preparation by Fries rearrangement of 2,3-dimethoxyphenyl acetate with aluminium chloride in nitrobenzene at r.t. (61%) [2742,2743].
- Preparation by partial methylation of gallacetophenone,
  - with methyl iodide,
    - in the presence of sodium methoxide in boiling methanol [2915,3065];
    - in the presence of potassium carbonate in refluxing acetone (47%) [3066] or in acetone–DMF mixture [3067].
  - with dimethyl sulfate,
    - in the presence of potassium carbonate in refluxing benzene (65%) [2820], (51%) [2811];
    - in the presence of 40% potassium hydroxide [2406,3058,3068].
- Preparation by partial methylation of 2,3-dihydroxy-4-methoxyacetophenone with methyl iodide in the presence of potassium hydroxide in methanol [2812,2815].
- Also obtained by selective demethylation of 2,3,4-trimethoxyacetophenone,
  - with boron trichloride in methylene chloride at 0° (88%) [3069];
  - with aluminium chloride in refluxing ethyl ether [1821] or in acetonitrile at 30° for 3 h (95%) [2747];
  - with aniline hydriodide in aniline for 7 h at 95° (54%) [2693];
  - with cupric bromide in refluxing chloroform–ethyl acetate mixture (4%) [2922].

m.p. 83° [2693,3057], 78–80° [3058], 78–79° [2406,2742,2743,3062,3066,3068], 78° [3067], 77–78° [2812,2915,3065], 77° [2820,3059–3061], 75–77° [2811], 72–76° [3069], 68–72° [2922],

^{1}H NMR [2922,3062,3069], ^{13}C NMR [1821], IR [3062,3069].

1-(2-Hydroxy-3,5-dimethoxyphenyl)ethanone

[17605-00-4] $C_{10}H_{12}O_4$ mol.wt. 196.20



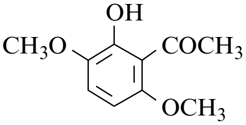
Syntheses

- Preparation by reaction of dimethyl sulfate on 2,5-di-hydroxy-3-methoxyacetophenone with potassium carbonate in refluxing acetone (54%) [2848].
- Preparation by photo-Fries rearrangement of 2,4-di-methoxyphenyl acetate in ethanol (61%) or in benzene (55%) [2198].

m.p. 84–86° [2848]; ^{1}H NMR [2198], IR [2198].

1-(2-Hydroxy-3,6-dimethoxyphenyl)ethanone

[52099-27-1] $C_{10}H_{12}O_4$ mol.wt. 196.20



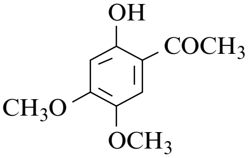
Synthesis

– Preparation by reaction of concentrated hydrochloric acid on 2-(benzyloxy)-3,6-dimethoxyacetophenone in acetic acid at 60° (99%) [2408].

m.p. 61° [2408].

1-(2-Hydroxy-4,5-dimethoxyphenyl)ethanone

[20628-06-2] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

– Preparation by partial methylation of 2,5-dihydroxy-4-methoxyacetophenone,
  - with methyl iodide in the presence of potassium carbonate in refluxing acetone (81%) [2850];
  - with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone (71%) [2418], in the presence of sodium methoxide in methanol (by-product) [2410,2733,2734,3070] or in the presence of sodium hydroxide in boiling aqueous ethanol (37%) [2742,2743];
  - with an excess of ethereal diazomethane in methanol (93%) [2740].

– Preparation by reaction of acetonitrile on 3,4-dimethoxyphenol with zinc chloride (Hoesch reaction) (47%) [3071].

– Also obtained (by-product) by reaction of acetyl chloride on 1,2,4-trimethoxybenzene with aluminium chloride in carbon disulfide at r.t. [2410,3070,3072].

– Also obtained by partial demethylation of 2,4,5-trimethoxyacetophenone with boiling aqueous hydrochloric acid [2410,3070] or aluminium chloride in acetonitrile for 6 h at 45° (50%) [2747].

– Preparation by reaction of boiling acetic acid on 3,4-dimethoxyphenyldiazonium borofluoride. The 3,4-dimethoxyphenyl acetate which was first formed was rearranged by the boron trifluoride produced during the reaction (62%) [3073].

– Also refer to: [3074].

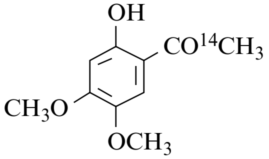
Isolation from natural sources

– From various plants belonging to the Polypodiaceae family, namely Inomotosou (*Pteris multifide* Poiret), Oobainomotosou (*Pteris cretica* L.) and Hitotsuba (*Pyrrosia"ingua* Farw.) [3075].

m.p. 115° [3070], 114–116° [2418], 114–115° [2733,2734], 112–114° [2740], 112° [2850,3071,3073], 111–112° [2742,2743]; b.p.$_{0.004}$ 125° [3071];
GC [3075], GC-MS [3075];
^{1}H NMR [2850], ^{13}C NMR [2421], IR [2850,3075].

1-(2-Hydroxy-4,5-dimethoxyphenyl)ethanone-*2*-^{14}*C*

[77184-92-0] $C_{10}H_{12}O_4$ mol.wt. 198.20



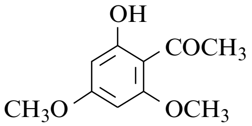
Synthesis

– Preparation by heating a mixture of 3,4-dimethoxyphenol, boron trifluoride-acetic acid complex and sodium acetate-2-^{14}C (250 μCi) at 100° for 25 min (54%) [3076].

m.p. 112–113° [3076];
specific radioactivity 1.50 μCi/mmol [3076].

1-(2-Hydroxy-4,6-dimethoxyphenyl)ethanone *(Xanthoxylin; Brevifolin)*

[90-24-4] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

– Preparation by reaction of acetonitrile on phloroglucinol dimethyl ether (Hoesch reaction) [2837,3077].

– Also obtained by partial methylation of phloroacetophenone,

  - with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone [2860,3078], (86%) [2860] or in solution of acetone–benzene mixture at reflux (78–83%) [2422,2936,3079], (65%) [3080];
  - with diazomethane in ethyl ether, at r.t. [2827,3081];
  - with methyl iodide in the presence of potassium carbonate in boiling acetone (83%) [2830], (6%) [2861].

– Also obtained by partial methylation of 2,4-dihydroxy-6-methoxyacetophenone [2845] or 2,6-di-hydroxy-4-methoxyacetophenone with diazomethane [2844,2845], (quantitative yield) [2844].

– Preparation by acetylation of phloroglucinol dimethyl ether,

  - with the boron trifluoride-acetic acid complex at 100° (24%) [2242];
  - with a mixture of acetic anhydride–acetic acid in the presence of boron trifluoride at 0° (54%) [3082];
  - with acetic anhydride in the presence of boron trifluoride in ethyl ether at 15° (49%) [3042];
  - with acetylating complex mixture at 60° (17%) [2746].

– Preparation by partial demethylation of phloroacetophenone trimethyl ether,

  - with hydriodic acid in acetic anhydride at r.t. (96%) [3083];
  - with aluminium chloride [2747,2832,2833,3084,3085], in nitrobenzene (sole product) [2832], in acetonitrile for 6 h at 45° (90%) [2747], in boiling ethyl ether (79%) [3084] or without solvent at 110° (30%) [3085];
  - with hydrobromic acid in acetic acid [3083].

- Also obtained by reaction of boron trifluoride etherate on 2,4-diacetyl-3,5-dimethoxyphenol in acetic acid (61%) [3042].
- Also obtained (major product) by reaction of acetyl chloride on phloroglucinol trimethyl ether with aluminium chloride in boiling petroleum ether [3086].
- Preparation by UV light irradiation of 3,5-dimethoxyphenyl acetate at 25° [2193] (photo-Fries rearrangement), in cyclohexane (62%), in isopropanol (45%) or in ethyl ether (32%).

Isolation from natural sources

- From the Bark of *Phytophthora* — and *Hendersonula* — infected *Citrus limon* [3087].
- From *Xanthoxylum piperitum* De Candolle [3088,3089], from the essential oils of *Xanthoxylum aubertia* (*Evodia aubertia*) (10%) [2921] and of *Xanthoxylum alatum* Roxb. (Rutaceae) [2921].
- From the root bark of *Fagara okinawaensis* Nakai (0.5%) [3081].
- From the leaves of *Hippomane mancinella* L. [2920].
- From the essential oil of *Artemisia brevifolia* Wallich [3090] or indigenous species of *Artemisia gallica* Willd. [3091].
- From the essential oil of *Eucalyptus Bakeri* Maiden [2919].
- From various species of *Geijera* [2919].
- From *Blumea balsamifera* DC [2918,3092].
- A new flavone, hinokiflavone was obtained from the leaves of *Chamaecyparis obtusa* Endlicher (Cupressaceae) [2259]. Hinokiflavone pentamethyl ether treated with alkali gave Xanthoxylin,
  - with potassium hydroxide [2260];
  - by boiling in methanolic barium hydroxide solution (77%) [3093].
- A flavonoid, Sciadopitysin, was obtained from the leaves of *Sciadopitys verticillata*. Xanthoxylin was obtained in high yield by degradation of sciadopitysin trimethyl ether in boiling methanolic barium hydroxide solution [3093,3094].
- A flavonoid, Tricin, was obtained from the Khapli wheat leaves *(Triticum Dicoccum).*
- Xanthoxylin was obtained by degradation of tricin trimethyl ether in boiling 80% solution of potassium hydroxide in ethanol [3095].
- In the steam distillates of resins from *Xanthorrhoea preissi*, *Xanthorrhoea reflexa*, *Xanthorrhoea tateana* F. Muell. and *Xanthorrhoea arborea* R. Br. [2584,2761,2862,2876].

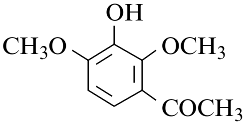
m.p. 87–88° [2432], 86° [3078], 85–88° [3086], 85° [2921], 84–85° [2746,2837], 83°5 [2920], 83° [2827,3079], 82–83° [2916,2918,2919,2936,3080,3081, 3085,3096], 82° [2214,2242,2860,2862,3082], 81°5 [3042], 81° [3084], 80°5–81° [3087], 80–85° [3095], 80–81° [2139,3077], 80° [3090], 79° [2584,2761], 78°5–79°5 [2830], 78–80° [3089], 78–79° [2422,2861];

b.p.$_{18}$ 175–185° [2761], b.p.$_{20}$ 185° [2584];

^{1}H NMR [2205,2830,3087], ^{13}C NMR [1821], IR [2205,2830,3078,3081,3087]; UV [2214,2920,3087], MS [2830,3087].

1-(3-Hydroxy-2,4-dimethoxyphenyl)ethanone

[23133-83-7] $C_{10}H_{12}O_4$ mol.wt. 196.20



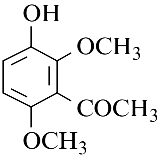
Syntheses

- Preparation from 2,6-dimethoxyphenol or its acetate by reaction of refluxing acetic anhydride in the presence of few drops of concentrated sulfuric acid, followed by saponification of 3-acetoxy-2,4-dimethoxyacetophenone formed with 2 N sodium hydroxide (good yield) [3058].
- Preparation from 3-acetoxy-2,4-dimethoxyacetophenone by hydrolysis with 2 N hydrochloric acid in refluxing methanol (78%) [2403].

m.p. 79–80° [2403,3058]; ^{1}H NMR [2403], IR [2403].

1-(3-Hydroxy-2,6-dimethoxyphenyl)ethanone

[56358-74-8] $C_{10}H_{12}O_4$ mol.wt. 196.20



Isolation from natural sources

- Identification in liquid wastes from eucalyptus wood and kraft lignin charring [2868].

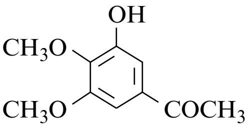
From Microorganisms

- Ketone identified from metabolism of 2,6-dimethoxyacetophenone in the rat [2374].

^{1}H NMR [2374], MS [2374].

1-(3-Hydroxy-4,5-dimethoxyphenyl)ethanone

[114012-82-7] $C_{10}H_{12}O_4$ mol.wt. 196.20

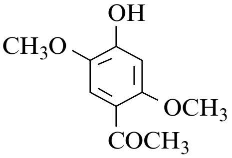


Isolation from natural sources

- Identified by flame-ionization gas-chromatography and gas chromatography–mass spectrometry into liquid wastes from eucalyptus wood and kraft lignin charring [2868].

1-(4-Hydroxy-2,5-dimethoxyphenyl)ethanone

[13909-71-2] $C_{10}H_{12}O_4$ mol.wt. 196.20



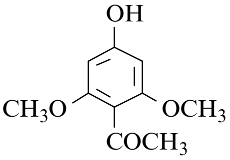
Syntheses

- Preparation by Fries rearrangement of 2,5-dimethoxyphenyl acetate with aluminium chloride [2410].
- Preparation by nuclear oxidation of 2,5-dimethoxyacetophenone with peracetic acid [2410].

m.p. 117–118° [2410].

1-(4-Hydroxy-2,6-dimethoxyphenyl)ethanone

[13246-14-5] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

- Preparation by reaction of acetic anhydride on phloroglucinol dimethyl ether with boron trifluoride,
  - in ethyl ether at 15° (49%) [3042];
  - in acetic acid at 0° (24%) [3082].
- Preparation by reaction of phloroglucinol dimethyl ether with boron trifluoride-acetic acid complex at 100° (40%) [2242].
- Preparation by reaction of acetonitrile on phloroglucinol dimethyl ether (Hoesch reaction) [2837,3077], (32%) [3077].
- Preparation by reaction of aluminium chloride on phloroacetophenone trimethyl ether in refluxing chlorobenzene [2837].
- Also obtained from 4-(benzyloxy)-2,6-dimethoxyacetophenone by heating with concentrated hydrochloric acid in acetic acid [2838].
- Also obtained by saponification of 4-(benzoyloxy)-2,6-dimethoxyacetophenone with 8% methanolic potassium hydroxide at r.t. [3077].

Isolation from natural sources

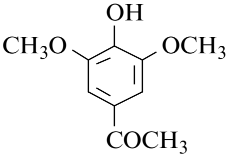
- From *Pancratium maritimum* (Amaryllidaceae) [3041].

m.p. 186° [2837], 185°5 [3077], 185° [2242], 184–185° [3082], 183–184° [2838], 76–78° [3041]. One of the reported melting points is obviously wrong.

^{1}H NMR [3041], ^{13}C NMR [3041], IR [3041], EIMS [3041].

1-(4-Hydroxy-3,5-dimethoxyphenyl)ethanone *(Acetosyringone)*

[2478-38-8] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

- Preparation by adding a solution of 4-hydroxy-3-iodo-5-methoxyacetophenone and cupric chloride in DMF to a solution of sodium methoxide in methanol and heating between 105° and 120° (86%) [2527].
- Preparation by reaction of sodium methoxide with 5-iodo-acetovanillone in methanol in the presence of copper catalyst (53%) [2570].
- Also obtained by debenzylation of 4-(benzyloxy)-3,5-dimethoxyacetophenone by means of a cold saturated solution of hydrogen bromide in acetic acid (52%) [3097].
- Also obtained by Fries rearrangement of 2,6-dimethoxyphenyl acetate with aluminium chloride in nitrobenzene at r.t. (8–14%) [2807,3098,3099].
- Also refer to: [1828].

Isolation from natural sources

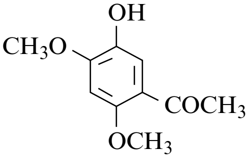
- From hairy roots cultures of *Nicotiana tabacum* and of *Atropa belladonna* [3100].

- In cell suspension cultures of *Hyoscyamus albus* [3101].
- From birch lignin sulfonic acid by treatment with hot aqueous "alkali" (0.8%) [2807].
- Identified by flame-ionization gas chromatography and gas chromatography–mass spectrometry into liquid wastes from eucalyptus wood and kraft lignin charring [2868].

m.p. 125° [2527], 123–124° [2570], 122–123° [3098], 121–122° [3099], 120°5–121°5 [2807], 120–125° [2572], 117° [3097];
^{1}H NMR [3100], UV[2269,3100], MS [3100].

1-(5-Hydroxy-2,4-dimethoxyphenyl)ethanone

[91061-75-5] $C_{10}H_{12}O_4$ mol.wt. 196.20



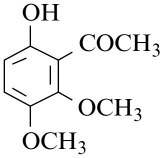
Syntheses

- Preparation by Fries rearrangement of 2,4-dimethoxyphenyl acetate,
  - with aluminium chloride in nitrobenzene at 25° [2410,3102], (50%) [3102];
  - with 36.2% boron trifluoride in acetic acid, first at r.t. overnight, then at 70° for 2 h (81%) [3103].
- Preparation by saponification of 5-acetyl-2,4-dimethoxyphenyl acetate with sodium hydroxide in dilute ethanol at 40° (90%) [2849].

m.p. 155–156° [3102,3103], 154° [2849].

1-(6-Hydroxy-2,3-dimethoxyphenyl)ethanone

[22248-13-1] $C_{10}H_{12}O_4$ mol.wt. 196.20



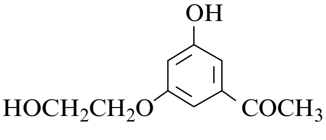
Syntheses

- Preparation by reaction of dimethyl sulfate on 3,6-di-hydroxy-2-methoxyacetophenone with potassium carbonate in boiling benzene (67%) [2781].
- Also refer to: [3104] (compound **III**).

b.p.$_{22}$ 162–163° [2781].

1-[3-Hydroxy-5-(2-hydroxyethoxy)phenyl]ethanone

[63437-86-5] $C_{10}H_{12}O_4$ mol.wt. 196.20



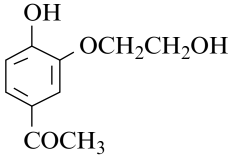
Synthesis

- Obtained by reaction of 2-chloroethanol with 3,5-di-hydroxyacetophenone in refluxing aqueous sodium hydroxide for 5 h under nitrogen atmosphere (29%) [3105].

m.p. 140° [3105].

1-[4-Hydroxy-3-(2-hydroxyethoxy)phenyl]ethanone

[63437-85-4] $C_{10}H_{12}O_4$ mol.wt. 196.20



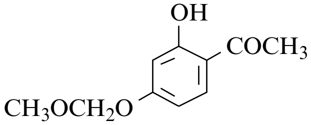
Synthesis

– Preparation by hydrogenolysis of 4-(benzyloxy)-3-(2-hydroxyethoxy)acetophenone in ethanol under hydrogen atmosphere in the presence of 5% Pd/C for 45 min (98%) [3105].

m.p. 108–109° [3105].

1-[2-Hydroxy-4-(methoxymethoxy)phenyl]ethanone

[65490-08-6] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

– Preparation by reaction of chloromethyl methyl ether,

- with resacetophenone in the presence of potassium carbonate in acetone at r.t. for 2.5 h (65%) [3106], at 20° for 20 h (70–75%) [3107] or at reflux for 3–6 h [3108];
- with resacetophenone disodium salt in a methanol/toluene mixture (27%) [3109].

N.B.: Preparation by selective methoxymethylation of resacetophenone (no accuracy) (91%) [3110].

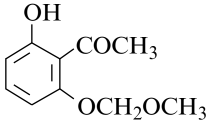
– Also refer to: [3111].

oily residue [3106], colourless oil [3110]; TLC [3107];
b.p.$_{0.15}$ 110° [3107], b.p.$_{0.2}$ 118–122° [3109], b.p.$_{3}$ 138° [3106].

CAUTION! Traces of acids or water in the residue may result in a violent decomposition of the material during distillation [3106]; m.p. 36° [3109];
^{1}H NMR [3106,3107,3110], ^{13}C NMR [3107], IR [3106,3107], MS [3107,3110].

1-[2-Hydroxy-6-(methoxymethoxy)phenyl]ethanone

[78646-28-3] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

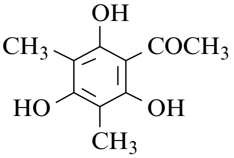
– Obtained by treatment of 2,6-dihydroxyacetophenone with methoxymethyl chloride in the presence of potassium carbonate in refluxing acetone for 2 h (85%) [3112].

– Also obtained by adding methoxymethyl chloride to a solution of 2,6-dihydroxyacetophenone monosodium salt prepared by reaction of sodium hydride with the keto phenol in DMF at r.t. (60%) [3113].

light yellow oil [3112]; ^{1}H NMR [3112], IR [3112], EIMS [3112].

1-(2,4,6-Trihydroxy-3,5-dimethylphenyl)ethanone

[13383-63-6] $C_{10}H_{12}O_4$ mol.wt. 196.20



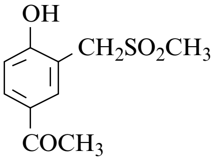
Syntheses

- Preparation by reaction of acetonitrile on 1,3-dimethyl-phloroglucinol (Hoesch reaction) [3114,3115], (66%) [3114].
- Preparation by Friedel–Crafts acylation of 1,3-dimethyl-phloroglucinol with acetic acid in the presence of boron trifluoride (63%) [3116].
- Also obtained by reaction of methyl iodide with phloroacetophenone in the presence of potassium hydroxide in 80% aqueous methanol (12%) [3117], of potassium hydroxide in anhydrous methanol (5%) [2437,2876] or sodium methoxide in methanol [2878].
- Also obtained by demethylation of 2-hydroxy-4,6-dimethoxy-3,5-dimethylacetophenone [3118].
- Also refer to: [3119–3121].

m.p. 226–229° [3116], 221–222° [3115], 220–222° [2876], 219° [3117], 218° [3114]; UV [2878,3117], MS [3114].

1-[4-Hydroxy-3-[(methylsulfonyl)methyl]phenyl]ethanone

[49640-12-2] $C_{10}H_{12}O_4S$ mol.wt. 228.27



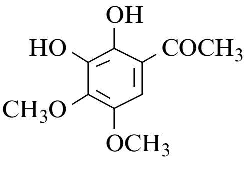
Syntheses

- Obtained by reaction of 3′-chloromethyl-4′-hydroxyacetophenone with magnesium methylsulfinate in refluxing aqueous methanol for 18 h (52%) [2544].
- Also refer to: [3122–3125].

m.p. 207–209° [2544].

1-(2,3-Dihydroxy-4,5-dimethoxyphenyl)ethanone

[119892-31-8] $C_{10}H_{12}O_5$ mol.wt. 212.20

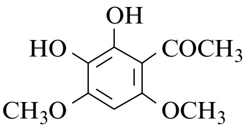


Synthesis

- Refer to: [3126] (Japanese patent).

1-(2,3-Dihydroxy-4,6-dimethoxyphenyl)ethanone

[21919-66-4] $C_{10}H_{12}O_5$ mol.wt. 212.20



Syntheses

- Preparation by hydrolysis of 3-acetoxy-2-hydroxy-4,6-di-methoxyacetophenone,

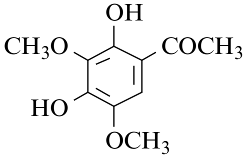
  - with 6 N hydrochloric acid in refluxing ethanol (92%) [3127];
  - with 10% potassium hydroxide at r.t. [3128,3129], (90%) [3128].

- Preparation by hydrolysis of 2-hydroxy-3,4,6-trimethoxyacetophenone with 30% hydrobromic acid in acetic acid at r.t. (42%) [2814].
- Preparation by catalytic hydrogenolysis of 2,3-bis(benzyloxy)-4,6-dimethoxy-acetophenone in the presence of 10% Pd/C in ethyl acetate (89%) [3130].
- Also obtained by reaction of hydrobromic acid with 3-ethoxy-2-hydroxy-4,6-dimethoxy-acetophenone in acetic acid at r.t. (major compound) [3131].
- Also obtained (by-product) by reaction of acetyl chloride with 1,2,3,5-tetramethoxybenzene in ethyl ether (3%) [3131].
- Also refer to: [3132,3133].

m.p. 165°2–166°5 [2814], 165–167°5 [3131], 164–165° [3128], 160–165° [3127], 160–162° [3130];
^{1}H NMR [3131], IR [3130,3131], MS [3131].

1-(2,4-Dihydroxy-3,5-dimethoxyphenyl)ethanone

[198203-68-8] $C_{10}H_{12}O_5$ mol.wt. 212.20



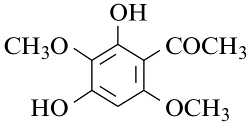
Synthesis

- Obtained (poor yields) by treatment of 4-hydroxy-3,5-di-methoxyacetophenone or 3,4,5-trimethoxy-acetophenone with alkaline hydrogen peroxide (pH 11) irradiating with UV light (254 nm) at 40° for 2 h (<1%) [3134].

N.B.: The silylated product was analyzed by gas chromatography–mass spectrometry (GC-MS).

1-(2,4-Dihydroxy-3,6-dimethoxyphenyl)ethanone

[7499-99-2] $C_{10}H_{12}O_5$ mol.wt. 212.20



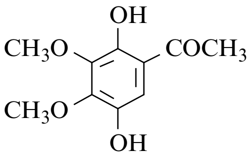
Syntheses

- Preparation by reaction of acetonitrile, (Hoesch reaction),
  - on 2,6-bis(benzyloxy)-1,4-dimethoxybenzene (47%) [3135], (71%) [3136];
  - on 2,5-dimethoxyresorcinol [2884,3137,3138].

m.p. 129° [2884,3138], 128–129° [3136], 125–130° [3135].

1-(2,5-Dihydroxy-3,4-dimethoxyphenyl)ethanone

[69616-56-4] $C_{10}H_{12}O_5$ mol.wt. 212.20



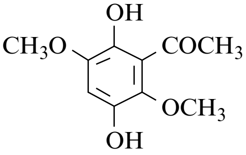
Synthesis

- Obtained from 2-hydroxy-3,4-dimethoxyacetophenone by persulfate oxidation (Elbs reaction) (30%) [2820], (10%) [3057].

m.p. 119–121° [3057], 119° [2820].

1-(2,5-Dihydroxy-3,6-dimethoxyphenyl)ethanone

[6212-45-9] $C_{10}H_{12}O_5$ mol.wt. 212.20



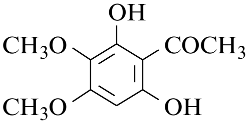
Syntheses

- Preparation by metallation of 2,5-dimethoxyhydroquinone-bis-[tetrahydropyranyl-(2)-ether], followed by treatment of the intermediate aryllithium compound with acetic anhydride in tetrahydrofuran at r.t. (82%) [2564].
- Preparation by reaction of acetic acid on 2,5-dimethoxy-hydroquinone diacetate with boron trifluoride etherate at 75° [2998,3139], (55%) [3139].
- Preparation by catalytic hydrogenolysis of 2,5-bis(benzyloxy)-3,6-dimethoxyacetophenone in the presence of palladium oxide in ethanol (96%) [2450].

m.p. 170° [3139], 94° [2450,2564].

1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)ethanone

[74556-32-4] $C_{10}H_{12}O_5$ mol.wt. 212.20



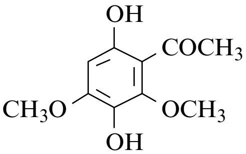
Syntheses

- Preparation by reaction of dimethyl sulfate on 2,3,4,6-tetrahydroxyacetophenone with sodium hydroxide in boiling ethanol (94%) [2443].
- Also obtained by reaction of acetyl chloride on 5-hydroxy-1,2,3-trimethoxybenzene (antiarol) with aluminium chloride in nitrobenzene [3140].
- Also obtained by reaction of acetyl chloride on 1,2,3,5-tetramethoxybenzene with aluminium chloride in nitrobenzene at r.t. [3140–3142].
- Preparation by cleavage of 2,6-dihydroxy-3,4-dimethoxyacetophenone mono-2,4-dinitrophenyl ether with piperidine by heating in a steam bath (76%) [3127].
- Also obtained by cleavage of 2,3,4-trimethoxy-6-isopropoxyacetophenone or 6-hydroxy-2,3,4-trimethoxyacetophenone with hydrobromic acid in acetic acid (23%) [3127].

m.p. 166–168° [2443], 162–163° [3141,3142], 160–161° [3140], 135°6–135°9 [3127].

1-(3,6-Dihydroxy-2,4-dimethoxyphenyl)ethanone

[6962-57-8] $C_{10}H_{12}O_5$ mol.wt. 212.20



Syntheses

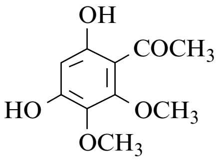
- Preparation by Fries rearrangement of 1,4-diacetoxy-2,6-di-methoxybenzene with aluminium chloride in nitrobenzene at r.t. [2446,3127,3143,3144], (57%) [3144], (47–56%) [3143] and (34%) [2446].
- Preparation by reaction of 2,6-dimethoxyhydroquinone with boron trifluoride-acetic acid complex at 28–30° (60%) [2242].
- Preparation from 2-hydroxy-4,6-dimethoxyacetophenone by persulfate oxidation (Elbs reaction) (36%) [3080], (7%) [2830].

- Also obtained (by-product) by reaction of acetyl chloride on 1,2,3,5-tetramethoxybenzene with aluminium chloride in carbon disulfide [3145].
- Preparation by hydrolysis of 3-acetoxy-6-hydroxy-2,4-dimethoxyacetophenone with refluxing 5% methanolic hydrochloric acid (major product) [3146] or refluxing 10% ethanolic hydrochloric acid [3127].
- Also refer to: [2777,3147].

m.p. 164–165° [3080], 162–163° [2446,3144–3146], 162–162°5 [3127], 162° [2242,3143], 161–162° [2830];
^{1}H NMR [2830,3146], ^{13}C NMR [2328], IR [2830,3146], UV [3146], MS [2830,3146].

1-(4,6-Dihydroxy-2,3-dimethoxyphenyl)ethanone

[103777-42-0] $C_{10}H_{12}O_5$ mol.wt. 212.20

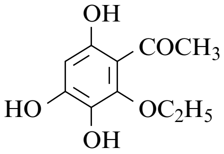


Synthesis

- Preparation from 4-(benzyloxy)-6-hydroxy-2,3-dimethoxyacetophenone by hydrogenolysis with hydrogen in the presence of Pd/C [3148].

1-(2-Ethoxy-3,4,6-trihydroxyphenyl)ethanone

[63635-41-6] $C_{10}H_{12}O_5$ mol.wt. 212.20



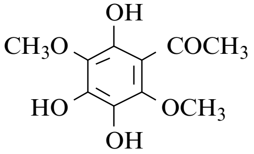
Synthesis

- Preparation by hydrogenolysis of 2-ethoxy-3,4,6-tris-(benzyloxy)acetophenone with 5% Pd/C in ethanol at r.t. (96%) [2442,3149].

m.p. 169° [2442,3149];
^{1}H NMR [2442,3149], IR [2442,3149].

1-(2,4,5-Trihydroxy-3,6-dimethoxyphenyl)ethanone

[15994-32-8] $C_{10}H_{12}O_6$ mol.wt. 228.20



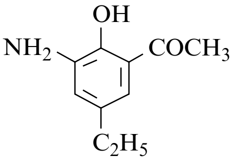
Syntheses

- Obtained by hydrogenolysis of 4-benzyloxy-2,5-dihydroxy-3,6-dimethoxyacetophenone with hydrogen in the presence of 30% Pd/C [3150].
- Also obtained by persulfate oxidation of 2,4-dihydroxy-3,6-dimethoxyacetophenone (Elbs reaction) [3151].
- Also refer to: [3152,3153].

m.p. 142–144° [3152], 140–142° [3154], 137°5–138°5 [3150], 131–134° [3155];
^{1}H NMR [3151], IR [3151,3154].

1-(3-Amino-5-ethyl-2-hydroxyphenyl)ethanone

[70977-78-5] $C_{10}H_{13}NO_2$ mol.wt. 179.22



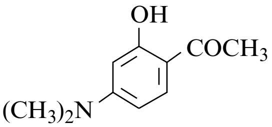
Synthesis

- Preparation by catalytic hydrogenation of 5-ethyl-2-hydroxy-3-nitroacetophenone in the presence of 5% Pd/C in ethanol at 25° (70%) [1898], (60%) [1897].

m.p. 50–51° [1898], 48–51° [1897].

1-[4-(Dimethylamino)-2-hydroxyphenyl]ethanone

[107070-69-9] $C_{10}H_{13}NO_2$ mol.wt. 179.22



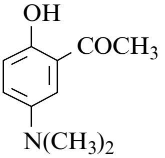
Syntheses

- Preparation by reaction of dimethyl sulfate with 4-amino-2-hydroxyacetophenone in the presence of sodium carbonate in boiling water for 40 min (49%) [3156].
- Also refer to: [3157,3158].

m.p. 118° [3156]; ^{1}H NMR [3156], ^{13}C NMR [3159], IR [3156].

1-[5-(Dimethylamino)-2-hydroxyphenyl]ethanone

[49619-68-3] $C_{10}H_{13}NO_2$ mol.wt. 179.22



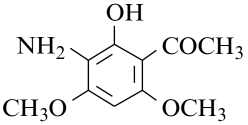
Synthesis

- Preparation by catalytic hydrogenation of 2-hydroxy-5-nitro-acetophenone with formalin in the presence of palladium chloride (82%) [2899].

m.p. 76°5–77°5 [2899]; ^{1}H NMR [2899], IR [2899].

1-(3-Amino-2-hydroxy-4,6-dimethoxyphenyl)ethanone

[81325-91-9] $C_{10}H_{13}NO_4$ mol.wt. 211.22



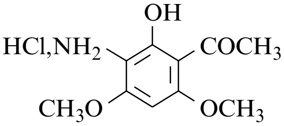
Synthesis

- Preparation by adding a hot solution of stannous chloride in hydrochloric acid to a solution of 2-acetoxy-4,6-dimethoxy-3-nitroacetophenone in ethanol containing zinc dust and heating the mixture in a steam bath (48%) [2139].

m.p. 118–119° [2139]; ^{1}H NMR [2139], IR [2139].

1-(3-Amino-2-hydroxy-4,6-dimethoxyphenyl)ethanone *(Hydrochloride)*

[81325-92-0] $C_{10}H_{13}NO_4$, HCl mol.wt. 247.68



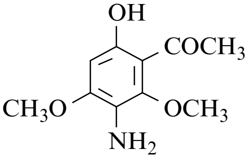
Synthesis

- Preparation by treatment of a solution of 2-hydroxy-4,6-di-methoxy-3-nitroacetophenone in ethanol with zinc dust and a solution of stannous chloride in hydrochloric acid, and heating in a steam bath (50%) [2139].

m.p. 171° [2139].

1-(3-Amino-6-hydroxy-2,4-dimethoxyphenyl)ethanone

[81325-95-3] $C_{10}H_{13}NO_4$ mol.wt. 211.22



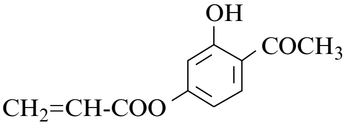
Synthesis

- Preparation by adding a solution of stannous chloride in hydrochloric acid to a hot solution of 6-hydroxy-2,4-di-methoxy-3-phenylazoacetophenone in ethanol containing zinc dust, and heating the mixture in a steam bath (73%) [2139].

m.p. 75° [2139]; ^{1}H NMR [2139].

1-[4-(Acryloyloxy)-2-hydroxyphenyl]ethanone

$C_{11}H_{10}O_4$ mol.wt. 206.20



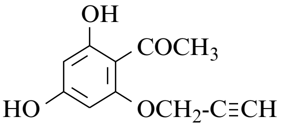
Synthesis

- Preparation by reaction of acryloyl chloride with resacetophenone in the presence of triethylamine in ethyl ether (25%) [2295].

m.p. 55–57° [2295]; ^{1}H NMR [2295], IR [2295].

1-[2,4-Dihydroxy-6-(2-propynyloxy)phenyl]ethanone

[53771-24-7] $C_{11}H_{10}O_4$ mol.wt. 206.20



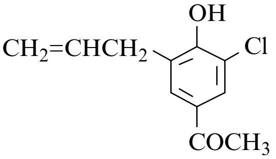
Synthesis

- Obtained (poor yield) by reaction of 2-propynyl bromide with phloroacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (5%) [3160].

m.p. 142–143° [3160]; UV [3160].

1-[3-Chloro-4-hydroxy-5-(2-propenyl)phenyl]ethanone

[51832-82-7] $C_{11}H_{11}ClO_2$ mol.wt. 210.66

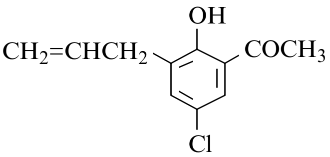


Synthesis

- Preparation by Claisen rearrangement of 4-(allyloxy)-3-chloroacetophenone [3161,3162].

1-[5-Chloro-2-hydroxy-3-(2-propenyl)phenyl]ethanone

[186956-46-7] $C_{11}H_{11}ClO_2$ mol.wt. 210.66



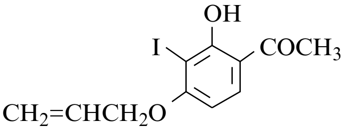
Synthesis

- Obtained by Claisen rearrangement of 2-(allyloxy)-5-chloroacetophenone in refluxing N,N-diethylaniline at 220° for 4 h [3163].

Colourless oil [3163]; b.p.$_{0.1}$ 140° [3163]; ^{1}H NMR [3163], IR [3163].

1-[2-Hydroxy-3-iodo-4-(2-propenyloxy)phenyl]ethanone

[72511-76-3] $C_{11}H_{11}IO_3$ mol.wt. 318.11



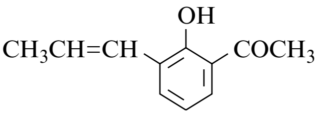
Synthesis

- Obtained by partial allylation of 2,4-dihydroxy-3-iodo-acetophenone with allyl bromide in the presence of potassium carbonate in refluxing acetone for 4–5 h (52%) [3164].

m.p. 91–92° [3164]; ^{1}H NMR [3164].

1-[2-Hydroxy-3-(1-propenyl)phenyl]ethanone

[67127-96-2] $C_{11}H_{12}O_2$ mol.wt. 176.22



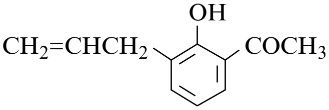
Synthesis

- Preparation by isomerization of 3-allyl-2-hydroxy-acetophenone using bis(benzonitrile)-palladous chloride in refluxing toluene (90%) [1897,3165].

b.p.$_{18}$ 153–155° [1897,3165].

1-[2-Hydroxy-3-(2-propenyl)phenyl]ethanone

[58621-39-9] $C_{11}H_{12}O_2$ mol.wt. 176.22



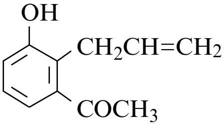
Syntheses

- Preparation by thermal Claisen rearrangement of 2-(allyl- oxy)acetophenone (m.p. 19–21°) [3166],
  - in refluxing N,N-diethylaniline at 220° for 4 h [3163];
  - in boiling dichlorobenzene (30%) [3167];
  - without solvent at 260–270° (85%) [3168];
  - without solvent at reflux under nitrogen atmosphere for 5 h [3169], (66%) [3166].
- Also obtained by photolysis of o-allylphenyl acetate in cyclohexane (major product) (photo-Fries rearrangement) [3170].
- Also refer to: [3171–3173].

light yellow liquid [3166], colourless oil [3163];
b.p.$_{0.3}$ 110° [3166], b.p.$_{20}$ 135–138° [3168], b.p.$_{0.1}$ 135–138° [3163], b.p. 258° [3172]; ^1H NMR [3163,3166,3167], ^{13}C NMR [3166], IR [3163,3166,3167], MS [3166].

1-[3-Hydroxy-2-(2-propenyl)phenyl]ethanone

[58621-37-7] $C_{11}H_{12}O_2$ mol.wt. 176.22



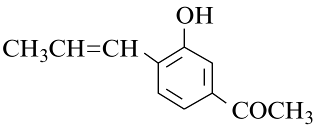
Syntheses

- Preparation by thermal Claisen rearrangement of 3-(allyloxy)acetophenone,
  - in boiling dichlorobenzene (10%) [3167];
  - in DMF at 220° (53%) [2357];
  - without solvent at 220° (43%) [2357].

oil [2357]; ^1H NMR [2357,3167], IR [2357,3167], MS [2357].

1-[3-Hydroxy-4-(1*E*)-1-propenylphenyl]ethanone

[430474-15-0] $C_{11}H_{12}O_2$ mol.wt. 176.22



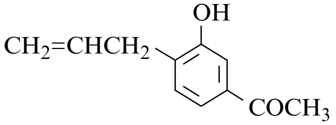
Synthesis

- Obtained by isomerization of 1-[3-hydroxy-4-(2-propenyl)phenyl]ethanone in the presence of polymer-supported iridium catalyst in THF at r.t.

(92% *trans*) [3174,3175].
^1H NMR [3175].

1-[3-Hydroxy-4-(2-propenyl)phenyl]ethanone

[58621-38-8] $C_{11}H_{12}O_2$ mol.wt. 176.22



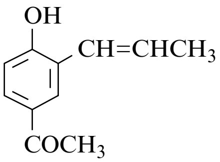
Syntheses

- Preparation by thermal Claisen rearrangement of 3-(allyloxy)acetophenone,
  - in boiling dichlorobenzene (50%) [3167];
  - in DMF at 220° (27%) [2357];
  - without solvent at 220° (22%) [2357].
- Also obtained by UV light irradiation of 3-(allyloxy)acetophenone in benzene or cyclohexane under nitrogen (8%) [3167].

m.p. 75–76° [3167], 62–64° [2357]; ^{1}H NMR [2357,3167], IR [2357,3167], MS [2357].

1-[4-Hydroxy-3-(1-propenyl)phenyl]ethanone

[53889-99-9] $C_{11}H_{12}O_2$ mol.wt. 176.22

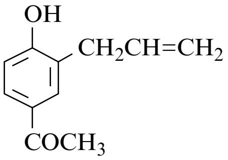


Synthesis

- Refer to: [3176].

1-[4-Hydroxy-3-(2-propenyl)phenyl]ethanone

[1132-05-4] $C_{11}H_{12}O_2$ mol.wt. 176.22



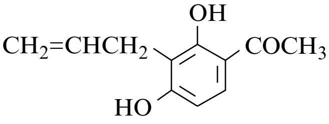
Syntheses

- Preparation by thermal Claisen rearrangement of 4-(allyloxy)acetophenone,
  - in boiling N,N-dimethylaniline [3177];
  - in diphenyl ether at 185° [3178] or at reflux (76%) [3179];
  - without solvent at 200–210° (78%) [3180], at 200–230°(96%) [3181] or at 260–270° (64%) [3168].
- Also obtained by UV light irradiation of 4-(allyloxy)acetophenone in benzene or cyclohexane under nitrogen (13%) [3167].

m.p. 115°9–116°7 [3179], 115–116° [3180], 115° [3177];
b.p.$_4$ 164–165° [3168], b.p.$_{21}$ 200–201° [3177];
^{1}H NMR [3167], IR [3167], UV [3179], MS [3181].

1-[2,4-Dihydroxy-3-(2-propenyl)phenyl]ethanone

[38987-00-7] $C_{11}H_{12}O_3$ mol.wt. 192.21



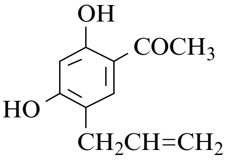
Syntheses

- Preparation by thermal Claisen rearrangement of 4-(allyloxy)-2-hydroxyacetophenone without solvent at 200–210° (85%) [3182].
- Also refer to: [3182–3184].

m.p. 133° [3182]; UV [3183].

1-[2,4-Dihydroxy-5-(2-propenyl)phenyl]ethanone

[38987-01-8] $C_{11}H_{12}O_3$ mol.wt. 192.21



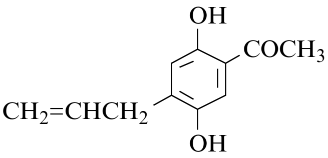
Syntheses

- Preparation by demethylation of 5-allyl-4-hydroxy-2-methoxyacetophenone with aluminium chloride in ethyl ether or acetonitrile [3183].
- Preparation by reaction of boron tribromide on 5-allyl-4-hydroxy-2-methoxyacetophenone in methylene chloride, first at −78°, then at −45° (52%) [2671, 2678,2679].

m.p. 74–76° [2671,2678,2679], 55° [3183]; ^{1}H NMR [2678,2679], UV [3183].

1-[2,5-Dihydroxy-4-(2-propenyl)phenyl]ethanone

[174901-51-0] $C_{11}H_{12}O_3$ mol.wt. 192.21

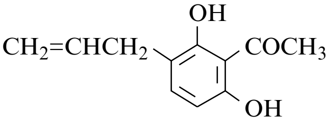


Synthesis

- Refer to: [3185].

1-[2,6-Dihydroxy-3-(2-propenyl)phenyl]ethanone

[17488-71-0] $C_{11}H_{12}O_3$ mol.wt. 192.21



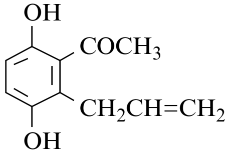
Syntheses

- Obtained from 8-acetyl-6-allyl-7-hydroxy-4-methyl-coumarin by alkaline degradation with 12% aqueous sodium hydroxide solution by heating in a water bath (80%) [3186].
- Also refer to: [2590,2997].

m.p. 63–65° [3186]; ^{13}C NMR [2590].

1-[3,6-Dihydroxy-2-(2-propenyl)phenyl]ethanone

[40815-79-0] $C_{11}H_{12}O_3$ mol.wt. 192.21



Syntheses

– Preparation by thermal Claisen rearrangement of 5-(allyloxy)-2-hydroxyacetophenone without solvent at 200–220° (36%) [3181] or at 220–230° (74–75%) [2357,3187].

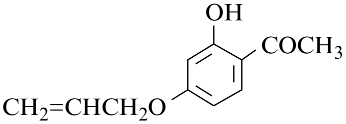
– Also obtained by thermal reaction of 2-acetyl-1,4-benzo-quinone,

- with allyltrimethylstannane in benzene (27%) or acetonitrile (18%) [2355];
- with allyltributylstannane in benzene (29%) [2355].

m.p. 107°5 [3187], 103–104° [2357], 78–80° [2355];
^{1}H NMR [2355,2357,3181], IR [2355,2357], MS [2355,2357,3181].

1-[2-Hydroxy-4-(2-propenyloxy)phenyl]ethanone

[40815-74-5] $C_{11}H_{12}O_3$ mol.wt. 192.21



Syntheses

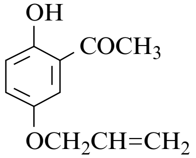
– Preparation by reaction of allyl bromide on resacetophenone with potassium carbonate,

- in refluxing methyl ethyl ketone (90%) [2671,2678,2679];
- in refluxing acetone (70%) [3182].

oil [2671,2678,2679]; b.p.$_9$ 156–157° [3182].

1-[2-Hydroxy-5-(2-propenyloxy)phenyl]ethanone

[40815-75-6] $C_{11}H_{12}O_3$ mol.wt. 192.21



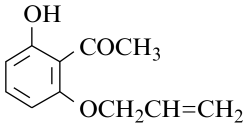
Synthesis

– Preparation by reaction of allyl bromide on quinacetophenone with potassium carbonate in refluxing acetone (86%) [2226,2357], (73%) [3187] or in refluxing methyl ethyl ketone (52%) [3181].

m.p. 59–60° [2226,2357,3187];
^{1}H NMR [2357,3181], IR [2357], MS [2357,3181].

1-[2-Hydroxy-6-(2-propenyloxy)phenyl]ethanone

[23226-84-8] $C_{11}H_{12}O_3$ mol.wt. 192.21



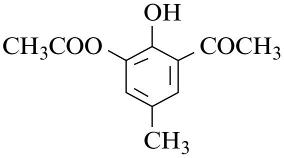
Synthesis

– Preparation by reaction of allyl bromide with 2,6-dihydroxy-acetophenone in the presence of potassium carbonate in refluxing acetone (52%) [2997].

m.p. 45°5–46°5 [2997]; b.p.$_{0.15}$ 128–132° [2997].

1-[3-(Acetyloxy)-2-hydroxy-5-methylphenyl]ethanone

[77347-23-0] $C_{11}H_{12}O_4$ mol.wt. 208.21



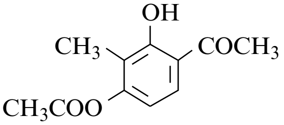
Synthesis

- Preparation by reaction of m-chloroperoxybenzoic acid with 2,6-diacetyl-4-methylphenol using chloroform and trifluoroacetic acid as solvent at r.t. (39%) [3188].

m.p. 80–81° [3188]; ^{1}H NMR [3188], MS [3188].

1-[4-(Acetyloxy)-2-hydroxy-3-methylphenyl]ethanone

[144224-86-2] $C_{11}H_{12}O_4$ mol.wt. 208.21



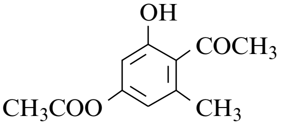
Syntheses

- Preparation by reaction of acetyl chloride with 2,4-di-hydroxy-3-methylacetophenone in the presence of triethylamine in methylene chloride at 0° for 2 h then at r.t. overnight (82%) [2891].
- Also refer to: [2675].

m.p. 71–73° [2891]; ^{1}H NMR [2891], ^{13}C NMR [2891], IR [2891].

1-[4-(Acetyloxy)-2-hydroxy-6-methylphenyl]ethanone

[57600-87-0] $C_{11}H_{12}O_4$ mol.wt. 208.21

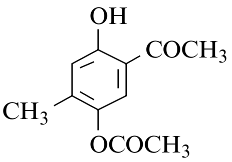


Synthesis

- Preparation by treatment of a mixture of alkylated resorcinols with acetic anhydride and acetic acid in the presence of zinc chloride at 140–145°, followed by suitable separation [3189].

1-[5-(Acetyloxy)-2-hydroxy-4-methylphenyl]ethanone

[126570-32-9] $C_{11}H_{12}O_4$ mol.wt. 208.21

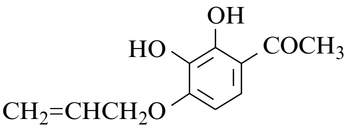


Syntheses

- Preparation by partial acetylation of 2,5-dihydroxy-4-methylacetophenone [2024].
- Also obtained (by-product) by Fries rearrangement of 2-methylhydroquinone diacetate with aluminium chloride [2024].

m.p. 109° [2024].

1-[2,3-Dihydroxy-4-(2-propenyloxy)phenyl]ethanone



$C_{11}H_{12}O_4$ mol.wt. 208.21

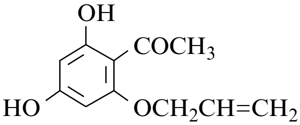
Synthesis

- Obtained by reaction of allyl bromide on gallacetophenone with sodium bicarbonate in refluxing acetone–ethanol mixture (16%) [2817].

m.p. 84° [2817].

1-[2,4-Dihydroxy-6-(2-propenyloxy)phenyl]ethanone

[76609-35-3]



$C_{11}H_{12}O_4$ mol.wt. 208.21

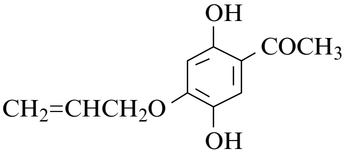
Synthesis

- Preparation by tosylation of phloroacetophenone with p-toluenesulfonyl chloride (2.2 mol) in acetone in the presence of potassium carbonate, followed by allylation with allyl bromide (1.2 mol) and final detosylation with methanolic potassium hydroxide [3190].

m.p. 144–145° [3190].

1-[2,5-Dihydroxy-4-(2-propenyloxy)phenyl]ethanone

[92831-82-8]



$C_{11}H_{12}O_4$ mol.wt. 208.21

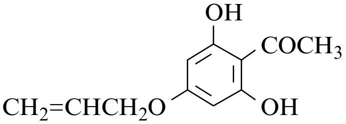
Synthesis

- Preparation from 4-(allyloxy)-2-hydroxyacetophenone by persulfate oxidation (Elbs reaction) (24%) [3191].

m.p. 79–80° [3191]; ^1H NMR [3191].

1-[2,6-Dihydroxy-4-(2-propenyloxy)phenyl]ethanone

[35028-03-6]



$C_{11}H_{12}O_4$ mol.wt. 208.21

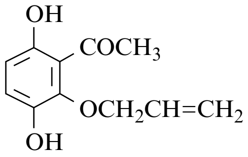
Synthesis

- Preparation from 2,4-diacetyl-5-(allyloxy)resorcinol by selective deacetylation by refluxing in 1 N sodium hydroxide for 1 h [2856].

m.p. 145–146° [2856]; ^1H NMR [2856].

1-[3,6-Dihydroxy-2-(2-propenyloxy)phenyl]ethanone

[33539-24-1] $C_{11}H_{12}O_4$ mol.wt. 208.21



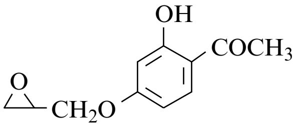
Syntheses

- Easy preparation by reduction of 2-acetyl-3-(allyloxy)-1,4-benzoquinone using conventional methods [2869].
- Obtained (low yield) by reaction of 2-acetyl-1,4-benzo-quinone with an excess of allyl alcohol at r.t., with exclusion of light [2869].

m.p. 68–69° [2869]; ^{1}H NMR [2869], IR [2869].

1-[2-Hydroxy-4-(oxiranylmethoxy)phenyl]ethanone

[61270-24-4] $C_{11}H_{12}O_4$ mol.wt. 208.21



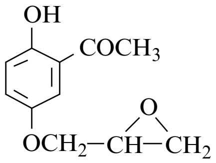
Synthesis

- Preparation by reaction of epichlorohydrin with resaceto-phenone in the presence of potassium hydroxide, in refluxing ethanol [2270] or in a concentrated aqueous solution at 120° [3192].

m.p. 78° [3192], 72–73° [2270].

1-[2-Hydroxy-5-(oxiranylmethoxy)phenyl]ethanone

[16139-53-0] $C_{11}H_{12}O_4$ mol.wt. 208.21



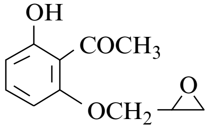
Synthesis

- Refer to: [3193,3194].

m.p. 76–79° [3194].

1-[2-Hydroxy-6-(oxiranylmethoxy)phenyl]ethanone

[16130-28-2] $C_{11}H_{12}O_4$ mol.wt. 208.21



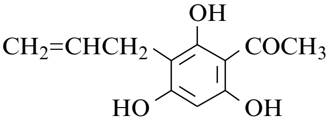
Synthesis

- Preparation by reaction of epichlorohydrin with 2,6-dihydroxyacetophenone in the presence of potassium hydroxide in refluxing ethanol [2270].

m.p. 61–63° [2270].

1-[2,4,6-Trihydroxy-3-(2-propenyl)phenyl]ethanone

[118062-86-5]

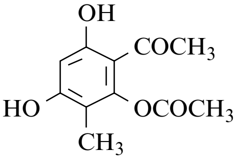


$C_{11}H_{12}O_4$ mol.wt. 208.21

Synthesis

- Refer to: [3195] (Japanese patent).

1-[2-(Acetyloxy)-4,6-dihydroxy-3-methylphenyl]ethanone



$C_{11}H_{12}O_5$ mol.wt. 224.21

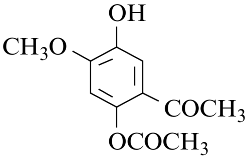
Synthesis

- Obtained (poor yield) by hydrolysis of the ozonide formed from 4,6-dihydroxy-2,3,7-trimethylbenzofuran (m.p. 178°(d)) with dilute ozone (7%) [3196] in ethyl acetate at −30° (13%) [3197].

m.p. 161–164° [3197].

1-[2-(Acetyloxy)-5-hydroxy-4-methoxyphenyl]ethanone

[144152-30-7]



$C_{11}H_{12}O_5$ mol.wt. 224.21

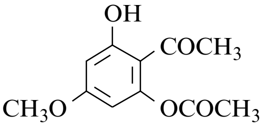
Synthesis

- Obtained by enzymatic deacylation of 2,5-diacetoxy-4-methoxyacetophenone with *Candida cylindracea* lipase in diisopropyl ether at 42–45° (65%) [2388,2897].

Pale yellow viscous oil [2897]; ^{1}H NMR [2897].

1-[2-(Acetyloxy)-6-hydroxy-4-methoxyphenyl]ethanone

[63013-36-5]

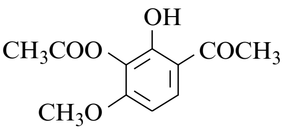


$C_{11}H_{12}O_5$ mol.wt. 224.21

Synthesis

- Refer to: [3198] (compound **5**) (48%).

1-[3-(Acetyloxy)-2-hydroxy-4-methoxyphenyl]ethanone



$C_{11}H_{12}O_5$ mol.wt. 224.21

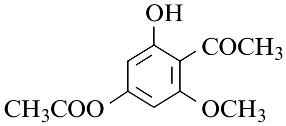
Syntheses

- Preparation by reaction of a mixture of acetic anhydride and acetic acid with 2,6-dimethoxyphenol in the presence of boron trifluoride at 30° (67%) [2813].
- Preparation by reaction of acetyl chloride with 2,3-di-hydroxy-4-methoxyacetophenone in pyridine at 0° (52%) [2403].

m.p. 123°4–125° [2813], 122–123° [2403]; ^{1}H NMR [2403], IR [2403].

1-[4-(Acetyloxy)-2-hydroxy-6-methoxyphenyl]ethanone

[29376-66-7]



$C_{11}H_{12}O_5$ mol.wt. 224.21

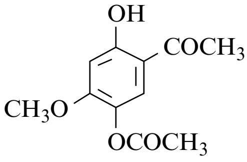
Synthesis

- Preparation by reaction of diazomethane with 4-acetoxy-2,6-dihydroxyacetophenone in tetrahydrofuran at r.t. (55%) [2905].

m.p. 86–89° [2905].

1-[5-(Acetyloxy)-2-hydroxy-4-methoxyphenyl]ethanone

[69470-86-6]



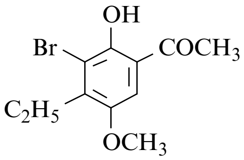
$C_{11}H_{12}O_5$ mol.wt. 224.21

Syntheses

- Preparation by Fries rearrangement of 2-methoxyhydroquinone diacetate with boron trifluoride in acetic acid (90%) [2849].
- Obtained by reaction of methyl iodide with 5-acetoxy-2,4-di-hydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (19%) [2906].

m.p. 104° [2849,2906].

1-(3-Bromo-4-ethyl-2-hydroxy-5-methoxyphenyl)ethanone



$C_{11}H_{13}BrO_3$ mol.wt. 273.13

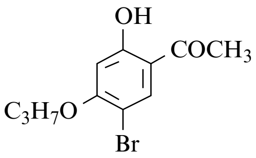
Synthesis

- Obtained by reaction of bromine on 4-ethyl-2-hydroxy-5-methoxyacetophenone in ethyl ether containing a trace of aluminium chloride [3199].

m.p. 83° [3199].

1-(5-Bromo-2-hydroxy-4-propoxyphenyl)ethanone

[57442-27-0]



$C_{11}H_{13}BrO_3$ mol.wt. 273.13

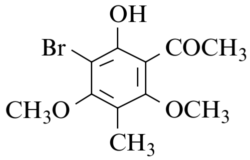
Synthesis

- Preparation by bromination of 2-hydroxy-4-propoxy-acetophenone (1 mol) in 80% acetic acid with bromine (1 mol) at r.t. (25–30°) (90%) [3200].

m.p. 118° [3200].

1-(3-Bromo-2-hydroxy-4,6-dimethoxy-5-methylphenyl)ethanone

[39701-15-0] $C_{11}H_{13}BrO_4$ mol.wt. 289.13



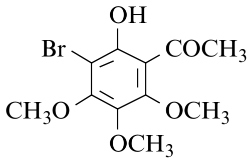
Syntheses

- Preparation by adding an aqueous solution of bromine and potassium bromide to an ethanolic solution of 2-hydroxy-4,6-dimethoxy-5-methylacetophenone (46%) [2873].

m.p. 75–76° [2873]; 1H NMR [2873], IR [2873].

1-(3-Bromo-2-hydroxy-4,5,6-trimethoxyphenyl)ethanone

$C_{11}H_{13}BrO_5$ mol.wt. 305.13



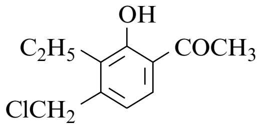
Syntheses

- Preparation by methylation of 3-bromo-2,5-dihydroxy-4,6-dimethoxyacetophenone [2917].
- Preparation by bromination of 6-hydroxy-2,3,4-trimethoxy-acetophenone [2917].

m.p. 89–90° [2917].

1-[4-(Chloromethyl)-3-ethyl-2-hydroxyphenyl]ethanone

[97582-38-2] $C_{11}H_{13}ClO_2$ mol.wt. 212.68



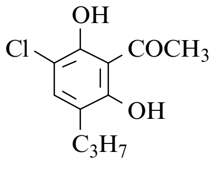
Synthesis

- Preparation by reaction of ethyl chloroformate with 4-(dimethylaminomethyl)-3-ethyl-2-hydroxyacetophenone [2550,2927], (71%) [2550].

m.p. 55–57° [2550]; b.p.$_{0.5}$ 120–130° [2927];
^{1}H NMR [2927], IR [2927].

1-(3-Chloro-2,6-dihydroxy-5-propylphenyl)ethanone

[102624-59-9] $C_{11}H_{13}ClO_3$ mol.wt. 228.68

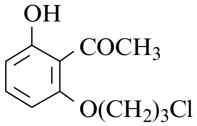


Syntheses

- Preparation according to literature procedures [1892] (compound **1c**).
- Also refer to: [3201].

1-[2-(3-Chloropropoxy)-6-hydroxyphenyl]ethanone

[105277-74-5] $C_{11}H_{13}ClO_3$ mol.wt. 228.68



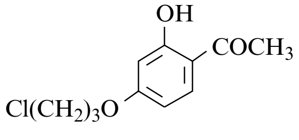
Synthesis

- Preparation by reaction of 1-bromo-3-chloropropane with 2,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 24 h (quantitative yield) [3202].

m.p. 60°5–62°5 [3202]; ^{1}H NMR [3202], MS [3202].

1-[4-(3-Chloropropoxy)-2-hydroxyphenyl]ethanone

[172739-45-6] $C_{11}H_{13}ClO_3$ mol.wt. 228.68



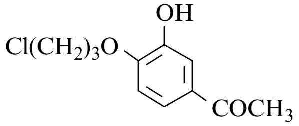
Syntheses

- Preparation by reaction of 1-bromo-3-chloropropane with resacetophenone [3203], in the presence of potassium carbonate in refluxing acetone for 5 h (84%) [3204] or for 16 h (69%) [3205].
- Also refer to: [3206].

m.p. 73–74° [3205], 73° [3204]; ^{1}H NMR [3205], IR [3205].

1-[4-(3-Chloropropoxy)-3-hydroxyphenyl]ethanone

[151719-65-2] $C_{11}H_{13}ClO_3$ mol.wt. 228.68



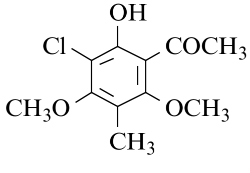
Syntheses

- Obtained by demethylation of 1-[4-(3-chloropropoxy)-3-methoxyphenyl]ethanone in concentrated sulfuric acid at 65° for 23 h (22%) [3207].
- Also refer to: [3208].

m.p. 101–103° [3207]; ^{1}H NMR [3207], MS [3207].

1-(3-Chloro-2-hydroxy-4,6-dimethoxy-5-methylphenyl)ethanone

[31913-64-1] $C_{11}H_{13}ClO_4$ mol.wt. 244.67



Synthesis

- Preparation by reaction of sulfuryl chloride with 6-hydroxy-2,4-dimethoxy-3-methylacetophenone in chloroform at –3° (59%) [3209].

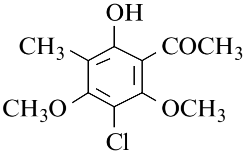
Isolation from natural sources

- Also obtained by alkaline hydrolysis of sordidone dimethyl ether (8-chloro-5,7-dimethoxy-2,6-di-methylchromone) [3209,3210] with refluxing 5% aqueous potassium hydroxide under nitrogen (83%) [3209]. Sordidone is a metabolite isolated from the lichen *Lecanora rupicola* (L) Zahlbr. (syn. *Lecanora sordida* Th. Fr.) [3209].

m.p. 94° [3209,3210]; ^{1}H NMR [3209], IR [3209], MS [3209].

1-(3-Chloro-6-hydroxy-2,4-dimethoxy-5-methylphenyl)ethanone

[23053-45-4] $C_{11}H_{13}ClO_4$ mol.wt. 244.67



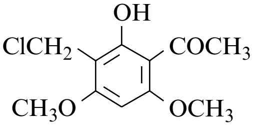
Syntheses

- Preparation by adding of ethereal solution of diazomethane to a solution of 3-chloro-2,4,6-trihydroxy-5-methyl-acetophenone in a mixture of acetone–ethyl ether at r.t. (76%) [2566].
- Preparation by reaction of sulfuryl chloride with 2-hydroxy-4,6-dimethoxy-3-methylacetophenone in chloroform at −3° (43%) [3209].
- Also obtained by alkaline hydrolysis of isosordidone dimethyl ether (6-chloro-5,7-dimethoxy-2,8-dimethylchromone) with refluxing 5% aqueous potassium hydroxide under nitrogen (55%) [3209].

m.p. 108–109° [2566], 108° [3209]; ^{1}H NMR [3209], IR [3209], MS [3209].

1-[3-(Chloromethyl)-2-hydroxy-4,6-dimethoxyphenyl]ethanone

[40356-82-9] $C_{11}H_{13}ClO_4$ mol.wt. 244.67



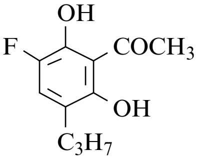
Synthesis

- Preparation by chloromethylation of 2-hydroxy-4,6-di-methoxyacetophenone with chloromethyl methyl ether in acetic acid for 1 h at r.t. (57%) [3211,3212].

m.p. 133–135° (d) [3211,3212].

1-(3-Fluoro-2,6-dihydroxy-5-propylphenyl)ethanone

[102624-71-5] $C_{11}H_{13}FO_3$ mol.wt. 212.22

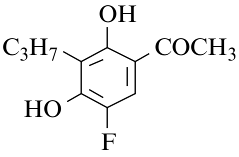


Syntheses

- Preparation according to literature procedures [1892] (compound **1f**).
- Also refer to: [3201].

1-(5-Fluoro-2,4-dihydroxy-3-propylphenyl)ethanone

[119257-53-3] $C_{11}H_{13}FO_3$ mol.wt. 212.22

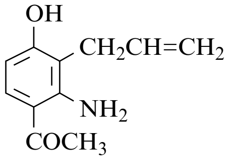


Synthesis

- Refer to: [3213].

1-[2-Amino-4-hydroxy-3-(2-propenyl)phenyl]ethanone

[118684-00-7] $C_{11}H_{13}NO_2$ mol.wt. 191.23



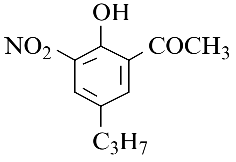
Synthesis

- Preparation by thermal Claisen rearrangement of 4-(allyloxy)-2-aminoacetophenone without solvent at 200° (54%) [2464].

Colourless oil [2464]; ^1H NMR [2464].

1-(2-Hydroxy-3-nitro-5-propylphenyl)ethanone

[70978-38-0] $C_{11}H_{13}NO_4$ mol.wt. 223.23



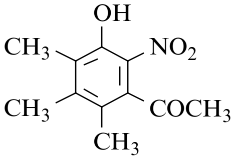
Synthesis

- Preparation by reaction of nitric acid (d = 1.42) on 2-hydroxy-5-propylacetophenone in concentrated sulfuric acid between −15° and −5° [1897,1898], (42%) [1897].

m.p. 67–69° [1898], 67–68° [1897].

1-(3-Hydroxy-4,5,6-trimethyl-2-nitrophenyl)ethanone

[13667-21-5] $C_{11}H_{13}NO_4$ mol.wt. 223.23



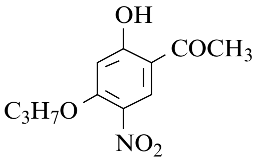
Synthesis

- Preparation by adding a solution of nitric acid (d = 1.38) in concentrated sulfuric acid to a solution of 5-hydroxy-2,3,4-trimethylacetophenone in acetic acid and carbon tetrachloride mixture between −5° and 0° (80%) [3214,3215].

m.p. 99–100° [3214,3215]; IR [3215].

1-(2-Hydroxy-5-nitro-4-propoxyphenyl)ethanone

[70668-14-3] $C_{11}H_{13}NO_5$ mol.wt. 239.23



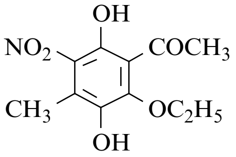
Synthesis

- Preparation by nitration of 2-hydroxy-4-propoxy-aceto-phenone in acetic acid with concentrated nitric acid at 0° [3216].

m.p. 104° [3216].

1-(2-Ethoxy-3,6-dihydroxy-4-methyl-5-nitrophenyl)ethanone

[43140-85-8] $C_{11}H_{13}NO_6$ mol.wt. 255.23



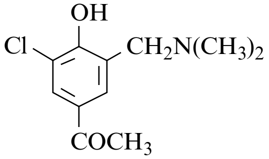
Synthesis

- Preparation by adding to a solution of 5-acetyl-2-methyl-3-nitro-1,4-benzoquinone in ethanol, a solution of pyrrolidine in ethanol. After stirring for 3 min, the solvent was eliminated, excess sulfurous acid was added, and the mixture was allowed to stand overnight (31%) [2583].

yellow viscous oil [2583]; ^{1}H NMR [2583], IR [2583].

1-[3-Chloro-4-hydroxy-5-[(dimethylamino)methyl]phenyl]ethanone

$C_{11}H_{14}ClNO_2$ mol.wt. 227.69



Synthesis

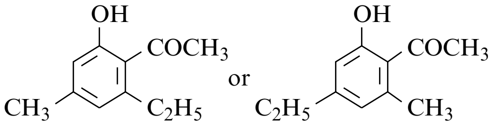
- Preparation by aminomethylation of 3-chloro-4-hydroxy-acetophenone with dimethylamine and formalin in water at 35–40° for 4 h (60%) [3217].

m.p. 112° [3217]; ^{1}H NMR [3217], IR [3217].

1-(2-Ethyl-6-hydroxy-4-methylphenyl)ethanone

or

1-(4-Ethyl-2-hydroxy-6-methylphenyl)ethanone



(I) (II)

$C_{11}H_{14}O_2$ mol.wt. 178.23

Syntheses

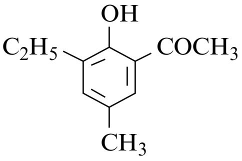
- Mixture obtained by Fries rearrangement of 3-ethyl-5-methylphenyl acetate with aluminium chloride,
  - at 160–170° for 3 h. The two isomeric ketones were separated via their semicarbazide derivatives. However, the respective structures of the isolated ketones have not been attributed. The melting point of one of them (**I**) or (**II**) is 93° (6% yield) and the melting point of the other (**II**) or (**I**) is 18–19° (30% yield) [3218];
  - without solvent at 130° (80%) [2004];
  - in nitrobenzene at 25° (85%) [2004].

N.B.: The 4-ethyl-2-hydroxy-6-methylacetophenone (**II**) is the most likely formula. However, one does not exclude to deal with a mixture (**I** + **II**), especially if working without solvent at 130° [2004].

b.p.$_8$ 170° [2004].

1-(3-Ethyl-2-hydroxy-5-methylphenyl)ethanone

[81591-15-3] $C_{11}H_{14}O_2$ mol.wt. 178.23



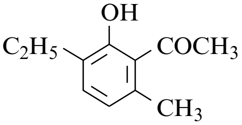
Syntheses

- Obtained by reaction of acetyl chloride on 2-ethyl-4-methylanisole with aluminium chloride in boiling carbon disulfide [2956].
- Also obtained by Fries rearrangement of 2-ethyl-4-methyl-phenyl acetate [2596,2907], (40%) [2907] or 2-ethyl-5-methylphenyl acetate (41%) [2596] with aluminium chloride at 130–140°.

b.p.$_{25}$ 144–146° [2956], b.p.$_{30}$ 153° [2596], b.p. 260° [2907].

1-(3-Ethyl-2-hydroxy-6-methylphenyl)ethanone

[81591-16-4] $C_{11}H_{14}O_2$ mol.wt. 178.23



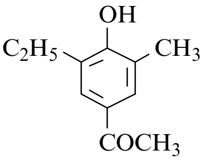
Syntheses

- Obtained (by-product) by reaction of acetyl chloride on 2-ethyl-5-methylanisole with aluminium chloride in carbon disulfide [2233].
- Preparation by Fries rearrangement of 2-ethyl-5-methyl-phenyl acetate by heating with aluminium chloride (30%) [2907].

b.p.$_{12}$ 137–138° [2233], b.p. 270° [2907].

1-(3-Ethyl-4-hydroxy-5-methylphenyl)ethanone

$C_{11}H_{14}O_2$ mol.wt. 178.23



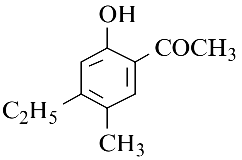
Syntheses

- Preparation by Fries rearrangement at high temperature of 2-ethyl-6-methylphenyl acetate with aluminium chloride (50%) [2233].
- Also obtained (poor yield) by Fries rearrangement of 4-dodecyl-2-ethyl-6-methylphenyl acetate with aluminium chloride (13%) [2957].

m.p. 101–102° [2957], 95°5–96°5 [2233]; b.p.$_{12}$ 180–200° [2957].

1-(4-Ethyl-2-hydroxy-5-methylphenyl)ethanone

[93351-16-7] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

- Obtained by oxidation of 6-ethyl-2,3,5-trimethylbenzofuran with chromium trioxide in acetic acid at 50° for 30 min, followed by saponification of the resulting keto ester with potassium hydroxide in boiling aqueous ethanol for 2 h (65%) [3219].

– Also obtained by Friedel–Crafts acylation of 2,5-diethyl-4-methylanisole (SM) (1 mol) with acetyl chloride (1.5 mol) in the presence of aluminium chloride (1.5 mol) in boiling carbon disulfide. There is elimination of the ortho ethyl group in SM during the reaction [2956].

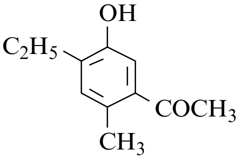
– Also obtained by dehydrogenation of 6-acetyl-3-ethyl-4-methyl-2-cyclohexen-1-one,

  - with a 5% palladium–barium sulfate catalyst at reflux [2612];
  - with a refluxing solution of bromine (16%) in acetic acid [2612,3220].

– Also refer to: [3221].

Yellow oil [2956]; m.p. 52° [2612,3219–3221];
b.p.$_{15}$ 144–147° [2956], b.p.$_{18}$ 154–155° [3219]; IR [3219].

1-(4-Ethyl-5-hydroxy-2-methylphenyl)ethanone



$C_{11}H_{14}O_2$ mol.wt. 178.23

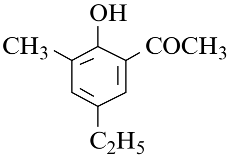
Syntheses

– Preparation by reaction of acetyl chloride on 2-ethyl-4-methylanisole with aluminium chloride in boiling carbon disulfide [2956].

– Preparation by reaction of aluminium chloride on 4-ethyl-5-methoxy-2-methylacetophenone [2956].

m.p. 120–121° [2956].

1-(5-Ethyl-2-hydroxy-3-methylphenyl)ethanone



$C_{11}H_{14}O_2$ mol.wt. 178.23

Syntheses

– Preparation by Fries rearrangement of 4-ethyl-2-methyl-phenyl acetate with aluminium chloride (77%) [223].

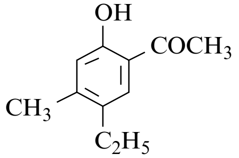
– Also obtained by heating the following phenyl esters with aluminium chloride at high temperature (in these reactions, there is elimination or migration of the bulky alkyl or arylalkyl group located in ortho position of the ester group),

  - from 2-allyl-4-ethyl-6-methylphenyl acetate (74%) [2957];
  - from 2-benzyl-4-ethyl-6-methylphenyl acetate (54%) [2957];
  - from 2-ethyl-6-methylphenyl acetate (noticeable quantity) [2233];
  - from 4-ethyl-2-methyl-6-propylphenyl acetate (12%) [2957].

b.p.$_{20}$ 120–136° [2957], b.p.$_{11}$ 129–131° [2233], b.p.$_{12}$ 130–132° [2233], b.p.$_{18}$ 130–142° [2957],
b.p.$_{13}$ 142–150° [2957].

1-(5-Ethyl-2-hydroxy-4-methylphenyl)ethanone

[27513-07-1] $C_{11}H_{14}O_2$ mol.wt. 178.23



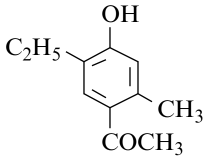
Syntheses

- Preparation by Fries rearrangement of 4-ethyl-3-methyl-phenyl acetate with aluminium chloride at 120° (quantitative yield) [2151], (70%) [2233], (52%) [3222].
- Also obtained (by-product) by reaction of acetyl chloride on 2,4-diethyl-5-methylanisole with aluminium chloride in carbon disulfide [2233].
- Preparation by chromic acid degradation of 5-ethyl-2,3,6-trimethylbenzofuran (60%) [3222].
- Preparation by dehydrogenation of 6-acetyl-4-ethyl-3-methyl-2-cyclohexen-1-one,
  - with a 5% palladium-barium sulfate catalyst at reflux [2612,3004];
  - with a refluxing solution of bromine (16%) in acetic acid [2612,3004].

m.p. 96°5–97° [2612], 96–97° [2151], 94–95° [2233], 94° [3222], 92–94° [3004]; b.p.$_{15}$ 142–143° [2151].

1-(5-Ethyl-4-hydroxy-2-methylphenyl)ethanone

$C_{11}H_{14}O_2$ mol.wt. 178.23



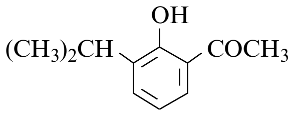
Synthesis

- Obtained (by-product) by Fries rearrangement of 2-ethyl-5-methylphenyl acetate with aluminium chloride at 130–140° [2596].

m.p. 117–118° [2596].

1-[2-Hydroxy-3-(1-methylethyl)phenyl]ethanone

[104175-18-0] $C_{11}H_{14}O_2$ mol.wt. 178.23



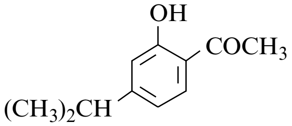
Synthesis

- Preparation by reaction of acetyl chloride with a suspension of aluminium tri-o-isopropylphenoxide in benzene in the presence of aluminium chloride, first at r.t. for 1 h, then on a water bath for 2 h (50%) [2945].

b.p.$_3$ 108–110° [2945].

1-[2-Hydroxy-4-(1-methylethyl)phenyl]ethanone

[91969-72-1] $C_{11}H_{14}O_2$ mol.wt. 178.23



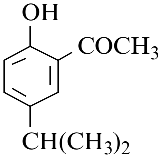
Synthesis

- Obtained by Fries rearrangement of 2-isopropylphenyl acetate with aluminium chloride without solvent at 140°, accompanied by an alkyl group migration (22%) [3223].

b.p.$_{12}$ 129–130° [3223].

1-[2-Hydroxy-5-(1-methylethyl)phenyl]ethanone

[1634-36-2] $C_{11}H_{14}O_2$ mol.wt. 178.23



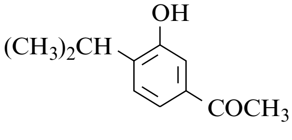
Syntheses

- Preparation by reaction of acetyl chloride on 4-isopropylanisole with aluminium chloride in carbon disulfide at r.t. (48%) [3224].
- Also refer to: [2227].

b.p.$_{10}$ 130–132° [3224].

1-[3-Hydroxy-4-(1-methylethyl)phenyl]ethanone

[1634-62-4] $C_{11}H_{14}O_2$ mol.wt. 178.23



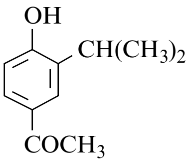
Synthesis

- Preparation by diazotization of 3-amino-4-isopropyl-acetophenone, followed by hydrolysis of the diazonium salt obtained (50%) [1951], (26%) [3224].

m.p. 100–101° [3224], 97–100° [1951].

1-[4-Hydroxy-3-(1-methylethyl)phenyl]ethanone

[1632-59-3] $C_{11}H_{14}O_2$ mol.wt. 178.23



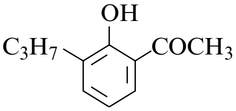
Syntheses

- Preparation by reaction of acetyl chloride on 2-isopropyl-phenol with aluminium chloride in boiling carbon disulfide (44%) [1951].
- Preparation by demethylation of 4-methoxy-3-isopropylacetophenone with boiling pyridinium chloride (30%) [3224].
- Also obtained (poor yield) by Fries rearrangement of 2-isopropylphenyl acetate with aluminium chloride, without solvent at 104° (14%) [3223] or in nitrobenzene at r.t. (13%) [3224].

m.p. 143° [3224], 140° [3223], 139–140° [1951].

1-(2-Hydroxy-3-propylphenyl)ethanone

[93915-84-5] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

– Preparation by hydrogenation of 3-allyl-2-hydroxyacetophenone in the presence of 10% Pd/C and sodium hypophosphite,

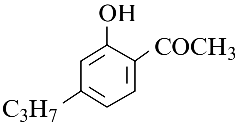
- in dilute ethanol at 20° (92%) [3225];
- in aqueous sodium hydroxide at 50° (88%) [3225].

– Refer to: [3226,3227] (Japanese patents).

^{1}H NMR [3225].

1-(2-Hydroxy-4-propylphenyl)ethanone

$C_{11}H_{14}O_2$ mol.wt. 178.23



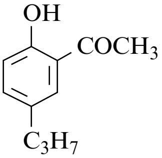
Synthesis

– Preparation by reaction of acetic acid with 3-propylphenol in the presence of zinc chloride for 5 h at 180° (reflux) (Nencki reaction) (40%) [3228].

b.p.$_{0.7}$ 93° [3228], b.p.$_{16}$ 128–131° [3228].

1-(2-Hydroxy-5-propylphenyl)ethanone

[1990-24-5] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

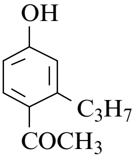
– Preparation by Fries rearrangement of 4-propylphenyl acetate with aluminium chloride [2957,3229] without solvent at 140° [3229].

– Preparation by reaction of acetyl chloride with 4-propylanisole in the presence of aluminium chloride in methylene chloride, first at 0°, then at reflux under nitrogen (86%) [3230].

Yellow liquid [3230];
b.p.$_{0.25}$ 80–85° [3229], b.p.$_{1.2}$ 98–105° [3230], b.p.$_{20}$ 145–147° [2957];
^{1}H NMR [3230]; $n_D^{20} = 1.5365$ [3229].

1-(4-Hydroxy-2-propylphenyl)ethanone

[104174-27-8] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

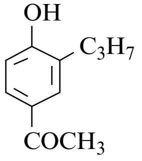
– Obtained by boiling 3-propylphenol with acetic anhydride in the presence of a little sulfuric acid [3228].

– Also refer to: [3231].

b.p.$_{18}$ 121–123° [3228].

1-(4-Hydroxy-3-propylphenyl)ethanone

[61270-28-8] $C_{11}H_{14}O_2$ mol.wt. 178.23



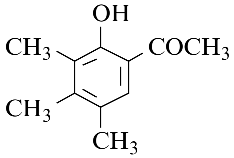
Syntheses

- Preparation by reaction of acetyl chloride on 2-propyl-phenol with aluminium chloride in nitrobenzene at r.t. [2309].
- Also obtained by catalytic hydrogenation of 3-allyl-4-hydroxyacetophenone in the presence of Pd/C in ethanol [2270,2671,2946], (90–100%) [2270,2946] or Raney nickel in ethyl acetate (80%) [3181].

m.p. 90–91° [2270], 89–90° [2946], 87° [2309]; b.p.$_{25}$ 210° [2309];
MS [3181]; pK_a [3181].

1-(2-Hydroxy-3,4,5-trimethylphenyl)ethanone

[58972-39-7] $C_{11}H_{14}O_2$ mol.wt. 178.23



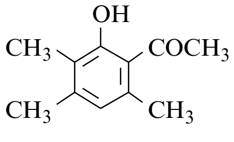
Syntheses

- Preparation by Fries rearrangement of 2,3,4-trimethylphenyl acetate with aluminium chloride at 130–140° (good yield) [2596].
- Preparation by reaction of acetyl chloride on 2,3,4-tri-methylphenol with aluminium chloride in carbon disulfide at r.t. (55%) [3232].
- Also obtained from various aryl esters by heating with aluminium chloride between 130° and 150°, the reaction being accompanied by a migration of methyl groups,
  - 2,3,5-trimethylphenyl acetate [2596,2965], (86%) [2596];
  - 2,4,5-trimethylphenyl acetate (major compound) [2596];
  - 2,4,6-trimethylphenyl acetate [2535,2596], (major compound) [2596].
- Also obtained (poor yield) by heating a mixture of 2,4,6-trimethylphenyl acetate and 4-methyl-phenyl chloroacetate with aluminium chloride (10%) [2535].
- Also obtained by isomerization of 2-hydroxy-3,4,6-trimethylacetophenone by heating with aluminium chloride [2965].

m.p. 43–44° [2596], 42° [2535,2965]; b.p.$_{11}$ 142–144° [2596], b.p. 275–276° [2596];
^{1}H NMR [3232], IR [3232].

1-(2-Hydroxy-3,4,6-trimethylphenyl)ethanone

[163429-79-6] $C_{11}H_{14}O_2$ mol.wt. 178.23

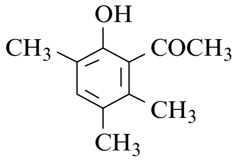


Syntheses

- Preparation by Fries rearrangement of 2,3,5-trimethylphenyl acetate (isopseudocumenol acetate) with aluminium chloride [2965], at 100° for 1.5 h (85%) [3233].
- Also refer to: [3234].

m.p. 46° [2965], 32–34° [3233]; ^{1}H NMR [3233], ^{13}C NMR [3233], IR [3233].

1-(2-Hydroxy-3,5,6-trimethylphenyl)ethanone



$C_{11}H_{14}O_2$ mol.wt. 178.23

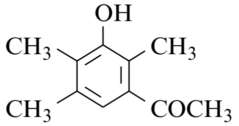
Synthesis

- Obtained (by-product) by Fries rearrangement of 2,4,5-trimethylphenyl acetate (pseudocumenol acetate) with aluminium chloride at 130–140° [2596].

b.p.$_{11}$ 145–146° [2596].

1-(3-Hydroxy-2,4,5-trimethylphenyl)ethanone

[99892-62-3]



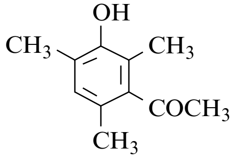
$C_{11}H_{14}O_2$ mol.wt. 178.23

Synthesis

- Preparation by rearrangement of 1α, 5α, 6α, 8α-tetramethyl-2α-*H*, 4α-*H* -3,9-dioxatricyclo [3.3.1.0 ^{2,4}] nonan-7-one by treatment with sodium ethoxide in ethanol (81%) [2979].

m.p. 115° [2979]; ^{1}H NMR [2979], IR [2979], MS [2979].

1-(3-Hydroxy-2,4,6-trimethylphenyl)ethanone



$C_{11}H_{14}O_2$ mol.wt. 178.23

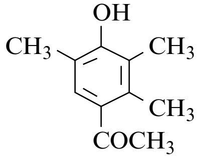
Synthesis

- Preparation by reaction of acetyl chloride on 2,4,6-trimethylanisole (mesitol methyl ether) with aluminium chloride in boiling carbon disulfide (20%) [2956].

m.p. 81–82° [2956].

1-(4-Hydroxy-2,3,5-trimethylphenyl)ethanone

[61405-64-9]



$C_{11}H_{14}O_2$ mol.wt. 178.23

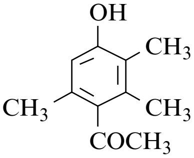
Syntheses

- Preparation by Fries rearrangement of 2,3,6-trimethylphenyl acetate with aluminium chloride at 165–170° for 15 min (94%) [3220].
- Also obtained by dehydrogenation of 4-acetyl-2,3,6-trimethyl-2-cyclohexen-1-one at reflux for 30 min in the presence of 5% palladium–barium sulfate [2612], (10%) [3220].

m.p. 132–133° [2612,3220]; IR [3220], UV [3220].

1-(4-Hydroxy-2,3,6-trimethylphenyl)ethanone

[156483-08-8] $C_{11}H_{14}O_2$ mol.wt. 178.23

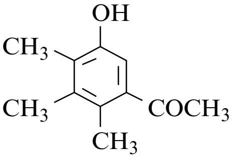


Synthesis

- Refer to: [3235].

1-(5-Hydroxy-2,3,4-trimethylphenyl)ethanone

[13667-28-2] $C_{11}H_{14}O_2$ mol.wt. 178.23



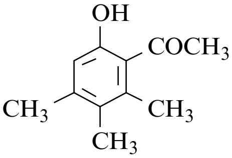
Syntheses

- Preparation from 2,3,4-trimethyl-5-nitroacetophenone via reduction with stannous chloride and following diazotization of the resulting 5-amino-2,3,4-trimethylacetophenone (68%) [3236], (53%) [3214].
- Also obtained by rearrangement of 3-acetyl-4,4,5-trimethyl-2,5-cyclohexadiene-1-one in 68% sulfuric acid at 40° (29%) [2989].

m.p. 168° [3214,3236]; ^{1}H NMR [2989], IR [2989,3236], MS [2989].

1-(6-Hydroxy-2,3,4-trimethylphenyl)ethanone

[27192-99-0] $C_{11}H_{14}O_2$ mol.wt. 178.23



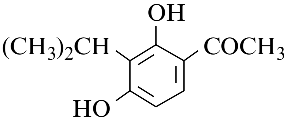
Syntheses

- Preparation by Fries rearrangement of 3,4,5-trimethylphenyl acetate with aluminium chloride without solvent at 130° [2596,3237], (50%) [3237].
- Preparation by reaction of acetyl chloride on 3,4,5-trimethylanisole with aluminium chloride in boiling carbon disulfide (45%) [1811].
- Also obtained via pyrolysis of 1,3,7,8-tetramethyl-2-oxabicyclo[4.2.0]octa-3,7-dien-5-one (2,6-dimethyl-4-pyrone–Butyne-2–Adduct) in refluxing o-dichlorobenzene [3237].

m.p. 83°5–84°5 [1811], 83–84° [2596], 58–60° [3237]; b.p.$_{13}$ 163–166° [2596]; ^{1}H NMR [3237], IR [3237], UV [3237], MS [3237].

1-[2,4-Dihydroxy-3-(1-methylethyl)phenyl]ethanone

[118604-45-8] $C_{11}H_{14}O_3$ mol.wt. 194.23

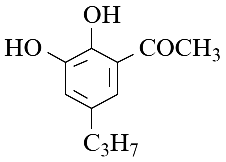


Synthesis

- Preparation by total demethylation of 2,4-dimethoxy-3-iso-propylacetophenone with 48% hydrobromic acid in refluxing acetic acid [3238].

1-(2,3-Dihydroxy-5-propylphenyl)ethanone

[86253-71-6] $C_{11}H_{14}O_3$ mol.wt. 194.23



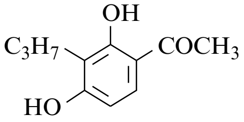
Synthesis

- Preparation by reaction of acetic acid with 2-methoxy-4-propylphenol in the presence of boron trifluoride at 150–155° (81%) [3230].

oil [3230].

1-(2,4-Dihydroxy-3-propylphenyl)ethanone

[40786-69-4] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

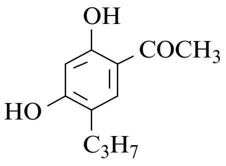
- Preparation by reaction of acetonitrile on 2-propylresorcinol (Hoesch reaction) (86%) [3182].
- Preparation by catalytic hydrogenation of 3-allyl-2,4-di-hydroxyacetophenone using palladium chloride as catalyst in ethanol (quantitative yield) [3182] or Raney nickel in ethyl acetate [2671].
- Preparation by total demethylation of 2,4-dimethoxy-3-propylacetophenone with 48% hydrobromic acid in refluxing acetic acid for 19 h (73%) [3238].
- Also refer to: [2326,2327,3184].

N.B.: Pr indicates the *propyl* group –C_3H_7 in Chem. Abstr., **92**, 6368x (1980) and **98**, 54239b (1983), an usual abbreviation. However, in the two references [2326,2327], Pr represented the *prenyl* group –$CH_2CH=C(CH_3)_2$. Therefore they concern the 2,4-dihydroxy-3-prenylacetophenone and not the above mentioned 2,4-dihydroxy-3-propylacetophenone.

m.p. 127–128° [3182].

1-(2,4-Dihydroxy-5-propylphenyl)ethanone

[63411-87-0] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

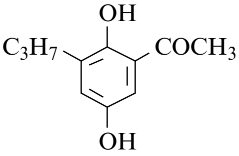
- Preparation by reaction of acetic acid on 4-propylresorcinol with zinc chloride (Nencki reaction) [1839,3018].
- Preparation by reaction of acetonitrile on 4-propylresorcinol (Hoesch reaction) (75%) [2676].
- Preparation by Fries rearrangement of 4-propylresorcinol diacetate in the presence of 4-propylresorcinol with aluminium chloride in nitrobenzene at 50° (quantitative yield) [3020].
- Also refer to: [2326,2327].

N.B.: Pr indicates the *propyl* group -C_3H_7 in Chem. Abstr., **92**, 6368x (1980) and **98**, 54239b (1983), an usual abbreviation. However, in the two references [2326,2327], Pr represented the *prenyl* group –$CH_2CH=C(CH_3)_2$. Therefore they concern the 2,4-dihydroxy-5-prenylacetophenone and not the above mentioned 2,4-dihydroxy-5-propylacetophenone.

m.p. 110–111° [1839], 108–109° [3020], 108° [2676].

1-(2,5-Dihydroxy-3-propylphenyl)ethanone

[83812-26-4] $C_{11}H_{14}O_3$ mol.wt. 194.23

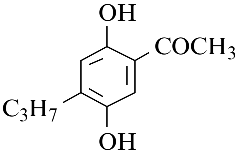


Synthesis

- Refer to: [2271,3239] (patents).

1-(2,5-Dihydroxy-4-propylphenyl)ethanone

$C_{11}H_{14}O_3$ mol.wt. 194.23



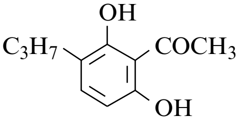
Synthesis

- Obtained (by-product) by reaction of acetyl chloride on 2-propylhydroquinone dimethyl ether with aluminium chloride in boiling carbon disulfide (3%) [3240].

m.p. 85° [3240].

1-(2,6-Dihydroxy-3-propylphenyl)ethanone

[53542-79-3] $C_{11}H_{14}O_3$ mol.wt. 194.23



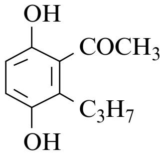
Syntheses

- Preparation from 2,6-dihydroxyacetophenone according to the method [3241], (64%) [3242].
- Also refer to: [1892,3201,3243–3246].

m.p. 84–85° [3242].

1-(3,6-Dihydroxy-2-propylphenyl)ethanone

[106627-41-2] $C_{11}H_{14}O_3$ mol.wt. 194.23



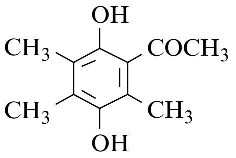
Synthesis

- Preparation by catalytic hydrogenation of 2-allyl-3,6-dihydroxyacetophenone using palladised strontium carbonate catalyst [3240] or Raney nickel (60%) [3181] in ethyl acetate.

m.p. 88° [3240] (monohydrate); MS [3181].

1-(2,5-Dihydroxy-3,4,6-trimethylphenyl)ethanone

[64794-45-2] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

- Obtained by oxidation of 2,4,5,7,8-pentamethyl-4*H*-1,3-benzodioxin-6-ol (PBD)* in aqueous media via 2-(1-hydroxyethyl)-3,5,6-trimethylbenzo-1,4-quinone, without or with aldehyde trapping (method A or B, respectively) [3247].

Oxidant	Method	Yield (%)
$FeCl_3.6\ H_2O$	A (B)	38 (85)
$K_2Cr_2O_7$	A (B)	38 (86)
$AgNO_3$	A (B)	44 (83)
H_2O_2	A (B)	44 (82)
$KMnO_4$	A (B)	32 (68)
NMMO**	- (B)	- (81)

N.B.:

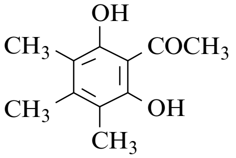
- PBD* is a novel 3-oxa-tocopherol-type stabilizer which is obtained as a mixture of two diastereoisomers by condensation of trimethylhydroquinone with acetaldehyde.
- NMMO** = N-methylmorpholine-N-oxide.
- Preparation by Fries rearrangement of 2,3,5-trimethylhydroquinone diacetate,
  - with aluminium chloride at 220° (51%) [3248];
  - with boron trifluoride–acetic acid complex, followed by saponification of the 3-acetoxy-6-hydroxy-2,4,5-trimethylacetophenone obtained [2909,2994,3249], (71%) [3249], (65%) [2994].
- Also obtained by hydrolysis of 3-(acetyloxy)-6-hydroxy-2,4,5-trimethylacetophenone with 5% methanolic hydrogen chloride (50%) [3250].
- Also refer to: [3251].

m.p. 152° [3248], 111° [2994], 110–113° [3247], 107–109° [3250], 107–108°5 [3249];

^{1}H NMR [3247,3249,3250], ^{13}C NMR [3247,3250], IR [3247,3249]
UV [3248,3249], MS [3249].

1-(2,6-Dihydroxy-3,4,5-trimethylphenyl)ethanone

[66842-24-8] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

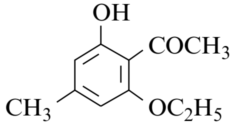
- Obtained by Fries rearrangement of trimethylhydroquinone diacetate with aluminium chloride at 220°, via a secondary rearrangement of the normal product (I) (50%) [3249].

- Also obtained by reaction of aluminium chloride on 5-acetoxy-2-hydroxy-3,4,6-trimethylacetophenone at 220° (53–55%) [3249].
- Also obtained by rearrangement of 2,5-dihydroxy-3,4,6-trimethylacetophenone (I) with aluminium chloride at 220° (29–34%) [3249].

m.p. 136–145° [3249]; ^{1}H NMR [3249], IR [3249], UV [3249], MS [3249].

1-(2-Ethoxy-6-hydroxy-4-methylphenyl)ethanone

[78274-02-9] $C_{11}H_{14}O_3$ mol.wt. 194.23



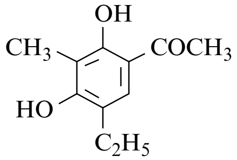
Syntheses

- Obtained by reaction of sodium ethoxide with 3,5-diacetyl-2,6-dimethyl-*4H*-pyran-4-one in ethanol at r.t. (20%) [3252].
- Also obtained by heating 3-acetyl-2,6-dimethyl-4H-pyran-4-one with sodium ethoxide in ethanol (17%) [2940].

m.p. 95° [2940,3252,3253]; ^{1}H NMR [3252], IR [3252], MS [2940,3252].

1-(5-Ethyl-2,4-dihydroxy-3-methylphenyl)ethanone

[140660-34-0] $C_{11}H_{14}O_3$ mol.wt. 194.23

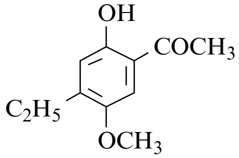


Synthesis

- Preparation by reaction of acetic acid on 4-ethyl-1,3-di-hydroxy-2-methylbenzene with zinc chloride at 95–100° (Nencki reaction) (24%) [2671]. Crystalline product [2671].

1-(4-Ethyl-2-hydroxy-5-methoxyphenyl)ethanone

$C_{11}H_{14}O_3$ mol.wt. 194.23



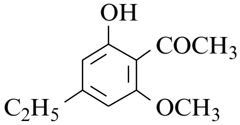
Syntheses

- Preparation from 2-ethylhydroquinone dimethyl ether,
  - by reaction with acetic acid in the presence of boron trifluoride (55%) [3199];
  - by reaction with acetyl chloride in the presence of aluminium chloride in boiling ethyl ether (27%) [2944].
- Preparation by partial methylation of 4-ethyl-2,5-hydroxyacetophenone with dimethyl sulfate in the presence of potassium carbonate in boiling acetone (47%) [2944].
- Also obtained (poor yield) by partial demethylation of 4-ethyl-2,5-dimethoxyacetophenone with 6% aqueous hydrobromic acid in acetic acid at r.t. (9%) [3199].

m.p. 60°4–61° [3199], 60° [2944]; UV [3199].

1-(4-Ethyl-2-hydroxy-6-methoxyphenyl)ethanone

[128546-82-7] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

- Refer to: [3254–3259] (Japanese papers) and [3260].

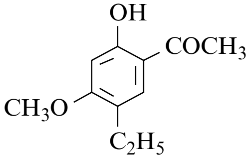
Isolation from natural sources

- From *Juniperus semiglobosa* Regel (Cupressaceae) [3261].

m.p. 85° [3261]; ^{1}H NMR [3261], IR [3261], UV [3261], MS [3261].

1-(5-Ethyl-2-hydroxy-4-methoxyphenyl)ethanone

[4223-85-2] $C_{11}H_{14}O_3$ mol.wt. 194.23

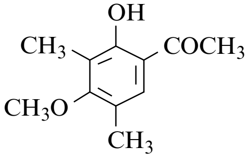


Syntheses

- Preparation by reaction of dimethyl sulfate [3018] or methyl iodide [3004] on 5-ethyl-2,4-dihydroxyacetophenone with potassium carbonate in refluxing acetone.
- Preparation by chromic acid degradation of 5-ethyl-6-methoxy-2,3-dimethylbenzofuran (21%) [3013].

m.p. 49–50° [3004], 48° [3013]; b.p.$_{20}$ 165–167° [3013].

1-(2-Hydroxy-4-methoxy-3,5-dimethylphenyl)ethanone



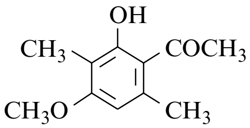
$C_{11}H_{14}O_3$ mol.wt. 194.23

Synthesis

- Preparation by treating 2,4-dihydroxy-3,5-dimethylacetophenone with diazomethane or with dimethyl sulfate and sodium hydroxide [2990].

m.p. 35–36° [2990]; b.p.$_{0.6}$ 90° [2990].

1-(2-Hydroxy-4-methoxy-3,6-dimethylphenyl)ethanone



$C_{11}H_{14}O_3$ mol.wt. 194.23

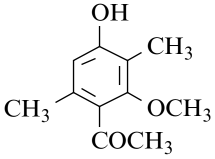
Synthesis

- Obtained by reaction of methyl iodide with 2,4-dihydroxy-6-methylacetophenone in the presence of potassium hydroxide (15%) [2330].

m.p. 90–91° [2330].

1-(4-Hydroxy-2-methoxy-3,6-dimethylphenyl)ethanone

[97761-88-1] $C_{11}H_{14}O_3$ mol.wt. 194.23

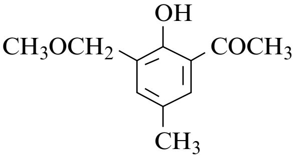


Synthesis

- **N.B.**: Mentioned in the Chem. Abstr. **103**, 85031d (1985). However, this compound does not appear in the original paper [3052] which concerns only some ketones derived of phloroglucinol.

1-[2-Hydroxy-3-(methoxymethyl)-5-methylphenyl]ethanone

[87165-63-7] $C_{11}H_{14}O_3$ mol.wt. 194.23



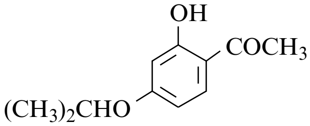
Syntheses

- Preparation from 3-chloromethyl-2-hydroxy-5-methyl-acetophenone (b.p.$_{0.4}$ 110–116°) [2494] by reaction with methanol in the presence of concentrated hydrochloric acid and iron powder at reflux for 3.5–4.5 h (82%) [3262], (49%) [2494].
- Also refer to: [3263].

m.p. 36–38° [3262]; b.p.$_{0.6}$ 95–103° [2494], b.p.$_{20}$ 160–165° [3262]; ^{1}H NMR [2494,3262], ^{13}C NMR [3262], IR [2494,3262].

1-[2-Hydroxy-4-(1-methylethoxy)phenyl]ethanone

[73473-62-8] $C_{11}H_{14}O_3$ mol.wt. 194.23

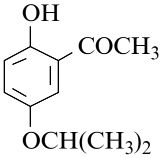


Synthesis

- Refer to: [3264] (Chinese reference) and [3265] (Japanese patent).

1-[2-Hydroxy-5-(1-methylethoxy)phenyl]ethanone

[152810-05-4] $C_{11}H_{14}O_3$ mol.wt. 194.23



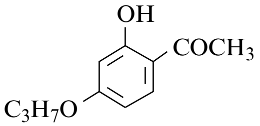
Syntheses

- Preparation by reaction of 2-bromopropane with quinacetophenone in the presence of sodium iodide and potassium carbonate in DMF at 60° for 24 h (51%) [3266].
- Also refer to: [2227,3267].

Oil [3266]; ^{1}H NMR [3266].

1-(2-Hydroxy-4-propoxyphenyl)ethanone

[55329-63-0] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

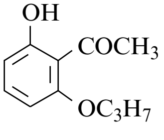
- Preparation by reaction of propyl iodide [2220,3002,3004] or propyl bromide [2220] on resacetophenone,
  - with potassium hydroxide in boiling ethanol [2220,3002];
  - with potassium carbonate in boiling acetone [3004].
- Also refer to: [2326,2327].

N.B.: Pr indicates the *propyl* group -C_3H_7 in Chem. Abstr., **92**, 6368x (1980) and **98**, 54239b (1983), an usual abbreviation. However, in the two references [2326,2327], Pr represented the *prenyl* group –$CH_2CH=C(CH_3)_2$. Therefore they concern the 2-hydroxy-4-prenyloxyacetophenone and not the above mentioned 2-hydroxy-4-propyloxyacetophenone.

m.p. 25° [2220]; b.p.$_{3–4}$ 136° [3002].

1-(2-Hydroxy-6-propoxyphenyl)ethanone

[14718-38-8] $C_{11}H_{14}O_3$ mol.wt. 194.23



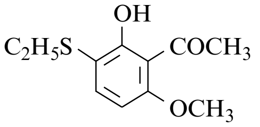
Synthesis

- Preparation by reaction of propyl iodide on 2,6-di-hydroxy-acetophenone with potassium carbonate in refluxing acetone (66%) [2999].

m.p. 70–71° [2999].

1-[3-(Ethylthio)-2-hydroxy-6-methoxyphenyl]ethanone

[126405-82-1] $C_{11}H_{14}O_3S$ mol.wt. 226.30



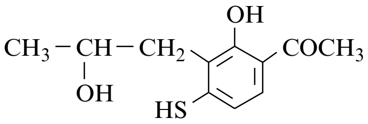
Synthesis

- Preparation by adding 2-hydroxy-3-iodo-6-methoxy-acetophenone and cuprous oxide to a solution of sodium ethyl sulfhydrate, previously prepared from ethanethiol and sodium hydride in DMF [2524].

m.p. 57° [2524]; ^{1}H NMR [2524], IR [2524].

1-[2-Hydroxy-3-(2-hydroxypropyl)-4-mercaptophenyl]ethanone

[167211-59-8] $C_{11}H_{14}O_3S$ mol.wt. 226.30

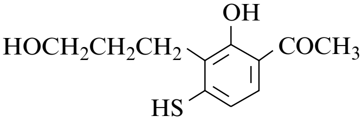


Synthesis

- Preparation in six steps from 3-allyl-2,4-dihydroxy-acetophenone [3268] (Japanese patent).

1-[2-Hydroxy-3-(3-hydroxypropyl)-4-mercaptophenyl]ethanone

[167211-71-4] $C_{11}H_{14}O_3S$ mol.wt. 226.30

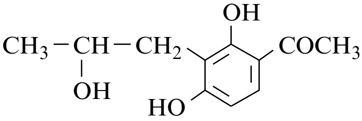


Synthesis

– Refer to: [3268] (Japanese patent).

1-[2,4-Dihydroxy-3-(2-hydroxypropyl)phenyl]ethanone

[167211-56-5] $C_{11}H_{14}O_4$ mol.wt. 210.23

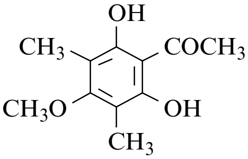


Synthesis

– Refer to: [3268] (Japanese patent).

1-(2,6-Dihydroxy-4-methoxy-3,5-dimethylphenyl)ethanone *(Mallophenone)*

[129399-54-8] $C_{11}H_{14}O_4$ mol.wt. 210.23



Syntheses

– Obtained by degradation of 5-hydroxy-7-methoxy-6,8-dimethylchromone (Leptorumol monomethyl ether) with 2 N potassium hydroxide (30%) [2437].

– Also refer to: [2847,3269].

Isolation from natural sources

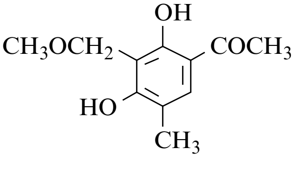
– From the pericarps of *Mallotus japonicus* Muell. Arg. (Euphorbiaceae) [3044].

m.p. 132° [2437], 131–132° [3044];

^{1}H NMR [2437,3044], ^{13}C NMR [3044], IR [2437,3044], UV [2437,2847,3044], MS [3044].

1-[2,4-Dihydroxy-3-(methoxymethyl)-5-methylphenyl]ethanone

[333763-54-5] $C_{11}H_{14}O_4$ mol.wt. 210.23

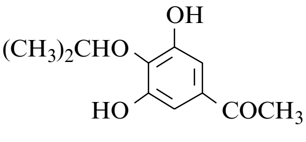


Isolation from natural sources

– From the culture filtrate of chilean strain of *Trichoderma pseudokoningii* [3270].

1-[3,5-Dihydroxy-4-(1-methylethoxy)phenyl]ethanone

[192625-58-4] $C_{11}H_{14}O_4$ mol.wt. 210.23



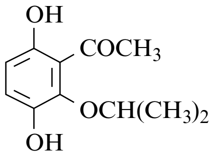
Synthesis

– Preparation by total demethylation of 3′,5′-dimethoxy-4′-isopropoxyacetophenone in two steps via formation of dimethyl acetal (80%) [3271].

^{1}H NMR [3271], MS [3271]; TLC [3271].

1-[3,6-Dihydroxy-2-(1-methylethoxy)phenyl]ethanone

[33539-22-9] $C_{11}H_{14}O_4$ mol.wt. 210.23



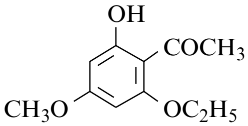
Syntheses

- Easy preparation by reduction of 2-acetyl-3-isopropoxy-1,4-benzoquinone using conventional methods [2869].
- Also obtained (low yield) by reaction of 2-acetyl-1,4-benzoquinone with an excess of isopropanol at r.t., with exclusion of light [2869].

m.p. 90–92° [2869]; ^{1}H NMR [2869], IR [2869].

1-(2-Ethoxy-6-hydroxy-4-methoxyphenyl)ethanone

[76554-79-5] $C_{11}H_{14}O_4$ mol.wt. 210.23



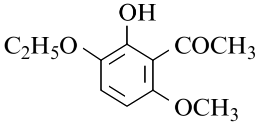
Syntheses

- Preparation by reaction of dimethyl sulfate on 6-ethoxy-2,4-dihydroxyacetophenone with potassium carbonate in boiling acetone (81%) [2851].
- Preparation by reaction of diethyl sulfate on 2,6-dihydroxy-4-methoxyacetophenone with potassium carbonate in boiling acetone [2851].
- Preparation by reaction of diazoethane on 2,6-dihydroxy-4-methoxyacetophenone [2845].

m.p. 134° [2851], 133–134° [2845].

1-(3-Ethoxy-2-hydroxy-6-methoxyphenyl)ethanone

[126405-76-3] $C_{11}H_{14}O_4$ mol.wt. 210.23



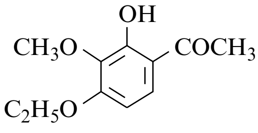
Synthesis

- Preparation by adding 2-hydroxy-3-iodo-6-methoxy-acetophenone and cuprous iodide to a solution of sodium ethoxide, previously prepared from ethyl alcohol and sodium hydride in DMF [2524].

m.p. 70° [2524]; ^{1}H NMR [2524], IR [2524].

1-(4-Ethoxy-2-hydroxy-3-methoxyphenyl)ethanone

[69616-59-7] $C_{11}H_{14}O_4$ mol.wt. 210.23



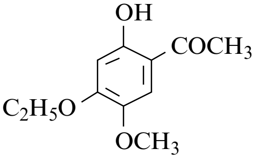
Synthesis

- Preparation by reaction of ethyl iodide with 2,4-dihydroxy-3-methoxyacetophenone in the presence of potassium carbonate in refluxing acetone (64%) [2818].

m.p. 76–77° [2818]; ^{1}H NMR [2818], IR [2818].

1-(4-Ethoxy-2-hydroxy-5-methoxyphenyl)ethanone

[75672-62-7] $C_{11}H_{14}O_4$ mol.wt. 210.23



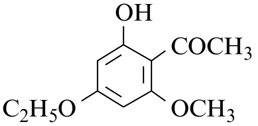
Synthesis

- Preparation by reaction of dimethyl sulfate with 4-ethoxy-2,5-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (82%) [3056].

m.p. 94–95° [3056].

1-(4-Ethoxy-2-hydroxy-6-methoxyphenyl)ethanone

[76554-80-8] $C_{11}H_{14}O_4$ mol.wt. 210.23



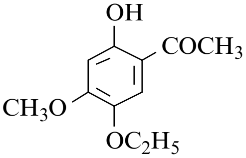
Syntheses

- Preparation by reaction of diazoethane on 2,4-dihydroxy-6-methoxyacetophenone [2845].
- Also refer to: [3272].

m.p. 56–57° [2845].

1-(5-Ethoxy-2-hydroxy-4-methoxyphenyl)ethanone

[75672-59-2] $C_{11}H_{14}O_4$ mol.wt. 210.23



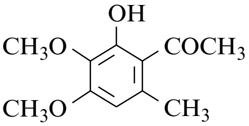
Synthesis

- Preparation by reaction of diethyl sulfate with 2,5-di-hydroxy-4-methoxyacetophenone in the presence of potassium carbonate in refluxing acetone (71%) [3056].

m.p. 101–102° [3056].

1-(2-Hydroxy-3,4-dimethoxy-6-methylphenyl)ethanone

[63542-37-0] $C_{11}H_{14}O_4$ mol.wt. 210.23



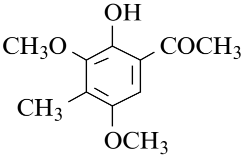
Syntheses

- Preparation by reaction of acetyl chloride on 3,4,5-trimethoxytoluene with aluminium chloride,
  - in ethyl ether at r.t. [3273,3274], (50%) [3273];
  - in refluxing methylene chloride (67%) [3035].

m.p. 94° [3274], 92° [3273], 77–78° [3035];
^{1}H NMR [3035], ^{13}C NMR [3035], IR [3035], MS [3035].

1-(2-Hydroxy-3,5-dimethoxy-4-methylphenyl)ethanone

[134255-78-0] $C_{11}H_{14}O_4$ mol.wt. 210.23



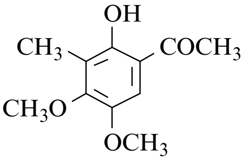
Synthesis

– Obtained by Fries rearrangement of 3-acetoxy-2,6-di-methoxytoluene (oil) with boron trifluoride etherate at 95° for 3 h (63%) [3275].

m.p. 73°5–75° [3275]; TLC [3275];
^{1}H NMR [3275], IR [3275], MS [3275].

1-(2-Hydroxy-4,5-dimethoxy-3-methylphenyl)ethanone

[134255-79-1] $C_{11}H_{14}O_4$ mol.wt. 210.23



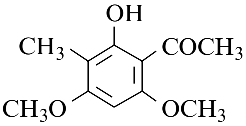
Synthesis

– Obtained by selective demethylation of 3-methyl-2,4,5-tri-methoxyacetophenone (pale yellow oil) with boron tri-chloride in methylene chloride at 0° for 80 min (85%) [3275].

m.p. 86–87° [3275]; ^{1}H NMR [3275], MS [3275].

1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)ethanone

[23121-32-6] $C_{11}H_{14}O_4$ mol.wt. 210.23



Syntheses

– Preparation by reaction of acetonitrile on 3,5-dimethoxy-2-methylphenol (Hoesch reaction) (68%) [2870].

– Preparation by reaction of methyl iodide, in the presence of potassium carbonate in boiling acetone,

- on 2,4-dihydroxy-6-methoxy-3-methylacetophenone [3036];
- on 2,6-dihydroxy-4-methoxy-3-methylacetophenone [3037];
- on phloroacetophenone [1821,2716,2861,2870,3276–3279], (37–42%) [3277,3279], (21–28%) [2861,2870,3278].

– Also obtained (by-product) by reaction of methyl iodide with phloroacetophenone in 10% methanolic potassium hydroxide (<3%) [2876].

– Preparation by partial methylation of 2,4,6-trihydroxy-3-methylacetophenone,

- with diazomethane in ethyl ether–methanol mixture at 0° (good yield) [2861];
- with dimethyl sulfate in the presence of potassium carbonate in boiling acetone [2877,3278], (71%) [3278];
- with methyl iodide, in the presence of potassium carbonate in boiling acetone [2870,2886,3278].

– Preparation by partial demethylation of 2,4,6-trimethoxy-3-methylacetophenone with aluminium chloride in acetonitrile at 30° for 6 h (90%) [2747].

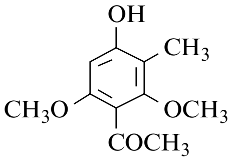
- Preparation by reaction of acetyl chloride,
  - with 2,4,6-trimethoxytoluene in the presence of aluminium chloride in ethyl ether at r.t. (51%) [2747];
  - with 2-hydroxy-4,6-dimethoxytoluene in the presence of aluminium chloride in nitrobenzene at r.t. [3054] according to [3280].
- Preparation by reaction of acetic anhydride and acetic acid,
  - on 3,5-dimethoxy-2-methylphenol with boron trifluoride at 20–30° (60%) [3042];
  - on 2,4,6-trimethoxytoluene with boron trifluoride at 20–30° or at 100° [3042].
- Also obtained on deacylation of 2,4-diacetyl-3,5-dimethoxy-6-methylphenol acetate with 10% hydrochloric acid in ethanol [3042].

Isolation from natural sources

- From the leaves and bark of *Acradenia franklinii* (Kippist) (Rutaceae) [3276].
- From the stem wood of *Euphorbia quinquecostata* Volk. (Euphorbiaceae) [3281].
- From *Euphorbia portulacoides* (Euphorbiaceae) [3282].

m.p. 145° [3042], 144–145° [2861], 144° [3278], 143–144° [2870], 143° [3276], 142–143° [3279], 142° [2886], 141°5–143° [2747], 141–143° [3277], 141–142° [1956,3036,3037], 140–141° [2876], 130–131° [3281];
^{1}H NMR [2877,3281,3282], ^{13}C NMR [1821,3281], IR [3276,3281],
UV [3276,3281], MS [3281].

1-(4-Hydroxy-2,6-dimethoxy-3-methylphenyl)ethanone



$C_{11}H_{14}O_4$ mol.wt. 210.23

Synthesis

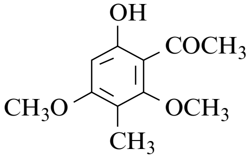
- Preparation by catalytic hydrogenolysis of 4-(benzyloxy)-2,6-dimethoxy-3-methylacetophenone with Pd/C in acetic acid (quantitative yield) [3051].

m.p. 121° [3051].

1-(6-Hydroxy-2,4-dimethoxy-3-methylphenyl)ethanone *(Bancroftinone)*

[14964-98-8]

$C_{11}H_{14}O_4$ mol.wt. 210.23



Syntheses

- Preparation by hydrolysis of 6-(benzoyloxy)-2,4-di-methoxy-3-methylacetophenone with 3 N aqueous-methanolic potassium hydroxide at r.t. (75%) [3051].
- Preparation by Fries rearrangement of 3,5-dimethoxy-4-methylphenyl acetate with aluminium chloride in nitrobenzene (75–77%) [3283,3284].
- Preparation by reaction of methyl iodide with 4,6-dihydroxy-2-methoxy-3-methylacetophenone in the presence of potassium carbonate in refluxing acetone (93%) [3043].

- Preparation by reaction of acetyl chloride on 3,5-dimethoxy-4-methylphenol with aluminium chloride in ethyl ether first at 0°, then at r.t. [2877,3285,3286], (40%) [2877].

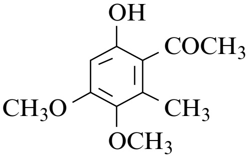
Isolation from natural sources

- From the leaf oils of *Backhousia bancroftii* F. M. Bailey and Muell. (Myrtaceae) as a major constituent [3287].
- The ketone was present to the extent of 35% in clove oil [2873].
- Swertisin was isolated from the whole herb of *Swertia japonica* Makino (Gentianaceae). Further, hydrolytic decomposition of its dimethyl ether with aqueous barium hydroxide gave a degradation product, the 3-C-β-D-glucopyranosyl-6-hydroxy-2,4-dimethoxyacetophenone. This one, by treatment with an excess of aqueous periodic acid followed by Clemmensen reduction gave 6-hydroxy-2,4-dimethoxy-3-methylacetophenone [3288].

m.p. 44–45° [2873], 38–39° [3284], 35° [3051];
oil [3043]; b.p.$_{0.2}$ 110–112° [3283], b.p.$_2$ 120–122° [2877], b.p.$_1$ 128–131° [3284];
GC [3287], GC/MS [3287];
^{1}H NMR [2873,3285,3287], ^{13}C NMR [3287], IR [2873], UV [2873], MS [2873,3287].

1-(6-Hydroxy-3,4-dimethoxy-2-methylphenyl)ethanone

[62615-64-9] $C_{11}H_{14}O_4$ mol.wt. 210.23



Syntheses

- Obtained by partial methylation of 2,5-dihydroxy-4-methoxy-6-methylacetophenone with dimethyl sulfate in acetone, in the presence of potassium carbonate (20%) [3050].
- Preparation by partial demethylation of 2,4,5-trimethoxy-6-methylacetophenone with boron trichloride in methylene chloride at 0° (85%) [3274].

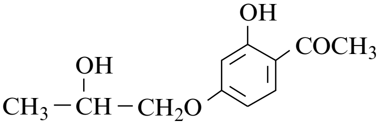
Isolation from natural sources

- By hydrolysis of 4,6,7-trimethoxy-5-methylcoumarin, isolated from *Leonotis nepetaefolia* [3274].

m.p. 77° [3274], 76–77° [3050]; ^{1}H NMR [3274], IR [3274], UV [3274].

1-[2-Hydroxy-4-(2-hydroxypropoxy)phenyl]ethanone

[149454-57-9] $C_{11}H_{14}O_4$ mol.wt. 210.23



Synthesis

- Obtained by reaction of resacetophenone with propylene oxide in the presence of sodium hydroxide in ethanol [3289].

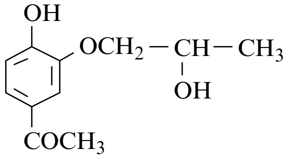
Isolation from natural sources

- From the aerial parts of *Urolepis hecatantha*, flowers and leaves of *Chromolaena arnottiana* (compound **11**) [3290].

^{1}H NMR [3290], MS [3290].

1-[4-Hydroxy-3-(2-hydroxypropoxy)phenyl]ethanone

[63437-94-5] $C_{11}H_{14}O_4$ mol.wt. 210.23



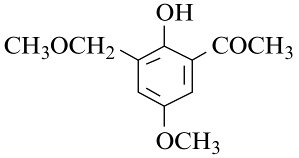
Synthesis

- Preparation by hydrogenolysis of 4-(benzyloxy)-3-(2-hydroxypropoxy)acetophenone in ethanol under hydrogen atmosphere in the presence of 5% Pd/C for 45 min (81%) [3105].

m.p. 143° [3105].

1-[2-Hydroxy-5-methoxy-3-(methoxymethyl)phenyl]ethanone

[87165-71-7] $C_{11}H_{14}O_4$ mol.wt. 210.23



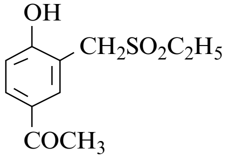
Synthesis

- Preparation from 3-chloromethyl-2-hydroxy-5-methoxy-acetophenone (m.p. 71°) by reaction with methanol in the presence of concentrated hydrochloric acid and iron powder at reflux for 3.5–4 h (69%) [2494].

b.p.$_{0.3}$ 110–114° [2494]; ^{1}H NMR [2494], IR [2494].

1-[3-[(Ethylsulfonyl)methyl]-4-hydroxyphenyl]ethanone

[56490-62-1] $C_{11}H_{14}O_4S$ mol.wt. 242.30



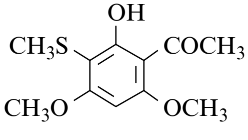
Synthesis

- Obtained by reaction of 3′-chloromethyl-4′-hydroxy-acetophenone with magnesium ethylsulfinate in refluxing aqueous methanol for 18 h (30%) [2544].

m.p. 137–141° [2544].

1-[2-Hydroxy-4,6-dimethoxy-3-(methylthio)phenyl]ethanone

[154389-63-6] $C_{11}H_{14}O_4S$ mol.wt. 242.30



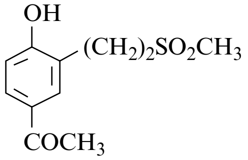
Syntheses

- Preparation by partial demethylation of 2,4,6-trimethoxy-3-methylthioacetophenone with aluminium chloride in acetonitrile for 1 h at 30° (95%) [2747].
- Also obtained by reaction of acetyl chloride with 2,4,6-tri-methoxy-1-(methylthio)benzene in the presence of aluminium chloride in ethyl ether at 0° (24%) [2747].

m.p. 143–145° [2747].

1-[4-Hydroxy-3-[2-(methylsulfonyl)ethyl]phenyl]ethanone

[56490-44-9] $C_{11}H_{14}O_4S$ mol.wt. 242.30



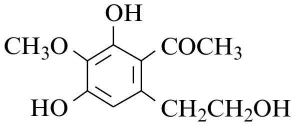
Synthesis

– Obtained by Fries rearrangement of 2-(methylsulfonylethyl)-phenyl acetate (m.p. 80–82°) with aluminium chloride in nitrobenzene, first at r.t. for 1 h, then at 50–60° for 1.5 h (53%) [2544].

m.p. 176–178° [2544].

1-[2,4-Dihydroxy-6-(2-hydroxyethyl)-3-methoxyphenyl]ethanone

[165186-29-8] $C_{11}H_{14}O_5$ mol.wt. 226.23

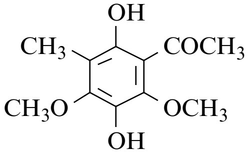


Isolation from natural sources

– One of seven metabolites produced by *Ophiosphaerella herpotricha* in liquid culture [3291].

1-(2,5-Dihydroxy-4,6-dimethoxy-3-methylphenyl)ethanone

$C_{11}H_{14}O_5$ mol.wt. 226.23



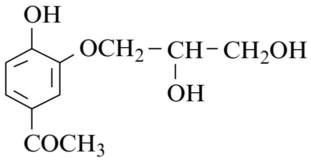
Syntheses

– Preparation by reaction of potassium persulfate on 2-hydroxy-4,6-dimethoxy-3-methylacetophenone in aqueous pyridine solution in the presence of potassium hydroxide [3292] or sodium hydroxide [2716,2886], (27–35%) [2886,3292], (10%) [2716].

m.p. 122–123° [2886], 121–122° [2716], 119–120° [3292];
^{1}H NMR [2716], UV [2886], MS [2716].

1-[3-(2,3-Dihydroxypropoxy)-4-hydroxyphenyl]ethanone

[70064-44-7] $C_{11}H_{14}O_5$ mol.wt. 226.23

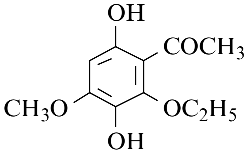


Synthesis

– Preparation by hydrogenolysis of 4-(benzyloxy)-3-(2,3-dihydroxypropoxy)acetophenone in ethanol under hydrogen atmosphere in the presence of 5% Pd/C for 45 min (99%) [3105].

m.p. 136° [3105].

1-(2-Ethoxy-3,6-dihydroxy-4-methoxyphenyl)ethanone



$C_{11}H_{14}O_5$ mol.wt. 226.23

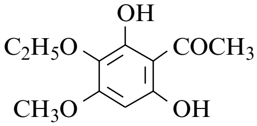
Synthesis

- Preparation by reaction of potassium persulfate on 2-ethoxy-6-hydroxy-4-methoxyacetophenone in dilute aqueous sodium hydroxide solution at 15–20° (25%) [2851].

m.p. 145° [2851].

1-(3-Ethoxy-2,6-dihydroxy-4-methoxyphenyl)ethanone

[105342-70-9]



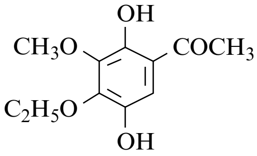
$C_{11}H_{14}O_5$ mol.wt. 226.23

Synthesis

- Obtained (by-product) by reaction of hydrobromic acid with 3-ethoxy-2-hydroxy-4,6-dimethoxyacetophenone in acetic acid at r.t. [3131].

m.p. 108°5–110° [3131];
^1H NMR [3131], IR [3131], MS [3131].

1-(4-Ethoxy-2,5-dihydroxy-3-methoxyphenyl)ethanone



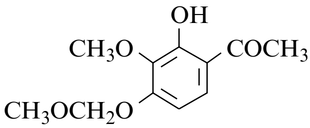
$C_{11}H_{14}O_5$ mol.wt. 226.23

Synthesis

- Preparation from 4-ethoxy-2-hydroxy-3-methoxyacetophenone by persulfate oxidation in 10% aqueous sodium hydroxide (Elbs reaction) [2818].

1-[2-Hydroxy-3-methoxy-4-(methoxymethoxy)phenyl]ethanone

[220504-99-4]



$C_{11}H_{14}O_5$ mol.wt. 226.23

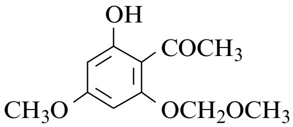
Synthesis

- Preparation in two steps: first, reaction of acetic acid with a mixture (ca. 1:1) of 1-O- and 2-O-methylp-yrogallol in the presence of zinc chloride at reflux for 6 h (Nencki reaction); then, methoxymethylation of the obtained product (31%) [3110].

^1H NMR [3110]; MS [3110].

1-[2-Hydroxy-4-methoxy-6-(methoxymethoxy)phenyl]ethanone

[186693-85-6] $C_{11}H_{14}O_5$ mol.wt. 226.23



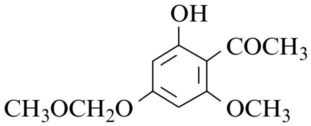
Synthesis

- Obtained by treatment of 2,6-dihydroxy-4-methoxy-acetophenone with methoxymethyl chloride [3293] in the presence of potassium carbonate in refluxing acetone for 2 h (86%) [3112].

m.p. 59–61°5 [3112]; ^{1}H NMR [3112], IR [3112], EIMS [3112].

1-[2-Hydroxy-6-methoxy-4-(methoxymethoxy)phenyl]ethanone

[404597-93-9] $C_{11}H_{14}O_5$ mol.wt. 226.23

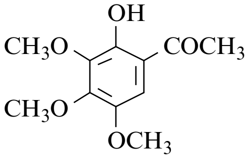


Synthesis

- Obtained by treatment of 2,4-di(methoxymethoxy)-6-methoxyacetophenone with silica gel in mild acetic medium (97%) [3294].

1-(2-Hydroxy-3,4,5-trimethoxyphenyl)ethanone

[30225-96-8] $C_{11}H_{14}O_5$ mol.wt. 226.23



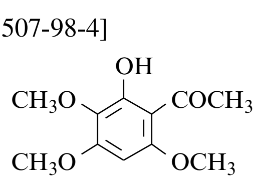
Syntheses

- Preparation by reaction of dimethyl sulfate on 2,5-di-hydroxy-3,4-dimethoxyacetophenone with potassium carbonate in refluxing benzene (73%) [2820].
- Preparation by reaction of acetyl chloride on 1,2,3,4-tetramethoxybenzene with aluminium chloride in refluxing carbon disulfide (70%) [3067,3295,3296] or in boiling ethyl ether [2770,3296], (76%) [2770].

m.p. 88° [2770,3295,3296], 86° [2820,3067]; ^{1}H NMR [3067].

1-(2-Hydroxy-3,4,6-trimethoxyphenyl)ethanone *(Xanthoxylone)*

[7507-98-4] $C_{11}H_{14}O_5$ mol.wt. 226.23



Syntheses

- Preparation by reaction of diazomethane on 2,4-dihydroxy-3,6-dimethoxyacetophenone in ethyl ether [2442,3149].
- Preparation by reaction of dimethyl sulfate,
  - on 2,6-dihydroxy-3,4-dimethoxyacetophenone disodium salt (95%) [2443];
  - on 2,4-dihydroxy-3,6-dimethoxyacetophenone with potassium carbonate in boiling acetone (75%) [3136];
  - on 2,3,6-trihydroxy-4-methoxyacetophenone in the presence of potassium carbonate in refluxing acetone [2881];
  - on 2,3,4,6-tetrahydroxyacetophenone [2443,3297].

- Also obtained by partial methylation of 2,4-dihydroxy-3,6-dimethoxyacetophenone [3298], according to [3136].
- Also obtained by Friedel–Crafts acylation of 1,2,3,5-tetramethoxybenzene [3299].
- Preparation by reaction of acetyl chloride on 1,2,3,5-tetramethoxybenzene with aluminium chloride,
    - in ethyl ether [2770,2820,3096,3131,3135,3300–3302], (81%) [2770], (64–70%) [2820,3096,3302], (53%) [3131], (35–36%) [2857,3135];
    - in carbon disulfide [3140,3141,3145,3297,3301,3303], (51%) [3297];
    - without solvent at 70° (44%) [3304].
- Preparation by reaction of acetic acid with 1,2,3,5-tetramethoxybenzene in the presence of boron trifluoride at 30° (81%) [3127].
- Also obtained by partial demethylation of 2,3,4,6-tetramethoxyacetophenone in the presence of aluminium chloride [2747,3305] in acetonitrile at 30° for 1 h (95%) [2747].
- Also refer to: [3306–3309].

Isolation from natural sources

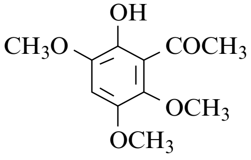
- From the New Zealand liverwort, *Plagiochila fasciculata* [3310].
- From *Croton aff. nepetifolius* Bail (Euphorbiaceae) [3311].
- From hydrolysis of Wogonin (5,7-dihydroxy-8-methoxyflavone). Wogonin was isolated in small amounts in the roots of *Scutellaria baicalensis* Georgi (Labiatae) [3305].
- From the fresh leaves of *Fagara okinawensis* Nakai (Rutaceae) [3096].

m.p. 125–126° [2443], 113–115° [3135], 113–114° [3136], 113° [2881], 112–114° [3131], 112–113° [2770,3096,3297,3301,3303,3305], 111°5–113°5 [2857], 111–112° [3140], 110–112° [3298], 110–111° [2442,3149], 109–114° [3127], 109°5–111°5 [3302], 109–111° [3304], 105–107° [3141,3145,3312], 103–105° [2820,3313];

^{1}H NMR [2442,2857,3096,3131,3149,3298,3302,3310,3311], ^{13}C NMR [3298]; IR [2442,2857,3096,3131,3149,3298,3302,3310,3311], UV [3096,3310]; MS [3298,3311].

1-(2-Hydroxy-3,5,6-trimethoxyphenyl)ethanone

[72424-28-3] $C_{11}H_{14}O_5$ mol.wt. 226.23



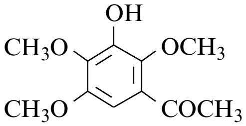
Synthesis

- Preparation by partial demethylation of 2,3,5,6-tetramethoxyacetophenone with aluminium chloride in ethyl ether in an ice bath (55%) [3300] or in acetonitrile for 6 h at 45° (25%) [2747].

m.p. 62–63°5 [3300]; ^{1}H NMR [3300].

1-(3-Hydroxy-2,4,5-trimethoxyphenyl)ethanone

[97565-35-0] $C_{11}H_{14}O_5$ mol.wt. 226.23



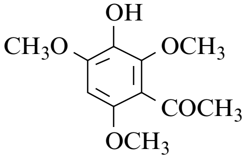
Isolation from natural sources

- Obtained by alkaline degradation of two octasubstituted flavones with 50% potassium hydroxide in refluxing ethanol under nitrogen atmosphere for 15 h. These flavones were isolated from the aerial parts of *Ageratum houstonianum* Mill (Asteraceae) (Eupatorieae) [3314],
  - From *agehoustin C* (3′-hydroxy-5,6,7,8,2′,4′,5′-heptamethoxyflavone) (m.p. 145°).
  - From *agehoustin D* (5,3′-dihydroxy-6,7,8,2′,4′,5′-hexamethoxyflavone) (m.p. 168–169°).

m.p. 98–100° [3314]; ^{1}H NMR [3314], IR [3314], UV [3314], MS [3314].

1-(3-Hydroxy-2,4,6-trimethoxyphenyl)ethanone

[103777-45-3] $C_{11}H_{14}O_5$ mol.wt. 226.23



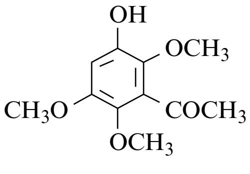
Syntheses

- Obtained by treatment of polygoacetophenoside with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 24 h. Then, the formed oil was hydrolyzed with 10% sulfuric acid on a water bath for 3 h [3309].
- Also obtained (by-product) by reaction of dimethyl sulfate on 3,6-dihydroxy-2,4-dimethoxy-acetophenone in aqueous sodium hydroxide solution at r.t. (3%) [3135].

m.p. 128–129° [3309], 113–117° [3135];
^{1}H NMR [3309], ^{13}C NMR [3309], IR [3309], MS [3309].

1-(3-Hydroxy-2,5,6-trimethoxyphenyl)ethanone

[73034-32-9] $C_{11}H_{14}O_5$ mol.wt. 226.23



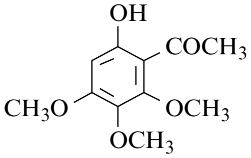
Synthesis

- Preparation by hydrolysis of 3-acetoxy-2,5,6-trimethoxy-acetophenone with 10% sodium hydroxide in methanol [3131,3300].

m.p. 71–72° [3131]; b.p.$_{0.1}$ 150–160° [3131];
^{1}H NMR [3131], IR [3131].

1-(6-Hydroxy-2,3,4-trimethoxyphenyl)ethanone

[22248-14-2] $C_{11}H_{14}O_5$ mol.wt. 226.23



Syntheses

- Preparation by Fries rearrangement of antiarol acetate (3,4,5-trimethoxyphenyl acetate) with aluminium chloride in nitrobenzene at r.t. (54%) [3144], (45%) [3135].
- Preparation by reaction of antiarol (3,4,5-trimethoxyphenol) with boron trifluoride–acetic acid complex at 28–30° (50%) [2242].
- Also obtained by reaction of acetyl chloride,
  - on antiarol benzyl ether with aluminium chloride in ethyl ether, followed by subsequent debenzylation of the keto compound obtained (22%) [3144];
  - on 1,2,3,5-tetramethoxybenzene with aluminium chloride in carbon disulfide at r.t. (9%) [3312].
- Also obtained by reaction of dimethyl sulfate,
  - on 3,6-dihydroxy-2,4-dimethoxyacetophenone,
    - with potassium carbonate in refluxing benzene [2820,3135,3144], (15–18%) [2820,3135] or in refluxing acetone–benzene mixture [3080,3146,3300], (70%) [3080], (36%) [3146];
    - with aqueous sodium hydroxide solution at r.t. (52%) [3135].
  - on 2,3,4,6-tetrahydroxyacetophenone [2443];
  - on 2,6-dihydroxy-3,4-dimethoxyacetophenone disodium salt, followed by acidification (<5%) [2443].
- Also obtained by alkaline degradation of 3′,4′,5,6,7-pentamethoxyflavone with potassium hydroxide in refluxing aqueous ethanol [2851].
- Also refer to: [2777,3315].

Isolation from natural sources

- From 5,6,7-trimethoxyflavone by alkaline hydrolysis in refluxing 50% methanolic potassium hydroxide. The 5,6,7-trimethoxyflavone is one of major constituents of the leaves of *Zeyhera tuberculosa* Bur. ex. Verlot (Bignoniaceae) [3146].
  Yellow oil [2851,3080,3146];

  m.p. 164–165° [2443], 105–107° [3312], 41–42° [3144], 32° [2242], 30°5–31°5 [3135].

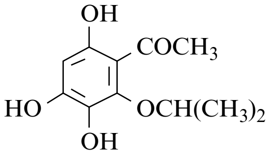
  There is a discrepancy between the different melting points indicated in literature.

  b.p.$_{0.35}$ 121–122° [3135], b.p.$_{1}$ 140° [2242], b.p.$_{20}$ 180–185° [3144], b.p.$_{14}$ 182–185° [3135], b.p.$_{27}$ 184–186° [2820];

  ^{1}H NMR [3146], ^{13}C NMR [2328], IR [3146], UV [3146], MS [3146].

1-[3,4,6-Trihydroxy-2-(1-methylethoxy)phenyl]ethanone

[60840-21-3] $C_{11}H_{14}O_5$ mol.wt. 226.23



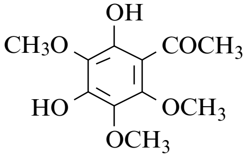
Synthesis

- Preparation by catalytic hydrogenolysis of 3,4,6-tris-(benzyloxy)-2-isopropoxyacetophenone in the presence of 5% Pd/C in ethanol at r.t. (94%) [2442,3149].

m.p. 137° [2442,3149];
^{1}H NMR [2442,3149], IR [2442,3149], MS [2442,3149].

1-(2,4-Dihydroxy-3,5,6-trimethoxyphenyl)ethanone

$C_{11}H_{14}O_6$ mol.wt. 242.23



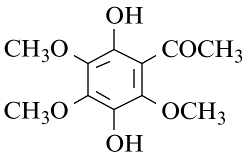
Synthesis

- Obtained by hydrolysis of 1-(2,4-diacetoxy-3,5,6-tri-methoxyphenyl)ethanone (SM) with aqueous 10% sodium hydroxide at r.t. for 10–15 min. SM was obtained by Friedel–Crafts acylation of 2,3,5,6-tetramethoxyphenyl acetate (m.p. 97–98°) with acetic anhydride/acetic acid in the presence of excess boron trifluoride at 50–60° for 5 h (65%, m.p. 82–84°) [3316].

N.B.: This ketone was not obtained by Friedel–Crafts reaction of 2,3,5,6-tetramethoxyphenol, and its acetate or benzyl ether with acetyl chloride and aluminium chloride in ethyl ether [3316,3317].

1-(2,5-Dihydroxy-3,4,6-trimethoxyphenyl)ethanone

[55742-65-9] $C_{11}H_{14}O_6$ mol.wt. 242.23

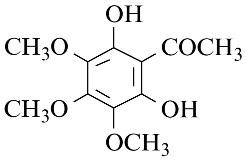


Syntheses

- Obtained by persulfate oxidation of 2-hydroxy-3,4,6-tri-methoxyacetophenone (Elbs reaction) [3307], (33%) [3299], (29%) [2820], (9%) [3318].
- Also obtained by reduction of 2-acetyl-3,5,6-trimethoxy-1,4-benzoquinone with zinc dust in acetic anhydride, followed by hydrolysis of the acetic ester formed with dilute sulfuric acid [2443].
- Also refer to: [3319–3322].

m.p. 174–176° [2443], 116–117° [2820], 115–117° [3299,3307].
One of the reported melting points is obviously wrong.

1-(2,6-Dihydroxy-3,4,5-trimethoxyphenyl)ethanone



$C_{11}H_{14}O_6$ mol.wt. 242.23

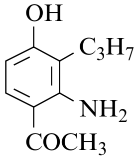
Synthesis

- Obtained by acylation of pentamethoxybenzene with acetyl chloride in the presence of aluminium chloride in ethyl ether, first for 14 h at r.t., then for 2 h at reflux (17%) [3323].

m.p. 86–88° [3323];
^{1}H NMR [3323], IR [3323], UV [3323].

1-(2-Amino-4-hydroxy-3-propylphenyl)ethanone

[87472-78-4] $C_{11}H_{15}NO_2$ mol.wt. 193.25



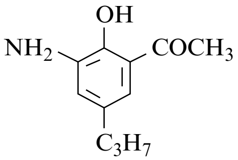
Synthesis

- Preparation by hydrogenation of 3-allyl-2-amino-4-hydroxyacetophenone in the presence of 5% Pd/C in ethanol (quantitative yield) [2464].

Viscous oil [2464]; ^{1}H NMR [2464].

1-(3-Amino-2-hydroxy-5-propylphenyl)ethanone

[70978-22-2] $C_{11}H_{15}NO_2$ mol.wt. 193.25



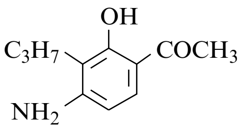
Synthesis

- Preparation by catalytic hydrogenation of 2-hydroxy-3-nitro-5-propylacetophenone in the presence of 5% Pd/C in ethanol at 25° [1897,1898], (77%) [1897].

m.p. 43–45° [1898], 42–43° [1897].

1-(4-Amino-2-hydroxy-3-propylphenyl)ethanone

[75452-54-9] $C_{11}H_{15}NO_2$ mol.wt. 193.25



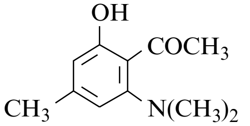
Synthesis

- Preparation by hydrolysis of 4-acetamido-2-hydroxy-3-propylacetophenone with 6 N hydrochloric acid in refluxing ethanol (95%) [2464].

Yellow oil [2464]; b.p.$_{0.15}$ 120–122° [2464]; ^{1}H NMR [2464].

1-[2-(Dimethylamino)-6-hydroxy-4-methylphenyl]ethanone

[97066-06-3] $C_{11}H_{15}NO_2$ mol.wt. 193.25



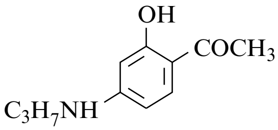
Synthesis

- Preparation by reaction of potassium hydroxide with 2-acetyl-3-dimethylamino-5-hydroxy-5-methyl-2-cyclo-hexenone in ethanol at 40° (41%) [2712].

Yellow oil [2712]; m.p. –5° [2712];
^{1}H NMR [2712], IR [2712], UV [2712], MS [2712].

1-[2-Hydroxy-4-(propylamino)phenyl]ethanone

[118684-26-7] $C_{11}H_{15}NO_2$ mol.wt. 193.25



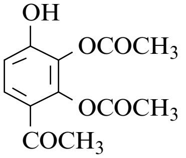
Synthesis

- A solution of 4-amino-2-hydroxyacetophenone and propionaldehyde in methanol was stored over 3 Å molecular sieves for 3 days. The solution of "2-hydroxy-4-propyl-iminoacetophenone" so obtained was then treated with hydrogen in the presence of 10% Pd/C (50%) [2464].

m.p. 73–75° [2464]; ^{1}H NMR [2464].

1-[2,3-Bis(acetyloxy)-4-hydroxyphenyl]ethanone

[144152-31-8] $C_{12}H_{12}O_6$ mol.wt. 252.22



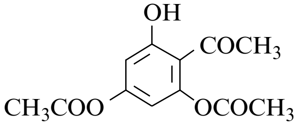
Synthesis

- Obtained by enzymatic deacylation of 2,3,4-triacetoxyacetophenone with porcine pancreas lipase in tetrahydrofuran at 42–45° (55%) [2388,2897].

Pale yellow viscous oil [2897]; ^{1}H NMR [2897].

1-[2,4-Bis(acetyloxy)-6-hydroxyphenyl]ethanone

[17820-33-6] $C_{12}H_{12}O_6$ mol.wt. 252.22



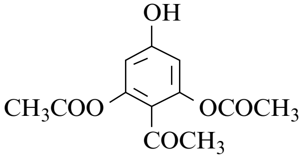
Syntheses

- Obtained (by-product) by reaction of acetic anhydride on phloroacetophenone with pyridine at r.t. (7%) [2368].
- Also obtained by photo-Fries rearrangement of 1,3,5-tri-acetoxybenzene in methanol (15%) [2343].

m.p. 86–87° [2853], 79–80° [2343];
^{1}H NMR [2343,2853], ^{13}C NMR [2368], IR [2343].

1-[2,6-Bis(acetyloxy)-4-hydroxyphenyl]ethanone

[17820-32-5] $C_{12}H_{12}O_6$ mol.wt. 252.22



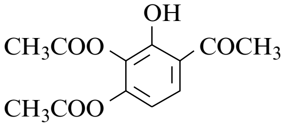
Syntheses

- Preparation by reaction of acetic anhydride with phloroacetophenone between 110° and 165° (40%) [2853].
- Also obtained by enzymatic deacylation of 2,4,6-triacetoxy-acetophenone with porcine pancreatic lipase in tetrahydrofuran at 42–45° (78%) [2388,2389,2897].

m.p. 154–155° [2853], 112° [2897]. One of the reported melting points is obviously wrong.
^{1}H NMR [2853,2897].

1-[3,4-Bis(acetyloxy)-2-hydroxyphenyl]ethanone

[27865-58-3] $C_{12}H_{12}O_6$ mol.wt. 252.22



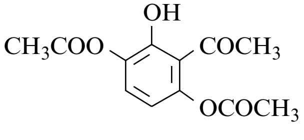
Syntheses

- Obtained by UV light irradiation of pyrogallol triacetate in methanol (15%) [2343].
- Also obtained by partial Fries rearrangement of pyrogallol triacetate with zinc chloride at 130–135° [3324].
- Also obtained by reaction of acetic anhydride on gallacetophenone with pyridine [2901].
- Also refer to: [2675].

m.p. 217–219° [3324], 110–112° [2343], 78–81° [2901];
^{1}H NMR [2343], IR [2343].

1-[3,6-Bis(acetyloxy)-2-hydroxyphenyl]ethanone

[104654-33-3] $C_{12}H_{12}O_6$ mol.wt. 252.22



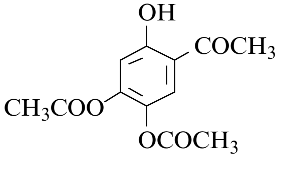
Synthesis

- Obtained by photo-Fries rearrangement of 1,2,4-triacetoxy-benzene in methanol (15%) [2343].

m.p. 116–118° [2343]; ^{1}H NMR [2343], IR [2343].

1-[4,5-Bis(acetyloxy)-2-hydroxyphenyl]ethanone

[42059-51-8] $C_{12}H_{12}O_6$ mol.wt. 252.22



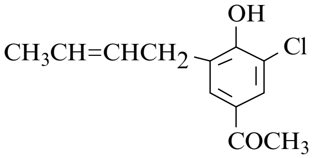
Syntheses

- Obtained by partial Fries rearrangement of 1,2,4 -triacetoxy-benzene with zinc chloride in acetic acid at 140° (21%) [2414,2415], (8%) [2899].

- Preparation by reaction of acetic anhydride with 2,4,5-tri-hydroxyacetophenone in the presence of pyridine at 35° (52%) [2899].

m.p. 165–166° [2414,2415], 100–102° [2899]; ^{1}H NMR [2899], IR [2899].

1-[3-(2-Butenyl)-5-chloro-4-hydroxyphenyl]ethanone



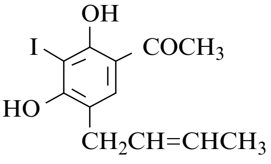
$C_{12}H_{13}ClO_2$ mol.wt. 224.69

Synthesis

- Obtained by heating 4-(2-butenyloxy)-3-chloroacetophenone for 3 h under nitrogen atmosphere (Claisen rearrangement) [3162].

1-[5-(2-Butenyl)-2,4-dihydroxy-3-iodophenyl]ethanone

[91664-19-6]



$C_{12}H_{13}IO_3$ mol.wt. 332.14

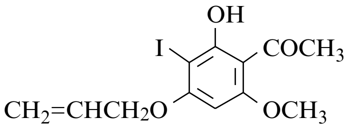
Synthesis

- Preparation by condensation of 2,4-dihydroxy-3-iodo-acetophenone with 1,3-butadiene in the presence of 85% orthophosphoric acid in xylene at 30–35° (75%) [3325].

m.p. 100–101° [3325]; ^{1}H NMR [3325].

1-[2-Hydroxy-3-iodo-6-methoxy-4-(2-propenyloxy)phenyl]ethanone

[74047-33-9]



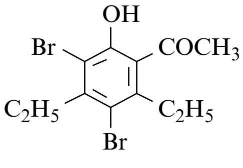
$C_{12}H_{13}IO_4$ mol.wt. 348.14

Synthesis

- Preparation by reaction of allyl bromide with 2,4-di-hydroxy-3-iodo-6-methoxy-acetophenone in the presence of potassium carbonate in refluxing acetone (58%) [2064].

m.p. 162–164° [2064]; ^{1}H NMR [2064].

1-(3,5-Dibromo-2,4-diethyl-6-hydroxyphenyl)ethanone



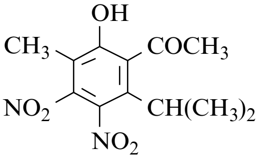
$C_{12}H_{14}Br_2O_2$ mol.wt. 350.05

Synthesis

- Preparation by reaction of potassium bromate and bromide on 2,4-diethyl-6-hydroxyacetophenone in solution of acetic acid–carbon tetrachloride mixture (quantitative yield) [2965].

m.p. 81° [2965].

1-[2-Hydroxy-3-methyl-6-(1-methylethyl)-4,5-dinitrophenyl]ethanone



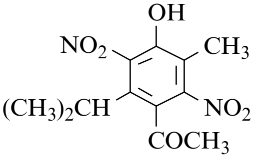
$C_{12}H_{14}N_2O_6$ mol.wt. 282.26

Synthesis

- Preparation by reaction of nitric acid (d = 1.4) on 2-hydroxy-3-methyl-5-nitro-6-isopropylacetophenone in acetic acid, between −5° and 0° (53%) [3326].

m.p. 119° [3326].

1-[4-Hydroxy-3-methyl-6-(1-methylethyl)-2,5-dinitrophenyl]ethanone



$C_{12}H_{14}N_2O_6$ mol.wt. 282.26

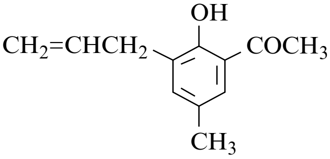
Synthesis

- Preparation by reaction of nitric acid (d = 1.4) on 4-hydroxy-5-methyl-3-nitro-2-isopropylacetophenone in acetic acid (59%) [3327].

m.p. 55° [3327].

1-[2-Hydroxy-5-methyl-3-(2-propenyl)phenyl]ethanone

[108293-73-8]



$C_{12}H_{14}O_2$ mol.wt. 190.24

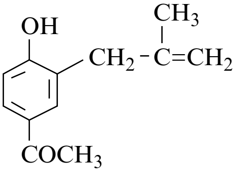
Synthesis

- Preparation by thermal Claisen rearrangement of 2-(allyloxy)-5-methylacetophenone without solvent at 190° (95%) [2513] or at 260–270° (84%) [3168].

Yellow oil [2513];
b.p.$_{0.15}$ 94–96° [2513], b.p.$_5$ 103–105° [3168];
^{1}H NMR [2513], ^{13}C NMR [2513], IR [2513], MS [2513].

1-[4-Hydroxy-3-(2-methyl-2-propenyl)phenyl]ethanone

[57899-03-3]



$C_{12}H_{14}O_2$ mol.wt. 190.24

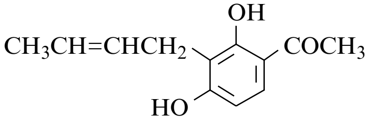
Synthesis

- Preparation by thermal Claisen rearrangement of 4-(β-methallyloxy)acetophenone in boiling N,N-di-methylaniline [3177].

m.p. 86° [3177]; b.p.$_{16}$ 196° [3177].

1-[3-(2-Butenyl)-2,4-dihydroxyphenyl]ethanone

[91664-16-3] $C_{12}H_{14}O_3$ mol.wt. 206.24



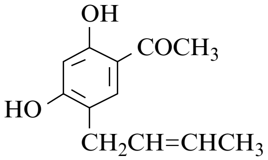
Synthesis

- Preparation by condensation of resacetophenone with 1,3-butadiene in the presence of 85% orthophosphoric acid in xylene at 30–35° (38%) [3325].

m.p. 164–165° [3325]; ^{1}H NMR [3325].

1-[5-(2-Butenyl)-2,4-dihydroxyphenyl]ethanone

[91664-17-4] $C_{12}H_{14}O_3$ mol.wt. 206.24



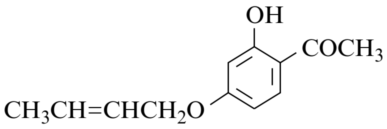
Syntheses

- Preparation by condensation of resacetophenone with 1,3-butadiene in the presence of 85% orthophosphoric acid in xylene at 30–35° (42%) [3325].
- Also obtained from 5-(2-butenyl)-2,4-dihydroxy-3-iodo-acetophenone by heating with zinc dust and concentrated hydrochloric acid in refluxing ethanol (80%) [3325].

m.p. 103–104° [3325]; ^{1}H NMR [3325], IR [3325].

1-[4-(2-Butenyloxy)-2-hydroxyphenyl]ethanone

[79557-72-5] $C_{12}H_{14}O_3$ mol.wt. 206.24



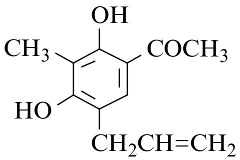
Synthesis

- Preparation by reaction of 3-chloro-1-butene with resacetophenone in the presence of potassium carbonate and sodium iodide in refluxing butanone (44%) [3184].

Oil [3184]; b.p.$_{0.1}$ 162–175° [3184].

1-[2,4-Dihydroxy-3-methyl-5-(2-propenyl)phenyl]ethanone

[77869-01-3] $C_{12}H_{14}O_3$ mol.wt. 206.24



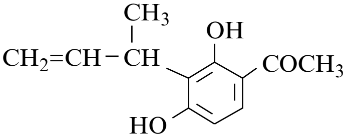
Synthesis

- Preparation by thermal Claisen rearrangement of 4-(allyloxy)-2-hydroxy-3-methylacetophenone in boiling N,N-dimethylaniline [3328].

m.p. 139° [3328]; IR [3328].

1-[2,4-Dihydroxy-3-(1-methyl-2-propenyl)phenyl]ethanone

[79557-73-6] $C_{12}H_{14}O_3$ mol.wt. 206.24



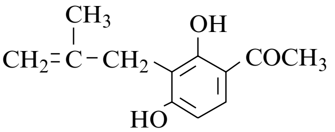
Syntheses

- Preparation by thermal Claisen rearrangement of 4-(2-butenyloxy)-2-hydroxyacetophenone without solvent at 180–190° under nitrogen (40%) [3184].
- Also obtained (by-product) by condensation of resacetophenone with 1,3-butadiene in the presence of 85% orthophosphoric acid in xylene at 30–35° (3%) [3325].

m.p. 147–149° [3325], 146–147° [3184]; ^{1}H NMR [3325].

1-[2,4-Dihydroxy-3-(2-methyl-2-propenyl)phenyl]ethanone

[118683-89-9] $C_{12}H_{14}O_3$ mol.wt. 206.24



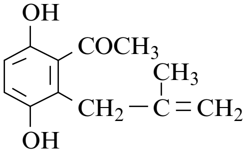
Synthesis

- Preparation by thermal Claisen rearrangement of 2-hydroxy-4-(2-methylprop-2-enoxy) acetophenone without solvent at 210° (24%) [2464].

White solid [2464]; ^{1}H NMR [2464].

1-[3,6-Dihydroxy-2-(2-methyl-2-propenyl)phenyl]ethanone

[127870-07-9] $C_{12}H_{14}O_3$ mol.wt. 206.24



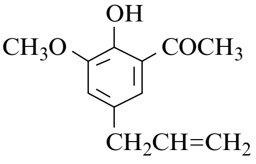
Synthesis

- Obtained by thermal reaction of 2-acetyl-1,4-benzoquinone (1 mol) with (2-methallyl)trimethylstannane (2 mol) in benzene or acetonitrile under argon atmosphere (77%) [2355].

m.p. 78–80° [2355]; ^{1}H NMR [2355], MS [2355].

1-[2-Hydroxy-3-methoxy-5-(2-propenyl)phenyl]ethanone

[23343-04-6] $C_{12}H_{14}O_3$ mol.wt. 206.24



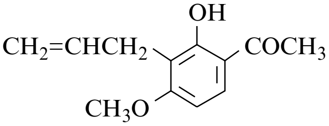
Synthesis

- Preparation by thermal Claisen rearrangement of 2-(allyloxy)-3-methoxyacetophenone [3329] without solvent at 210° [3330], (77%) [3330], (38%) [3329].

m.p. 43–44° [3330], 42–43° [3329];
^{1}H NMR [3329], IR [3329], UV [3329].

1-[2-Hydroxy-4-methoxy-3-(2-propenyl)phenyl]ethanone

[117156-86-2] $C_{12}H_{14}O_3$ mol.wt. 206.24



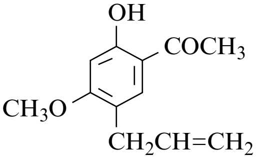
Syntheses

- Preparation by thermal Claisen rearrangement of 2′-(allyloxy)-4′-methoxyacetophenone in boiling N,N-di-methylaniline (80%) [3331].
- Preparation by reaction of dimethyl sulfate on 3-allyl-resacetophenone in 10% aqueous potassium hydroxide at 30° [3182].

m.p. 61° [3182], 59° [3331]; ^{1}H NMR [3331], IR [3331], UV [3331].

1-[2-Hydroxy-4-methoxy-5-(2-propenyl)phenyl]ethanone

[117713-79-8] $C_{12}H_{14}O_3$ mol.wt. 206.24

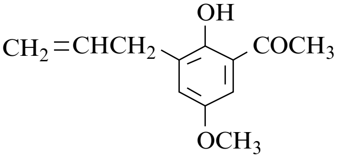


Synthesis

- Preparation by reaction of methyl bromide on 5-allyl-2,4-dihydroxyacetophenone with potassium carbonate and potassium iodide (41%) [2678,2679].

1-[2-Hydroxy-5-methoxy-3-(2-propenyl)phenyl]ethanone

[186956-47-8] $C_{12}H_{14}O_3$ mol.wt. 206.24



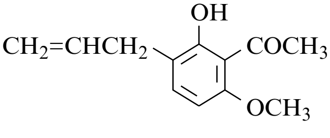
Synthesis

- Obtained by Claisen rearrangement of 2-(allyloxy)-5-methoxyacetophenone in refluxing N,N-diethyl-aniline at 220° for 4 h [3163].

colourless oil [3163]; b.p.$_{0.1}$ 145° [3163];
^{1}H NMR [3163], IR [3163].

1-[2-Hydroxy-6-methoxy-3-(2-propenyl)phenyl]ethanone

[17488-68-5] $C_{12}H_{14}O_3$ mol.wt. 206.24

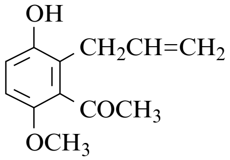


Syntheses

- Preparation by reaction of dimethyl sulfate on 3-allyl-2,6-dihydroxyacetophenone with potassium carbonate in benzene in a water bath (89%) [3186].
- Also obtained by thermal Claisen rearrangement of 2-(allyloxy)-6-methoxyacetophenone at 215–220°, in a sealed tube (35%) [3186].

b.p.$_2$ 122–124° [3186], b.p.$_3$ 127° [3186]; d^{20} = 1.0283 [3186];
n_D^{20} = 1.5602 and 1.5598 [3186].

1-[3-Hydroxy-6-methoxy-2-(2-propenyl)phenyl]ethanone



$C_{12}H_{14}O_3$ mol.wt. 206.24

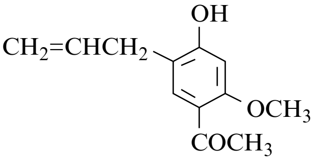
Synthesis

- Preparation by thermal Claisen rearrangement of 5-(allyloxy)-2-methoxyacetophenone at 230° (74%) [3187].

m.p. 104° [3187].

1-[4-Hydroxy-2-methoxy-5-(2-propenyl)phenyl]ethanone

[117705-59-6]



$C_{12}H_{14}O_3$ mol.wt. 206.24

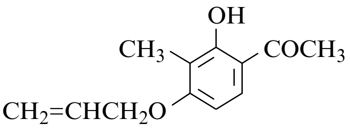
Synthesis

- Preparation by Claisen rearrangement of 4-(allyloxy)-2-methoxyacetophenone at 210–215° (60%) [3182], (24%) [2671,2678, 2679].

m.p. 136° [3182], <25° [2671]; ^{1}H NMR [2678,2679].

1-[2-Hydroxy-3-methyl-4-(2-propenyloxy)phenyl]ethanone

[77036-77-2]



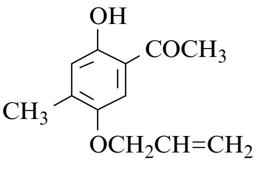
$C_{12}H_{14}O_3$ mol.wt. 206.24

Synthesis

- Preparation by reaction of allyl bromide on 2,4-di-hydroxy-3-methylacetophenone with potassium carbonate in refluxing acetone [3332].

1-[2-Hydroxy-4-methyl-5-(2-propenyloxy)phenyl]ethanone

[76267-82-8]



$C_{12}H_{14}O_3$ mol.wt. 206.24

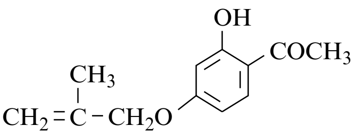
Synthesis

- Preparation by reaction of allyl bromide with 2,5-dihydroxy-4-methylacetophenone in the presence of potassium carbonate in refluxing acetone [2699].

Greenish yellow liquid [2699].

1-[2-Hydroxy-4-[(2-methyl-2-propenyl)oxy]phenyl]ethanone

[118683-88-8]



$C_{12}H_{14}O_3$ mol.wt. 206.24

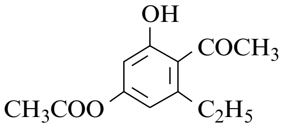
Synthesis

- Preparation by reaction of 3-chloro-2-methylpropene with resacetophenone in the presence of potassium carbonate in refluxing acetone (75%) [2464].

White solid [2464]; ^{1}H NMR [2464].

1-[4-(Acetyloxy)-2-ethyl-6-hydroxyphenyl]ethanone

[57600-88-1] $C_{12}H_{14}O_4$ mol.wt. 222.24

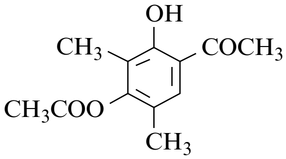


Synthesis

- Preparation by treatment of a mixture of alkylated resorcinols with acetic anhydride and acetic acid in the presence of zinc chloride at 140–145°, followed by suitable separation [3189].

1-[4-(Acetyloxy)-2-hydroxy-3,5-dimethylphenyl]ethanone

$C_{12}H_{14}O_4$ mol.wt. 222.24



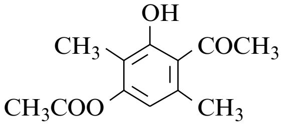
Synthesis

- Preparation by reaction of acetic anhydride with 2,4-di-hydroxy-3,5-dimethylacetophenone in the presence of pyridine [2990].

m.p. 95–96° [2990].

1-[4-(Acetyloxy)-2-hydroxy-3,6-dimethylphenyl]ethanone

[57600-89-2] $C_{12}H_{14}O_4$ mol.wt. 222.24

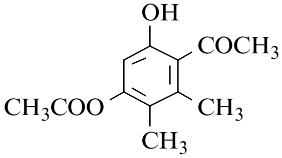


Synthesis

- Preparation by treatment of a mixture of alkylated resorcinols with acetic anhydride and acetic acid in the presence of zinc chloride at 140–145°, followed by suitable separation [3189].

1-[4-(Acetyloxy)-6-hydroxy-2,3-dimethylpheny]ethanone

[57600-90-5] $C_{12}H_{14}O_4$ mol.wt. 222.24

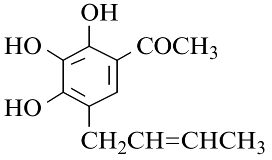


Synthesis

- Preparation by treatment of a mixture of alkylated resorcinols with acetic anhydride and acetic acid in the presence of zinc chloride at 140–145°, followed by suitable separation [3189].

1-[5-(2-Butenyl)-2,3,4-trihydroxyphenyl]ethanone

[91664-14-1] $C_{12}H_{14}O_4$ mol.wt. 222.24

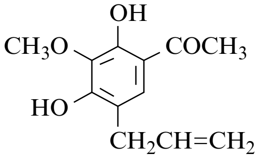


Synthesis

- Preparation by condensation of gallacetophenone with 1,3-butadiene in the presence of 85% orthophosphoric acid in xylene at 30–35° (60%) [3325].

m.p. 93–94° [3325]; ^{1}H NMR [3325].

1-[2,4-Dihydroxy-3-methoxy-5-(2-propenyl)phenyl]ethanone



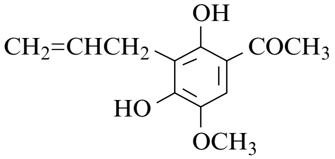
$C_{12}H_{14}O_4$ mol.wt. 222.24

Synthesis

- Preparation by heating 4-(allyloxy)-2-hydroxy-3-methoxyacetophenone at 220° under reduced pressure (Claisen rearrangement) (70%) [2817].

m.p. 94° [2817].

1-[2,4-Dihydroxy-5-methoxy-3-(2-propenyl)phenyl]ethanone



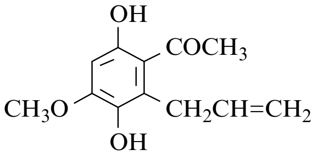
$C_{12}H_{14}O_4$ mol.wt. 222.24

Synthesis

- Preparation by thermal Claisen rearrangement of 4-(allyloxy)-2-hydroxy-5-methoxyacetophenone without solvent at 180° (85%) [2824].

m.p. 118° [2824].

1-[3,6-Dihydroxy-4-methoxy-2-(2-propenyl)phenyl]ethanone



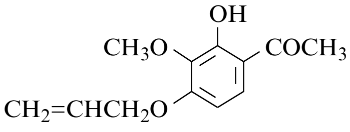
$C_{12}H_{14}O_4$ mol.wt. 222.24

Synthesis

- Preparation by heating 5-(allyloxy)-2-hydroxy-4-methoxy-acetophenone in glycerol at 200° (Claisen rearrangement) (81%) [2849].

m.p. 114° [2849].

1-[2-Hydroxy-3-methoxy-4-(2-propenyloxy)phenyl]ethanone



$C_{12}H_{14}O_4$ mol.wt. 222.24

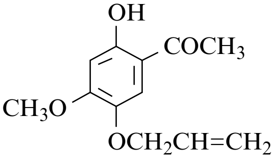
Syntheses

- Preparation by reaction of allyl bromide on 2,4-di-hydroxy-3-methoxyacetophenone with potassium carbonate in boiling acetone (70%) [2817].
- Preparation by reaction of dimethyl sulfate on 4-(allyloxy)-2,3-dihydroxyacetophenone with potassium carbonate in boiling acetone (80%) [2817].

m.p. 63–64° [2817].

1-[2-Hydroxy-4-methoxy-5-(2-propenyloxy)phenyl]ethanone

$C_{12}H_{14}O_4$ mol.wt. 222.24



Synthesis

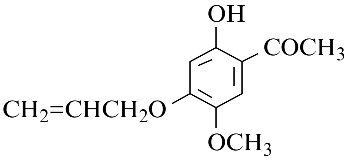
- Preparation by reaction of allyl bromide on 2,5-dihydroxy-4-methoxyacetophenone with potassium carbonate in boiling acetone (82%) [2849].

m.p. 80° [2849].

1-[2-Hydroxy-5-methoxy-4-(2-propenyloxy)phenyl]ethanone

[91497-16-4]

$C_{12}H_{14}O_4$ mol.wt. 222.24



Syntheses

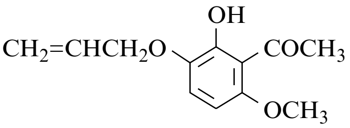
- Preparation by partial methylation of 4-(allyloxy)-2,5-dihydroxyacetophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone (90%) [3191].
- Preparation by reaction of allyl bromide with 2,4-di-hydroxy-5-methoxyacetophenone in the presence of potassium carbonate in boiling acetone (60%) [2824].

m.p. 50–51° [3191], 50° [2824].

1-[2-Hydroxy-6-methoxy-3-(2-propenyloxy)phenyl]ethanone

[126405-78-5]

$C_{12}H_{14}O_4$ mol.wt. 222.24



Synthesis

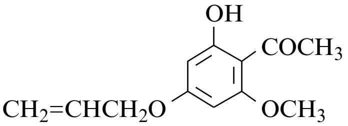
- Preparation by adding 2-hydroxy-3-iodo-6-methoxy-acetophenone and cuprous iodide to a solution of sodium allyloxide, previously prepared from allyl alcohol and sodium hydride in DMF (45%) [2524].

Oil [2524]; ^{1}H NMR [2524], IR [2524].

1-[2-Hydroxy-6-methoxy-4-(2-propenyloxy)phenyl]ethanone

[74047-37-3]

$C_{12}H_{14}O_4$ mol.wt. 222.24



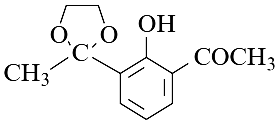
Syntheses

- Preparation by reaction of allyl bromide with 2,4-di-hydroxy-6-methoxyacetophenone in the presence of potassium carbonate in refluxing acetone (61%) [2573].
- Also refer to: [3333].

m.p. 74–76° [2573]; ^{1}H NMR [2573].

1-[2-Hydroxy-3-(2-methyl-1,3-dioxolan-2-yl)phenyl]ethanone

[103867-84-1] $C_{12}H_{14}O_4$ mol.wt. 222.24



Syntheses

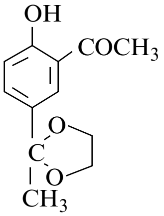
– Preparation by UV light irradiation of ethylene acetal of 2-acetoxyacetophenone in hexane,

- with potassium carbonate (76%) [3334];
- without potassium carbonate (10%) [3334,3335].

Oil [3335]; ^{1}H NMR [3335], IR [3335], UV [3335].

1-[2-Hydroxy-5-(2-methyl-1,3-dioxolan-2-yl)phenyl]ethanone

[103867-87-4] $C_{12}H_{14}O_4$ mol.wt. 222.24



Syntheses

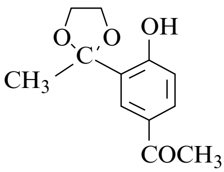
– Preparation by UV light irradiation of ethylene acetal of the 4-acetoxyacetophenone in hexane,

- with potassium carbonate (82%) [3334];
- without potassium carbonate (21%) [3334,3335].

m.p. 56–57° [3335];
^{1}H NMR [3335], IR [3335], UV [3335].

1-[4-Hydroxy-3-(2-methyl-1,3-dioxolan-2-yl)phenyl]ethanone

[103867-88-5] $C_{12}H_{14}O_4$ mol.wt. 222.24



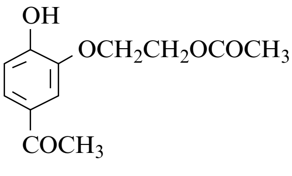
Synthesis

– Obtained (by-product) by UV light irradiation of ethylene acetal of 2-acetoxyacetophenone in hexane, with or without potassium carbonate (8–9%) [3334,3335].

m.p. 78–80° [3335];
^{1}H NMR [3335], IR [3335], UV [3335].

1-[3-[2-(Acetyloxy)ethoxy]-4-hydroxyphenyl]ethanone

[63437-82-1] $C_{12}H_{14}O_5$ mol.wt. 238.24



Syntheses

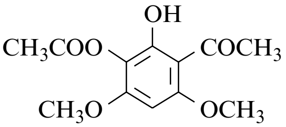
– Preparation by Fries rearrangement of 2-(2-acetoxy-ethoxy)phenyl acetate (m.p. 142–146°) with aluminium chloride in nitrobenzene for 48 h at 20° (55%) [3105].

– Also refer to: [3336].

m.p. 103–104° [3105].

1-[3-(Acetyloxy)-2-hydroxy-4,6-dimethoxyphenyl]ethanone

[21919-65-3] $C_{12}H_{14}O_6$ mol.wt. 254.24



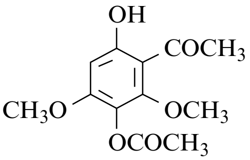
Syntheses

- Preparation by reaction of acetic anhydride with 2,3-di-hydroxy-4,6-dimethoxyacetophenone in the presence of sodium acetate at 60° (80%) [3128].
- Preparation by reaction of aluminium chloride with 3-acetoxy-2,4,6-trimethoxy-acetophenone in nitrobenzene at 100° [3128,3129], (52%) [3128].

m.p. 180° [3128].

1-[3-(Acetyloxy)-6-hydroxy-2,4-dimethoxyphenyl]ethanone

[21919-63-1] $C_{12}H_{14}O_6$ mol.wt. 254.24



Syntheses

- Preparation by reaction of acetic anhydride with 3,6-di-hydroxy-2,4-dimethoxyacetophenone in the presence of sodium acetate at 60° [3128,3129], (80%) [3128].
- Preparation by Fries rearrangement of 2,6-dimethoxy-hydroquinone diacetate with aluminium chloride at 120–125° (80%) [3146].

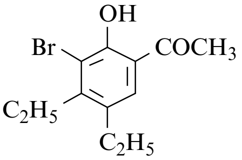
Isolation from natural sources

- From *Euphorbia portulacoides* (Euphorbiaceae) [3337].

m.p. 112° [3128], 109–110° [3146];
^{1}H NMR [3146], IR [3146], UV [3146], MS [3146].

1-(3-Bromo-4,5-diethyl-2-hydroxyphenyl)ethanone

$C_{12}H_{15}BrO_2$ mol.wt. 271.15



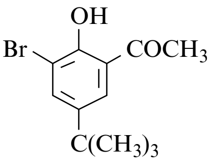
Synthesis

- Preparation by reaction of potassium bromate and bromide on 4,5-diethyl-2-hydroxyacetophenone (quantitative yield) [2965].

m.p. 59° [2965].

1-[3-Bromo-5-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone

[105340-27-0] $C_{12}H_{15}BrO_2$ mol.wt. 271.15



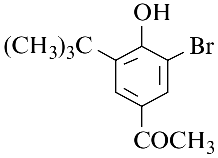
Synthesis

- Preparation by Fries rearrangement of 2-bromo-4-tert-butyl-phenyl acetate with aluminium chloride without solvent at 110° (54%) [3338].

b.p.$_3$ 142° [3338].

1-[3-Bromo-5-(1,1-dimethylethyl)-4-hydroxyphenyl]ethanone

[153356-10-6] $C_{12}H_{15}BrO_2$ mol.wt. 271.15

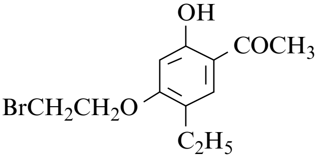


Synthesis

– Refer to: [3339] (Japanese patent).

1-[4-(2-Bromoethoxy)-5-ethyl-2-hydroxyphenyl]ethanone

[117706-54-4] $C_{12}H_{15}BrO_3$ mol.wt. 287.15



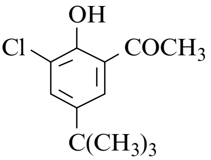
Synthesis

– Preparation by reaction of 2-bromoethyl bromide on 5-ethyl-2,4-dihydroxyacetophenone with potassium carbonate and potassium iodide at reflux (43%) [2678,2679].

m.p. 58–59° [2678,2679]; ^{1}H NMR [2678,2679].

1-[3-Chloro-5-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone

$C_{12}H_{15}ClO_2$ mol.wt. 226.70



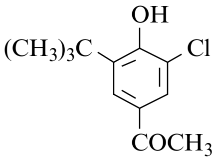
Synthesis

– Preparation by Fries rearrangement of 4-tert-butyl-2-chloro-phenyl acetate with aluminium chloride at 110–130° (82–85%) [2152,3340].

b.p.$_{33}$ 142° [3340].

1-[3-Chloro-5-(1,1-dimethylethyl)-4-hydroxyphenyl]ethanone

[153356-01-5] $C_{12}H_{15}ClO_2$ mol.wt. 226.70

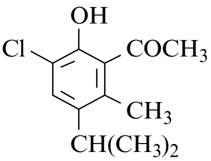


Synthesis

– Refer to: [3339] (Japanese patent).

1-[3-Chloro-2-hydroxy-6-methyl-5-(1-methylethyl)phenyl]ethanone

$C_{12}H_{15}ClO_2$ mol.wt. 226.70



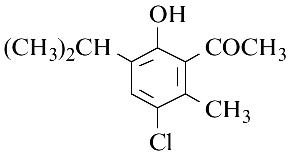
Synthesis

– Preparation by reaction of acetyl chloride on 2-chloro-5-methyl-4-isopropylanisole with aluminium chloride in carbon disulfide at r.t. (35%) [3341].

Colourless oil [3341]; b.p.$_{12}$ 142° [3341].

1-[3-Chloro-6-hydroxy-2-methyl-5-(1-methylethyl)phenyl]ethanone

$C_{12}H_{15}ClO_2$ mol.wt. 226.70



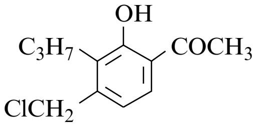
Syntheses

- Preparation by Fries rearrangement of 4-chloro-5-methyl-2-isopropylphenyl acetate with aluminium chloride at 155° [2011].
- Also obtained by reaction of acetyl chloride on 4-chloro-5-methyl-2-isopropylanisole with aluminium chloride in carbon disulfide at r.t. (17%) [2542].

b.p.$_{3}$ 127–135° [2011], b.p.$_{21}$ 151–152° [2542].

1-[4-(Chloromethyl)-2-hydroxy-3-propylphenyl]ethanone

[97582-36-0] $C_{12}H_{15}ClO_2$ mol.wt. 226.70



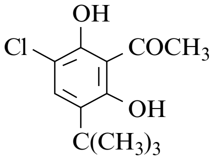
Syntheses

- Preparation by reaction of ethyl chloroformate with 4-(dimethylaminomethyl)-2-hydroxy-3-propylacetophenone in toluene (68–82%) [2550,2927].
- Also refer to: [3342,3343].

m.p. 55–57° [2550]; b.p.$_{0.2}$ 105–125° [2927]; ^{1}H NMR [2927], IR [2927].

1-[3-Chloro-5-(1,1-dimethylethyl)-2,6-dihydroxyphenyl]ethanone

[120034-10-8] $C_{12}H_{15}ClO_3$ mol.wt. 242.70

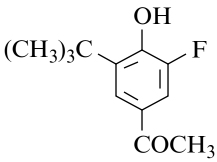


Synthesis

- Preparation by reaction of tert-butyl chloride with 3-chloro-2,6-dihydroxyacetophenone in the presence of sulfuric acid [1892,3243].

1-[3-(1,1-Dimethylethyl)-5-fluoro-4-hydroxyphenyl]ethanone

[153356-03-7] $C_{12}H_{15}FO_2$ mol.wt. 210.25

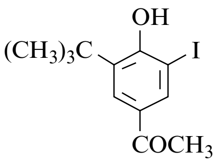


Synthesis

- Refer to: [3339] (Japanese patent).

1-[3-(1,1-Dimethylethyl)-4-hydroxy-5-iodophenyl]ethanone

[153356-02-6] $C_{12}H_{15}IO_2$ mol.wt. 318.15

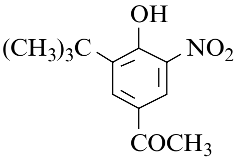


Synthesis

- Refer to: [3339] (Japanese patent).

1-[3-(1,1-Dimethylethyl)-4-hydroxy-5-nitrophenyl]ethanone

[153356-04-8] $C_{12}H_{15}NO_4$ mol.wt. 237.26

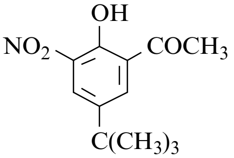


Synthesis

– Refer to: [3339] (Japanese patent).

1-[5-(1,1-Dimethylethyl)-2-hydroxy-3-nitrophenyl]ethanone

[100245-06-5] $C_{12}H_{15}NO_4$ mol.wt. 237.26



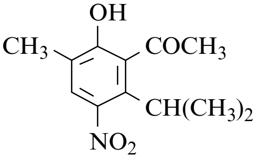
Synthesis

– Obtained (poor yield) by nitration of 5-tert-butyl-2-hydroxy-acetophenone at −20° using standard reagents (2%) [1897].

m.p. 80–81° [1897].

1-[2-Hydroxy-3-methyl-6-(1-methylethyl)-5-nitrophenyl]ethanone

$C_{12}H_{15}NO_4$ mol.wt. 237.26



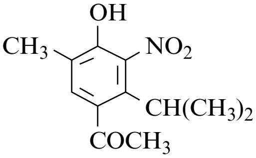
Synthesis

– Preparation by reaction of nitric acid (d = 1.4) on 2-hydroxy-3-methyl-6-isopropylacetophenone in acetic acid between −5° and 0° (67%) [3326].

m.p. 151° [3326].

1-[4-Hydroxy-5-methyl-2-(1-methylethyl)-3-nitrophenyl]ethanone

$C_{12}H_{15}NO_4$ mol.wt. 237.26



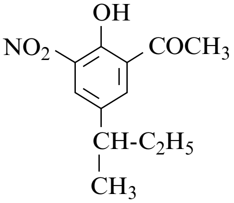
Synthesis

– Preparation by reaction of nitric acid (d = 1.4) on 4-hydroxy-5-methyl-2-isopropylacetophenone in acetic acid at −5° (69%) [3327].

m.p. 157° [3327].

1-[2-Hydroxy-5-(1-methylpropyl)-3-nitrophenyl]ethanone

[84942-36-9] $C_{12}H_{15}NO_4$ mol.wt. 237.26



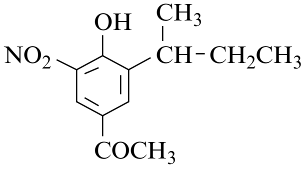
Synthesis

– Preparation by reaction of 100% nitric acid on 5-sec-butyl-2-hydroxyacetophenone in acetic acid at r.t. (89%) [1852].

b.p.$_{0.05}$ 137–139° [1852].

1-[4-Hydroxy-3-(1-methylpropyl)-5-nitrophenyl]ethanone

[150313-75-0] $C_{12}H_{15}NO_4$ mol.wt. 237.26

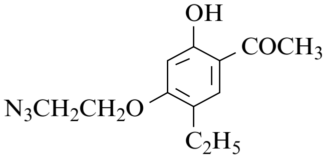


Synthesis

– Refer to: [3344].

1-[4-(2-Azidoethoxy)-5-ethyl-2-hydroxyphenyl]ethanone

[117706-27-1] $C_{12}H_{15}N_3O_3$ mol.wt. 249.27



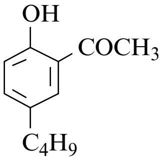
Synthesis

– Preparation by reaction of sodium azide with 4-(2-bromo-ethoxy)-5-ethyl-2-hydroxyacetophenone in N,N-dimethyl-formamide at r.t. [2678,2679].

^{1}H NMR [2678,2679], MS [2678,2679].

1-(5-Butyl-2-hydroxyphenyl)ethanone

[50743-14-1] $C_{12}H_{16}O_2$ mol.wt. 192.26



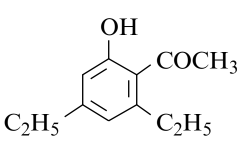
Syntheses

– Preparation by Fries rearrangement of p-butylphenyl acetate with aluminium chloride at 130° (80%) [2899].
– Preparation by reaction of acetyl chloride on 4-butylphenol with aluminium chloride in ethylene dichloride at 110–120° (63%) [2625].

Oil [2899]; b.p.$_{1.5}$ 105–109° [2899], b.p.$_4$ 119–123° [2625].

1-(2,4-Diethyl-6-hydroxyphenyl)ethanone

$C_{12}H_{16}O_2$ mol.wt. 192.26



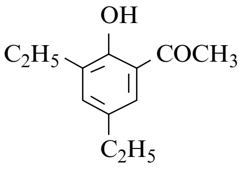
Synthesis

– Preparation by Fries rearrangement of 3,5-diethylphenyl acetate with aluminium chloride [2233], between 120° and 150° (quantitative yield) [2965].

b.p.$_{12}$ 140° [2233].

1-(3,5-Diethyl-2-hydroxyphenyl)ethanone

$C_{12}H_{16}O_2$ mol.wt. 192.26

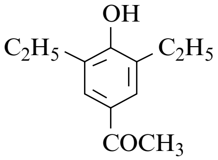


Synthesis

– Preparation by Fries rearrangement of 2,4-diethylphenyl acetate with aluminium chloride (67%) [2233].

b.p.$_{12}$ 138–140° [2233].

1-(3,5-Diethyl-4-hydroxyphenyl)ethanone



$C_{12}H_{16}O_2$ mol.wt. 192.26

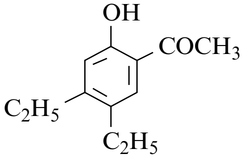
Synthesis

- Preparation by Fries rearrangement of 2,6-diethylphenyl acetate with aluminium chloride (60%) [2233].

m.p. 92–92°5 [2233].

1-(4,5-Diethyl-2-hydroxyphenyl)ethanone

[56394-40-2]



$C_{12}H_{16}O_2$ mol.wt. 192.26

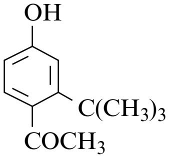
Syntheses

- Preparation by Fries rearrangement of 3,4-diethylphenyl acetate with aluminium chloride without solvent at 120–150° (90–100%) [2943,2965].
- Also obtained by isomerization of 2,4-diethyl-6-hydroxy-acetophenone by heating with aluminium chloride without solvent between 140° and 180° (quantitative yield) [2965].

b.p.$_{0.75}$ 134–137° [2943].

1-[2-(1,1-Dimethylethyl)-4-hydroxyphenyl]ethanone

[155982-91-5]



$C_{12}H_{16}O_2$ mol.wt. 192.26

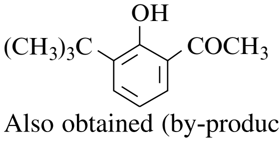
Syntheses

- Preparation from 4-hydroxyacetophenone by reaction,
  - with isobutylene in the presence of sulfuric acid in autoclave at 65° [3345];
  - with tert-butyl chloride in the presence of aluminium chloride [3345].

Crystalline compound [3345].

1-[3-(1,1-Dimethylethyl)-2-hydroxyphenyl]ethanone

[24242-55-5]



$C_{12}H_{16}O_2$ mol.wt. 192.26

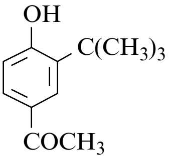
Syntheses

- Obtained by UV light irradiation of 2-tert-butylphenyl acetate in benzene (26%) [3346,3347].
- Also obtained (by-product) by reaction of acetyl chloride on bromomagnesium 2-tert-butylphenolate in toluene at r.t. (<3%) [3348].

pale yellow oil [3346–3348]; b.p.$_5$ 87° [3347];
^{1}H NMR [3347,3348], IR [3347,3348], MS [3348].

-1-[3-(1,1-Dimethylethyl)-4-hydroxyphenyl]ethanone

[16928-01-1] $C_{12}H_{16}O_2$ mol.wt. 192.26



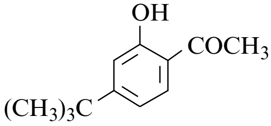
Syntheses

- Obtained by UV light irradiation of,
  - 3,5-di-tert-butyl-4-hydroxyacetophenone in cyclohexane solution (photochemical partial dealkylation) (> 95%) [3346,3347];
  - 2-tert-butylphenyl acetate in benzene solution (photo-Fries rearrangement) (24%) [3346,3347].

m.p. 175–176° [3346,3347]; ^{1}H NMR [3346,3347], IR [3347].

1-[4-(1,1-Dimethylethyl)-2-hydroxyphenyl]ethanone

[113027-08-0] $C_{12}H_{16}O_2$ mol.wt. 192.26

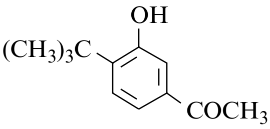


Syntheses

- Obtained by Fries rearrangement of 3-tert-butylphenyl acetate,
  - in the presence of hafnium triflate in 12 M lithium perchlorate in nitromethane at 50° (60%) [3349];
  - in the presence of aluminium chloride without solvent at 145° (68%) [3350].
- Also obtained by Friedel–Crafts acylation of 3-tert-butylphenol with acetyl chloride in the presence of hafnium triflate in 12 M lithium perchlorate in nitromethane (76%) [3349].
- Also obtained by acylation of 3-tert-butylphenol with acetic acid in the presence of hafnium triflate in 12 M lithium perchlorate in nitromethane at 50° (66%) [3351].
- Also refer to: [3352] (Japanese patent).

1-[4-(1,1-Dimethylethyl)-3-hydroxyphenyl]ethanone

[18606-87-6] $C_{12}H_{16}O_2$ mol.wt. 192.26



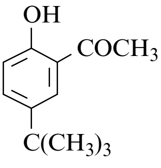
Synthesis

- Preparation by diazotization of 3-amino-4-tert-butyl-acetophenone, followed by hydrolysis of the diazonium salt obtained [3353].

m.p. 127° [3353].

1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]ethanone

[57373-81-6] $C_{12}H_{16}O_2$ mol.wt. 192.26



Syntheses

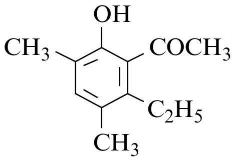
- Preparation by reaction of acetyl chloride on 4-tert-butylanisole with aluminium chloride,
  - in methylene chloride, first at 0°, then at r.t. (55%) [3354];

  - in ethylene dichloride, followed by demethylation of the acylanisole obtained with 4% hydrobromic acid in refluxing acetic acid (47–52%) [3355].

- Also obtained by Friedel–Crafts acylation of 4-tert-butylphenol with acetic anhydride in nitrobenzene in the presence of aluminium chloride at 60° (30%) [2518].
- Preparation by Fries rearrangement of 4-tert-butylphenyl acetate with aluminium chloride [2164,3355–3357],
  - without solvent at 120° (57%) [3356];
  - in 1,2,3-trichloropropane at 120° (60%) [3357] or in boiling nitroethane (22%) [2164].
- Preparation by Fries rearrangement of 4-tert-butylphenyl acetate in methylene chloride in the presence of zirconium chloride at r.t. for 48 h (52%). The same reaction performed in a simple ultrasound cleaning bath at r.t. for 10 h leads to 78% yield [2616].
- Also obtained *via* an intermolecular photo-Fries rearrangement, by irradiation of a solution of pinacolone and 4-tert-butylphenol in benzene for 5 h (42%) [3358].
- Also obtained by reaction of aluminium chloride on 4-(1,1,3,3-tetramethylbutyl) phenyl acetate at 120°, in 1,2,3-trichloropropane (60%) [3357] or in 1,1,2,2-tetrachloroethane (36%) [3357].

m.p. 26° [2518];
b.p.$_2$ 105–106° [2164], b.p.$_5$ 122–123° [2164], b.p.$_{12}$ 130° [3356];
^{1}H NMR [3354], IR [3354].

1-(2-Ethyl-6-hydroxy-3,5-dimethylphenyl)ethanone

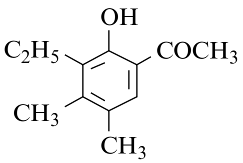


$C_{12}H_{16}O_2$ mol.wt. 192.26

Synthesis

- Obtained (by-product) by Fries rearrangement of 5-ethyl-2,4-dimethylphenyl acetate with aluminium chloride at 130–140° [2233].

1-(3-Ethyl-2-hydroxy-4,5-dimethylphenyl)ethanone



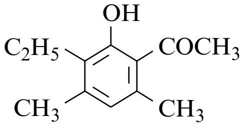
$C_{12}H_{16}O_2$ mol.wt. 192.26

Synthesis

- Obtained (by-product) by heating some esters with aluminium chloride. There is simultaneously displacement and rearrangement of alkyl groups during the Fries reaction from 2-ethyl-4,6-dimethyl-phenyl acetate or from 2-ethyl-4,5-dimethylphenyl acetate [2233].

1-(3-Ethyl-2-hydroxy-4,6-dimethylphenyl)ethanone

[69051-59-8] $C_{12}H_{16}O_2$ mol.wt. 192.26

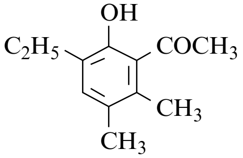


Synthesis

- Preparation by Fries rearrangement of 2-ethyl-3,5-dimethyl-phenyl acetate [3359].

1-(3-Ethyl-2-hydroxy-5,6-dimethylphenyl)ethanone

$C_{12}H_{16}O_2$ mol.wt. 192.26



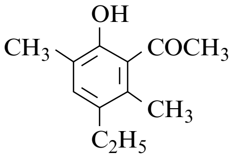
Syntheses

- Preparation by reaction of acetyl chloride on 2-ethyl-4,5-dimethylanisole with aluminium chloride in boiling carbon disulfide (50%) [2233].
- Preparation by heating 2-ethyl-4,5-dimethylphenyl acetate with aluminium chloride (major product) [2233].

b.p.$_{11}$ 143–145° [2233], b.p.$_{12}$ 145–147° [2233].

1-(3-Ethyl-6-hydroxy-2,5-dimethylphenyl)ethanone

$C_{12}H_{16}O_2$ mol.wt. 192.26



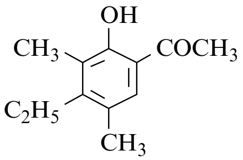
Synthesis

- Obtained by Fries rearrangement of 4-ethyl-2,5-dimethyl-phenyl acetate with aluminium chloride at 130° for 30 min (57%) [3219].

Pale yellow oil [3219]; b.p.$_{16}$ 156–158° [3219];
n_D^{22} = 1.5410 [3219]; IR [3219].

1-(4-Ethyl-2-hydroxy-3,5-dimethylphenyl)ethanone

$C_{12}H_{16}O_2$ mol.wt. 192.26



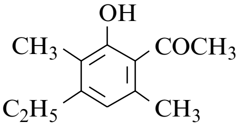
Syntheses

- Obtained by heating some esters* with aluminium chloride. There is simultaneously displacement and rearrangement of alkyl groups during the Fries reaction,

- 2-ethyl-4,6-dimethylphenyl acetate (major product) [2233];
- 4-ethyl-2,5-dimethylphenyl acetate (75%) [2956];
- 4-ethyl-2,6-dimethylphenyl acetate (by-product) [2233];
- 5-ethyl-2,4-dimethylphenyl acetate (major product) [2233].

m.p. 52–53° [2233];
b.p.$_{12}$ 145–147° [2233], b.p.$_{12}$ 146–152° [2956], b.p.$_{11}$ 153–155° [2233].

1-(4-Ethyl-2-hydroxy-3,6-dimethylphenyl)ethanone



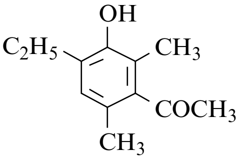
$C_{12}H_{16}O_2$ mol.wt. 192.26

Synthesis

- Obtained by oxidation of 6-ethyl-2,3,4,7-tetramethyl-benzofuran with chromium trioxide in acetic acid at 50° for 30 min, followed by saponification of the resulting keto ester with potassium hydroxide in boiling aqueous ethanol for 2 h (40%) [3219].

b.p.$_{20}$ 165–167° [3219];
n_D^{22} = 1.562 [3219]; IR [3219].

1-(4-Ethyl-3-hydroxy-2,6-dimethylphenyl)ethanone



$C_{12}H_{16}O_2$ mol.wt. 192.26

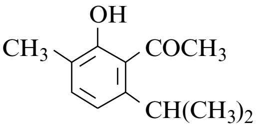
Synthesis

- Obtained by reaction of acetyl chloride on 6-ethyl-2,4-dimethylanisole with aluminium chloride in boiling carbon disulfide [2233].

b.p.$_{15}$ 178–182° [2233].

1-[2-Hydroxy-3-methyl-6-(1-methylethyl)phenyl]ethanone

[162853-19-2]



$C_{12}H_{16}O_2$ mol.wt. 192.26

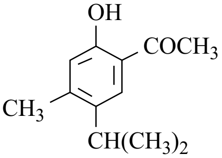
Syntheses

- Preparation by Fries rearrangement of carvacryl acetate,
  - without solvent at 120°, with aluminium chloride (80–86%) [2947,2948], with titanium tetrachloride (86%) [2948], with stannic chloride (80%) [2948] or with zinc chloride (60%) [2948];
  - with aluminium chloride in nitrobenzene at 60° (67%) [2947], in toluene or xylene at 100° (58–61%) [2947].
- Preparation by reaction of acetyl chloride on carvacrol with aluminium chloride,
  - in nitrobenzene at r.t. (49%) [3326];
  - in nitrobenzene in the presence of phosphorous oxychloride and magnesium chloride at r.t. (17%) [3326].
- Also obtained by UV irradiation of a carvacryl acetate solution in methanol/water (2:1) at 254 nm at r.t. under nitrogen atmosphere (51%) [2963].

Viscous oil [2963]; m.p. 100–101° [3326];
^{1}H NMR [2963], ^{13}C NMR [2963], IR [2963], MS [2963].

1-[2-Hydroxy-4-methyl-5-(1-methylethyl)phenyl]ethanone

[52774-08-0] $C_{12}H_{16}O_2$ mol.wt. 192.26



Syntheses

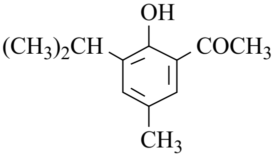
- Preparation by Fries rearrangement of 3-methyl-4-isopropyl-phenyl acetate, with titanium tetrachloride in nitromethane at 20° (95%) [3360] or with aluminium chloride at 125° (11%) [3341,3361].
- Preparation by reaction of acetic anhydride on 3-methyl-4-isopropylphenol (p-thymol) with 70% perchloric acid at 125–135° (32%) [2306].

N.B.: All the results of references [2306,3341,3361] were erroneous. Only the Fries rearrangement using titanium tetrachloride leads to the expected ketone and with a good yield [3360]. The ^{1}H NMR spectra confirms the above structure [3360]. In addition, the reported melting point (29°) [3360] is in good agreement with those generally measured for o-hydroxy-ketones (below 80°) compared to those of p-hydroxyketones which are considerably higher (usually 120–200°).

m.p. 122°5 [3341,3361], 110–115° [2306], 29° [3360];
b.p.$_{17}$ 153° [3360], b.p.$_{15}$ 188–192° [3341];
^{1}H NMR [3360] (Sadtler: standard n° 52738 M);
IR [3341,3360] (Sadtler: standard n° 79797 K); UV [3360], MS [3360].

1-[2-Hydroxy-5-methyl-3-(1-methylethyl)phenyl]ethanone

[35158-31-7] $C_{12}H_{16}O_2$ mol.wt. 192.26



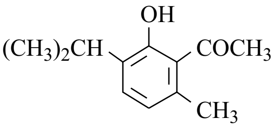
Synthesis

- Preparation by reaction of ethyl acetoacetate with 2,5-di-methyl-2-hexenal in the presence of pyridine and piperidine as catalysts in refluxing benzene [2958–2960].

yellow oil [2958–2960]; b.p.$_{1-2}$ 70–80° [2958–2960];
IR [2958–2960], UV [2958–2960], MS [2958–2960].

1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]ethanone

[105337-34-6] $C_{12}H_{16}O_2$ mol.wt. 192.26

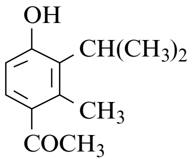


Syntheses

- Preparation by reaction of acetic acid on thymol with aluminium chloride at reflux (80%) [3362].
- Preparation by Fries rearrangement of thymyl acetate,
  - without solvent at 120° with titanium tetrachloride (90%) [2948], with aluminium chloride (84–86%) [2947,2948], with stannic chloride (84%) [2948] or with zinc chloride (62%) [2948];
  - with aluminium chloride in nitrobenzene (68%) [2947], in toluene or xylene (59–60%) [2947].
- Also obtained by photo-Fries rearrangement of thymyl acetate in dilute methanol under nitrogen atmosphere at r.t. (48%) [2963].

viscous oil [2963]; b.p.$_{12}$ 119° [3362];
^{1}H NMR [2963], ^{13}C NMR [2963], IR [2963], MS [2963].

1-[4-Hydroxy-2-methyl-3-(1-methylethyl)phenyl]ethanone



$C_{12}H_{16}O_2$ mol.wt. 192.26

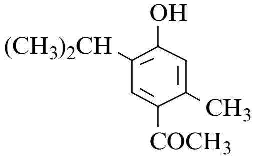
Syntheses

- Preparation by demethylation of 4-methoxy-2-methyl-3-iso-propylacetophenone with boiling pyridinium chloride (33%) [3224].
- Also obtained by Fries rearrangement of 3-methyl-2-isopropylphenyl acetate with aluminium chloride in nitrobenzene at r.t. (10%) [3224].

m.p. 128° [3224].

1-[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]ethanone

[37847-35-1]



$C_{12}H_{16}O_2$ mol.wt. 192.26

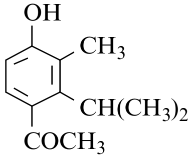
Syntheses

- Preparation by reaction of acetyl chloride on thymol, with aluminium chloride, in nitrobenzene [2309,2662,3363–3366], (97–100%) [2662,3363,3364], (75%) [2151] or with zinc chloride [2310,2311].
- Preparation by Fries rearrangement of thymyl acetate in nitrobenzene,
  - with aluminium chloride, at 40–47° (87–95%) [2082,2151,3367–3369], (65–75%) [2086], (51–55%) [3370] or at 20–25° (80–87%) [2151,2948];
  - at 25°, with titanium tetrachloride (88%), stannic chloride (78%) or zinc chloride (58%) [2948].
- Preparation from 4-methoxy-2-methyl-5-isopropylacetophenone by demethylation with pyridinium chloride at reflux (73–75%) [2542,3371,3372].
- Also obtained (by-product) by reaction of aluminium chloride on p-thymyl acetate without solvent at 140° (15%) [3373].
- Also obtained by UV light irradiation of thymyl acetate in methanol at 25° (41%) [2315].
- Also obtained (by-product) by reaction of acetyl chloride on 5-methyl-2-isopropylanisole with aluminium chloride in carbon disulfide at r.t. (6%) [3372].
- Also obtained by irradiation of thymyl acetate in methanol at 254 nm under nitrogen atmosphere at r.t. (24%) [2963].

m.p. 152–154° [2963], 135° [2315], 125° [2082,2151,2309,2662, 3370–3372],
122°5–125° [3367,3369], 122°5 [3373], 122° [3368]. There is a discrepancy between the different melting points indicated in literature.
b.p.$_{18}$ 204–207° [3371], b.p.$_{0.8}$ 204–207° [3372];
^{1}H NMR [2315,2963,3368],
^{13}C NMR [2963], IR [2315,2963,3368], MS [2963,3368].

1-[4-Hydroxy-3-methyl-2-(1-methylethyl)phenyl]ethanone

[61405-65-0] $C_{12}H_{16}O_2$ mol.wt. 192.26

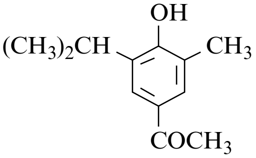


Synthesis

– Refer to: [3374] (Japanese patent).

1-[4-Hydroxy-3-methyl-5-(1-methylethyl)phenyl]ethanone

[713-23-5] $C_{12}H_{16}O_2$ mol.wt. 192.26

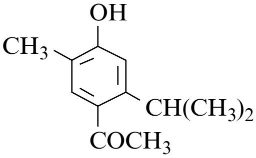


Synthesis

– Refer to: [3375] (Japanese patent).

1-[4-Hydroxy-5-methyl-2-(1-methylethyl)phenyl]ethanone

[37847-37-3] $C_{12}H_{16}O_2$ mol.wt. 192.26



Syntheses

- Preparation by reaction of acetyl chloride on carvacrol with aluminium chloride in nitrobenzene,
  - at r.t. [2662,3224,3327,3376,3377], (71%) [2662], (52%) [3377] and (31–39%) [3327,3376];
  - at 50° (80–90%) [3378].
- Preparation by Fries rearrangement of 2-methyl-5-isopropylphenyl acetate in nitrobenzene at r.t.,
  - with aluminium chloride (84–90%) [2151,2948], (20%) [3327];
  - with stannic chloride or titanium tetrachloride (86%) [2948];
  - with zinc chloride (58%) [2948].
- Preparation by reaction of pyridinium chloride on 4-methoxy-5-methyl-2-isopropylacetophenone [3224].
- Also obtained by irradiation of carvacryl acetate in methanol at 254 nm under nitrogen atmosphere at r.t. (15%) [2963].

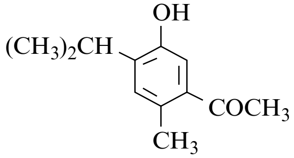
m.p. 127° [3327], 120° [2151,3378], 101° [2662,3376], 86–88° [2963].

There is a discrepancy between the different melting points indicated in literature.

^1H NMR [2963], ^{13}C NMR [2963], IR [2963], MS [2963].

1-[5-Hydroxy-2-methyl-4-(1-methylethyl)phenyl]ethanone

[126570-37-4] $C_{12}H_{16}O_2$ mol.wt. 192.26



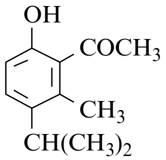
Synthesis

- Preparation by reaction of acetyl chloride with isothymol methyl ether in the presence of aluminium chloride in nitrobenzene at r.t. [3368].

m.p. 106–107° [3368]; ^1H NMR [3368], IR [3368].

1-[6-Hydroxy-2-methyl-3-(1-methylethyl)phenyl]ethanone

[105337-35-7] $C_{12}H_{16}O_2$ mol.wt. 192.26



Syntheses

- Preparation by reaction of aluminium chloride on 3-tert-butyl-2-hydroxy-6-methyl-5-isopropylacetophenone in nitromethane at 20° (84%) [3360].
- Claimed to be prepared,

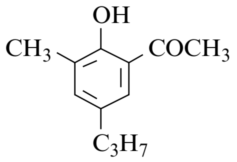
- by Fries rearrangement of 3-methyl-4-isopropylphenyl acetate with aluminium chloride, without solvent at 90°(85%) [3373] or at 125° (43%) [3341] and in nitrobenzene at 90° (>50%) [3373];
- by reaction of acetyl chloride on 3-methyl-4-isopropylanisole with aluminium chloride in boiling carbon disulfide (41%) [3361];
- by reaction of acetyl chloride on 3-methyl-4-isopropylphenetole with aluminium chloride in carbon disulfide at r.t. (37%) [3341];
- by heating 6-methoxy-2-methyl-3-isopropylacetophenone with pyridinium chloride at reflux [3361].

N.B.: All the results of references [3341,3361,3373] were erroneous. Only the first route was correct. The ^1H NMR spectra confirms the above structure [3360].

m.p. 70° [3360]; amber-coloured liquid [3341,3361];
b.p.$_{14}$ 150° [3341], b.p.$_{19}$ 153–154° [3361]; n_D^{20} = 1.5410 [3341,3361];
^1H NMR [3360] (Sadtler: standard n° 52739 M);
IR [3341,3360] (Sadtler: standard n° 79798 K);
UV [3360], MS [3360].

1-(2-Hydroxy-3-methyl-5-propylphenyl)ethanone

$C_{12}H_{16}O_2$ mol.wt. 192.26

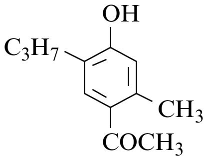


Syntheses

- Preparation by Fries rearrangement of 2-methyl-4-propyl-phenyl acetate with aluminium chloride without solvent at 100–110° [2957].
- Also obtained by reaction of aluminium chloride on 2-ethyl-6-methyl-4-propylphenyl acetate, with elimination of ethyl group (8%) [2957].

b.p.$_{12}$ 136° [2957].

1-(4-Hydroxy-2-methyl-5-propylphenyl)ethanone



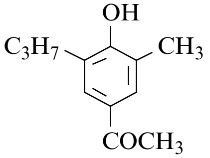
$C_{12}H_{16}O_2$ mol.wt. 192.26

Synthesis

- Preparation by Fries rearrangement of 5-methyl-2-propyl-phenyl acetate with aluminium chloride in nitrobenzene at 20° (83%) [2151].

m.p. 113° [2151]; b.p.$_{18}$ 194° [2151].

1-(4-Hydroxy-3-methyl-5-propylphenyl)ethanone



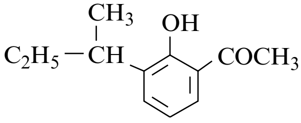
$C_{12}H_{16}O_2$ mol.wt. 192.26

Syntheses

- Preparation by Fries rearrangement of 2-methyl-6-propyl-phenyl acetate with aluminium chloride without solvent at 130–140° [2957].
- Also obtained by reaction of aluminium chloride on 4-ethyl-2-methyl-6-propylphenyl acetate, with elimination of ethyl group (12%) [2957].

m.p. 101° [2957].

1-[2-Hydroxy-3-(1-methylpropyl)phenyl]ethanone



$C_{12}H_{16}O_2$ mol.wt. 192.26

Synthesis

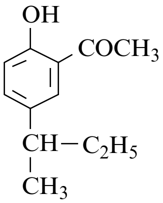
- Obtained (poor yield) by reaction of acetyl chloride with a suspension of aluminium o-sec-butylphenoxide in benzene in the presence of aluminium chloride, first at r.t. for 12 h, then on a water bath for 2 h (11%) [2945].

m.p. 121° [2945].

N.B.: This o-hydroxyketone should be liquid. The authors probably intended to write b.p.$_3$ 121° instead of m.p. 121°. This b.p. would be in agreement with those of the other homologous o-hydroxyketones, that have been prepared by the authors [2945].

1-[2-Hydroxy-5-(1-methylpropyl)phenyl]ethanone

[84942-39-2] $C_{12}H_{16}O_2$ mol.wt. 192.26

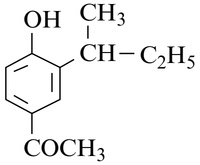


Syntheses

- Preparation by reaction of acetyl chloride with a suspension of aluminium p-sec-butylphenoxide in benzene in the presence of aluminium chloride, first at r.t. for 12 h, then on a water bath for 2 h (75%) [2945].
- Preparation by Fries rearrangement of 4-sec-butylphenyl acetate with aluminium chloride at 120° (84%) [1852].

b.p.$_{0.15}$ 86–87° [1852], b.p.$_3$ 115–119° [2945].

1-[4-Hydroxy-3-(1-methylpropyl)phenyl]ethanone



$C_{12}H_{16}O_2$ mol.wt. 192.26

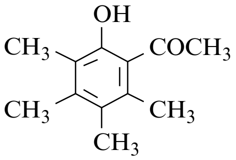
Synthesis

- Obtained (poor yield) by reaction of acetyl chloride with a suspension of aluminium o-sec-butylphenoxide in benzene in the presence of aluminium chloride, first at r.t. for 12 h, then on a water bath for 2 h (8%) [2945].

m.p. 123° [2945].

1-(2-Hydroxy-3,4,5,6-tetramethylphenyl)ethanone

[118078-21-0] $C_{12}H_{16}O_2$ mol.wt. 192.26

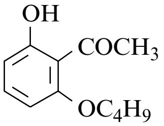


Synthesis

- Preparation by acetylation of 2,3,4,5-tetramethylphenol [2025] according to [3379].

1-(2-Butoxy-6-hydroxyphenyl)ethanone

[63438-68-6] $C_{12}H_{16}O_3$ mol.wt. 208.26



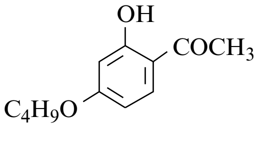
Synthesis

- Obtained by reaction of n-butyl iodide with 2,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (40%) [3242].

m.p. 59–60° [3242].

1-(4-Butoxy-2-hydroxyphenyl)ethanone

[57221-60-0] $C_{12}H_{16}O_3$ mol.wt. 208.26



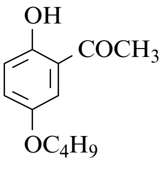
Synthesis

- Preparation by reaction of butyl iodide on resacetophenone with potassium hydroxide in boiling ethanol [2220,3002], (14%) [3002].

m.p. 43° [3002], 42° [2220].

1-(5-Butoxy-2-hydroxyphenyl)ethanone

[152810-06-5] $C_{12}H_{16}O_3$ mol.wt. 208.26

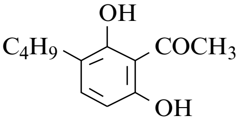


Synthesis

- Refer to: [2227].

1-(3-Butyl-2,6-dihydroxyphenyl)ethanone

[63411-82-5] $C_{12}H_{16}O_3$ mol.wt. 208.26



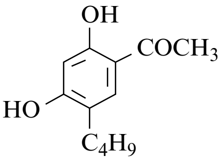
Synthesis

- Preparation from 2,6-dihydroxyacetophenone according to the method [3241], (70%) [3242].

m.p. 72–73° [3242].

1-(5-Butyl-2,4-dihydroxyphenyl)ethanone

[81468-73-7] $C_{12}H_{16}O_3$ mol.wt. 208.26



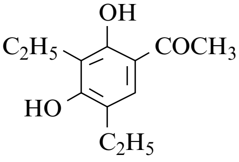
Syntheses

- Preparation by reaction of acetonitrile on 4-n-butylresorcinol (Hoesch reaction) (80%) [2676].
- Preparation from 5-n-butyl-2,4-dimethoxyacetophenone by demethylation with boron tribromide in methylene chloride at r.t. (64%) [2671,2678,2679].
- Preparation by reaction of acetic acid on 4-n-butylresorcinol with zinc chloride (Nencki reaction) [1839,3018].

m.p. 95–96° [2676], 95° [1839].

1-(3,5-Diethyl-2,4-dihydroxyphenyl)ethanone

$C_{12}H_{16}O_3$ mol.wt. 208.26



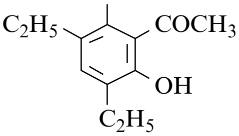
Synthesis

- Obtained by reaction of acetic acid with 2,4-diethylresorcinol in the presence of zinc chloride at 140° for 15 min (Nencki reaction) [3380].

m.p. 115° [3380].

1-(3,5-Diethyl-2,6-dihydroxyphenyl)ethanone

[37467-65-5] $C_{12}H_{16}O_3$ mol.wt. 208.26

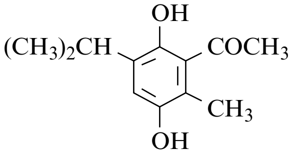


Synthesis

- Preparation by reaction of acetic anhydride with 4,6-diethyl-resorcinol in the presence of boron trifluoride–acetic acid complex for 2 h at 100° [2997].

m.p. 76–77° [2997].

1-[2,5-Dihydroxy-6-methyl-3-(1-methylethyl)phenyl]ethanone



$C_{12}H_{16}O_3$ mol.wt. 208.26

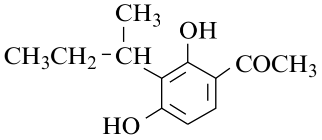
Synthesis

- Obtained by reaction of acetyl chloride on 2-methyl-5-isopropylhydroquinone dimethyl ether with aluminium chloride in carbon disulfide at 35–40° (6%) [2542].

b.p.$_{17}$ 148–149° [2542].

1-[2,4-Dihydroxy-3-(1-methylpropyl)phenyl]ethanone

[79557-74-7]



$C_{12}H_{16}O_3$ mol.wt. 208.26

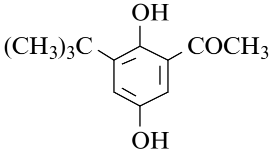
Synthesis

- Preparation by hydrogenation of 2,4-dihydroxy-3-(1-methyl-2-propenyl)acetophenone in ethanol using 5% Pd/C as catalyst [3184].

m.p. 174–175° [3184].

1-[3-(1,1-Dimethylethyl)-2,5-dihydroxyphenyl]ethanone

[35205-23-3]



$C_{12}H_{16}O_3$ mol.wt. 208.26

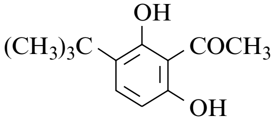
Synthesis

- Obtained (trace) by reaction of di-tert-butyl diperoxyoxalate with tert-butyl-1,4-benzoquinone in acetaldehyde; the solution was kept in the dark at r.t. (<1%) [3381].

m.p. 141–142° [3381];
^{1}H NMR [3381], IR [3381], UV [3381].

1-[3-(1,1-Dimethylethyl)-2,6-dihydroxyphenyl]ethanone

[91124-33-3]



$C_{12}H_{16}O_3$ mol.wt. 208.26

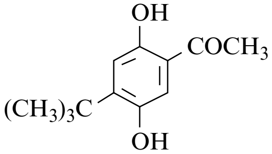
Synthesis

- Preparation by reaction of 2,6-dihydroxyacetophenone with tert-butanol in the presence of concentrated sulfuric acid in benzene at 55° in a sealed tube [3382].

m.p. 183–186° [3382]; ^{1}H NMR [3382].

1-[4-(1,1-Dimethylethyl)-2,5-dihydroxyphenyl]ethanone

[35205-24-4] $C_{12}H_{16}O_3$ mol.wt. 208.26



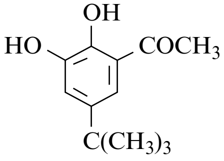
Syntheses

- Preparation by reaction of acetic acid with 2-tert-butyl-hydroquinone in the presence of boron trifluoride at 80–90° (65%) [3383].
- Preparation by demethylation of 4-tert-butyl-2,5-dimethoxy-acetophenone with boron tribromide in methylene chloride at r.t. (58%) [3384].
- Also obtained (trace) by reaction of di-tert-butyl diperoxyoxalate with tert-butyl-1,4-benzoquinone in acetaldehyde; the solution was kept in the dark at r.t. (<1%) [3381].
- Also refer to: [3251,3385].

m.p. 195–196°5 [3381], 193°5–195°5 [3384];
^{1}H NMR [3381,3384], IR [3381,3384], UV [3381], MS [3384].

1-[5-(1,1-Dimethylethyl)-2,3-dihydroxyphenyl]ethanone

[84296-64-0] $C_{12}H_{16}O_3$ mol.wt. 208.26



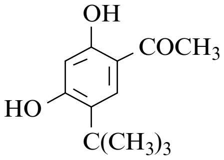
Syntheses

- Obtained by irradiation of a benzene solution of 4-tert-butyl-o-benzoquinone in the presence of a large excess of acetaldehyde (20%) [3386].
- Also obtained by treatment of a benzene solution of 4-tert-butyl-o-benzoquinone and acetaldehyde in the presence of di-tert-butyl diperoxyoxalate at 38° (26%) [3386].

m.p. 82–83° [3386]; ^{1}H NMR [3386], IR [3386].

1-[5-(1,1-Dimethylethyl)-2,4-dihydroxyphenyl]ethanone

[140660-31-7] $C_{12}H_{16}O_3$ mol.wt. 208.26



Synthesis

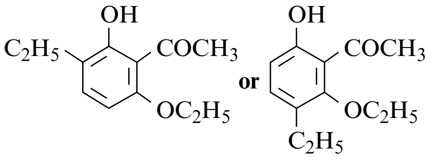
- Preparation by reaction of tert-butyl alcohol with resacetophenone in the presence of zinc chloride at 95° (53%) [2671].

Oil [2671].

1-(6-Ethoxy-3-ethyl-2-hydroxyphenyl)ethanone

or

1-(2-Ethoxy-3-ethyl-6-hydroxyphenyl)ethanone



$C_{12}H_{16}O_3$ mol.wt. 208.26

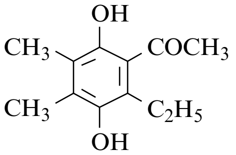
Syntheses

- Obtained by partial ethylation of 2-acetyl-4-ethylresorcinol with diethyl sulfate in refluxing 2 N sodium hydroxide for 30 min [3015].
- Also obtained by ethylation of 2-acetyl-resorcinol [3015].

m.p. 84° [3015].

1-(2-Ethyl-3,6-dihydroxy-4,5-dimethylphenyl)ethanone

[396639-83-1] $C_{12}H_{16}O_3$ mol.wt. 208.26



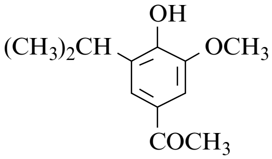
Synthesis

- Obtained by diethylcadmium alkylation of 5-acetyl-2,3-di-methylbenzoquinone (71%) [3387].

m.p. 76° [3387];
^{1}H NMR [3387], ^{13}C NMR [3387].

1-[4-Hydroxy-3-methoxy-5-(1-methylethyl)phenyl]ethanone

[133393-99-4] $C_{12}H_{16}O_3$ mol.wt. 208.26

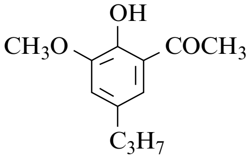


Synthesis

- Preparation from guaiacol by acetylation and isopropylation [3388].

1-(2-Hydroxy-3-methoxy-5-propylphenyl)ethanone

[23343-03-5] $C_{12}H_{16}O_3$ mol.wt. 208.26



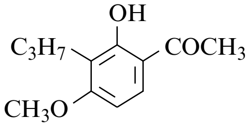
Syntheses

- Preparation by partial demethylation of 2,3-dimethoxy-5-propylacetophenone with aluminium chloride in refluxing methylene chloride (67%) [3230].
- Preparation by catalytic hydrogenation of 5-allyl-2-hydroxy-3-methoxyacetophenone [3329,3330] in the presence of 5% palladium on barium sulfate (97%) [3330].

m.p. 18–19° [3330], 17–19° [3329]; b.p.$_{0.2}$ 105–120° [3230];
^{1}H NMR [3230,3329], IR [3329], UV [3329].

1-(2-Hydroxy-4-methoxy-3-propylphenyl)ethanone

[72018-33-8] $C_{12}H_{16}O_3$ mol.wt. 208.26



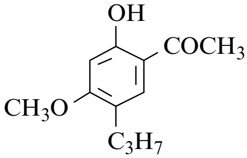
Synthesis not yet described

- Refer to: [2326,2327].

N.B.: Pr indicates the *propyl* group -C_3H_7 in Chem. Abstr., **92**, 6368x (1980) and **98**, 54239b (1983), an usual abbreviation. However, in the two references [2326,2327], Pr represented the *prenyl* group–$CH_2CH{=}C(CH_3)_2$. Therefore they concern the 2-hydroxy-4-methoxy-3-prenylacetophenone and not the above mentioned 2-hydroxy-4-methoxy-3-propylacetophenone.

1-(2-Hydroxy-4-methoxy-5-propylphenyl)ethanone

[72018-35-0] $C_{12}H_{16}O_3$ mol.wt. 208.26



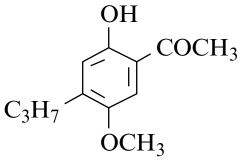
Syntheses

- Preparation by reaction of dimethyl sulfate on 2,4-di-hydroxy-5-propylacetophenone with calcinated potassium carbonate in refluxing acetone [3018].
- Also refer to: [2326,2327].

N.B.: Pr indicates the *propyl* group–C_3H_7 in Chem. Abstr., **92**, 6368x (1980) and **98**, 54239b (1983), an usual abbreviation. However, in the two references [2326,2327], Pr represented the *prenyl* group–$CH_2CH{=}C(CH_3)_2$. Therefore they concern the 2-hydroxy-4-methoxy-5-prenylacetophenone and not the above mentioned 2-hydroxy-4-methoxy-3-propylacetophenone.

1-(2-Hydroxy-5-methoxy-4-propylphenyl)ethanone

$C_{12}H_{16}O_3$ mol.wt. 208.26



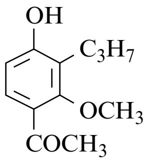
Synthesis

- Preparation by reaction of acetyl chloride on 2-propylhydroquinone dimethyl ether with aluminium chloride in boiling carbon disulfide (69%) [3240].

b.p.$_1$ 150–155° [3240].

1-(4-Hydroxy-2-methoxy-3-propylphenyl)ethanone

[151027-43-9] $C_{12}H_{16}O_3$ mol.wt. 208.26

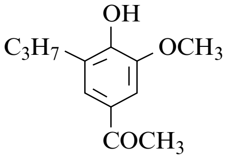


Synthesis

- Refer to: [3389,3390].

1-(4-Hydroxy-3-methoxy-5-propylphenyl)ethanone

[54514-40-8] $C_{12}H_{16}O_3$ mol.wt. 208.26

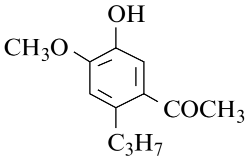


Synthesis

- Refer to: [3391,3392].

1-(5-Hydroxy-4-methoxy-2-propylphenyl)ethanone

[23343-08-0] $C_{12}H_{16}O_3$ mol.wt. 208.26



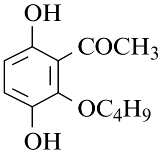
Syntheses

- Preparation by Fries rearrangement of dihydroeugenol acetate with aluminium chloride in nitrobenzene at 5° (13%) [3329].
- Also refer to: [3393].

m.p. 68° [3329]; b.p.$_1$ 138° [3329]; ^{1}H NMR [3329], IR [3329], UV [3329].

1-(2-Butoxy-3,6-dihydroxyphenyl)ethanone

[33539-23-0] $C_{12}H_{16}O_4$ mol.wt. 224.26



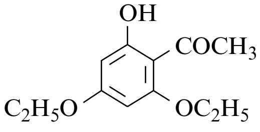
Syntheses

- Easy preparation by reduction of 2-acetyl-3-butoxy-1,4-benzoquinone using conventional methods [2869].
- Also obtained in low yield by reaction of 2-acetyl-1,4-benzoquinone with an excess of butanol at r.t., with exclusion of light [2869].

m.p. 62°5–63°5 [2869]; ^{1}H NMR [2869], IR [2869].

1-(2,4-Diethoxy-6-hydroxyphenyl)ethanone

[26207-59-0] $C_{12}H_{16}O_4$ mol.wt. 224.26



Syntheses

- Obtained from phloroacetophenone triethyl ether by heating with aluminium chloride [3085].
- Also obtained by reaction of acetonitrile on phloroglucinol diethyl ether (Hoesch reaction) (18%) [3042].
- Also obtained by reaction of ethyl iodide [3394,3395] or diethyl sulfate [3396] on phloroacetophenone with potassium carbonate in refluxing acetone (18%) [3394].

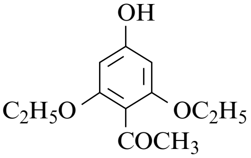
Isolation from natural sources

- By treatment of Sotetsuflavone pentaethyl ether with a methanolic barium hydroxide suspension [3396]. Sotetsuflavone was extracted of the plants of Coniferae and allied orders.

- By reaction of Kayaflavone triethyl ether with barium hydroxide octahydrate in refluxing methanol (55%) [3397]. Kayaflavone was isolated from dried leaves of *Torreya nucifera*.

m.p. 86–87° [3394], 85° [3042,3085], 83–84° [3396], 81–83° [3397].

1-(2,6-Diethoxy-4-hydroxyphenyl)ethanone



$C_{12}H_{16}O_4$ mol.wt. 224.26

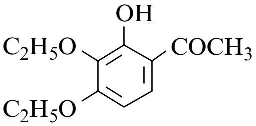
Synthesis

- Preparation by reaction of acetonitrile on phloroglucinol diethyl ether (Hoesch reaction) (29%) [3042].

m.p. 186–187° [3042].

1-(3,4-Diethoxy-2-hydroxyphenyl)ethanone

[6342-86-5]



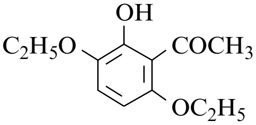
$C_{12}H_{16}O_4$ mol.wt. 224.26

Syntheses

- Refer to: [3398–3400].

1-(3,6-Diethoxy-2-hydroxyphenyl)ethanone

[88771-47-5]



$C_{12}H_{16}O_4$ mol.wt. 224.26

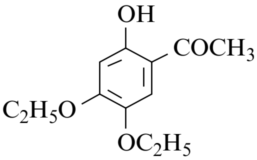
Synthesis

- Preparation by hydrogenolysis procedure on 2-(benzyloxy)-3,6-diethoxyacetophenone with 10% Pd/C as catalyst, and ethanol as solvent at 40° (99%) [1884].

m.p. 64° [1884]; ^{1}H NMR [1884], IR [1884].

1-(4,5-Diethoxy-2-hydroxyphenyl)ethanone

[80938-23-4]



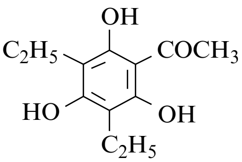
$C_{12}H_{16}O_4$ mol.wt. 224.26

Syntheses

- Refer to: [3398,3399].

1-(3,5-Diethyl-2,4,6-trihydroxyphenyl)ethanone

[19687-48-0]



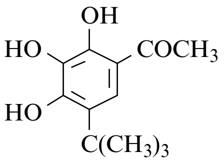
$C_{12}H_{16}O_4$ mol.wt. 224.26

Syntheses

- Preparation by reaction of acetyl chloride with 2,4-diethyl-phloroglucinol in the presence of aluminium chloride in nitrobenzene (28%) [3401].
- Preparation by reaction of acetonitrile on 2,4-diethyl-phloroglucinol (Hoesch reaction) (39%) [3402].

m.p. 106–109° [3401], 102–105° [3402];
^{1}H NMR [3401], UV [3401,3402], MS [3401].

1-[5-(1,1-Dimethylethyl)-2,3,4-trihydroxyphenyl]ethanone



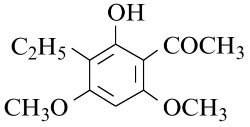
$C_{12}H_{16}O_4$ mol.wt. 224.26

Synthesis

- Preparation by reaction of tert-butyl chloride with gallacetophenone in the presence of ferric chloride in acetic acid and heating on a steam bath (52%) [2268].

m.p. 174° [2268]; UV [2268].

1-(3-Ethyl-2-hydroxy-4,6-dimethoxyphenyl)ethanone



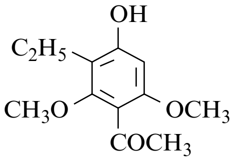
$C_{12}H_{16}O_4$ mol.wt. 224.26

Synthesis

- Preparation by reaction of acetonitrile on 2-ethyl-3,5-di-methoxyphenol (Hoesch reaction) [3277,3403], (55%) [3277].

m.p. 111° [3403], 66–68° [3277].

1-(3-Ethyl-4-hydroxy-2,6-dimethoxyphenyl)ethanone



$C_{12}H_{16}O_4$ mol.wt. 224.26

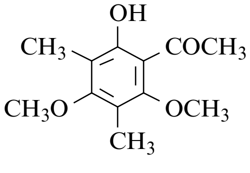
Synthesis

- Obtained (poor yield) by reaction of acetonitrile on 2-ethyl-3,5-dimethoxyphenol (Hoesch reaction) (7%) [3277].

m.p. 184–186° [3277].

1-(2-Hydroxy-4,6-dimethoxy-3,5-dimethylphenyl)ethanone

[21722-31-6]



$C_{12}H_{16}O_4$ mol.wt. 224.26

Syntheses

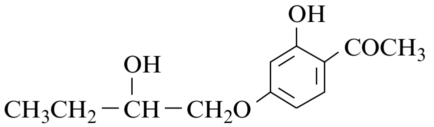
- Preparation by adding of an ethereal solution of diazomethane to a methanolic solution of phloroacetophenone (25%) [2437]; this compound was obtained from 3,5-dimethylphloroacetophenone or from 2,6-dihydroxy-4-methoxy-3,5-dimethylacetophenone in the same conditions [2437].
- Also obtained (by-product) from phloroacetophenone by reaction with methyl iodide in the presence of potassium carbonate in refluxing acetone (6%) [3279].

Isolation from natural sources

- By chromatography of *Melaleuca cajeputi* oil; this oil was obtained from the leaves of *Melaleuca cajeputi* Powell (Myrtaceae) (10%) [3118].
  Crystalline compound [3118]; m.p. 51–52° [3279], 51° [2437];
  ^{1}H NMR [2437,3118], IR [2437,3118], UV [2437,3118], MS [3118].

1-[2-Hydroxy-4-(2-hydroxybutoxy)phenyl]ethanone

[149454-53-5]

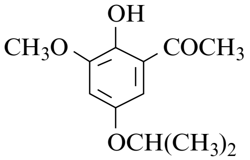


$C_{12}H_{16}O_4$ mol.wt. 224.26

Synthesis

- Obtained by reaction of resacetophenone with 1,2-butylene oxide in the presence of sodium hydroxide in ethanol [3289].

1-[2-Hydroxy-3-methoxy-5-(1-methylethoxy)phenyl]ethanone



$C_{12}H_{16}O_4$ mol.wt. 224.26

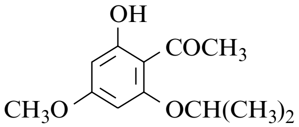
Synthesis

- Preparation by partial alkylation of 2,5-dihydroxy-3-methoxyacetophenone with isopropyl sulfate in alkaline medium (41%) [2848].

m.p. 70–72° [2848].

1-[2-Hydroxy-4-methoxy-6-(1-methylethoxy)phenyl]ethanone

[76554-78-4]



$C_{12}H_{16}O_4$ mol.wt. 224.26

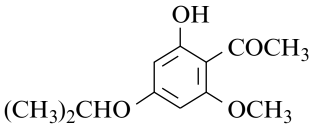
Syntheses

- Preparation by partial demethylation of 2,4-dimethoxy-6-isopropoxyacetophenone with aluminium bromide in acetonitrile at 0° (95%) [2747].
- Also refer to: [3272].

m.p. 65–66° [2747].

1-[2-Hydroxy-6-methoxy-4-(1-methylethoxy)phenyl]ethanone

[119136-15-1]



$C_{12}H_{16}O_4$ mol.wt. 224.26

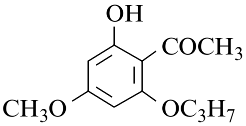
Synthesis

- Preparation by action of 2-bromopropane with 2,4-di-hydroxy-6-methoxyacetophenone in the presence of potassium carbonate in DMF for 2 h at 100–110° (87%) [2830].

m.p. 71–72° [2830]; ^{1}H NMR [2830], IR [2830], MS [2830].

1-(2-Hydroxy-4-methoxy-6-propoxyphenyl)ethanone

[76554-77-3] $C_{12}H_{16}O_4$ mol.wt. 224.26

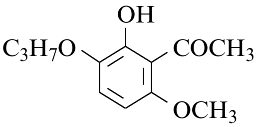


Synthesis

- Refer to: [3272].

1-(2-Hydroxy-6-methoxy-3-propoxyphenyl)ethanone

[126405-77-4] $C_{12}H_{16}O_4$ mol.wt. 224.26



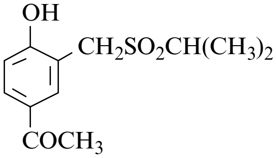
Synthesis

- Preparation by adding 2-hydroxy-3-iodo-6-methoxyacetophenone and cuprous iodide to a solution of sodium propoxide, previously prepared from propyl alcohol and sodium hydride in DMF [2524].

m.p. 90° [2524]; ^{1}H NMR [2524], IR [2524].

1-[4-Hydroxy-3-[[(1-methylethyl)sulfonyl]methyl]phenyl]ethanone

[56490-64-3] $C_{12}H_{16}O_4S$ mol.wt. 256.32



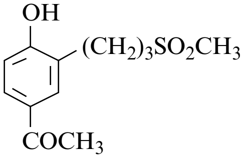
Synthesis

- Obtained by reaction of 3-chloromethyl-4-hydroxyacetophenone with magnesium isopropylsulfinate in refluxing dilute methanol for 18 h (83%) [2544].

m.p. 96°5–100° [2544].

1-[4-Hydroxy-3-[3-(methylsulfonyl)propyl]phenyl]ethanone

[56490-61-0] $C_{12}H_{16}O_4S$ mol.wt. 256.32



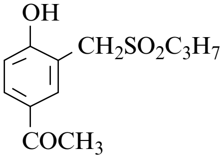
Synthesis

- Obtained by Fries rearrangement of 2-(methylsulfonyl-propyl)phenyl acetate with aluminium chloride in nitrobenzene, first at r.t. for 1 h, then at 50–60° for 1.5 h (48%) [2544].

m.p. 140–141° [2544].

1-[4-Hydroxy-3-[(propylsulfonyl)methyl]phenyl]ethanone

[56490-63-2] $C_{12}H_{16}O_4S$ mol.wt. 256.32

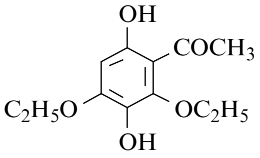


Synthesis

- Obtained by reaction of 3-chloromethyl-4-hydroxyacetophenone with magnesium propylsulfinate in refluxing aqueous methanol for 18 h (36%) [2544].

m.p. 73°5–76° [2544].

1-(2,4-Diethoxy-3,6-dihydroxyphenyl)ethanone



$C_{12}H_{16}O_5$ mol.wt. 240.26

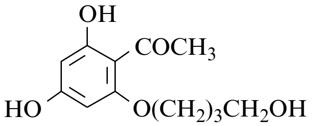
Synthesis

- Preparation from 2,4-diethoxy-6-hydroxyacetophenone by persulfate oxidation [2851,3394,3395], (Elbs reaction) (21%) [3394].

m.p. 130–131° [3394].

1-[2,4-Dihydroxy-6-(4-hydroxybutoxy)phenyl]ethanone

[121379-45-1]



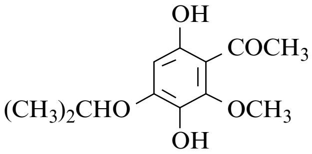
$C_{12}H_{16}O_5$ mol.wt. 240.26

Isolation from natural sources

- From the bulbs of *Dioscorea bulbifera* [3404].

1-[3,6-Dihydroxy-2-methoxy-4-(1-methylethoxy)phenyl]ethanone

[119136-16-2]



$C_{12}H_{16}O_5$ mol.wt. 240.26

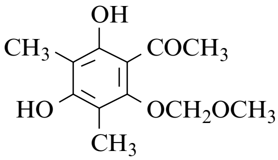
Synthesis

- Preparation from 4-(allyloxy)-2-hydroxy-6-methoxy-acetophenone by persulfate oxidation (Elbs reaction) (20%) [2830].

oil [2830]; ^{1}H NMR [2830], IR [2830], MS [2830].

1-[2,4-Dihydroxy-6-(methoxymethoxy)-3,5-dimethylphenyl]ethanone

[175465-97-1]



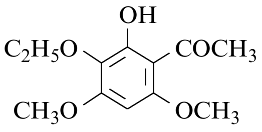
$C_{12}H_{16}O_5$ mol.wt. 240.26

Synthesis

- Refer to: [3405] (Japanese patent).

1-(3-Ethoxy-2-hydroxy-4,6-dimethoxyphenyl)ethanone

[89880-47-7]



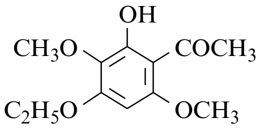
$C_{12}H_{16}O_5$ mol.wt. 240.26

Synthesis

- Obtained (by-product) by reaction of acetyl chloride with 1,2,3,5-tetramethoxybenzene in the presence of aluminium chloride in ethyl ether (9–12%) [3131,3302].

m.p. 74–75° [3131], 71°5–72°5 [3302];
^{1}H NMR [3131,3302], IR [3131,3302], MS [3131].

1-(4-Ethoxy-2-hydroxy-3,6-dimethoxyphenyl)ethanone



$C_{12}H_{16}O_5$ mol.wt. 240.26

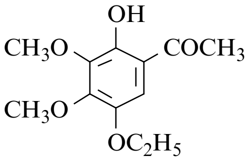
Synthesis

- Preparation by Friedel–Crafts acylation of 1,3-diethoxy-2,5-dimethoxybenzene with acetyl chloride in the presence of aluminium chloride in ether (81%) [3313].

m.p. 106–107° [3313].

1-(5-Ethoxy-2-hydroxy-3,4-dimethoxyphenyl)ethanone

[69616-62-2] $C_{12}H_{16}O_5$ mol.wt. 240.26

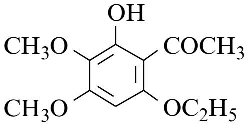


Synthesis

- Preparation by reaction of ethyl iodide with 2,5-dihydroxy-3,4-dimethoxyacetophenone in the presence of potassium carbonate in refluxing acetone (57%) [2818].

oil [2818]; b.p.$_{0.1}$ 120° [2818];
^{1}H NMR [2818], IR [2818].

1-(6-Ethoxy-2-hydroxy-3,4-dimethoxyphenyl)ethanone



$C_{12}H_{16}O_5$ mol.wt. 240.26

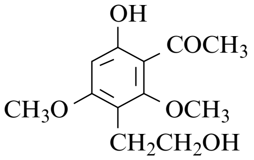
Synthesis

- Preparation by reaction of acetyl chloride on antiarol ethyl ether with aluminium chloride [3406].

m.p. 97–98° [3406].

1-[6-Hydroxy-3-(2-hydroxyethyl)-2,4-dimethoxyphenyl]ethanone

[34288-73-8] $C_{12}H_{16}O_5$ mol.wt. 240.26



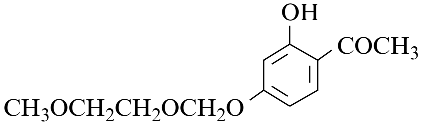
Synthesis

- Obtained from 6-(2′-hydroxyethyl)-5,7-dimethoxy-2-methylchromanone by degradation with potassium hydroxide in refluxing ethanol under nitrogen (32%) [3407].

m.p. 133–134° [3407]; ^{1}H NMR [3407], UV [3407].

1-[2-Hydroxy-4-[(2-methoxyethoxy)methoxy]phenyl]ethanone

[123253-31-6] $C_{12}H_{16}O_5$ mol.wt. 240.26



Syntheses

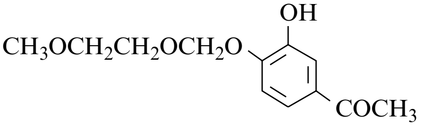
- Preparation by reaction of 2-methoxyethoxy-methyl chloride with resacetophenone,

- in the presence of N,N-diisopropylethylamine,
  in DMF at 0° for 1.5 h (83%) [3408] or
  in methylene chloride at r.t. for 20 h (48%) [3409];
- in the presence of potassium carbonate in acetone for 20 h at 20° (65–70%) [3107];
- also obtained in two steps: first, by adding sodium hydride (11 mmol) to a solution of resacetophenone (10 mmol) in DMF during 15–30 min at 20°; then, addition of methoxyethoxymethyl chloride (10 mmol) to the mixture between 0° and 5° (80–85%) [3107].

Pasty solid [3409]; TLC [3107];
^{1}H NMR [3107,3408,3409], ^{13}C NMR [3107,3408], IR [3107,3409], MS [3107,3408].

1-[3-Hydroxy-4-[(2-methoxyethoxy)methoxy]phenyl]ethanone

[101140-09-4] $C_{12}H_{16}O_5$ mol.wt. 240.26



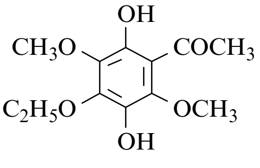
Synthesis

– Preparation by action of sodium methoxide with the crude 3-(benzoyloxy)-4-(2-methoxy-ethoxy) methoxyacetophenone (SM) in THF at r.t. in a nitrogen atmosphere for 30 min (92%). SM was previously prepared from 3-(benzoyloxy)-4-hydroxyacetophenone by etherification with 2-methoxyethoxymethyl chloride in the presence of N,N-diisopropylethylamine in methylene chloride at r.t. [3410].

colourless oil [3410]; ^{1}H NMR [3410], IR [3410], MS [3410].

1-(4-Ethoxy-2,5-dihydroxy-3,6-dimethoxyphenyl)ethanone

$C_{12}H_{16}O_6$ mol.wt. 256.26



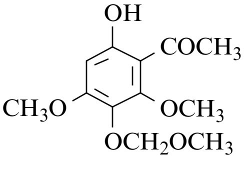
Synthesis

– Preparation from 2-hydroxy-4-ethoxy-3,6-dimethoxy-acetophenone by persulfate oxidation (Elbs reaction) (31%) [3313].

m.p. 73–74° [3313].

1-[6-Hydroxy-2,4-dimethoxy-3-(methoxymethoxy)phenyl]ethanone

[104481-00-7] $C_{12}H_{16}O_6$ mol.wt. 256.26

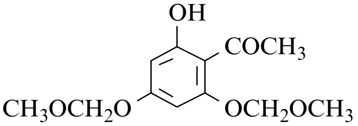


Synthesis

– Obtained from 3,6-dihydroxy-2,4-dimethoxyacetophenone by methoxymethylation with chloromethyl methyl ether in the presence of diisopropylamine in methylene chloride [3411,3412].

1-[2-Hydroxy-4,6-bis(methoxymethoxy)phenyl]ethanone

[65490-09-7] $C_{12}H_{16}O_6$ mol.wt. 256.26



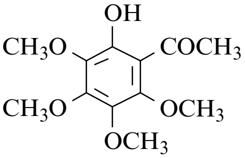
Syntheses

- Obtained by reaction of methoxymethyl chloride with phloroacetophenone in the presence of potassium carbonate,
  - in acetone at r.t. for 2.5 h (47%) [3106];
  - in refluxing acetone for 15 min (43%) [3413] or for 1 h (60%) [3294].
- Also refer to: [3414–3420].

m.p. 52° [3413], 45–46°5 [3106]; ^{1}H NMR [3106,3413], IR [3106,3413], UV [3413].

1-(2-Hydroxy-3,4,5,6-tetramethoxyphenyl)ethanone

[3162-28-5] $C_{12}H_{16}O_6$ mol.wt. 256.26



Syntheses

- Obtained by partial demethylation of 2,3,4,5,6-pentamethoxyacetophenone (**I**),
  - with boron trifluoride etherate in acetic acid at 80°. (**I**) was obtained by metallation of pentamethoxybenzene, followed by treatment of the intermediate aryllithium compound with acetic anhydride at r.t. (85% yield) [2564];
  - with aluminium chloride in ethyl ether at r.t. for 3 h (21%) [3318];
  - with aluminium chloride in acetonitrile for 6 h at 30° (75%) [2747].
- Also obtained by acylation of pentamethoxybenzene with acetyl chloride in the presence of aluminium chloride in ethyl ether [2770,2820,3323,3421], (25–34%) [2770,2820] or first for 14 h at r.t., then for 2 h at reflux (14%) [3323].
- Also obtained by adding a methanolic solution of 2,5-dihydroxy-3,4,6-trimethoxyacetophenone to an ethereal solution of diazomethane and keeping the mixture overnight in a refrigerator [3299,3307], (80%) [3299].
- Also refer to: [3422–3427].

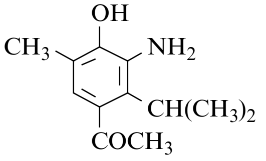
Isolation from natural sources

- By alkaline degradation of two substituted flavones with potassium hydroxide in boiling aqueous ethanol for 17–20 h under nitrogen,
  (a) From *Lucidin dimethyl ether* (5,6,7,8-tetramethoxy-3′,4′-methylenedioxy flavone) (m.p. 171–172°) (SM) (60% yield) [3428]. SM was obtained from two origins:
- Isolation from ground root of *Lindera lucida* (Lauraceae).
- Also prepared by methylation of *Lucidin* (5,7-dihydroxy-6,8-dimethoxy-3′,4′-methylenedioxy-flavone) (m.p. 255–257°), itself isolated from ground root of *Lindera lucida.*

(b) From 5,6,7,8-tetramethoxyflavone (m.p. 112–113°) (SM), [3429], (53% yield) [3428]. SM was also isolated from the above mentioned plant.

Yellow oil [3323,3421], light orange oil [3428], oil [2564], liquid [3299,3307]; b.p.$_2$ 115° [3299,3307], b.p.$_{0.2}$ 130° [3428], b.p.$_{14}$ 183° [2820], b.p.$_{15}$ 183° [3421]; ^{1}H NMR [3323,3429], IR [3323,3429], UV [3323,3428,3429].

1-[3-Amino-4-hydroxy-5-methyl-2-(1-methylethyl)phenyl]ethanone



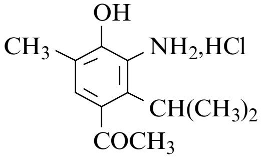
$C_{12}H_{17}NO_2$ mol.wt. 207.27

Syntheses

- Preparation by reduction of 4-hydroxy-5-methyl-3-nitro-2-isopropylacetophenone,
  - with sodium hydrosulfite in aqueous sodium hydroxide solution at 80–90° (92%) [3327];
  - with tin in dilute hydrochloric acid (46%) [3327].

m.p. 100° [3327].

1-[3-Amino-4-hydroxy-5-methyl-2-(1-methylethyl)phenyl]ethanone *(Hydrochloride)*



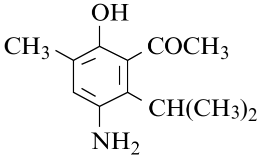
$C_{12}H_{17}NO_2$, HCl mol.wt. 243.73

Synthesis

- Preparation by reaction of hydrochloric acid on 3-amino-4-hydroxy-5-methyl-2-isopropylacetophenone in ethyl ether [3327].

m.p. 199–200° (d) [3327].

1-[3-Amino-6-hydroxy-5-methyl-2-(1-methylethyl)phenyl]ethanone



$C_{12}H_{17}NO_2$ mol.wt. 207.27

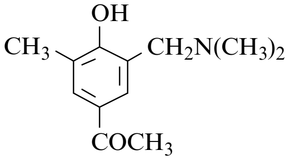
Syntheses

- Preparation by reduction of 2-hydroxy-3-methyl-5-nitro-6-isopropylacetophenone,
  - with sodium hydrosulfite in aqueous sodium hydroxide solution (87%) [3326];
  - with tin in dilute hydrochloric acid (50%) [3326].

m.p. 117° [3326].

1-[3-[(Dimethylamino)methyl]-4-hydroxy-5-methylphenyl]ethanone

[82506-14-7]



$C_{12}H_{17}NO_2$ mol.wt. 207.27

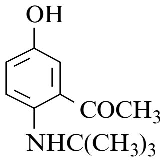
Synthesis

- Preparation by aminomethylation of 4-hydroxy-3-methyl-acetophenone with dimethylamine and formalin in water at 35–40° for 4 h (64%) [3217].

m.p. 44° [3217]; ^{1}H NMR [3217], IR [3217].

1-[2-[(1,1-Dimethylethyl)amino]-5-hydroxyphenyl]ethanone

[63609-62-1] $C_{12}H_{17}NO_2$ mol.wt. 207.27



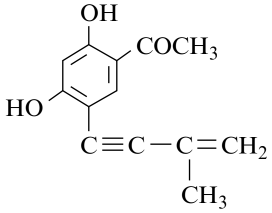
Synthesis

– Obtained by UV irradiation of a solution of 3-methyl-N-tert-butylanthranilium perchlorate (SM) in 10% aqueous acetonitrile (64%) [3430]. SM was prepared according to [3431].

yellow oil [3430]; TLC [3430];
^{1}H NMR [3430], ^{13}C NMR [3430], IR [3430], MS [3430].

1-[2,4-Dihydroxy-5-(3-methyl-3-buten-1-ynyl)phenyl]ethanone

[193333-25-4] $C_{13}H_{12}O_3$ mol.wt. 216.24



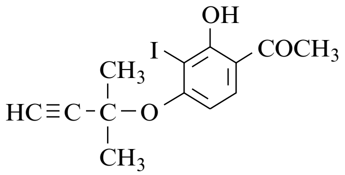
Synthesis

– Obtained (by-product) by reaction of boron tribromide (4 equiv.) with 2,4-bis(benzyloxy)-5-(3-hydroxy-3-methyl-butynyl)acetophenone in methylene chloride for 5 min at 0° (14%) [3432].

m.p. 110–112° [3432]; ^{1}H NMR [3432].

1-[4-[(1,1-Dimethyl-2-propynyl)oxy]-2-hydroxy-3-iodophenyl]ethanone

[82538-73-6] $C_{13}H_{13}IO_3$ mol.wt. 344.15



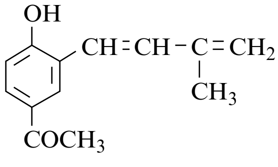
Synthesis

– Preparation by reaction of 3-chloro-3-methylbut-1-yne with 2,4-dihydroxy-3-iodoacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (65%) [2064].

m.p. 100–101° [2064]; ^{1}H NMR [2064].

1-[4-Hydroxy-3-(3-methyl-1,3-butadienyl)phenyl]ethanone

$C_{13}H_{14}O_2$ mol.wt. 202.10

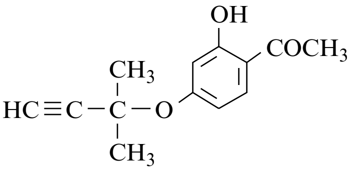


Isolation from natural sources

– From the roots of *Helianthella uniflora* (Heliantheae) [3433].

m.p. 137–138° [3433];
^{1}H NMR [3433], IR [3433], UV [3433], MS [3433].

1-[4-[(1,1-Dimethyl-2-propynyl)oxy]-2-hydroxyphenyl]ethanone



$C_{13}H_{14}O_3$ mol.wt. 218.25

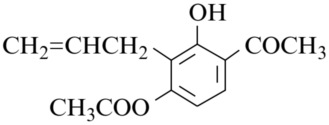
Synthesis

- Obtained by treatment of resacetophenone with 3-chloro-3-methylbutyne in DMF in the presence of potassium carbonate and potassium iodide for 12 h at 75° (29%) [3434].

m.p. 62°5 [3434]; ^{1}H NMR [3434], IR [3434].

1-[4-(Acetyloxy)-2-hydroxy-3-(2-propenyl)phenyl]ethanone

[149810-10-6]



$C_{13}H_{14}O_4$ mol.wt. 234.25

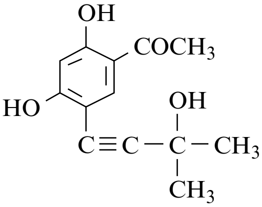
Synthesis

- Preparation by reaction of acetyl chloride with 3-allyl-2,4-di-hydroxyacetophenone in the presence of triethylamine in methylene chloride for 1 h at 0° and at 10° for 30 min (62%) [2891].

m.p. 56–57° [2891]; ^{1}H NMR [2891], ^{13}C NMR [2891], IR [2891].

1-[2,4-Dihydroxy-5-(3-hydroxy-3-methyl-1-butynyl)phenyl]ethanone

[193333-24-3]



$C_{13}H_{14}O_4$ mol.wt. 234.25

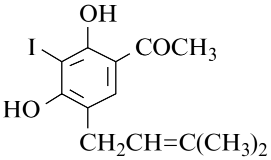
Synthesis

- Obtained (by-product) by reaction of boron tribromide (4 equiv.) with 2,4-bis(benzyloxy)-5-(3-hydroxy-3-methyl-butynyl)acetophenone in methylene chloride for 5 min at 0° (18%) [3432].

m.p. 148–150° [3432]; ^{1}H NMR [3432].

1-[2,4-Dihydroxy-3-iodo-5-(3-methyl-2-butenyl)phenyl]ethanone

[82538-74-7]



$C_{13}H_{15}IO_3$ mol.wt. 346.16

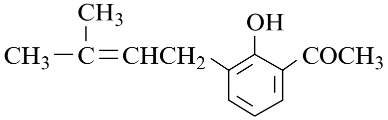
Synthesis

- Obtained by reaction of 2-methylbut-3-en-2-ol with 2,4-di-hydroxy-3-iodoacetophenone in the presence of boron trifluoride etherate in dioxane at 35–40° (15%) [2064].

m.p. 135–137° [2064]; ^{1}H NMR [2064].

1-[2-Hydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[310402-63-2] $C_{13}H_{16}O_2$ mol.wt. 204.27

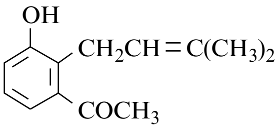


Synthesis

– Refer to: [3435] (compound **76**).

1-[3-Hydroxy-2-(3-methyl-2-butenyl)phenyl]ethanone

[154520-54-4] $C_{13}H_{16}O_2$ mol.wt. 204.27

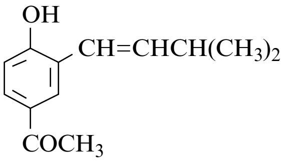


Isolation from natural sources

– From the aerial parts of *Helichrysum stoechas* (L.) grown in Libya [3436].

1-[4-Hydroxy-3-(3-methyl-1-butenyl)phenyl]ethanone

[35816-89-8] $C_{13}H_{16}O_2$ mol.wt. 204.27



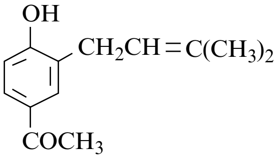
Synthesis

– Preparation by migration of a C=C double bond in 4-hydroxy-3-(2-isopentenyl) acetophenone by treatment with potassium hydroxide in triethylene glycol (Triglykol) at 150° (70%) [3437] (allylic/propenylic rearrangement).

b.p.$_1$ 120° [3437]; ^{1}H NMR [3437].

1-[4-Hydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[26932-05-8] $C_{13}H_{16}O_2$ mol.wt. 204.27



Syntheses

– Obtained by reaction of 2-methyl-3-buten-2-ol with 4-hydroxyacetophenone in the presence of boron trifluoride etherate [2060,3438], (15%) [3438].

– Preparation by thermal Claisen rearrangement of 4-(2,2-di-methylallyloxy)acetophenone in diethylaniline at 165° (79%) [3437].

Isolation from natural sources

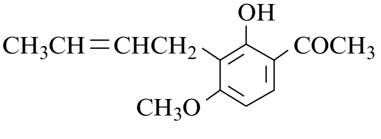
- From the aerial parts of *Ophryosporus chilca* (Compositae, tribe Eupatorieae) [3439].
- From the roots of *Flourensia cernua* DC (Compositae) [3440].
- From *Artemis campestris* silbsp. glutinosa (Compositae) [3441].
- From *Senecio phylolleptus* Cuatr., *Senecio viridis* Phil. and *Senecio nutans* sch. Bip. (Asteraceae) native of northern Chile [3442].
- From *Senecio nutans* sch. Bip. [3443].

- From the aerial parts of *Stevia hyssopifolia* Phil. var. hyssopifolia [3444].
- From the aerial parts of *Baccharis santelicis* Phil. (Compositae, tribe Cistereae, subtribe Baccharidinae) [3433].
- From sliced yacon tubers after inoculation with the bacterium *Pseudomonas cichorii* and incubation at 20° for 3 days, then extraction with acetone. Yacon (*Polymnia sonchifolia*) (Compositae) is cultivated in South America and has recently been introduced into Japan [3445].
- From the aerial parts of *Helichrysum italicum* (Compositae) (major compound) [3446].
- From the aerial parts of *Helichrysum stoechas* (Compositae) [3447] (trace amounts) [3446].
- From the aerial parts of *Werneria poposa* [3448].
- From the roots of Helianthella uniflora (Compositae) [3433,3449].
- From the leaves of Ageratina altissima (L) K & K (Compositae) [3450].
- Also refer to: [3451].

Amorphous [3445]; TLC [3439];
m.p. 94–95° [3438], 93–94° [3433,3437], 92–93° [3447], 90–91° [3442], 90° [3441];
^{1}H NMR [3433,3438,3441,3442,3445–3448],
^{13}C NMR [3442,3445,3446,3448], IR [3433,3438,3441,3442,3445–3447],
UV [3433,3438,3441,3445–3447],
MS [3433,3441,3442,3445–3448].

1-[3-(2-Butenyl)-2-hydroxy-4-methoxyphenyl]ethanone

[91664-24-3] $C_{13}H_{16}O_3$ mol.wt. 220.27

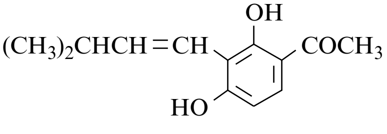


Synthesis

N.B.: In the paper [3325], the formula of the compound **13** displayed page 131, which is the formula of the titled compound $C_{13}H_{16}O_3$, is incomplete and erroneous. It actually deals with another compound $C_{14}H_{18}O_4$, the 1-[3-(2-butenyl)-2-hydroxy-4,6-dimethoxyphenyl]ethanone, which is described in the experimental part. This is detailed just below the formula, page 131.

1-[2,4-Dihydroxy-3-(3-methyl-1-butenyl)phenyl]ethanone

[80190-95-0] $C_{13}H_{16}O_3$ mol.wt. 220.27

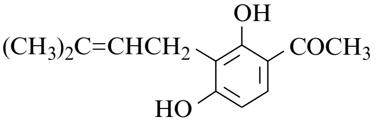


Syntheses

- Obtained by reaction of 2-methylbut-3-en-2-ol with resacetophenone in the presence of boron trifluoride etherate [3452] according to the method [3453].
- Also refer to: [3454] (Chinese paper).

1-[2,4-Dihydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[19825-40-2]



$C_{13}H_{16}O_3$ mol.wt. 220.27

Syntheses

- Preparation by reaction of prenyl bromide on resacetophenone with potassium hydroxide solution at r.t. [3455–3457], (21–25%) [3455,3456].
- Preparation by reaction of 2-methylbut-3-en-2-ol with resacetophenone in the presence of boron trifluoride etherate in dioxane at r.t. [2326,3453,3458], (33%) [3458], (13%) [3453].
- Isoprenylation of resacetophenone with prenyl bromide by photochemical method in the presence of benzoyl peroxide in dry benzene for 8 h (40%) [3455].
- Preparation from 6-acetyl-3-phenylthio-2,2-dimethylchroman-5-ol,
  - by reaction with potassium naphthalenide in tetrahydrofuran at r.t. (59%) [3459] or by reaction with lithium naphthalenide in tetrahydrofuran at –32° for 30 min (38%) [3459];
  - by electrolysis using a mercury cathode, and acetonitrile-tetraethylammonium bromide electrolyte (49%) [3459].
- Also obtained by reaction of potassium naphthalenide with 6-acetyl-2,2-dimethyl-3-phenyl-sulfonylchroman-5-ol in tetrahydrofuran at r.t. (25%) [3459].

Isolation from natural sources

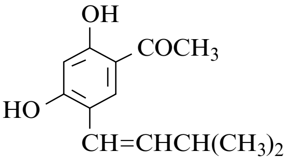
- By cleavage of isobavachin with alkali. The isobavachin is a flavonoid compound obtained from *Psoralea Corylifolia* Linn. [3460].

m.p. 162° [3457], 157–158° [3456], 155–156° [3453,3460], 149–151° [3459], 148° [3458];

^{1}H NMR [2326,2327,3457–3460], IR [3456–3458,3460], UV [2326,3456], MS [3459].

1-[2,4-Dihydroxy-5-(3-methyl-1-butenyl)phenyl]ethanone

[41347-54-0]



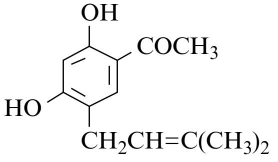
$C_{13}H_{16}O_3$ mol.wt. 220.27

Synthesis

- Obtained by reaction of 2-methylbut-3-en-2-ol with resacetophenone in the presence of boron trifluoride etherate [3452] according to the method [3453].

1-[2,4-Dihydroxy-5-(3-methyl-2-butenyl)phenyl]ethanone

[28437-37-8] $C_{13}H_{16}O_3$ mol.wt. 220.27



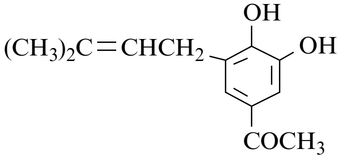
Syntheses

- Preparation by reaction of 2-methylbut-3-en-2-ol with resacetophenone in the presence of boron trifluoride etherate [2326,3453,3458], (20–29%) [3437,3453].
- Preparation from 2,4-dihydroxy-3-iodo-5-prenyl-acetophenone by elimination of iodine with zinc dust and concentrated hydrochloric acid in refluxing ethanol (79%) [2064].
- Preparation by reaction of prenyl bromide with resacetophenone in potassium hydroxide solution at r.t. [3457].

m.p. 146° [3457], 144–145° [2064,3453]; ^{1}H NMR [2326,2327,3457], IR [3457], UV [2326].

1-[3,4-Dihydroxy-5-(3-methyl-2-butenyl)phenyl]ethanone

[186966-70-1] $C_{13}H_{16}O_3$ mol.wt. 220.27



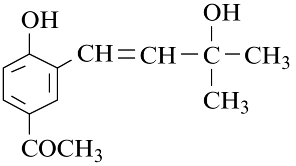
Synthesis

- Obtained (poor yield) by rearrangement of 4-(dimethyl-allyloxy)-3-hydroxyacetophenone in the presence of montmorillonite KSF (<10%) [3461].

1-[4-Hydroxy-3-(3-hydroxy-3-methyl-1-butenyl)phenyl]ethanone

[35816-94-5]
[26931-61-3] *(E)*

$C_{13}H_{16}O_3$ mol.wt. 220.27



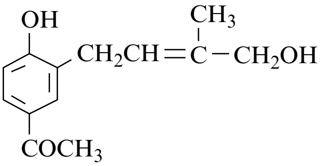
Isolation from natural sources

- From the aerial parts of *Ophryosporus floribundus* (Compositae, tribe Eupatorieae) [3462].
- From the leaves and the roots of *Ageratina altissima* (L.) K. et R. (Compositae) [3450].
- From the roots of *Helianthella uniflora* (tribe Heliantheae) [3433].

^{1}H NMR [3433], IR [3433], UV [3433], MS [3433].

1-[4-Hydroxy-3-(4-hydroxy-3-methyl-2-butenyl)phenyl]ethanone

[68034-24-2] $C_{13}H_{16}O_3$ mol.wt. 220.27

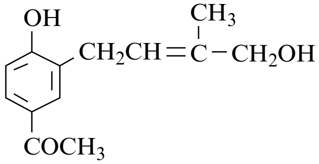


Isolation from natural sources

- From the aerial parts of *Artemisia campestris*, L., subsp. glutinosa (Gay ex Besser), Batt (Compositae) [3463].

1-[4-Hydroxy-3-(4-hydroxy-3-methyl-2-butenyl)phenyl]ethanone *(E)*

[73869-86-0] $C_{13}H_{16}O_3$ mol.wt. 220.27



Synthesis

- Obtained by alkaline hydrolysis of 3-[4-acetoxyisopent-2(*E*)-enyl]-4-hydroxyacetophenone (SM) (viscous oil) with 10% potassium hydroxide in methanol (quantitative yield) [3464]. SM was isolated from the *Artemisia campestris* L., subsp. glutinosa (Gay ex Besser), Batt.

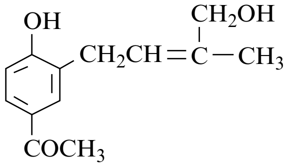
Isolation from natural sources

- From the aerial parts of *Artemisia campestris* L., subsp. glutinosa (Gay ex Besser), Batt (Compositae), (1.1%) [3465], (11.2%) [3441].
- From *Artemisia monosperma* [3466].

m.p. 84–85° [3465];
^{1}H NMR [3441,3465], IR [3465], UV [3465], MS [3441,3465].

1-[4-Hydroxy-3-(4-hydroxy-3-methyl-2-butenyl)phenyl]ethanone *(Z)*

[123614-13-1] $C_{13}H_{16}O_3$ mol.wt. 220.27



Synthesis

- Obtained by alkaline hydrolysis of 3-[4-acetoxyisopent-2(*Z*)-enyl]-4-hydroxyacetophenone (SM) (viscous oil) with 10% potassium hydroxide in methanol (quantitative yield) [3464]. SM was isolated from the *Artemisia campestris* L., subsp. glutinosa (Gay ex Besser), Batt.

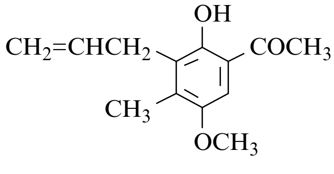
Isolation from natural sources

- From the aerial parts of *Artemisia campestris* L., subsp. glutinosa (Gay ex Besser), Batt (Compositae) [3463], (44.8%) [3441].
- From *Artemisia monosperma* [3466].

Viscid oil [3441]; ^{1}H NMR [3441], IR [3441], UV [3441], MS [3441].

1-[2-Hydroxy-5-methoxy-4-methyl-3-(2-propenyl)phenyl]ethanone

[43037-66-7] $C_{13}H_{16}O_3$ mol.wt. 220.27

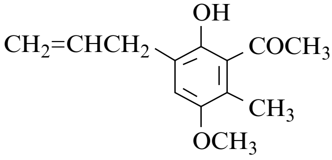


Synthesis

- Preparation by thermal Claisen rearrangement of 2-(allyloxy)-5-methoxy-4-methylacetophenone in N,N-dimethylaniline at 170° (42%) [3467].

1-[2-Hydroxy-5-methoxy-6-methyl-3-(2-propenyl)phenyl]ethanone

[43037-68-9]



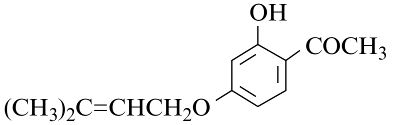
$C_{13}H_{16}O_3$ mol.wt. 220.27

Synthesis

- Obtained by thermal Claisen rearrangement of 2-(allyloxy)-5-methoxy-4-methylacetophenone in N,N-dimethylaniline at 170°, with a [2368,,3466] shift of the aromatic acetyl substituent (22%) [3467].

1-[2-Hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]ethanone

[24672-83-1]



$C_{13}H_{16}O_3$ mol.wt. 220.27

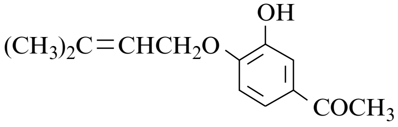
Syntheses

- Preparation by reaction of prenyl bromide on resacetophenone [3468],
  - in the presence of potassium carbonate in refluxing acetone [2326,3455,3456,3469], (69%) [3456], (40%) [3455];
  - in the presence of potassium hydroxide in methanol, at 0° (14%) or at r.t. (4%) [3456];
  - by photochemical method in the presence of benzoyl peroxide in dry benzene for 8 h (10%) [3455].

m.p. 46–47° [3456]; ^{1}H NMR [2326,2327,3456], IR [3456], UV [2326,3456].

1-[3-Hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]ethanone

[186966-69-8]



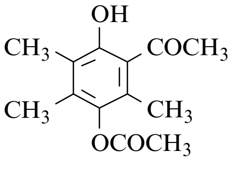
$C_{13}H_{16}O_3$ mol.wt. 220.27

Synthesis

- Preparation by reaction of 3,3-dimethylallyl chloride (2.5 equiv.) with 4-acetylcatechol in the presence of sodium carbonate and catalytic amounts of TBAI in DMF at r.t. (73%) [3461].

1-[3-(Acetyloxy)-6-hydroxy-2,4,5-trimethylphenyl]ethanone

[66901-79-9]



$C_{13}H_{16}O_4$ mol.wt. 236.27

Syntheses

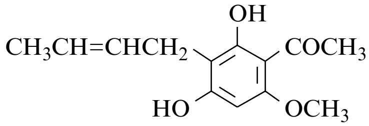
- Preparation by reaction of boron trifluoride–acetic acid complex,
  - with trimethylhydroquinone [2994,3249], (90%) [3249];
  - with trimethylhydroquinone diacetate in ethylene dichloride *via* a Fries rearrangement (99%) [3250].
- Also refer to: [3251].

m.p. 75° [2994]; ^{13}C NMR [3250].

1-[3-(2-Butenyl)-2,4-dihydroxy-6-methoxyphenyl]ethanone

[91664-22-1]

$C_{13}H_{16}O_4$ mol.wt. 236.27



Synthesis

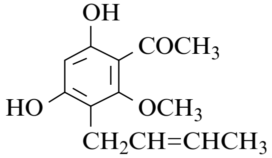
- Preparation by condensation of 2,4-dihydroxy-6-methoxyacetophenone with 1, 3-butadiene in the presence of 85% orthophosphoric acid in xylene at 30–35° (37%) [3325].

m.p. 134–135° [3325]; ^{1}H NMR [3325].

1-[3-(2-Butenyl)-4,6-dihydroxy-2-methoxyphenyl]ethanone

[91664-23-2]

$C_{13}H_{16}O_4$ mol.wt. 236.27



Synthesis

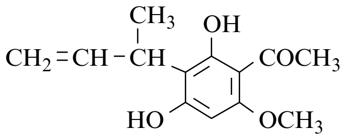
- Preparation by condensation of 2,4-dihydroxy-6-methoxy-acetophenone with 1,3-butadiene in the presence of 85% orthophosphoric acid in xylene at 30–35° (41%) [3325].

m.p. 128–129° [3325]; ^{1}H NMR [3325].

1-[2,4-Dihydroxy-6-methoxy-3-(1-methyl-2-propenyl)phenyl]ethanone

[91664-20-9]

$C_{13}H_{16}O_4$ mol.wt. 236.27



Synthesis

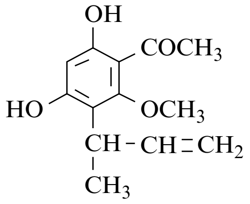
- Obtained, or else its isomer (see below), (by-product) by condensation of 2,4-dihydroxy-6-methoxyacetophenone with 1,3-butadiene in the presence of 85% orthophosphoric acid in xylene at 30–35° (7%) [3325].

m.p. 130–140° [3325]; ^{1}H NMR [3325].

1-[4,6-Dihydroxy-2-methoxy-3-(1-methyl-2-propenyl)phenyl]ethanone

[91664-21-0]

$C_{13}H_{16}O_4$ mol.wt. 236.27



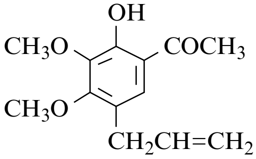
Synthesis

- Obtained, or else its isomer (see above), (by-product) by condensation of 2,4-dihydroxy-6-methoxyacetophenone with 1,3-butadiene in the presence of 85% orthophosphoric acid in xylene at 30–35° (7%) [3325].

m.p. 130–140° [3325]; ^{1}H NMR [3325].

1-[2-Hydroxy-3,4-dimethoxy-5-(2-propenyl)phenyl]ethanone

[75254-93-2] $C_{13}H_{16}O_4$ mol.wt. 236.27

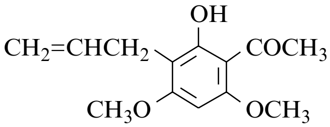


Synthesis

- Preparation by thermal Claisen rearrangement of 2-(allyloxy)-3,4-dimethoxyacetophenone without solvent at 190–200° [3470].

1-[2-Hydroxy-4,6-dimethoxy-3-(2-propenyl)phenyl]ethanone

[35109-98-9] $C_{13}H_{16}O_4$ mol.wt. 236.27



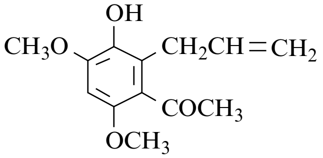
Synthesis

- Preparation by thermal Claisen rearrangement of 2-(allyloxy)-4,6-dimethoxyacetophenone in refluxing N,N-dimethylaniline (90%) [3078].

m.p. 85–87° [3078]; ^{1}H NMR [3078], IR [3078].

1-[3-Hydroxy-4,6-dimethoxy-2-(2-propenyl)phenyl]ethanone

[100612-87-1] $C_{13}H_{16}O_4$ mol.wt. 236.27



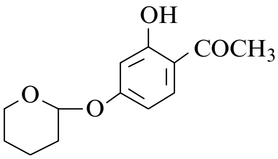
Syntheses

- Preparation by thermal Claisen rearrangement of 5-(allyloxy)-2,4-dimethoxyacetophenone,
  - in boiling quinoline (65%) [2849];
  - in glycerol at 200° (87%) [2849];
  - by pyrolysis at 160° for 2 h under nitrogen [3471].
- Preparation by reaction of methyl iodide on 2-allyl-3,6-dihydroxy-4-methoxyacetophenone with potassium carbonate in acetone (41%) [2849].

m.p. 110° [2849].

1-[2-Hydroxy-4-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone

[111841-07-7] $C_{13}H_{16}O_4$ mol.wt. 236.27



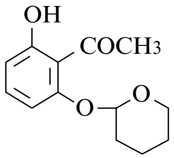
Syntheses

- Obtained by reaction of 2,3-dihydropyran with resaceto-phenone in the presence of concentrated hydrochloric acid (some drops) at r.t. overnight (33%) [3472]. In later runs, p-toluenesulfonic acid was used as catalyst.
- Also refer to: [3473].

m.p. 76–78° [3472].

1-[2-Hydroxy-6-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone

[63854-17-1] $C_{13}H_{16}O_4$ mol.wt. 236.27



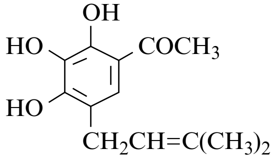
Syntheses

- Obtained by reaction of 2,3-dihydropyran with 2,6-dihydroxyacetophenone in the presence of p-toluenesulfonic acid in dioxane at r.t. for 3 h (64%) [3474] or for 20 h (69%) [2368].
- Also refer to: [3473].

Pale yellow crystals [3474];
^{1}H NMR [2368,3474], ^{13}C NMR [2368,3474], MS [3474].

1-[2,3,4-Trihydroxy-5-(3-methyl-2-butenyl)phenyl]ethanone

[35817-18-6] $C_{13}H_{16}O_4$ mol.wt. 236.27



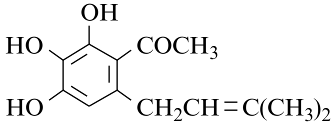
Synthesis

- Obtained by reaction of 2-methyl-3-buten-2-ol with gallacetophenone in the presence of boron trifluoride etherate in dioxane [3437,3438] at 50–60° (21%) [3438].

m.p. 74–75° [3438]; ^{1}H NMR [3438], IR [3438], UV [3438].

1-[2,3,4-Trihydroxy-6-(3-methyl-2-butenyl)phenyl]ethanone

[149876-26-6] $C_{13}H_{16}O_4$ mol.wt. 236.27

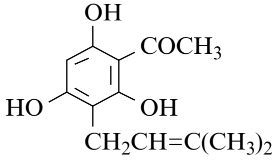


Synthesis

- **N.B.**: The titled ketone is mentioned in Chem. Abstr. Vol. **119**, 1993-FORMULA INDEX, p. 1525F under the reference 172870a. However, the original publication [3475] obtained from this reference does not include the expected ketone. This publication [3475] concerns only an isomeric ketone, the 1-[2,3,4-tri-hydroxy-**5**-(3-methyl-2-butenyl)phenyl]ethanone [35817-18-6] already described [2018], p. 240.

1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[27364-71-2] $C_{13}H_{16}O_4$ mol.wt. 236.27



Syntheses

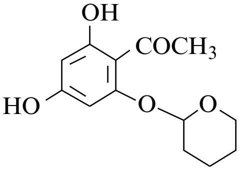
- Preparation from 2,4-dihydroxy-6-methoxy-5-iso-pentenylacetophenone *(Acronylin)* by demethylation with aluminium chloride in refluxing benzene [3476].
- Also obtained by reaction of 2-methylbut-3-en-2-ol with phloroacetophenone in the presence of boron trifluoride etherate in dioxane at 20° (10%) [3477].

- Also obtained by reaction of prenyl bromide with phloroacetophenone in solution of methanol in the presence of potassium hydroxide (26%) [3117] or sodium methoxide at r.t. (5%) [2878].

m.p. 182° [3476], 172° [2878,3117], 171–173° [3477], 169–171° [2879]; ^{1}H NMR [2879,3476], UV [3477].

1-[2,4-Dihydroxy-6-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone

[136257-86-8] $C_{13}H_{16}O_5$ mol.wt. 252.27



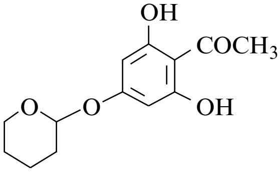
Syntheses

- Preparation by reaction of 3,4-dihydro-2*H*-pyran,
  - on phloroacetophenone with p-toluenesulfonic acid in dioxane at r.t. (10%) [2368];
  - on 4-acetoxy-2,6-dihydroxyacetophenone with p-toluenesulfonic acid in dioxane (32–49%) [2368].

White solid [2368]; ^{1}H NMR [2368], ^{13}C NMR [2368].

1-[2,6-Dihydroxy-4-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone

[136257-85-7] $C_{13}H_{16}O_5$ mol.wt. 252.27



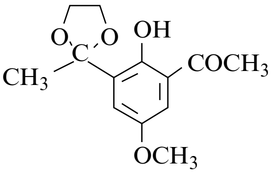
Syntheses

- Obtained by reaction of 3,4-dihydro-2*H*-pyran,
  - on phloroacetophenone with p-toluenesulfonic acid in dioxane at r.t. (20%) [2368];
  - on 4-acetoxy-2,6-dihydroxyacetophenone with p-toluene sulfonic acid in dioxane (8–11%) [2368].

White solid [2368]; ^{1}H NMR [2368], ^{13}C NMR [2368].

1-[2-Hydroxy-5-methoxy-3-(2-methyl-1,3-dioxolan-2-yl)phenyl]ethanone

[103867-85-2] $C_{13}H_{16}O_5$ mol.wt. 252.27

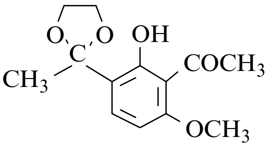


Synthesis

- Preparation by UV light irradiation of ethylene acetal of 2-acetoxy-5-methoxyacetophenone with potassium carbonate in hexane (85%) [3334].

1-[2-Hydroxy-6-methoxy-3-(2-methyl-1,3-dioxolan-2-yl)phenyl]ethanone

[103867-86-3] $C_{13}H_{16}O_5$ mol.wt. 252.27

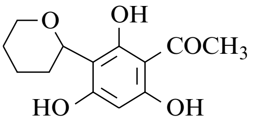


Synthesis

- Preparation by UV light irradiation of ethylene acetal of 2-acetoxy-4-methoxyacetophenone with potassium carbonate in hexane (52%) [3334].

m.p. 120–123° [3334]; ^{1}H NMR [3334], IR [3334].

1-[2,4,6-Trihydroxy-3-(tetrahydro-2*H*-pyran-2-yl)phenyl]ethanone



$C_{13}H_{16}O_5$ mol.wt. 252.27

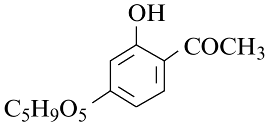
Synthesis

- Obtained by reaction of 3,4-dihydro-2*H*-pyran on phloroacetophenone with p-toluenesulfonic acid in dioxane at r.t. (21%) [2368].

^1H NMR [2368], ^{13}C NMR [2368].

1-[2-Hydroxy-4-(β-D-xylopyranosyloxy)phenyl]ethanone

[54918-29-5]



$C_{13}H_{16}O_7$ mol.wt. 284.14

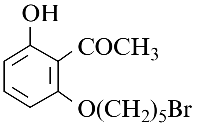
Synthesis

- Preparation by reaction of sodium on 2′,4′-dih-droxy-acetophenone-4′-β-tri-O-acetyl-D-xylopyranoside in methanol (66%) [1936].

m.p. 204–205° [1936];
$(\alpha)_D^{17} = -70°$ (0.4%, THF) [1936].

1-[2-[(5-Bromopentyl)oxy]-6-hydroxyphenyl]ethanone

[28862-10-4]



$C_{13}H_{17}BrO_3$ mol.wt. 301.18

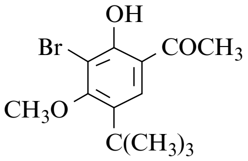
Synthesis

- Preparation by reaction of 1,5-dibromopentane with 2,6-di-hydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone [2270].

m.p. 35–36° [2270].

1-[3-Bromo-5-(1,1-dimethylethyl)-2-hydroxy-4-methoxyphenyl]ethanone

[127371-47-5]



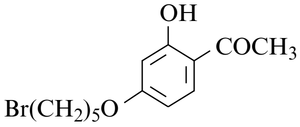
$C_{13}H_{17}BrO_3$ mol.wt. 301.18

Synthesis

- Refer to: [3478] (patent).

1-[4-[(5-Bromopentyl)oxy]-2-hydroxyphenyl]ethanone

[40785-72-6]



$C_{13}H_{17}BrO_3$ mol.wt. 301.18

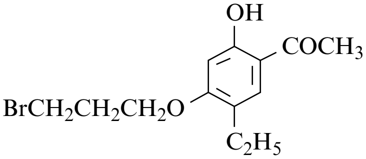
Synthesis

- Preparation by reaction of 1,5-dibromopentane with resacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (34%) [2270].

m.p. 64–66° [2270].

1-[4-(3-Bromopropoxy)-5-ethyl-2-hydroxyphenyl]ethanone

[117706-55-5]



$C_{13}H_{17}BrO_3$ mol.wt. 301.18

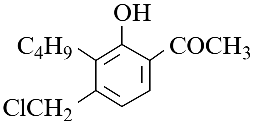
Synthesis

- Preparation by reaction of 3-bromopropyl bromide on 5-ethyl-2,4-dihydroxyacetophenone with potassium carbonate and potassium iodide, at reflux (80%) [2678,2679].

m.p. 126–127° [2678,2679]; ^{1}H NMR [2678,2679].

1-[3-Butyl-4-(chloromethyl)-2-hydroxyphenyl]ethanone

[107223-43-8]



$C_{13}H_{17}ClO_2$ mol.wt. 240.73

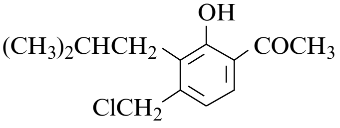
Synthesis

- Preparation by reaction of ethyl chloroformate with 3-n-butyl-4-(dimethylaminomethyl)-2-hydroxyacetophenone in toluene (78%) [2550].

m.p. 42–44° [2550].

1-[4-(Chloromethyl)-2-hydroxy-3-(2-methylpropyl)phenyl]ethanone

[97582-41-7]



$C_{13}H_{17}ClO_2$ mol.wt. 240.73

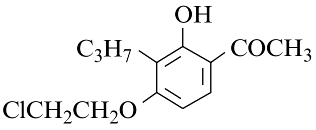
Synthesis

- Preparation by reaction of ethyl chloroformate with 3-iso-butyl-4-(dimethylaminomethyl)-2-hydroxyacetophenone in toluene [2550,2927], (42%) [2550].

Oil [2550]; ^{1}H NMR [2927], IR [2927].

1-[4-(2-Chloroethoxy)-2-hydroxy-3-propylphenyl]ethanone

[104074-07-9]



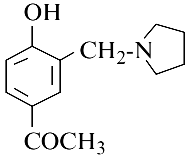
$C_{13}H_{17}ClO_3$ mol.wt. 256.73

Synthesis

- Preparation by action of 2-chloroethyl p-toluenesulfonate with 2,4-dihydroxy-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in boiling acetone for 16 h (36%) [3479].

m.p. 72–73°5 [3479]; ^{1}H NMR [3479].

1-[4-Hydroxy-3-(1-pyrrolidinylmethyl)phenyl]ethanone



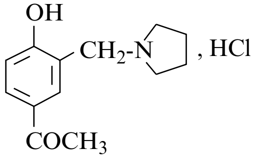
$C_{13}H_{17}NO_2$ mol.wt. 219.28

Synthesis

- Preparation by reaction of 4-hydroxyacetophenone with formaldehyde and pyrrolidine in 75% ethanol at r.t. (60%) [3480] (Mannich reaction).

m.p. 97–98° [3480].

1-[4-Hydroxy-3-(1-pyrrolidinylmethyl)phenyl]ethanone *(Hydrochloride)*



$C_{13}H_{17}NO_2$, HCl mol.wt. 255.74

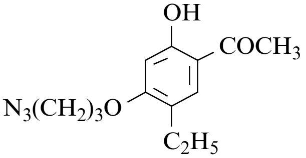
Synthesis

- Preparation from 4-hydroxy-3-(1-pyrrolidinylmethyl)-acetophenone [3480].

m.p. 202–203° [3480].

1-[4-(3-Azidopropoxy)-5-ethyl-2-hydroxyphenyl]ethanone

[117706-26-0]



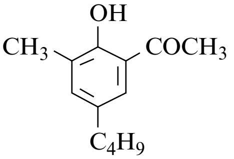
$C_{13}H_{17}N_3O_3$ mol.wt. 263.30

Synthesis

- Preparation by reaction of sodium azide with 4-(3-bromo-propoxy)-5-ethyl-2-hydroxyacetophenone in DMF at r.t. [2678,2679].

^{1}H NMR [2678,2679], MS [2678,2679].

1-(5-Butyl-2-hydroxy-3-methylphenyl)ethanone



$C_{13}H_{18}O_2$ mol.wt. 206.28

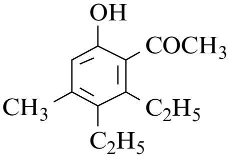
Synthesis

- Preparation by Fries rearrangement of 4-butyl-2-methyl-phenyl acetate with aluminium chloride without solvent at 100–110° (64%) [2957].

b.p.$_{12}$ 152–154° [2957].

1-(2,3-Diethyl-6-hydroxy-4-methylphenyl)ethanone

[27193-00-6]



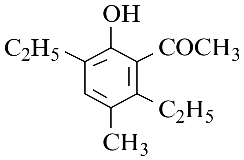
$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

- Obtained via pyrolysis of 7,8-diethyl-1,3-dimethyl-2-oxabicyclo [4.2.0] octa-3,7-dien-5-one (2,6-dimethyl-4-pyrone - Hexyne-3 - Adduct) in refluxing o-dichloro-benzene [3237].

IR [3237], UV [3237], MS [3237].

1-(2,5-Diethyl-6-hydroxy-3-methylphenyl)ethanone

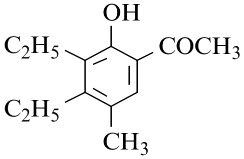


$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

- Obtained (small amounts) by Fries rearrangement of 2,5-diethyl-4-methylphenyl acetate with aluminium chloride without solvent at 130° [2956].

1-(3,4-Diethyl-2-hydroxy-5-methylphenyl)ethanone



$C_{13}H_{18}O_2$ mol.wt. 206.28

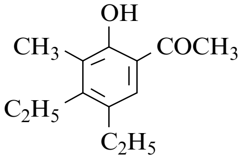
Syntheses

- Obtained by heating some esters with aluminium chloride. There is simultaneously displacement and rearrangement of alkyl groups during the Fries reaction,

- 2,4-diethyl-5-methylphenyl acetate (65%) [2233];
- 2,5-diethyl-4-methylphenyl acetate (at 130°) (60%) [2956];
- 2,6-diethyl-4-methylphenyl acetate (60%) [2233].

b.p.$_{12}$ 136–137° [2233], b.p.$_{11}$ 143–145° [2233], b.p.$_{15}$ 154–162° [2956].

1-(4,5-Diethyl-2-hydroxy-3-methylphenyl)ethanone



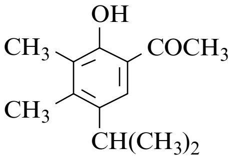
$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

- Obtained by heating 2,4-diethyl-6-methylphenyl acetate with aluminium chloride at high temperature. In this reaction, a migration of an ethyl group occurs [2233].

m.p. 50–51° [2233].

1-[3,4-Dimethyl-2-hydroxy-5-(1-methylethyl)phenyl]ethanone



$C_{13}H_{18}O_2$ mol.wt. 206.28

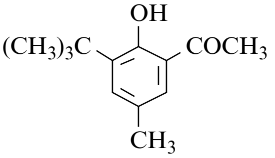
Synthesis

- Preparation by Fries rearrangement of 2,3-dimethyl-4-iso-propylphenyl acetate with aluminium chloride at 140–150° (37%) [3373].

m.p. 132° [3373]; b.p.$_{22}$ 176–180° [3373].

1-[3-(1,1-Dimethylethyl)-2-hydroxy-5-methylphenyl]ethanone

[14813-18-4] $C_{13}H_{18}O_2$ mol.wt. 206.28



Syntheses

- Preparation by Fries rearrangement of 2-tert-butyl-4-methyl-phenyl acetate in the presence of aluminium chloride in cyclohexane at <105° (47%) [3481].
- The reaction of acetyl chloride with a pentane solution of $[AlCH_3(dbmp)_2]$ leads to acylation of one of the (dbmp) ligands and affords $[AlCH_3(dbmp)(bhmap)]$. Hydrolysis of this aluminium complex with a saturated aqueous solution of ammonium chloride gives the ketone attempted (65%) [3482].

N.B.: Hdbmp = 2,6-di-tert-butyl-4-methylphenol and Hbhmap = 3-tert-butyl-2-hydroxy-5-methylacetophenone.

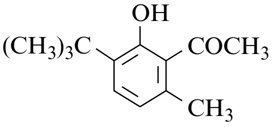
- Also obtained by reaction of p-tert-butyl alcohol with 2-hydroxy-5-methylacetophenone in concentrated sulfuric acid at r.t. for 24 h (49%) [3483].
- Also refer to: [3484].

m.p. 97–98° [3481], 58° [3482]. One of the reported melting points is obviously wrong.

X-ray crystallography [3482]; GC/MS [3483];
^{1}H NMR [3482,3483], ^{13}C NMR [3482], IR [3482], MS [3482].

1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methylphenyl]ethanone

$C_{13}H_{18}O_2$ mol.wt. 206.28



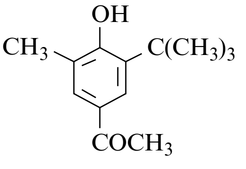
Synthesis

- Claimed to be prepared by Fries rearrangement of 2-tert-butyl-5-methylphenyl acetate with aluminium chloride, without solvent, at 110° (76%) [3485].

b.p.$_8$ 92° [3485].

1-[3-(1,1-Dimethylethyl)-4-hydroxy-5-methylphenyl]ethanone

[18606-50-3] $C_{13}H_{18}O_2$ mol.wt. 206.28

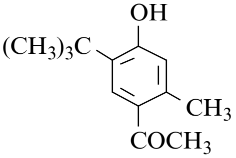


Syntheses

- Preparation by reaction of acetyl chloride or acetic anhydride with 2-tert-butyl-6-methylphenol in the presence of aluminium chloride, and saponification of keto ester obtained [3353].
- Also obtained (poor yield) by oxidation of 2-tert-butyl-4-ethyl-6-methylphenol. The oxidation was carried out by bubbling air at 80–100° into a solution of 2-tert-butyl-4-ethyl-6-methylphenol in cumene containing cobalt phthalate and cumene hydroperoxide as initiator (4%) [3486].

m.p. 126° [3486], 123–125° [3353].

1-[5-(1,1-Dimethylethyl)-4-hydroxy-2-methylphenyl]ethanone



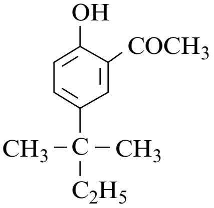
$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

- Preparation by Fries rearrangement of 2-tert-butyl-5-methyl-phenyl acetate with aluminium chloride in nitrobenzene at 25° (22%) [3485].

m.p. 124° [3485].

1-[5-(1,1-Dimethylpropyl)-2-hydroxyphenyl]ethanone



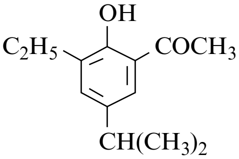
$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

- Preparation by Fries rearrangement of 4-tert-pentylphenyl acetate with aluminium chloride at 120° (57%) [3356].

b.p.$_{12}$ 165° [3356].

1-[3-Ethyl-2-hydroxy-5-(1-methylethyl)phenyl]ethanone



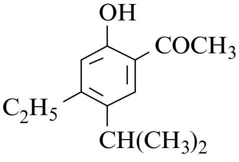
$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

- Preparation by chromic acid degradation of 7-ethyl-2,3-dimethyl-5-isopropylbenzofuran (44%) [3224].

b.p.$_{10}$ 139–140° [3224].

1-[4-Ethyl-2-hydroxy-5-(1-methylethyl)phenyl]ethanone



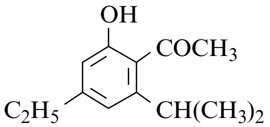
$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

- Preparation by chromic acid degradation of 6-ethyl-2,3-dimethyl-5-isopropylbenzofuran (44%) [3224].

b.p.$_{11}$ 152–153° [3224].

1-[4-Ethyl-2-hydroxy-6-(1-methylethyl)phenyl]ethanone



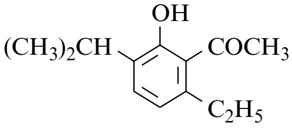
$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

- Preparation by chromic acid degradation of 6-ethyl-2,3-dimethyl-4-isopropylbenzofuran (60%) [3224].

m.p. 113° [3224]; b.p.$_{14}$ 167–168° [3224].

1-[6-Ethyl-2-hydroxy-3-(1-methylethyl)phenyl]ethanone



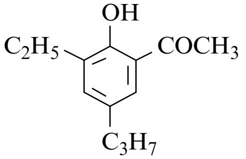
$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

- Preparation by chromic acid degradation of 4-ethyl-2,3-dimethyl-7-isopropylbenzofuran (60%) [3224].

b.p.$_{11}$ 145° [3224].

1-(3-Ethyl-2-hydroxy-5-propylphenyl)ethanone



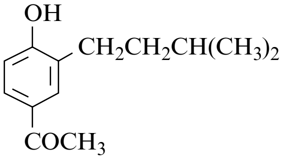
$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

- Preparation by Fries rearrangement of 2-ethyl-4-propyl-phenyl acetate with aluminium chloride [2957].

b.p.$_{18}$ 140–141° [2957].

1-[4-Hydroxy-3-(3-methylbutyl)phenyl]ethanone



$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

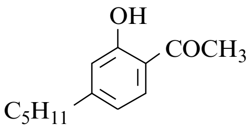
- Preparation by Fries rearrangement of 2-isopentylphenyl acetate [3449,3487].

Isolation from natural sources

- Also obtained by catalytic hydrogenation of Tremetone in the presence of Pd/C. Tremetone has been isolated from the *White Snakeroot* plant (Eupatorium urticaefolium) [3449,3487].

1-(2-Hydroxy-4-pentylphenyl)ethanone

[60441-58-9]



$C_{13}H_{18}O_2$ mol.wt. 206.28

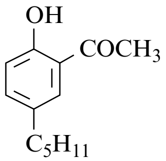
Syntheses

- Preparation by Fries rearrangement of 3-pentylphenyl acetate with aluminium chloride at 130° (50%) [3488].

- Also obtained by demethylation of a mixture of 2-methoxy-4-pentylacetophenone and 4-methoxy-2-pentylaceto-phenone (**I**) with boron tribromide in methylene chloride at 0° and separation of isomers by chromatography (21%). The mixture of anisoles (**I**) was obtained by reaction of acetic anhydride with 3-pentylanisole in the presence of aluminium chloride in refluxing carbon disulfide [3489].

oil [3488,3489]; b.p.$_{0.5}$ 130° [3489]; ^{1}H NMR [3488,3489].

1-(2-Hydroxy-5-pentylphenyl)ethanone



$C_{13}H_{18}O_2$ mol.wt. 206.28

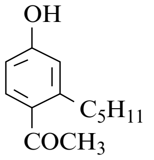
Synthesis

- Preparation by reaction of acetic acid on 4-pentylphenol with boron trifluoride at 140–150° (87%) [3490].

b.p.$_7$ 145–148° [3490]; n_D^{25} = 1.5249 [3490].

1-(4-Hydroxy-2-pentylphenyl)ethanone

[60441-59-0] $C_{13}H_{18}O_2$ mol.wt. 206.28

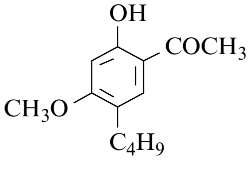


Synthesis

- Preparation by demethylation of a mixture of 2-methoxy-4-pentylacetophenone and 4-methoxy-2-pentylacetophenone (**I**) with boron tribromide in methylene chloride at 0° and separation of isomers by chromatography (46%). The mixture of anisoles (**I**) was obtained by reaction of acetic anhydride with 3-pentylanisole in the presence of aluminium chloride in refluxing carbon disulfide [3489].

m.p. 58–59° [3489]; ^{1}H NMR [3489].

1-(5-Butyl-2-hydroxy-4-methoxyphenyl)ethanone



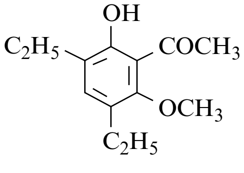
$C_{13}H_{18}O_3$ mol.wt. 222.28

Synthesis

- Preparation by reaction of dimethyl sulfate on 5-butyl-2,4-dihydroxyacetophenone with potassium carbonate in boiling acetone [3018].

1-(3,5-Diethyl-2-hydroxy-6-methoxyphenyl)ethanone

[37467-70-2] $C_{13}H_{18}O_3$ mol.wt. 222.28



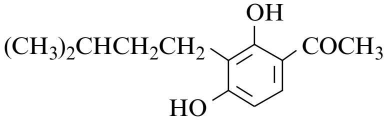
Synthesis

- Preparation by reaction of methyl iodide with 3,5-diethyl-2,6-dihydroxyacetophenone in the presence of potassium carbonate in boiling acetone (46%) [2997].

Oil [2997]; MS [2997].

1-[2,4-Dihydroxy-3-(3-methylbutyl)phenyl]ethanone

[50773-37-0] $C_{13}H_{18}O_3$ mol.wt. 222.28



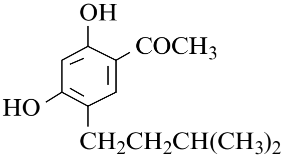
Synthesis

- Preparation by catalytic hydrogenation of 3-(dimethyl-allyl)-2,4-dihydroxacetophenone with platinic oxide as a catalyst in ethanol [3457].

m.p. 115° [3457]; ^{1}H NMR [3457], IR [3457].

1-[2,4-Dihydroxy-5-(3-methylbutyl)phenyl]ethanone

[56146-52-2] $C_{13}H_{18}O_3$ mol.wt. 222.28



Syntheses

- Obtained by Fries rearrangement of 4-isoamylresorcinol diacetate with aluminium chloride in nitrobenzene at 50–55° [3449,3487], (11%) [3487].

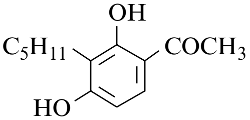
Isolation from natural sources

- Also obtained by hydrogenolysis of 6-hydroxytremetone in the presence of 10% Pd/C in ethanol at r.t. [3449,3487], (62%) [3487]. The 6-hydroxytremetone has been isolated from the *Eupatorium urticaefolium* Reichard (Compositae) (white snakeroot).

m.p. 93–95° [3487], 92°5–94° [3449]; IR [3487].

1-(2,4-Dihydroxy-3-pentylphenyl)ethanone

[111224-14-7] $C_{13}H_{18}O_3$ mol.wt. 222.28

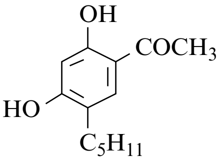


Synthesis

- Refer to: [3014].

1-(2,4-Dihydroxy-5-pentylphenyl)ethanone

[97304-17-1] $C_{13}H_{18}O_3$ mol.wt. 222.28



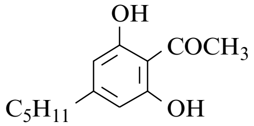
Syntheses

- Preparation by reaction of acetonitrile on 4-n-amylresorcinol (Hoesch reaction) (76%) [2676].
- Preparation from 2,4-dimethoxy-5-pentylacetophenone by demethylation with boron tribromide in methylene chloride at r.t. (53%) [2678,2679].

m.p. 110–111° [2676].

1-(2,6-Dihydroxy-4-pentylphenyl)ethanone

[67895-11-8] $C_{13}H_{18}O_3$ mol.wt. 222.28

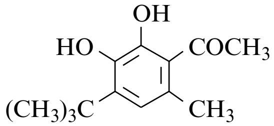


Synthesis

– Preparation by metallation of olivetol tetrahydropyranyl ether with n-butyllithium in tetrahydrofuran under nitrogen, followed by adding cuprous bromide in this solution. The condensation of 2,6-dihydroxy-4-pentylphenylcopper tetrahydropyranyl ether so obtained with acetyl chloride, connected with elimination of protective group under mild conditions gave the expected compound (80%) [3491].

1-[4-(1,1-Dimethylethyl)-2,3-dihydroxy-6-methylphenyl]ethanone

[84297-01-8] $C_{13}H_{18}O_3$ mol.wt. 222.28



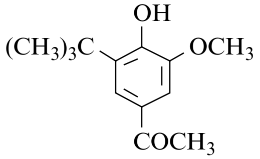
Synthesis

– Obtained by UV irradiation of a benzene solution of 3-tert-butyl-5-methyl-o-benzoquinone in the presence of a large excess of acetaldehyde (12%) [3386].

m.p. 72–73° [3386]; ^{1}H NMR [3386], IR [3386].

1-[3-(1,1-Dimethylethyl)-4-hydroxy-5-methoxyphenyl]ethanone

[153356-09-3] $C_{13}H_{18}O_3$ mol.wt. 222.28



Syntheses

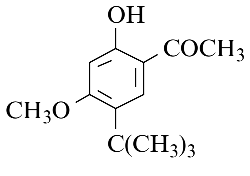
– Obtained by partial methylation of 3,4-dihydroxy-5-tert-butylacetophenone with diazomethane [1952].
– Also refer to: [3339].

N.B.: *(Catechol O-methyltransferase test).*

– Also obtained from 3,4-dihydroxy-5-tert-butylacetophenone in propylene glycol by incubation for 2 h at 37° with 0.5 M phosphate buffer (pH = 7.9), 0.5 M magnesium chloride, S-adenosylmethionine and catechol O-methyltransferase (enzyme). This enzyme solution was prepared from the blood of an adult male rat [1952].

1-[5-(1,1-Dimethylethyl)-2-hydroxy-4-methoxyphenyl]ethanone

[127371-46-4] $C_{13}H_{18}O_3$ mol.wt. 222.28

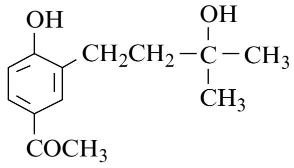


Synthesis

– Refer to: [3478] (patent).

1-[4-Hydroxy-3-(3-hydroxy-3-methylbutyl)phenyl]ethanone

[81944-40-3] $C_{13}H_{18}O_3$ mol.wt. 222.28



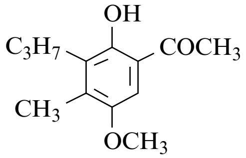
Isolation from natural sources

- From Senecio species of the North of Chile (*Senecio phylolleptus* Cuatr., *Senecio viridi*s Phil., *Senecio nutans* Sch. Bip.) [3442].
- From the aerial parts of *Werneria poposa* [3448].
- Also refer to: [3492].

m.p. 100–101° [3442]; ^{1}H NMR [3442,3448], ^{13}C NMR [3442,3448], IR [3442], MS [3442,3448].

1-(2-Hydroxy-5-methoxy-4-methyl-3-propylphenyl)ethanone

[43037-71-4] $C_{13}H_{18}O_3$ mol.wt. 222.28

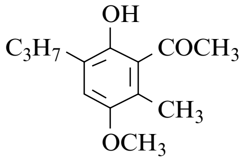


Synthesis

- Preparation by catalytic hydrogenation of 3-allyl-2-hydroxy-5-methoxy-4-methylacetophenone, previously obtained by thermal Claisen rearrangement of 2-(allyloxy)-5-methoxy-4-methylacetophenone in N,N-dimethylaniline at 170° (42%) [3467].

1-(2-Hydroxy-5-methoxy-6-methyl-3-propylphenyl)ethanone

[43037-72-5] $C_{13}H_{18}O_3$ mol.wt. 222.28

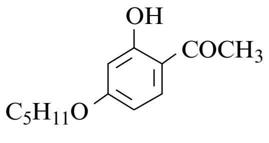


Synthesis

- Preparation by catalytic hydrogenation of 3-allyl-2-hydroxy-5-methoxy-6-methylacetophenone, previously obtained by thermal Claisen rearrangement of 2-(allyloxy)-5-methoxy-4-methylacetophenone—via a [2368,,3466] sigmatropic rearrangement—in N,N-dimethylaniline at 170° (22%) [3467].

1-[2-Hydroxy-4-(pentyloxy)phenyl]ethanone

[101002-29-3] $C_{13}H_{18}O_3$ mol.wt. 222.28

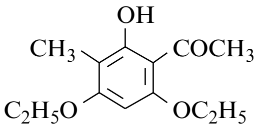


Syntheses

- Preparation by partial alkylation of resacetophenone with pentyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [3493].
- Also refer to: [3494].

m.p. 36° [3493].

1-(4,6-Diethoxy-2-hydroxy-3-methylphenyl)ethanone



$C_{13}H_{18}O_4$ mol.wt. 238.28

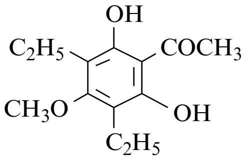
Synthesis

- Preparation by reaction of ethyl iodide or diethyl sulfate on 2,4,6-trihydroxy-3-methylacetophenone with potassium carbonate in boiling acetone (77%) [3051].

m.p. 147° [3051].

1-(3,5-Diethyl-2,6-dihydroxy-4-methoxyphenyl)ethanone

[175785-86-1]



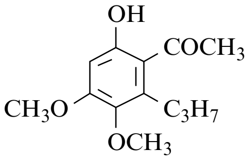
$C_{13}H_{18}O_4$ mol.wt. 238.28

Synthesis

- Refer to: [3495] (Japanese patent).

N.B.: Di-Na salt [175785-89-4] [3495].

1-(3,4-Dimethoxy-6-hydroxy-2-propylphenyl)ethanone



$C_{13}H_{18}O_4$ mol.wt. 238.28

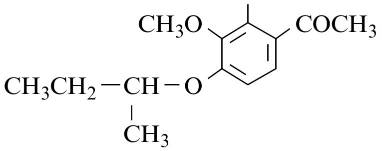
Syntheses

- Preparation by catalytic hydrogenation of 6-(benzyloxy)-3,4-dimethoxy-2-propenylacetophenone in the presence of Pd/C in methanol [2849].
- Also obtained by addition of 6-(benzyloxy)-3,4-dimethoxy-2-propenylacetophenone in ethyl ether to calcium in liquid ammonia, and addition of ammonium chloride before evaporation of solvents [2849].

Yellow oil [2849]; b.p.$_{0.2}$ 160° [2849]; UV [2849].

1-[2-Hydroxy-3-methoxy-4-(1-methylpropoxy)phenyl]ethanone

[94245-10-0]



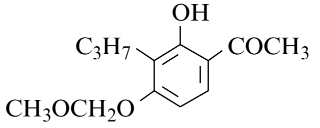
$C_{13}H_{18}O_4$ mol.wt. 238.28

Synthesis

- Refer to: [3496] (Indian patent).

1-[2-Hydroxy-4-(methoxymethoxy)-3-propylphenyl]ethanone

[200355-19-7] $C_{13}H_{18}O_4$ mol.wt. 238.28



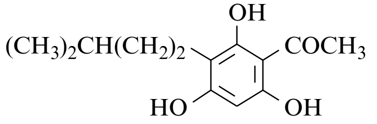
Synthesis

- Preparation by treatment of 2,4-dihydroxy-3-propyl-acetophenone according to the procedure [3106], (83%) [3497].

b.p.$_{1}$ 152–154° [3497]; ^{1}H NMR [3497], IR [3497].

1-[2,4,6-Trihydroxy-3-(3-methylbutyl)phenyl]ethanone

[39652-85-2] $C_{13}H_{18}O_4$ mol.wt. 238.28



Syntheses

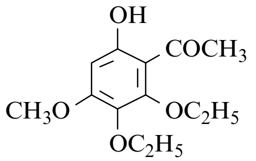
- Preparation by reaction of acetonitrile on 2-isoamyl-phloroglucinol (Hoesch reaction) (93%) [3498].
- Preparation by reaction of 2-isoamylphloroglucinol with boron trifluoride–acetic acid complex at 28–30° (70%) [2242].
- Preparation by catalytic hydrogenolysis of 2-acetyl-4,4-bis-(3-methylbut-2-enyl)cyclohexane-1,3,5-trione at r.t. and pressure in the presence of 10% Pd/C in ethanol (93%) [3477].
- Preparation by catalytic hydrogenation of 2′,4′,6′-trihydroxy-3′-(3-methylbut-2-enyl)acetophenone [3477].
- Also obtained by catalytic hydrogenation of 7-acetyl-4,6-dihydroxy-2-isopropenylcoumaran in the presence of platinic oxide (Adams catalyst) in methanol (72%) [2847].
- Also obtained by catalytic hydrogenation of 9-acetyl-2,5-dihydro-3-methyl-1-benzoxepin-6,8-diol in ethyl acetate in the presence of Adams catalyst (25%) [3499].
- Also obtained (by-product) by reaction of isoamyl iodide with phloroacetophenone in the presence of sodium methoxide in methanol (6%) [2878].

m.p. 190–192° [3499], 190° [2242], 188° [2878,3498], 185° [2847],184–185°5 [3477], 183–184° [3500], 130–131° (compound **VIII**) [2879]. One of the reported melting points is obviously wrong.

b.p.$_{0.01}$ 190° [3498]; ^{1}H NMR [2879,3499], IR [3499], UV [3477].

1-(2,3-Diethoxy-6-hydroxy-4-methoxyphenyl)ethanone

$C_{13}H_{18}O_5$ mol.wt. 254.28

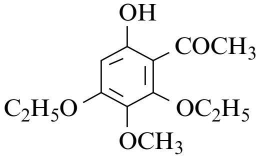


Synthesis

- Preparation by reaction of diethyl sulfate on 2-ethoxy-3,6-dihydroxy-4-methoxyacetophenone with potassium carbonate in boiling acetone [2851].
- oil [2851].

1-(2,4-Diethoxy-6-hydroxy-3-methoxyphenyl)ethanone

[18086-01-6] $C_{13}H_{18}O_5$ mol.wt. 254.28



Syntheses

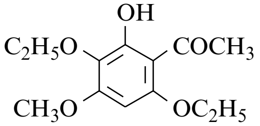
– Preparation by reaction of dimethyl sulfate on 2,4-diethoxy-3,6-dihydroxyacetophenone with potassium carbonate in refluxing acetone [2851,3394,3395], (47%) [3394].

– The same ketone was also obtained by alkaline degradation of 4′,5,7-triethoxy-3′, 6-dimethoxyflavone with potassium hydroxide in refluxing dilute ethanol [2851].

Oil [2851,3394].

1-(3,6-Diethoxy-2-hydroxy-4-methoxyphenyl)ethanone

[105342-72-1] $C_{13}H_{18}O_5$ mol.wt. 254.28



Syntheses

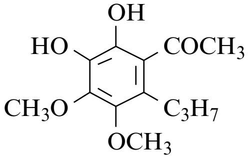
– Preparation from 1,4-diethoxy-2,6-dimethoxybenzene by reaction,

- with acetyl chloride in the presence of aluminium chloride in ethyl ether cooled in an ice bath (89%) [3501];
- with acetic acid in the presence of boron trifluoride for 5 h at 80° (50%) [3131].

m.p. 104–105° [3501], 101–103° [3131].

1-(2,3-Dihydroxy-4,5-dimethoxy-6-propylphenyl)ethanone

[76576-61-9] $C_{13}H_{18}O_5$ mol.wt. 254.28



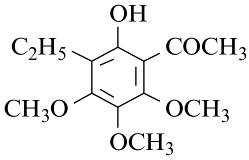
Synthesis

– Preparation by treatment of acetyldihydrodillapiole with boron trifluoride (almost quantitative yield) [3502].

m.p. 110° [3502];
^{1}H NMR [3502], IR [3502], MS [3502].

1-(3-Ethyl-2-hydroxy-4,5,6-trimethoxyphenyl)ethanone

$C_{13}H_{18}O_5$ mol.wt. 254.28

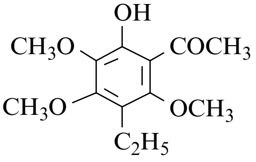


Synthesis

– Preparation by Fries rearrangement of 2-ethyl-3,4,5-tri-methoxyphenyl acetate with aluminium chloride in nitrobenzene at r.t. (25%) [3135].

b.p.$_{0.15}$ 98–102° [3135];
n_D^{25} = 1.5293 [3135].

1-(3-Ethyl-6-hydroxy-2,4,5-trimethoxyphenyl)ethanone



$C_{13}H_{18}O_5$ mol.wt. 254.28

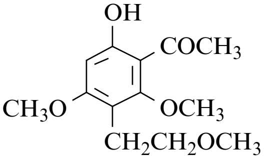
Syntheses

- Preparation by Fries rearrangement of 4-ethyl-2,3,5-tri-methoxyphenyl acetate with aluminium chloride in nitrobenzene at r.t. (51%) [3135].
- Preparation by reaction of acetyl chloride on 1-ethyl-2,3,4,6-tetramethoxybenzene with aluminium chloride in ethyl ether between –20° and –15°, followed by standing overnight at r.t. (46%) [3135].

Clear, orange coloured oil [3135]; b.p.$_{0.3}$ 117–118°5 [3135]; n_D^{25} = 1.5421 [3135].

1-[6-Hydroxy-2,4-dimethoxy-3-(2-methoxyethyl)phenyl]ethanone

[34288-74-9]



$C_{13}H_{18}O_5$ mol.wt. 254.28

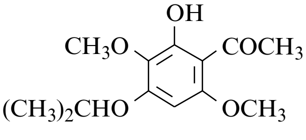
Synthesis

- Obtained by reaction of methyl iodide with 2-hydroxy-4,6-dimethoxy-5-(2-hydroxyethyl) acetophenone (m.p. 133–134°) in chloroform in the presence of silver oxide at r.t. overnight (25%) [3407].

m.p. 94–94°5 [3407];
^{1}H NMR [3407], UV [3407].

1-[2-Hydroxy-3,6-dimethoxy-4-(1-methylethoxy)phenyl]ethanone

[93344-52-6]



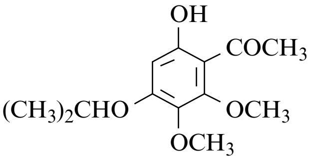
$C_{13}H_{18}O_5$ mol.wt. 254.28

Syntheses

- Refer to: [3503] (compound **11**) and [3504] (Japanese paper).

1-[6-Hydroxy-2,3-dimethoxy-4-(1-methylethoxy)phenyl]ethanone

[119136-17-3]



$C_{13}H_{18}O_5$ mol.wt. 254.28

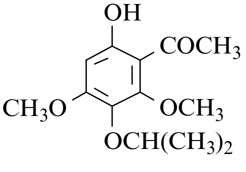
Synthesis

- Preparation by reaction of dimethyl sulfate with 2,5-di-hydroxy-4-isopropoxy-6-methoxyacetophenone in the presence of potassium carbonate in refluxing acetone (82%) [2830].

colourless oil [2830]; ^{1}H NMR [2830], IR [2830], MS [2830].

1-[6-Hydroxy-2,4-dimethoxy-3-(1-methylethoxy)phenyl]ethanone

[96501-84-7] $C_{13}H_{18}O_5$ mol.wt. 254.28



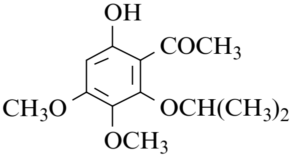
Synthesis

- Obtained by partial isopropylation of 2,5-dihydroxy-4,6-di-methoxyacetophenone with isopropyl bromide, according to [3505], (compound **27**) (44%) [3506].

Brown oil [3506]; ^{1}H NMR [3506].

1-[6-Hydroxy-3,4-dimethoxy-2-(1-methylethoxy)phenyl]ethanone

[188927-29-9] $C_{13}H_{18}O_5$ mol.wt. 254.28



Synthesis

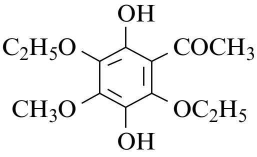
- Preparation by treatment of 3,4,6-trimethoxy-2-isopropoxy-acetophenone (m.p. 69–71°) with aluminium bromide in acetonitrile at 0° for 10–15 min (80%) [3507].

N.B.: The partial demethylation realized with aluminium bromide–sodium iodide system at 0° for 10 min gave a 94% yield.

m.p. 65–67° [3507].

1-(3,6-Diethoxy-2,5-dihydroxy-4-methoxyphenyl)ethanone

$C_{13}H_{18}O_6$ mol.wt. 270.28



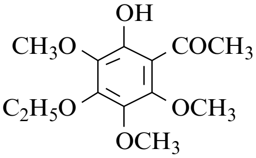
Synthesis

- Preparation from 3,6-diethoxy-2-hydroxy-4-methoxy-acetophenone by persulfate oxidation (Elbs reaction) (33%) [3501].

m.p. 131–132° [3501].

1-(4-Ethoxy-2-hydroxy-3,5,6-trimethoxyphenyl)ethanone

$C_{13}H_{18}O_6$ mol.wt. 270.28



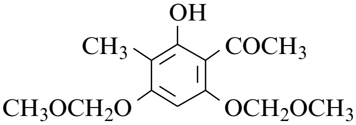
Synthesis

- Preparation by Friedel–Crafts acylation of 1-ethoxy-2,3,5,6-tetramethoxybenzene with acetyl chloride in the presence of aluminium chloride (46%) [3313].

b.p.$_{0.1}$ 117–119° [3313].

1-[2-Hydroxy-4,6-bis(methoxymethoxy)-3-methylphenyl]ethanone

[106929-57-1] $C_{13}H_{18}O_6$ mol.wt. 270.28



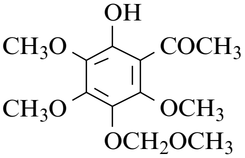
Synthesis

- Preparation by reaction of chloromethyl methyl ether with 2,4,6-trihydroxy-3-methylacetophenone in cooled acetone in the presence of potassium carbonate for 1 h (34%) [3508].

m.p. 77–78° [3508]; ^{1}H NMR [3508].

1-[2-Hydroxy-3,4,6-trimethoxy-5-(methoxymethoxy)phenyl]ethanone

[173217-34-0] $C_{13}H_{18}O_7$ mol.wt. 286.28

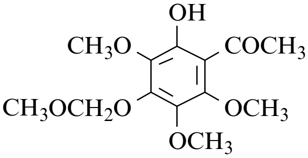


Synthesis

- Preparation by reaction of chloromethyl methyl ether with 2,5-dihydroxy-3,4,6-trimethoxyacetophenone in methylene chloride in the presence of N,N-diisopropylethylamine at r.t. for 3–4 h [3319].

1-[2-Hydroxy-3,5,6-trimethoxy-4-(methoxymethoxy)phenyl]ethanone

[176662-07-0] $C_{13}H_{18}O_7$ mol.wt. 286.28

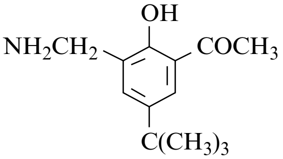


Synthesis

- Obtained by stirring a solution of 2,4-dihydroxy-3,5,6-tri-methoxyacetophenone, N,N-diisopropylethylamine and chloromethyl methyl ether in methylene chloride at 5° for 40–50 min [3316].

1-[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone

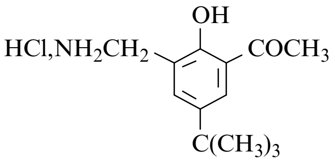
[75060-91-2] $C_{13}H_{19}NO_2$ mol.wt. 221.30



Synthesis

- Refer to: [3509].

1-[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone *(Hydrochloride)*



$C_{13}H_{19}NO_2$, HCl mol.wt. 268.77

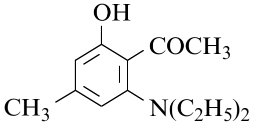
Synthesis

- Preparation by hydrolysis of 2-acetyl-4-tert-butyl-6-(N-chloroacetylaminomethyl) phenol with concentrated hydrochloric acid in refluxing ethanol (89%) [3354].

m.p. 191–193° [3354].

1-[2-(Diethylamino)-6-hydroxy-4-methylphenyl]ethanone

[97066-07-4] $C_{13}H_{19}NO_2$ mol.wt. 221.30



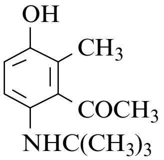
Synthesis

- Preparation by reaction of potassium hydroxide with 2-acetyl-3-diethylamino-5-hydroxy-5-methyl-2-cyclo-hexenone in ethanol at 40° (75%) [2712].

Yellow oil [2712];
^{1}H NMR [2712], IR [2712], UV [2712], MS [2712].

1-[6-[(1,1-Dimethylethyl)amino]-3-hydroxy-2-methylphenyl]ethanone

[158013-69-5] $C_{13}H_{19}NO_2$ mol.wt. 221.30



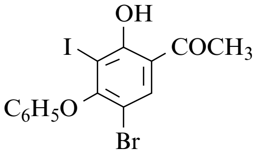
Synthesis

- Obtained from N-tert-butyl-2-acetyl-4-methyl quinol imine by refluxing overnight in trifluoroethanol (TFE) (quantitative yield) [3510].

Red oil [3510]; ^{1}H NMR [3510],
^{13}C NMR [3510], IR [3510], MS [3510].

1-(5-Bromo-2-hydroxy-3-iodo-4-phenoxyphenyl)ethanone

[145489-48-1] $C_{14}H_{10}BrIO_3$ mol.wt. 433.04



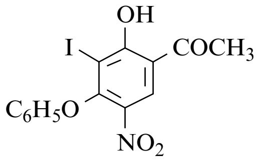
Synthesis

- Preparation by hypervalent iodine oxidation of 5-bromo-resacetophenone with iodosobenzene diacetate in the presence of potassium hydroxide in methanol via the rearrangement of iodonium ylide previously formed (35%) [3511].

m.p. 160–161° [3511]; ^{1}H NMR [3511], MS [3511].

1-(2-Hydroxy-3-iodo-5-nitro-4-phenoxyphenyl)ethanone

[145489-93-6] $C_{14}H_{10}INO_5$ mol.wt. 399.14



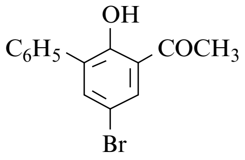
Syntheses

- Preparation by hypervalent iodine oxidation of 5-nitro-resacetophenone with iodosobenzene diacetate in the presence of potassium hydroxide in methanol via the rearrangement of iodonium ylide previously formed (77%) [3511].
- Preparation by thermal rearrangement of 4-acetyl-3-hydroxy-6-nitro-2-phenyliodonio-phenolate (SM) in refluxing acetonitrile for 30 min (47%) [3512]. SM was obtained by reaction of iodosobenzene diacetate with 5-nitroresacetophenone in methanol in the presence of potassium hydroxide at 0° for 30 min (45%, m.p. 145–147°).

m.p. 190–191° [3511], 180–185° [3512];
^{1}H NMR [3511,3512], IR [3511,3512], MS [3512].

1-(5-Bromo-2-hydroxy[1,1′-biphenyl]-3-yl)ethanone

$C_{14}H_{11}BrO_2$ mol.wt. 291.14



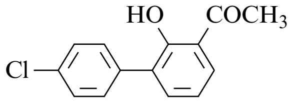
Synthesis

- Preparation by treatment of 1-(2-hydroxy[1,1′-biphenyl]-3-yl)ethanone in acetic acid with NBS at 85° for 2 h (quantitative yield) [1804].

^{1}H NMR [1804].

1-(4′-Chloro-2-hydroxy[1,1′-biphenyl]-3-yl)ethanone

[77893-89-1] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



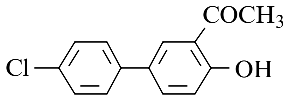
Synthesis

- Obtained by Fries rearrangement of 2-acetoxy-4′-chloro-biphenyl with aluminium chloride at 150° for 30 min (26%) [3513].

m.p. 76–78° [3513].

1-(4′-Chloro-4-hydroxy[1,1′-biphenyl]-3-yl)ethanone

[86608-89-1] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



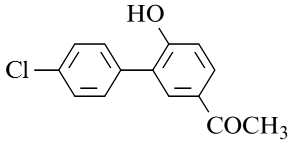
Synthesis

N.B.: It must be pointed out that in the referenced paper [3514], the described product [(chloro-4 phenyl)-5 hydroxy-2 phenyl]-1 ethanone is not consistent with the mentioned starting material, (chloro-4 phenoxy)-4 phenol.

In such a case, the obtained product should be the 1-[5-(4-chlorophenoxy)-2-hydroxyphenyl]ethanone. One of the authors of this paper, Daniel Humbert, has confirmed this typing mistake [3515].

1-(4′-Chloro-6-hydroxy[1,1′-biphenyl]-3-yl)ethanone

[77893-88-0] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



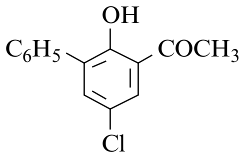
Synthesis

- Preparation by Fries rearrangement of 2-acetoxy-4′-chloro-biphenyl with aluminium chloride at 150° for 30 min (60%) [3513].

m.p. 165°5–167°5 [3513].

1-(5-Chloro-2-hydroxy[1,1′-biphenyl]-3-yl)ethanone

$C_{14}H_{11}ClO_2$ mol.wt. 246.69

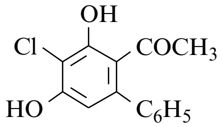


Synthesis

- Obtained by treatment of 1-(2-hydroxy[1,1′-biphenyl]-3-yl)ethanone in DMF solution with NCS at r.t. overnight (96%) (crude product) [1804].

1-(4-Chloro-3,5-dihydroxy[1,1′-biphenyl]-2-yl)ethanone

[76538-42-6] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



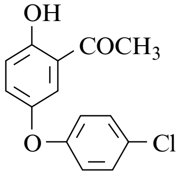
Synthesis

- Preparation by adding lithium chloride to a solution of 3-acetoxy-6-acetyl-2,6-dichloro-5-phenyl-2-cyclohexenone in DMF and heating for 2 h at 100° (83%) [3516].

m.p. 111–112° [3516]; ^{1}H NMR [3516], IR [3516], MS [3516].

1-[5-(4-Chlorophenoxy)-2-hydroxyphenyl]ethanone

[73051-30-6] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



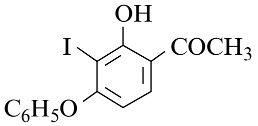
Syntheses

- Preparation by acetylation of 4-phenoxyphenol with,
  - 39.7% (w/w) boron trifluoride in acetic acid at 75° for 24 h (71%) [3514];
  - 35–37% boron trifluoride in acetic acid at 90° for 3 days (quantitative yield) [3515].
- Also refer to: [3517].

m.p. 88° [3514,3517].

1-(2-Hydroxy-3-iodo-4-phenoxyphenyl)ethanone

[144691-35-0] $C_{14}H_{11}IO_3$ mol.wt. 354.14



Syntheses

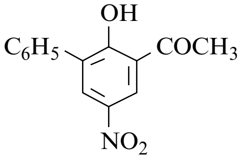
- Preparation by thermal rearrangement of 4-acetyl-3-hydroxy-2-phenyliodonio-phenolate (SM) in refluxing acetonitrile for 30 min (70%). SM was obtained by reaction of iodosobenzene diacetate with resacetophenone in methanol in the presence of potassium hydroxide at 0° for 30 min (40%, m.p. 81–85°) [3512].
- Preparation by reaction of resacetophenone with iodosobenzene diacetate in refluxing methanol (55%) [3518].
- Preparation by hypervalent iodine oxidation of resacetophenone with iodosobenzene diacetate in the presence of potassium hydroxide in methanol *via* the rearrangement of iodonium ylide previously formed (20%) [3511].

m.p. 116–118° [3511], 101–103° [3518], 72° [3512]. There is a discrepancy between the different melting points indicated in literature.

^{1}H NMR [3511,3512,3518], IR [3518], MS [3512].

1-(2-Hydroxy-5-nitro[1,1′-biphenyl]-3-yl)ethanone

[360791-69-1] $C_{14}H_{11}NO_4$ mol.wt. 257.25



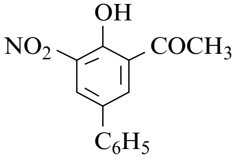
Synthesis

- Obtained by treatment of 3-bromo-2-hydroxy-5-nitro-acetophenone and phenylboronic acid mixture in THF with tetrakis(triphenylphosphine)palladium (0) and 10% aqueous sodium carbonate. The mixture was heated at 80° for 3 h. Then, 10% Pd/C catalyst was added and heating continued for 1.5 h (70%) (compound **32**) [1804].

^{1}H NMR [1804], ^{13}C NMR [1804].

1-(4-Hydroxy-5-nitro[1,1′-biphenyl]-3-yl)ethanone

[84942-37-0] $C_{14}H_{11}NO_4$ mol.wt. 257.25



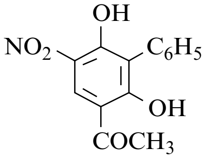
Synthesis

- Preparation by reaction of 100% nitric acid with 3-acetyl-4-hydroxybiphenyl in acetic acid at r.t. (90%) [1852].

m.p. 113–114° [1852].

1-(2,6-Dihydroxy-5-nitro[1,1′-biphenyl]-3-yl)ethanone

[160246-79-7] $C_{14}H_{11}NO_5$ mol.wt. 273.25



Syntheses

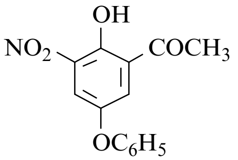
– Obtained by irradiation of 4-acetyl-3-hydroxy-6-nitro-2-phenyliodonio-phenolate (1 mmol),
  - in benzene suspension (65%) [3512];
  - in the presence of cyclohexene (3–5 mmol) in methylene chloride/acetonitrile solution (1:1) for 4 h (20–30%) [3512].

N.B.: The irradiations were performed with a 250 W low pressure Hg lamp.

m.p. 160–161° [3512]; ^{1}H NMR [3512], IR [3512], MS [3512].

1-(2-Hydroxy-3-nitro-5-phenoxyphenyl)ethanone

[84942-38-1] $C_{14}H_{11}NO_5$ mol.wt. 273.25



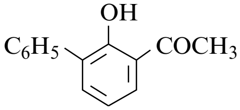
Synthesis

– Preparation by reaction of 100% nitric acid with 2-hydroxy-5-phenoxyacetophenone in acetic acid at r.t. (73%) [1852].

m.p. 126–127° [1852].

1-(2-Hydroxy[1,1′-biphenyl]-3-yl)ethanone

[21424-82-8] $C_{14}H_{12}O_2$ mol.wt. 212.25



Syntheses

– Preparation by Fries rearrangement of 2-acetoxybiphenyl with aluminium chloride,

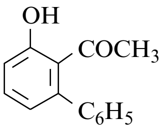
- without solvent between 130° and 160° [3519–3522];
- in boiling o-dichlorobenzene for 30 min (15%) [3523].

– Also obtained by UV light irradiation (254 nm) of 2-acetoxybiphenylyl in benzene (11%) [3524].

3^{1}H NMR [3523], IR [3523].

1-(3-Hydroxy[1,1′-biphenyl]-2-yl)ethanone

[136819-93-7] $C_{14}H_{12}O_2$ mol.wt. 212.25



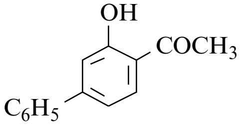
Synthesis

– Obtained by adding potassium fluoride to a solution of 1-phenyl-2-octene-1,5,7-trione in dry toluene and the whole refluxed overnight (32%) [3525].

Colourless oil [3525];
^{1}H NMR [3525], IR [3525], MS [3525].

1-(3-Hydroxy[1,1′-biphenyl]-4-yl)ethanone

[32101-38-5] $C_{14}H_{12}O_2$ mol.wt. 212.25



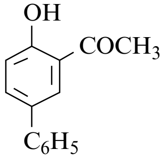
Syntheses

- Preparation by reaction of acetyl chloride with 3-methoxy-biphenyl in the presence of aluminium chloride in refluxing methylene chloride for 11 h (50%) [3526].
- Preparation by aromatization of 2-acetyl-3-chloro-5-phenyl-2-cyclohexenone in the presence of Pd/C in refluxing cyclohexene for 4 h (70%) [3527].
- Preparation by dehydrogenation of 6-acetyl-3-phenyl-2-cyclohexen-1-one with refluxing 16% bromine solution in acetic acid [2612].
- Also refer to: [2018].

m.p. 91° [2612], 90°5–91°5 [3526], 90–90°5 [3527].

1-(4-Hydroxy[1,1′-biphenyl]-3-yl)ethanone

[14031-80-2] $C_{14}H_{12}O_2$ mol.wt. 212.25



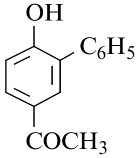
Syntheses

- Preparation by Fries rearrangement of 4-acetoxybiphenyl,
  - with aluminium chloride without solvent at 120–140° [1852,3528,3529], (48%) [1852] or in tetrachloroethane at 140° [3521,3522,3530,3531], (46%) [3531];
  - with titanium tetrachloride in nitrobenzene at r.t. (6%) [3379].
- Also obtained by photo-Fries rearrangement of 4-acetoxybiphenylyl with 254 nm light in benzene (61%) [3524].
- Also obtained—via an intermolecular photo-Fries rearrangement—by irradiation of a solution of pinacolone and p-phenylphenol in benzene for 5 h (42%) [3358].
- Preparation by catalytic hydrogenation of 2-(benzyloxy)-5-phenylacetophenone in the presence of 10% Pd/C in 95% ethanol at r.t. under 35 psi for 2 h (84%) [3379].
- Preparation by adding an ethereal solution of methyl lithium to a solution of 5-phenylsalicylic acid in 1,2-dimethoxyethane under nitrogen and maintained at 10–12° (85%) [3379].
- Preparation by reaction of acetic acid with 4-hydroxybiphenyl in the presence of boron trifluoride–acetic acid complex (good yield) [3521,3522].
- Also refer to: [3532–3535].

m.p. 61°5–62° [3530], 61–62° [1852,3528], 60–61° [3379], 59–61° [3531]; IR [3379].

1-(6-Hydroxy[1,1′-biphenyl]-3-yl)ethanone

[20281-51-0] $C_{14}H_{12}O_2$ mol.wt. 212.25



Syntheses

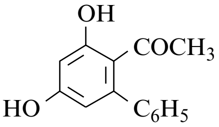
- Preparation by Fries rearrangement of 2-acetoxybiphenyl with aluminium chloride,
  - without solvent between 130° and 160° [3519,3520,3536], (60–62%) [3519,3520];
  - in nitrobenzene (58%) [3537] according to [2151];
  - in boiling o-dichlorobenzene for 30 min (80%) [3523].
- Also obtained by UV light irradiation (254 nm) of 2-acetoxybiphenyl in benzene (6%) [3524].
- Preparation by direct condensation of 2-hydroxybiphenyl with acetic acid in the presence of boron trifluoride [3538].
- Also refer to: [2117].

m.p. 184°1–184°4 [3523], 177° [3537], 173° [3538], 172–173° [3519], 167–168°5 [3520];

^{1}H NMR [3523], IR [3523]; GC-MS [3523].

1-(3,5-Dihydroxy[1,1′-biphenyl]-2-yl)ethanone

[54439-83-7] $C_{14}H_{12}O_3$ mol.wt. 228.25



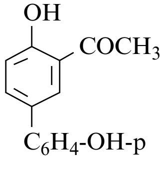
Synthesis

- Obtained (by-product) from 3-methyl-3′-phenyl-5,5′-di-isoxazolyl-methane by performing hydrogenolysis and subsequent hydrolysis with hydrochloric acid (12%) [2686].

m.p. 139° [2686]; ^{1}H NMR [2686], MS [2686].

1-(4,4′-Dihydroxy[1,1′-biphenyl]-3-yl)ethanone

[52189-90-9] $C_{14}H_{12}O_3$ mol.wt. 228.25



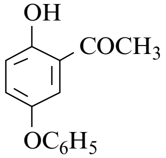
Syntheses

- Preparation by Fries rearrangement,
  - of 4-acetoxy-4′-hydroxybiphenyl with aluminium chloride without solvent for 6 h at 168–170° (57%) [3539];
  - of 4,4′-diacetoxybiphenyl with aluminium chloride in tetrachloroethane for 6 h at 160° (34%) [3539].
- Also obtained by irradiation of 4,4′-diphenoquinone [bi(cyclohexa-2,5-dienylidene)-4,4′-dione] in acetaldehyde for 2 days (28%) [3539].

m.p. 193° [3539]; ^{1}H NMR [3539], IR [3539], MS [3539].

1-(2-Hydroxy-5-phenoxyphenyl)ethanone

[56926-34-2] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

- Preparation by Fries rearrangement of 4-acetoxydiphenyl oxide in the presence of aluminium chloride at 120° (90%) [1852].
- Preparation by catalytic hydrogenolysis of 2-(benzyloxy)-5-phenoxyacetophenone at r.t. at 35 psi in the presence of 10% Pd/C in ethanol (85%) [3379].
- Preparation by adding an etheral solution of methyllithium to a solution of 5-phenoxysalicylic acid in 1,2-dimethoxyethane (79%) [3379].

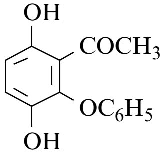
m.p. 160–161° [1852], 71–73° [3379].

One of the reported melting points is obviously wrong.

^{1}H NMR [3379].

1-(3,6-Dihydroxy-2-phenoxyphenyl)ethanone

[52095-10-0] $C_{14}H_{12}O_4$ mol.wt. 244.25



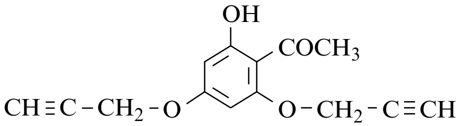
Synthesis

- Preparation by reaction of phenol on 2-acetylquinone with pyridine in benzene (40%) [2020].

m.p. 68–70° [2020]; ^{1}H NMR [2020], IR [2020].

1-[2-Hydroxy-4,6-bis(2-propynyloxy)phenyl]ethanone

[53771-23-6]



$C_{14}H_{12}O_4$ mol.wt. 244.25

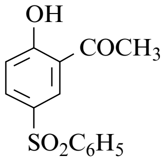
Synthesis

- Obtained by reaction of 2-propynyl bromide with phloroacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone in an atmosphere of nitrogen (12%) [3160].

m.p. 111° [3160]; IR [3160], UV [3160].

1-[2-Hydroxy-5-(phenylsulfonyl)phenyl]ethanone

[146575-61-3] $C_{14}H_{12}O_4S$ mol.wt. 276.31

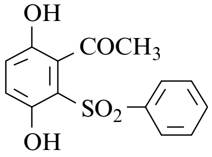


Synthesis

- Preparation by Fries rearrangement of 4-phenylsulfonylphenyl acetate with aluminium chloride at 190° (52%) [2478,2479].

1-[3,6-Dihydroxy-2-(phenylsulfonyl)phenyl]ethanone

[145746-54-9] $C_{14}H_{12}O_5S$ mol.wt. 292.31



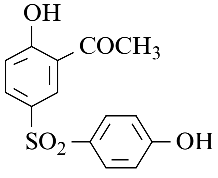
Synthesis

- Preparation by shaking an aqueous solution of sodium benzenesulfinate with a solution of 2-acetyl-1,4-benzoquinone and trifluoroacetic acid in methylene chloride for 4 h at r.t. (68%) [3540].

m.p. 182–183° [3540]; ^{1}H NMR [3540], IR [3540], MS [3540].

1-[2-Hydroxy-5-[(4-hydroxyphenyl)sulfonyl]phenyl]ethanone

[147816-50-0] $C_{14}H_{12}O_5S$ mol.wt. 292.31

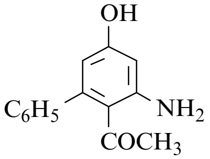


Syntheses

- Obtained by treatment of bisphenol-S diacetate (4,4′-diacetoxydiphenyl sulfone) with aluminium chloride (10 mol) at 160° (23%) [3541].
- Also obtained by UV light irradiation (254 nm) of bisphenol-S diacetate in acetonitrile for 5.5 h (18%) [3541].

1-(3-Amino-5-hydroxy[1,1′-biphenyl]-2-yl)ethanone

[54439-91-7] $C_{14}H_{13}NO_2$ mol.wt. 227.26



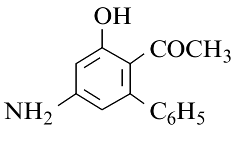
Synthesis

- Preparation from 5-methyl-3″-phenyl-3,5′-diisoxazolylmethane by performing hydrogenolysis and subsequent hydrolysis with hydrochloric acid (major product) [2686].

m.p. 192° [2686]; ^{1}H NMR [2686], MS [2686].

1-(5-Amino-3-hydroxy[1,1′-biphenyl]-2-yl)ethanone

[54439-90-6] $C_{14}H_{13}NO_2$ mol.wt. 227.26



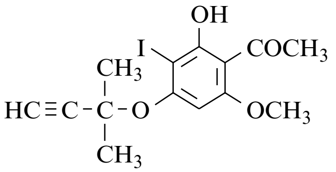
Synthesis

- Obtained (by-product) from 3′-methyl-5-phenyl-3,5′-di-isoxazolyl-methane by performing hydrogenolysis and subsequent hydrolysis with hydrochloric acid (4%) [2686].

m.p. 118° [2686]; ^{1}H NMR [2686], MS [2686].

1-[4-[(1,1-Dimethyl-2-propynyl)oxy]-2-hydroxy-3-iodo-6-methoxyphenyl]ethanone

[82538-75-8] $C_{14}H_{15}IO_4$ mol.wt. 374.17



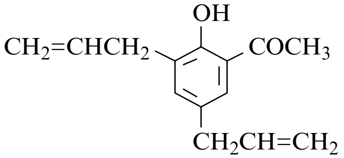
Synthesis

– Preparation by reaction of 3-chloro-3-methylbut-1-yne with 2,4-dihydroxy-3-iodo-6-methoxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (56%) [2064].

m.p. 110–111° [2064]; ^{1}H NMR [2064].

1-[2-Hydroxy-3,5-bis(2-propenyl)phenyl]ethanone

[35158-35-1] $C_{14}H_{16}O_2$ mol.wt. 216.28



Syntheses

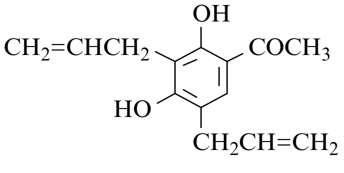
– Preparation by thermal Claisen rearrangement of 3-allyl-2-(allyloxy)acetophenone in N,N-diethylaniline for 4 h at 220° (93%) [2997].

– Preparation by reaction of ethyl acetoacetate with 2-allyl-2,6-heptadienal in the presence of pyridine and piperidine as catalysts, in benzene at 60–70° (33%). The 2-allyl-2,6-heptadienal was obtained by self condensation of 4-pentenal in the presence of 15% potassium hydroxide solution [2958–2960].

oil [2958–2960,2997];
b.p.$_{1-2}$ 90–95° [2958,2959,2960], b.p.$_{1.2}$ 100–120° [2997];
IR [2958–2960], UV [2958–2960].

1-[2,4-Dihydroxy-3,5-bis(2-propenyl)phenyl]ethanone

[40815-80-3] $C_{14}H_{16}O_3$ mol.wt. 232.28



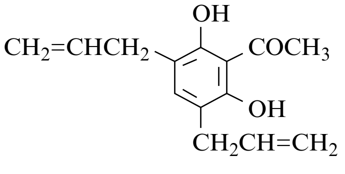
Syntheses

– Preparation by thermal Claisen rearrangement,
  - of 3-allyl-4-(allyloxy)-2-hydroxyacetophenone without solvent at 210° (20%) [3182];
  - of 3′-acetyl-4′,6′-bis(allyloxy)acetophenone in refluxing N,N-dimethylaniline (33%) [3331] or in refluxing phenyl ether (11%) [3331];
  - of 5′-acetyl-2′,4′-bis(allyloxy)-3′-bromoacetophenone in refluxing N,N-dimethylaniline (16%) [3331];
  - of 3′-acetyl-2′,4′-bis(allyloxy)acetophenone in refluxing N,N-dimethylaniline (18%) [3331];
  - of 2′,4′-bis(allyloxy)acetophenone in refluxing N,N-dimethylaniline (40%) [3331].

m.p. 90° [3331], 89–90° [3182]; ^{1}H NMR [3331], IR [3331], UV [3331], MS [3331].

1-[2,6-Dihydroxy-3,5-bis(2-propenyl)phenyl]ethanone

[37467-66-6]



$C_{14}H_{16}O_3$ mol.wt. 232.28

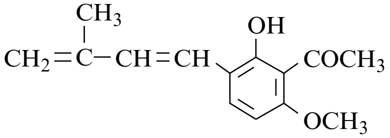
Syntheses

- Preparation by thermal Claisen rearrangement of,
  - 2′,6′-bis(allyloxy)acetophenone in refluxing N,N-dimethylaniline (45%) [3542] or in N,N-diethylaniline for 4 h at 220° [2997];
  - 3′-acetyl-4′,6′-bis(allyloxy)acetophenone in refluxing N,N-dimethylaniline or in phenyl ether (6–7%) [3331];
  - 3′-acetyl-2′,4′-bis(allyloxy)acetophenone in refluxing N,N-dimethylaniline (6%) [3331].

m.p. 57°5–58°5 [3542], 55–56° [2997]; b.p.$_{0.03}$ 150° [3542];
^{1}H NMR [3331], IR [3331], UV [3331], MS [3331].

1-[2-Hydroxy-6-methoxy-3-(3-methyl-1,3-butadienyl)phenyl]ethanone *(Z)*

[141215-43-2]



$C_{14}H_{16}O_3$ mol.wt. 232.28

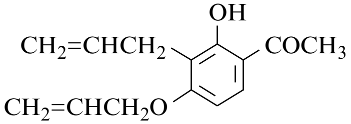
Synthesis

- Preparation by reaction of methylmagnesium iodide with 8-acetyl-7-methoxycoumarin in refluxing tetrahydrofuran during 1 h under nitrogen atmosphere (40%) [3543].

m.p. 112° [3543]; ^{1}H NMR [3543], IR [3543], UV [3543], MS [3543].

1-[2-Hydroxy-3-(2-propenyl)-4-(2-propenyloxy)phenyl]ethanone

[40903-02-4]



$C_{14}H_{16}O_3$ mol.wt. 232.28

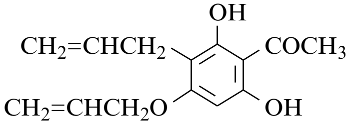
Synthesis

- Preparation by reaction of allyl bromide with 3-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (62%) [3182].

m.p. 34°5 [3182].

1-[2,6-Dihydroxy-3-(2-propenyl)-4-(2-propenyloxy)phenyl]ethanone

[53771-28-1] $C_{14}H_{16}O_4$ mol.wt. 248.28



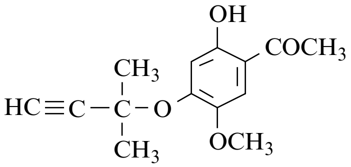
Synthesis

- Preparation by thermal Claisen rearrangement of 2,4-bis-(allyloxy)-6-hydroxyacetophenone in N-methylpiperazine at reflux in an atmosphere of nitrogen (53%) [3160] or without solvent in a sealed vial at 130° (33%) [3160].

m.p. 102–103°5 [3160]; UV [3160].

1-[4-[(1,1-Dimethyl-2-propynyl)oxy]-2-hydroxy-5-methoxyphenyl]ethanone

[70662-40-7] $C_{14}H_{16}O_4$ mol.wt. 248.28



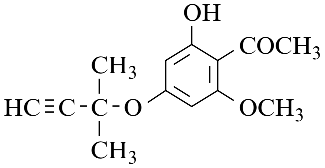
Synthesis

- Obtained by treatment of 2,4-dihydroxy-5-methoxy-acetophenone with 3-chloro-3-methyl-1-butyne in the presence of potassium carbonate and potassium iodide in DMF for 40 h at 80–85° (15%) [3544].

Yellow oil [3544]; TLC [3544]; ^{1}H NMR [3544], UV [3544].

1-[4-[(1,1-Dimethyl-2-propynyl)oxy]-2-hydroxy-6-methoxyphenyl]ethanone

[31273-60-6] $C_{14}H_{16}O_4$ mol.wt. 248.28



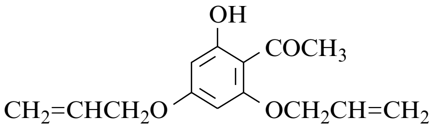
Synthesis

- Preparation by reaction of 2-methyl-2-chlorobut-3-yne on 2,4-dihydroxy-6-methoxy-acetophenone with potassium carbonate and potassium iodide in refluxing acetone (80%) [3545].

m.p. 107–108° [3545].

1-[2-Hydroxy-4,6-bis(2-propenyloxy)phenyl]ethanone

[53771-27-0] $C_{14}H_{16}O_4$ mol.wt. 248.28



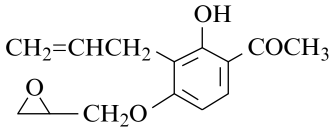
Synthesis

- Obtained by reaction of allyl bromide with phloroacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (10%) [3160].

m.p. 62°5 [3160]; UV [3160].

1-[2-Hydroxy-4-(oxiranylmethoxy)-3-(2-propenyl)phenyl]ethanone

[40785-92-0] $C_{14}H_{16}O_4$ mol.wt. 248.28



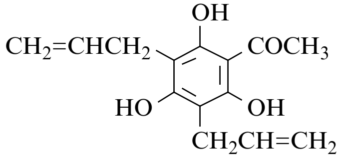
Synthesis

– Preparation by reaction of epichlorohydrin with 3-allyl-2,4-dihydroxyacetophenone in the presence of potassium hydroxide in refluxing ethanol [2270].

m.p. 67°5–68°5 [2270].

1-[2,4,6-Trihydroxy-3,5-bis(2-propenyl)phenyl]ethanone

[53771-29-2] $C_{14}H_{16}O_4$ mol.wt. 248.28



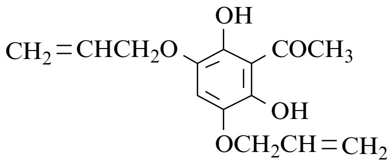
Synthesis

– Preparation by thermal Claisen rearrangement of 2,4-bis-(allyloxy)-6-hydroxyacetophenone in N,N-diethylaniline at reflux (215°) under nitrogen (87%) [3160].

m.p. 67–68° [3160]; UV [3160].

1-[2,6-Dihydroxy-3,5-bis(2-propenyloxy)phenyl]ethanone

[73331-27-8] $C_{14}H_{16}O_5$ mol.wt. 264.28

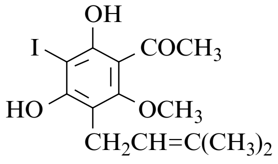


Synthesis

N.B.: In the paper [3542], the formula of the compound **59** displayed page 180, which is the formula of the titled compound $C_{14}H_{16}O_5$, does not fit at all with the one of the actually prepared compound. The Claisen rearrangement of 2,6-bis(allyloxy)acetophenone **58** leads to the formation of 3,5-diallyl-2,6-dihydroxyacetophenone $C_{14}H_{16}O_3$, page 183.

1-[2,4-Dihydroxy-3-iodo-6-methoxy-5-(3-methyl-2-butenyl)phenyl]ethanone

[82538-76-9] $C_{14}H_{17}IO_4$ mol.wt. 376.19



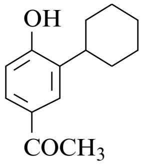
Synthesis

– Obtained by reaction of 2-methylbut-3-en-2-ol with 2,4-di-hydroxy-3-iodo-6-methoxyacetophenone in the presence of boron trifluoride etherate in dioxane at 35–40° (11%) [2064].

m.p. 136–137° [2064]; ^{1}H NMR [2064].

1-(3-Cyclohexyl-4-hydroxyphenyl)ethanone

[23299-00-5] $C_{14}H_{18}O_2$ mol.wt. 218.30



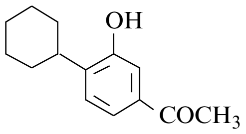
Synthesis

- Preparation by Fries rearrangement of 2-cyclohexylphenyl acetate with aluminium chloride in nitrobenzene at 85° [1951,3546], (55%) [1951].

m.p. 148–149° [3546], 147–148° [1951].

1-(4-Cyclohexyl-3-hydroxyphenyl)ethanone

[73898-21-2] $C_{14}H_{18}O_2$ mol.wt. 218.30



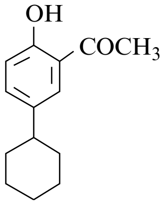
Synthesis

- Preparation by diazotization of 3-amino-4-cyclohexyl-acetophenone, followed by hydrolysis of diazonium salt obtained (48%) [1951].

m.p. 171° (Sadtler), 165–167° [1951];
^{1}H NMR (Sadtler: standard n° 44602 M); IR (Sadtler: standard n° 71630 K).

1-(5-Cyclohexyl-2-hydroxyphenyl)ethanone

[55168-33-7] $C_{14}H_{18}O_2$ mol.wt. 218.30



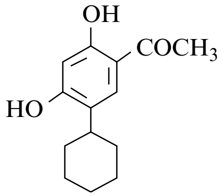
Syntheses

- Preparation by reaction of acetic acid with p-cyclohexylphenol in the presence of boron trifluoride at 75° for 24 h (91%) [3514].
- Preparation by Fries rearrangement of p-cyclohexylphenyl acetate with aluminium chloride without solvent at 140° [2899] or at 170° [3547], (47%) [2899].

Colourless oil [2899,3547]; m.p. < 50° [3514];
^{1}H NMR [2899].

1-(5-Cyclohexyl-2,4-dihydroxyphenyl)ethanone

[159977-36-3] $C_{14}H_{18}O_3$ mol.wt. 234.30

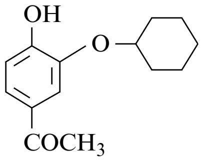


Synthesis

- Preparation by reaction of acetic acid with 4-cyclohexylresorcinol in the presence of boron trifluoride etherate at 105–108° for 15 min, followed by hydrolysis of the obtained boron difluoride chelate (m.p. 207–208°) with boiling aqueous ethanol for 15–20 min (89%) [3548].

m.p. 145–146° [3548]; IR [3548], UV [3548].

1-[3-(Cyclohexyloxy)-4-hydroxyphenyl]ethanone



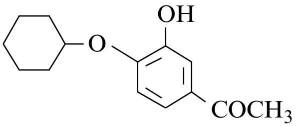
$C_{14}H_{18}O_3$ mol.wt. 234.30

Syntheses

- Preparation by catalytic hydrogenolysis of 4-(benzyloxy)-3-cyclohexyloxyacetophenone in the presence of Pd/C in ethanol at r.t. (82%) [2248].
- Also obtained (poor yield) by reaction of cyclohexyl bromide with 3,4-dihydroxyacetophenone in the presence of sodium hydroxide in a refluxing mixture of ethanol and methanol (2%) [2248].

m.p. 88° [2248].

1-[4-(Cyclohexyloxy)-3-hydroxyphenyl]ethanone



$C_{14}H_{18}O_3$ mol.wt. 234.30

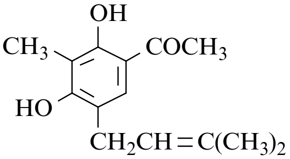
Synthesis

- Obtained (poor yield) by reaction of cyclohexyl bromide with 3,4-dihydroxyacetophenone in the presence of sodium hydroxide in a refluxing mixture of ethanol and methanol (8%) [2248].

m.p. 103° [2248].

1-[2,4-Dihydroxy-3-methyl-5-(3-methyl-2-butenyl)phenyl]ethanone

[74727-08-5]



$C_{14}H_{18}O_3$ mol.wt. 234.30

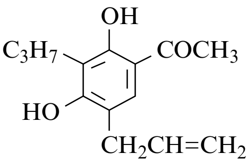
Synthesis

- Obtained by prenylation of 2,4-dihydroxy-3-methyl-acetophenone with 2-methyl-3-buten-2-ol in dioxane in the presence of boron trifluoride etherate for 1 h at r.t. (41%) [3549].

m.p. 117–118° [3549]; TLC [3549];
^{1}H NMR [3549], IR [3549].

1-[2,4-Dihydroxy-5-(2-propenyl)-3-propylphenyl]ethanone

[99370-48-6]



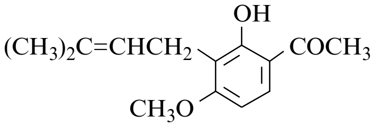
$C_{14}H_{18}O_3$ mol.wt. 234.30

Synthesis

- Preparation by thermal Claisen rearrangement of 4-(allyloxy)-2-hydroxy-3-propylacetophenone without solvent at 210° (79%) [2671].

1-[2-Hydroxy-4-methoxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[52601-06-6] $C_{14}H_{18}O_3$ mol.wt. 234.30



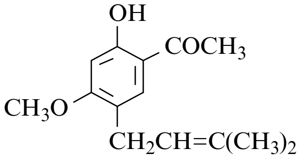
Synthesis

- Preparation by reaction of 2-methylbut-3-en-2-ol with 2-hydroxy-4-methoxyacetophenone in the presence of boron trifluoride etherate [2326].

^1H NMR [2326,2327], UV [2326].

1-[2-Hydroxy-4-methoxy-5-(3-methyl-2-butenyl)phenyl]ethanone

[28448-83-1] $C_{14}H_{18}O_3$ mol.wt. 234.30



Syntheses

- Preparation by reaction of dimethyl sulfate with 2,4-di-hydroxy-5-prenylacetophenone in the presence of potassium carbonate in refluxing acetone (89%) [3453].
- Preparation by reaction of 2-methylbut-3-en-2-ol with 2-hydroxy-4-methoxyacetophenone in the presence of boron trifluoride etherate [2326].

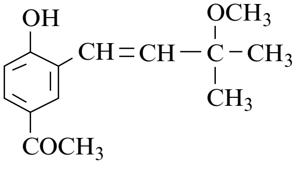
Isolation from natural sources

- By treatment of bavachinin with 50% potassium hydroxide aqueous solution at 180–200° (80%). The bavachinin is a flavonoid compound obtained from *Psoralea Corylifolia* Linn. [3460].

Oil [3453,3460]; ^1H NMR [2326,2327,3460], IR [3460], UV [2326,3460].

1-[4-Hydroxy-3-(3-methoxy-3-methyl-1-butenyl)phenyl]ethanone *(E)*

[26931-60-2] $C_{14}H_{18}O_3$ mol.wt. 234.30



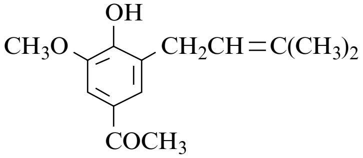
Isolation from natural sources

- From the roots of *Helianthella uniflor*a (tribe Heliantheae) [3433].

m.p. 124° [3433];
^1H NMR [3433], IR [3433], UV [3433], MS [3433].

1-[4-Hydroxy-3-methoxy-5-(3-methyl-2-butenyl)phenyl]ethanone

[73869-90-6] $C_{14}H_{18}O_3$ mol.wt. 234.30



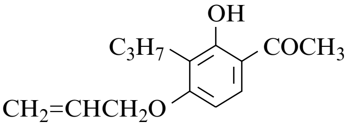
Synthesis

- Obtained by Claisen rearrangement of 3-methoxy-4-(3-methyl-2-butenyloxy) acetophenone (m.p. 41°5) in diethylaniline for 5 h at 170° (85%) [3434].

m.p. 65° [3434]; ^1H NMR [3434], IR [3434].

1-[2-Hydroxy-4-(2-propenyloxy)-3-propylphenyl]ethanone

[99370-47-5] $C_{14}H_{18}O_3$ mol.wt. 234.30



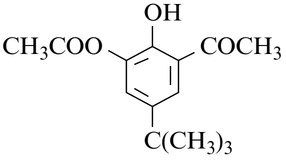
Synthesis

- Preparation by reaction of allyl bromide with 2,4-di-hydroxy-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing methyl ethyl ketone (78%) [2671].

Crystalline compound [2671].

1-[3-(Acetyloxy)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone

[84297-04-1] $C_{14}H_{18}O_4$ mol.wt. 250.29



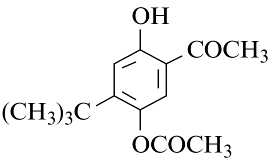
Syntheses

- Obtained by treatment of a benzene solution of 5-tert-butyl-2,3-dihydroxyacetophenone and acetaldehyde in the presence of di-tert-butyl diperoxyoxalate at 38° (25%) [3386].
- Also obtained by treatment of a benzene solution of 4-tert-butyl-o-benzoquinone and acetaldehyde in the presence of di-tert-butyl diperoxyoxalate at 38° (6%) [3386].

Pale yellow oil [3386]; ^{1}H NMR [3386], IR [3386].

1-[5-(Acetyloxy)-4-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone

[107188-26-1] $C_{14}H_{18}O_4$ mol.wt. 250.29



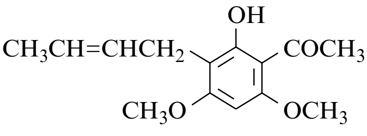
Synthesis

- Preparation by reaction of acetic anhydride with 2-tert-butylhydroquinone in the presence of boron trifluoride–acetic acid complex between 60° and 90° [3251].

m.p. 86°5–87°5 [3251].

1-[3-(2-Butenyl)-2-hydroxy-4,6-dimethoxyphenyl]ethanone

$C_{14}H_{18}O_4$ mol.wt. 250.29



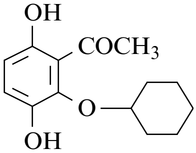
Synthesis

- Preparation by condensation of 2-hydroxy-4,6-di-methoxyacetophenone with 1,3-butadiene in the presence of 85% orthophosphoric acid in xylene at 30–35° (80%) [3325].

m.p. 101–103° [3325]; ^{1}H NMR [3325].

1-[2-(Cyclohexyloxy)-3,6-dihydroxyphenyl]ethanone

[33537-80-3] $C_{14}H_{18}O_4$ mol.wt. 250.29



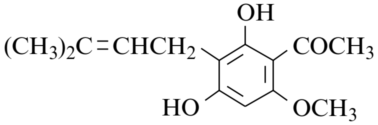
Syntheses

- Easy preparation by reduction of 2-acetyl-3-cyclohexyloxy-1,4-benzoquinone using conventional methods [2869].
- Also obtained (low yield) by reaction of 2-acetyl-1,4-benzoquinone with an excess of cyclohexanol at r.t., with exclusion of light [2869].

m.p. 75–76°5 [2869]; ^{1}H NMR [2869], IR [2869].

1-[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[30403-01-1] $C_{14}H_{18}O_4$ mol.wt. 250.29



Synthesis

- Preparation by reaction of prenyl bromide with 2,4-dihydroxy-6-methoxy-acetophenone in the presence of methanolic potassium hydroxide (major product) [2834], (27%) [2835].

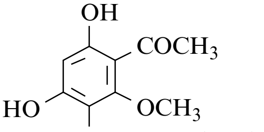
Isolation from natural sources

- From the rhizomes of *Remirea maritima* Aubl. (Cyperaceae) [2834,3550].

m.p. 173°5–174° [3550], 173–174° [2834], 170–171° [2835]; TLC [2835]; ^{1}H NMR [2834,2835], UV [2834,2835].

1-[4,6-Dihydroxy-2-methoxy-3-(3-methyl-2-butenyl)phenyl]ethanone
(Acronylin)

[27364-64-3] $C_{14}H_{18}O_4$ mol.wt. 250.29



Syntheses

- Preparation by prenylation of 2,4-dihydroxy-6-methoxy-acetophenone with 2-methyl-3-buten-2-ol in the presence of boron trifluoride etherate [2834].
- Preparation by dehalogenation of 2,4-dihydroxy-3-iodo-6-methoxy-5-prenylacetophenone in the presence of zinc dust and concentrated hydrochloric acid in refluxing ethanol (75%) [2064].

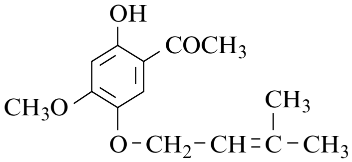
Isolation from natural sources

- From the bark of *Acronychia laurifolia* BL (Rutaceae) [3476].

m.p. 128–129° [3476,3550], 127–128° [2834];
^{1}H NMR [2834,3476], IR [3476], UV [2834,3476], MS [3476].

1-[2-Hydroxy-4-methoxy-5-[(3-methyl-2-butenyl)oxy]phenyl]ethanone

[142608-87-5]



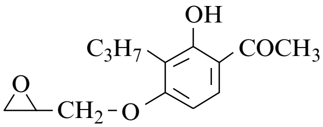
$C_{14}H_{18}O_4$ mol.wt. 250.29

Synthesis

- Preparation by prenylation of 2,5-dihydroxy-4-methoxy-acetophenone [3551].

1-[2-Hydroxy-4-(oxiranylmethoxy)-3-propylphenyl]ethanone

[57161-85-0]



$C_{14}H_{18}O_4$ mol.wt. 250.29

Syntheses

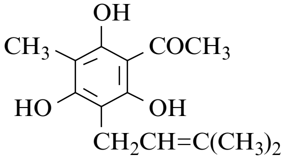
- Preparation by reaction of epichlorohydrin with 2,4-di-hydroxy-3-propylacetophenone in the presence of,

- potassium hydroxide in refluxing ethanol (64%) [2270];
- potassium carbonate in boiling 2-butanol for 10 h (62%) [3479].

m.p. 54–55° [2270], 52–56°5 [3479]; b.p.$_{0.5}$ 170–175° [2270]; GLC [3479]; ^{1}H NMR [3479].

1-[2,4,6-Trihydroxy-3-methyl-5-(3-methyl-2-butenyl)phenyl]ethanone

[55380-58-0]



$C_{14}H_{18}O_4$ mol.wt. 250.29

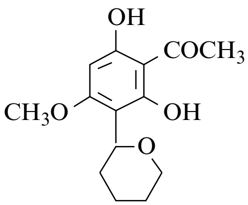
Synthesis

- Obtained by reaction of 2-methylbut-3-en-2-ol with methylphloroacetophenone in the presence of boron trifluoride etherate in dioxane (16%) [3401].

m.p. 115–117° [2879,3401], 114–116° [3500];
^{1}H NMR [2879,3401], UV [3401], MS [3401].

1-[2,6-Dihydroxy-4-methoxy-3-(tetrahydro-2*H*-pyran-2-yl)phenyl]ethanone

[136258-10-1]



$C_{14}H_{18}O_5$ mol.wt. 266.29

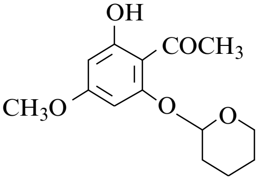
Synthesis

- Preparation by reaction of 3,4-dihydro-2*H*-pyran on 2,6-dihydroxy-4-methoxyacetophenone with p-toluene-sulfonic acid in dioxane, first at 0°, then at r.t. (29%) [2368].

m.p. 71–76° [2368]; ^{1}H NMR [2368], ^{13}C NMR [2368].

1-[2-Hydroxy-4-methoxy-6-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone

[136258-09-8] $C_{14}H_{18}O_5$ mol.wt. 266.29



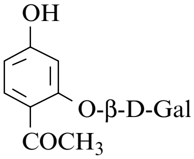
Synthesis

- Preparation by reaction of 3,4-dihydro-2*H*-pyran on 2,6-dihydroxy-4-methoxyacetophenone with p-toluene-sulfonic acid in dioxane, first at 0°, then at r.t. (29%) [2368].

m.p. 88–92° [2368]; ^{1}H NMR [2368], ^{13}C NMR [2368].

1-[2-(β-D-Galactopyranosyloxy)-4-hydroxyphenyl]ethanone

$C_{14}H_{18}O_8$ mol.wt. 314.29



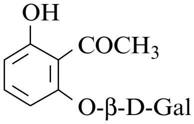
Synthesis

- Preparation from 2-(2,3,4,6-tetra-O-acetyl-β-D-galactopyranosyloxy)-4-hydroxyacetophenone by boiling in 0.2 N methanolic sodium methoxide solution for 3 min (45%) [3552].

m.p. 114–115° [3552]; monohydrate [3552];
$(\alpha)_D^{20} = -73°$ (c = 1 in water) [3552].

1-[2-(β-D-Galactopyranosyloxy)-6-hydroxyphenyl]ethanone

[88087-04-1] $C_{14}H_{18}O_8$ mol.wt. 314.29



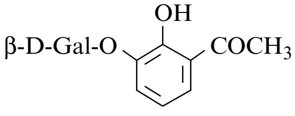
Synthesis

- Obtained by deprotection of 2-(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyloxy)-6-hydroxyacetophenone with 0.5 N methanolic sodium methoxide at r.t. overnight (20%) [2842].

m.p. 226–229° [2842];
$(\alpha)_D^{20} = -4°8$ (c = 1.67 in pyridine) [2842]; ^{1}H NMR [2842].

1-[3-(β-D-Galactopyranosyloxy)-2-hydroxyphenyl]ethanone

[88086-97-9] $C_{14}H_{18}O_8$ mol.wt. 314.29



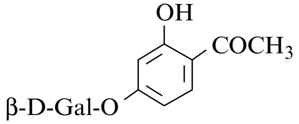
Synthesis

- Obtained from 3-(2,3,4,6-tetra-O-acetyl-β-D-galactopyrano-syloxy)-2-hydroxyacetophenone by treatment with 0.1 N methanolic sodium methoxide for 3 h at r.t. [2842].

m.p. 179–181° [2842]; dihydrate [2842];
$(\alpha)_D^{20} = -7°$ (c = 1 in pyridine) [2842]; ^{1}H NMR [2842].

1-[4-(β-D-Galactopyranosyloxy)-2-hydroxyphenyl]ethanone

[54918-26-2] $C_{14}H_{18}O_8$ mol.wt. 314.29



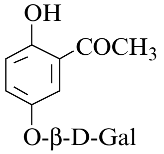
Syntheses

- Obtained from 4-(2,3,4,6-tetra-O-acetyl-β-D-galactopyrano-syloxy)-2-hydroxyacetophenone by treatment with methanolic sodium methoxide at r.t. [1936,2842], (87%) (monohydrate) [1936] or in boiling methanol for 3 min [3552].

m.p. 207° [2842], 205–207° [3552], 198° [1936];
$(\alpha)_D^{18} = -62°$ (c = 2.3 in pyridine) [1936];
$(\alpha)_D^{22} = -68°3$ (c = 1 in water) (monohydrate) [3552];
$(\alpha)_D^{22} = -73°$ (c = 1 in water) (anhydrous) [3552].

1-[5-(β-D-Galactopyranosyloxy)-2-hydroxyphenyl]ethanone

[88086-99-1] $C_{14}H_{18}O_8$ mol.wt. 314.29



Synthesis

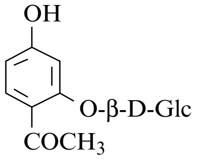
- Obtained from 5-(2,3,4,6-tetra-O-acetyl-β-D-galactopyrano-syloxy)-2-hydroxyacetophenone by treatment with 0.1 N methanolic sodium hydroxide solution for 2 h (22%) [2842].

m.p. 209–211° [2842]; ^{1}H NMR [2842].

1-[2-(β-D-Glucopyranosyloxy)-4-hydroxyphenyl]ethanone

(Cynanoneside B; Bungeiside B)

[149561-88-6] $C_{14}H_{18}O_8$ mol.wt. 314.29



Synthesis

- Preparation from 2-(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyloxy)-4-hydroxyacetophenone by boiling in 0.2 N methanolic sodium methoxide solution for 3 min (65%) [3553].

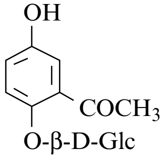
Isolation from natural sources

- From the roots of *Cynanchum bungei* DECNE (Asclepiadaceae) [3554].
- From the roots of *Cynanchum taiwanianum* (Asclepiadaceae) [3555].

Amorphous white powder [3554], Amorphous solid [3555];
m.p. 178–180° [3553];
$(\alpha)_D^{20} = -88°5$ (c = 1 in water) [3553],
$(\alpha)_D^{20} = -11°0$ (c = 1 in ethanol) [3555],
$(\alpha)_D = -17°3$ (c = 0.7 in methanol) [3554]; TLC [3553];
^{1}H NMR [3554,3555], ^{13}C NMR [3554,3555], IR [3555], UV [3555], MS [3554,3555].

1-[2-(β-D-Glucopyranosyloxy)-5-hydroxyphenyl]ethanone *(Bungeiside A)*

[149475-52-5] $C_{14}H_{18}O_8$ mol.wt. 314.29



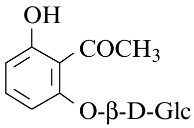
Isolation from natural sources

- From the roots of *Cynanchum bungei* DECNE (Asclepiadaceae) [3554].

white amorphous powder [3554];
$(\alpha)_D$ = −40°6 (c = 5 in methanol) [3554];
^{1}H NMR [3554], ^{13}C NMR [3554], IR [3554], MS [3554].

1-[2-(β-D-Glucopyranosyloxy)-6-hydroxyphenyl]ethanone

$C_{14}H_{18}O_8$ mol.wt. 314.29

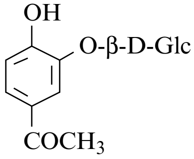


Synthesis

- Refer to: [3556].

1-[3-(β-D-Glucopyranosyloxy)-4-hydroxyphenyl]ethanone

[55483-00-6] $C_{14}H_{18}O_8$ mol.wt. 314.29



Synthesis

- Preparation by bubbling dry ammonia into an ice cooled solution of 3-(tetra-O-acetyl-β-D-glucopyranosyloxy)-4-hydroxyacetophenone in methanol for 40 min. Then, the solution was set aside at r.t. for 15 h (72%) [3410].

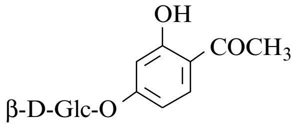
Isolation from natural sources

- From the leaves of *Picea pungens* Engelm. (Colorado spruce) [3557,3558].
- From the leaves of *Picea pungens* Engelm. var. glauca Beiss. [3559].
- From shoots of *Picea abies* [3560].
- From white spruce of *Picea glauca* (Moench) Voss [3559].
- From galls and shoots of *Picea glauca* (Pinaceae) [3561].

m.p. 198–199° [3557], 190–195° [3410], 190–191° [3559];
$(\alpha)_D^{24}$ = −96°8 (c = 0.53 in water) [3557], $(\alpha)_D$ = −88°9 (c = 0.53 in water) [3410],
$(\alpha)_D^{30}$ = −85.47 (c = 1.17 in water) [3559]; GC [3560], GC/MS [3560,3561];
^{1}H NMR [3410,3562], IR [3410], MS [3410].

1-[4-(β-D-Glucopyranosyloxy)-2-hydroxyphenyl]ethanone

[54918-24-0] $C_{14}H_{18}O_8$ mol.wt. 314.29



Syntheses

- Preparation by deacetylation of 4-(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyloxy)-2-hydroxyacetophenone,

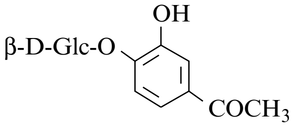
- with sodium methoxide in methanol (92%) [3563], (70–80%) [2528], (60–70%) [3553];
- with barium hydroxide in aqueous solution for 16 h (84%) [2736].

– Also refer to: [1936,3564].

m.p. 201–202° [2736,3553], 198–200° [3563];
$(\alpha)_D^{20}$ = −86°9 (c = 50 in acetone) [3563]; TLC [3553].

1-[4-(β-D-Glucopyranosyloxy)-3-hydroxyphenyl]ethanone *(Cynanoneside A)*

[17063-43-3] $C_{14}H_{18}O_8$ mol.wt. 314.29



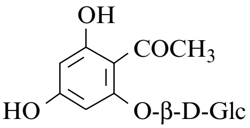
Isolation from natural sources

- From the shoots of *Picea abies* [3560].
- From the roots of *Cynanchum taiwanianum* (Asclepiadaceae) [3555].
- From galls and shoots of *Picea glauca* (Pinaceae) [3561].

Amorphous solid [3555];
$(\alpha)_D^{20}$ = −5°0 (c = 1 in ethanol) [3555];
GC [3560]; GC/MS [3560,3561];
^{1}H NMR [3555], ^{13}C NMR [3555], IR [3555], UV [3555], MS [3555].

1-[2-(β-D-Glucopyranosyloxy)-4,6-dihydroxyphenyl]ethanone

[26089-54-3] $C_{14}H_{18}O_9$ mol.wt. 330.29



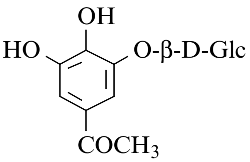
Isolation from natural sources

- From the aerial parts of *Artemisia stolonifera* (Compositae) [3565].

m.p. 201–203° [3565];
^{1}H NMR [3565], ^{13}C NMR [3565], IR [3565], UV [3565].

1-[3-(β-D-Glucopyranosyloxy)-4,5-dihydroxyphenyl]ethanone

[448949-59-5] $C_{14}H_{18}O_9$ mol.wt. 330.29

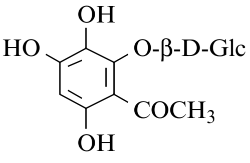


Isolation from natural sources

- From dried root of *Polygonum cuspidatum* SIEB. et ZUCC. (Polygonaceae) [3566].
- Also refer to: [3565].

1-[2-(β-D-Glucopyranosyloxy)-3,4,6-trihydroxyphenyl]ethanone *(Lalioside)*

[116964-03-5] $C_{14}H_{18}O_{10}$ mol.wt. 346.29



Isolation from natural sources

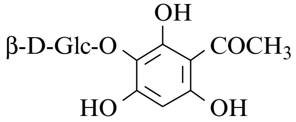
- From *Lawsonia inermis* [3567].

spectroscopic data [3567].

1-[3-(β-D-Glucopyranosyloxy)-2,4,6-trihydroxyphenyl]ethanone

(Polygoacetophenoside)

[110906-84-8] $C_{14}H_{18}O_{10}$ mol.wt. 346.29



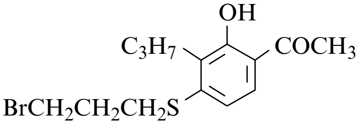
Isolation from natural sources

- From the leaves and stems of *Polygonum multiflorum* (Polygonaceae) [3309].

m.p. 214–216° [3309];
^{1}H NMR [3309], ^{13}C NMR [3309], IR [3309];
UV [3309], MS [3309], HRMS [3309].

1-[4-[(3-Bromopropyl)thio]-2-hydroxy-3-propylphenyl]ethanone

[125617-43-8] $C_{14}H_{19}BrO_2S$ mol.wt. 331.27



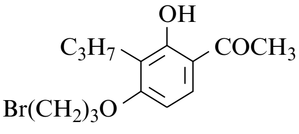
Synthesis

- Preparation by reaction of 1,3-dibromopropane with 2-hydroxy-4-mercapto-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing methyl ethyl ketone [2271].

Yellow oil [2271].

1-[4-(3-Bromopropoxy)-2-hydroxy-3-propylphenyl]ethanone

[40786-20-7] $C_{14}H_{19}BrO_3$ mol.wt. 315.21



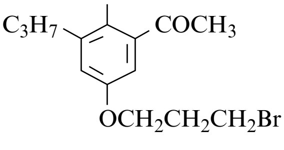
Synthesis

- Preparation by reaction of 1,3-dibromopropane with 2,4-di-hydroxy-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone [2270].

b.p.$_{0.02}$ 172–180° [2270].

1-[5-(3-Bromopropoxy)-2-hydroxy-3-propylphenyl]ethanone

[125617-25-6] $C_{14}H_{19}BrO_3$ mol.wt. 315.21



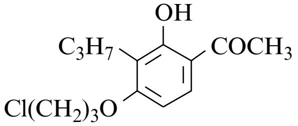
Synthesis

- Preparation by reaction of 1,3-dibromopropane with 2,5-di-hydroxy-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing methyl ethyl ketone [2271].

m.p. 69–70° [2271].

1-[4-(3-Chloropropoxy)-2-hydroxy-3-propylphenyl]ethanone

[79558-02-4] $C_{14}H_{19}ClO_3$ mol.wt. 270.76



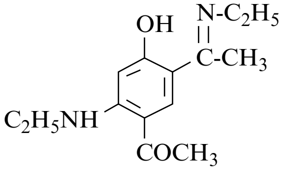
Syntheses

- Preparation by reaction of 1-bromo-3-chloropropane with 2,4-dihydroxy-3-propylacetophenone in the presence of potassium carbonate in refluxing acetone for 5 h (80%) [3204], (67%) [3479].
- Also refer to: [3568].

m.p. 39–41° [3479], 37–38° [3204]; GLC [3479]; ^{1}H NMR [3479].

1-[2-(Ethylamino)-5-[1-(ethylimino)ethyl]-4-hydroxyphenyl]ethanone

[115349-97-8] $C_{14}H_{20}N_2O_2$ mol.wt. 248.33



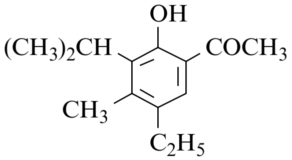
Synthesis

- Obtained by heating a mixture of 4,6-diacetylresorcinol, aqueous ethylamine and concentrated hydrochloric acid as catalyst, in an autoclave from 8 to 72 h [3569].

m.p. 153–154° [3569]; ^{1}H NMR [3569], MS [3569].

1-[5-Ethyl-2-hydroxy-4-methyl-3-(1-methylethyl)phenyl]ethanone

$C_{14}H_{20}O_2$ mol.wt. 220.31



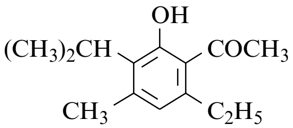
Synthesis

- Preparation by chromic degradation of 5-ethyl-2,3,6-tri-methyl-7-isopropylbenzofuran (52%) [3224].

b.p.$_{16}$ 164–167° [3224].

1-[6-Ethyl-2-hydroxy-4-methyl-3-(1-methylethyl)phenyl]ethanone

$C_{14}H_{20}O_2$ mol.wt. 220.31



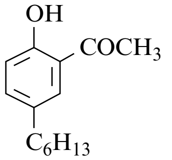
Synthesis

- Preparation by chromic degradation of 4-ethyl-2,3,6-tri-methyl-7-isopropylbenzofuran (46%) [3224].

b.p.$_{18}$ 165–166° [3224].

1-(5-Hexyl-2-hydroxyphenyl)ethanone

[55168-32-6] $C_{14}H_{20}O_2$ mol.wt. 220.31



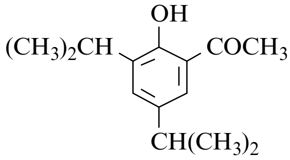
Synthesis

- Preparation by Fries rearrangement of p-n-hexylphenyl acetate with aluminium chloride without solvent (15%) [2899].

yellow oil [2899]; b.p.$_{0.7}$ 110° [2899]; MS [2899].

1-[2-Hydroxy-3,5-bis(1-methylethyl)phenyl]ethanone

[35158-23-7] $C_{14}H_{20}O_2$ mol.wt. 220.31



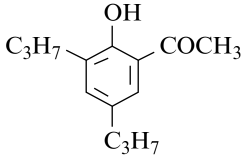
Synthesis

- Preparation by reaction of ethyl acetoacetate with 5-methyl-2-isopropyl-2-hexenal in the presence of pyridine and piperidine as catalysts in refluxing benzene (40%). The 5-methyl-2-isopropyl-2-hexenal was obtained by self-condensation of isovaleraldehyde in the presence of 15% potassium hydroxide solution [2958–2960].

m.p. 37° [2958–2960]; b.p.$_{0.05-0.10}$ 70–80° [2958–2960];
^{1}H NMR [2958–2960], IR [2958–2960], UV [2958–2960].

1-(2-Hydroxy-3,5-dipropylphenyl)ethanone

[35198-96-0] $C_{14}H_{20}O_2$ mol.wt. 220.31



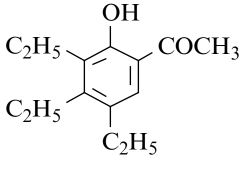
Synthesis

- Preparation by reaction of ethyl acetoacetate with 2-n-propyl-2-heptenal in the presence of pyridine and piperidine as catalysts in refluxing benzene (42%).
- The 2-n-propyl-2-heptenal was obtained by self-condensation of n-valeraldehyde in the presence of 15% potassium hydroxide solution [2958–2960].

liquid [2958–2960]; b.p.$_{0.075}$ 80–90° [2958–2960];
^{1}H NMR [2958–2960], IR [2958–2960], UV [2958–2960].

1-(3,4,5-Triethyl-2-hydroxyphenyl)ethanone

$C_{14}H_{20}O_2$ mol.wt. 220.31



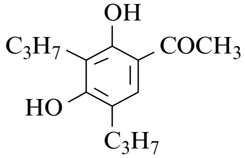
Synthesis

- Preparation by Fries rearrangement of 2,4,6-triethylphenyl acetate with aluminium chloride *via* a migration of two ethyl groups (65%) [2233].

b.p.$_{12}$ 153–155° [2233].

1-(2,4-Dihydroxy-3,5-dipropylphenyl)ethanone

[72018-36-1] $C_{14}H_{20}O_3$ mol.wt. 236.31



Syntheses

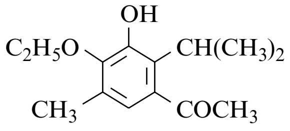
- Preparation by hydrogenation of 2,4-dihydroxy-3,5-di-propenylacetophenone in ethanol using 10% PdO/C as catalyst (98%) [3184].
- Also refer to: [2326,2327].

N.B.: Pr indicates the *propyl* group -C_3H_7 in Chem. Abstr., **92**, 6368x (1980) and **98**, 54239b (1983), as usual abbreviation. However, in the two references [2326,2327], Pr represented the *prenyl* group –$CH_2CH=C(CH_3)_2$. Therefore they concern the 2,4-dihydroxy-3,5-diprenylacetophenone and not the above mentioned 2,4-dihydroxy-3,5-dipropylacetophenone.

m.p. 98–99° [3184].

1-[4-Ethoxy-3-hydroxy-5-methyl-2-(1-methylethyl)phenyl]ethanone

$C_{14}H_{20}O_3$ mol.wt. 236.31



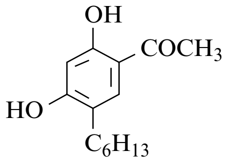
Synthesis

- Preparation by diazotization of 3-amino-4-ethoxy-5-methyl-2-isopropylacetophenone, followed by hydrolysis of the diazonium salt obtained (44%) [3327].

m.p. 88° [3327].

1-(5-Hexyl-2,4-dihydroxyphenyl)ethanone

[63411-88-1] $C_{14}H_{20}O_3$ mol.wt. 236.31



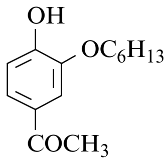
Syntheses

- Preparation by reaction of acetonitrile on 4-n-hexylresorcinol (Hoesch reaction) (84%) [2676].
- Preparation from 5-hexyl-2,4-dimethoxyacetophenone by demethylation with boron tribromide in methylene chloride at r.t. (70%) [2678,2679].

m.p. 86–87° [2676].

1-[3-(Hexyloxy)-4-hydroxyphenyl]ethanone

$C_{14}H_{20}O_3$ mol.wt. 236.31



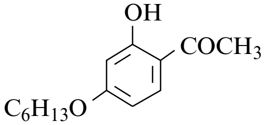
Synthesis

- Preparation from 4-(benzyloxy)-3-hexyloxyacetophenone by catalytic debenzylation on Pd/C (89%) [2946].

m.p. 48° [2946].

1-[4-(Hexyloxy)-2-hydroxyphenyl]ethanone

[143286-85-5] $C_{14}H_{20}O_3$ mol.wt. 236.31



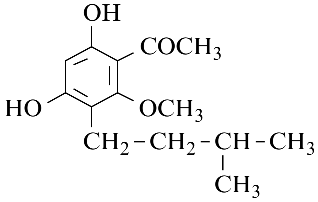
Synthesis

– Preparation by partial alkylation of resacetophenone with hexyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [3493].

m.p. 22° [3493].

1-[4,6-Dihydroxy-2-methoxy-3-(3-methylbutyl)phenyl]ethanone

[14764-76-2] $C_{14}H_{20}O_4$ mol.wt. 252.31



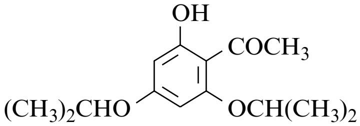
Synthesis

– Preparation from acronylin (4,6-dihydroxy-2-methoxy-3-isopentenylacetophenone) by catalytic reduction with Pd/C as catalyst in ethyl acetate [3476].

m.p. 143° [3476]; ^{1}H NMR [3476].

1-[2-Hydroxy-4,6-bis(1-methylethoxy)phenyl]ethanone

[93344-48-0] $C_{14}H_{20}O_4$ mol.wt. 252.31



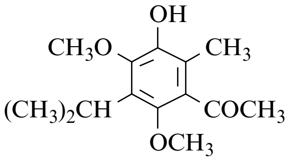
Synthesis

– Preparation by reaction of 2-bromopropane with phloroacetophenone in the presence of potassium carbonate in DMF at reflux (67%) [2830].

dark red oil [2830]; ^{1}H NMR [2830], IR [2830], MS [2830].

1-[3-Hydroxy-4,6-dimethoxy-2-methyl-5-(1-methylethyl)phenyl]ethanone

[159848-01-8] $C_{14}H_{20}O_4$ mol.wt. 252.31

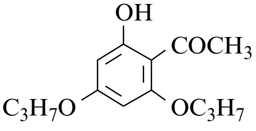


Synthesis

– Obtained by reaction of 3-chloroperbenzoic acid with 2,4-dimethoxy-6-methyl-3-isopropylacetophenone in refluxing ethylene dichloride for 48 h (34%) [3570].

m.p. 98–100° [3570]; ^{1}H NMR [3570].

1-[2-Hydroxy-4,6-bis(propyloxy)phenyl]ethanone



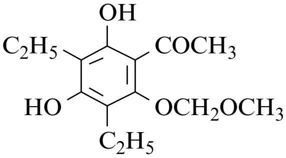
$C_{14}H_{20}O_4$ mol.wt. 252.31

Synthesis

- Preparation by catalytic hydrogenation of 2-hydroxy-4,6-bis(2-propynyloxy)acetophenone in the presence of 10% Pd/C in methanol [3160].

1-[3,5-Diethyl-2,4-dihydroxy-6-(methoxymethoxy)phenyl]ethanone

[175785-90-7]



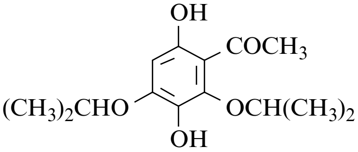
$C_{14}H_{20}O_5$ mol.wt. 268.31

Synthesis

- Refer to: [3495] (Japanese patent).

1-[3,6-Dihydroxy-2,4-bis(1-methylethoxy)phenyl]ethanone

[93344-49-1]



$C_{14}H_{20}O_5$ mol.wt. 268.31

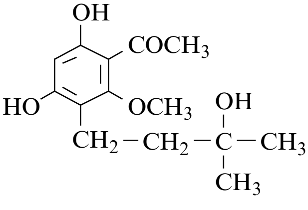
Synthesis

- Preparation from 2-hydroxy-4,6-diisopropoxy-acetophenone by persulfate oxidation (Elbs reaction) (12%) [2830].

oil [2830]; ^1H NMR [2830], IR [2830], MS [2830].

1-[4,6-Dihydroxy-3-(3-hydroxy-3-methylbutyl)-2-methoxyphenyl]ethanone

[153399-41-8]



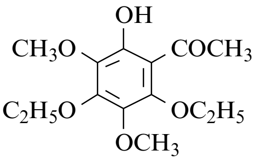
$C_{14}H_{20}O_5$ mol.wt. 268.31

Synthesis

- Obtained by catalytic hydrogenation of 4,6-bis(benzyloxy)-3-(3-hydroxy-3-methylbutynyl)-2-methylacetophenone in methanol in the presence of Pd/C at 20° [3571].

m.p. 156–158° [3571]; ^1H NMR [3571].

1-(2,4-Diethoxy-6-hydroxy-3,5-dimethoxyphenyl)ethanone



$C_{14}H_{20}O_6$ mol.wt. 284.31

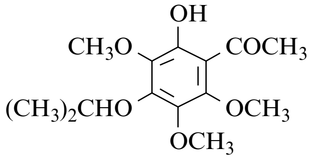
Synthesis

- Preparation by Friedel–Crafts acylation of 1, 3-diethoxy-2,4,5-trimethoxybenzene with acetyl chloride in the presence of aluminium chloride (60%) [3313].

b.p.$_{0.2}$ 119–121° [3313].

1-[2-Hydroxy-3,5,6-trimethoxy-4-(1-methylethoxy)phenyl]ethanone

[56002-87-0]



$C_{14}H_{20}O_6$ mol.wt. 284.31

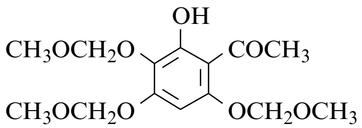
Synthesis

- Obtained (poor yield) by Friedel–Crafts acylation of 3-iso-propyloxy-2,4,5-trimethoxyphenol with acetyl chloride in ethyl ether in the presence of aluminium chloride (9%) [3317].

^{1}H NMR [3317].

1-[2-Hydroxy-3,4,6-tris(methoxymethoxy)phenyl]ethanone

[53000-17-2]



$C_{14}H_{20}O_8$ mol.wt. 316.31

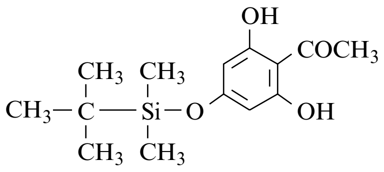
Synthesis

- Obtained by reaction of chloromethyl methyl ether with 2,3,4,6-tetrahydroxyacetophenone in ethanol in the presence of sodium ethoxide under nitrogen, first at 0°, then at 40° (13%) [2882].

Yellow oil [2882]; ^{1}H NMR [2882], IR [2882].

1-[4-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-2,6-dihydroxyphenyl]ethanone

[139140-13-9]



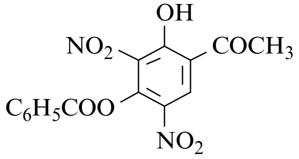
$C_{14}H_{22}O_4Si$ mol.wt. 282.41

Synthesis

- Obtained by sonication of 2′,4′,6′-(tri-tert-butyl-dimethylsilyloxy)acetophenone in solution of methanol and carbon tetrachloride (1:1) at 50–55° for 12 h (85%) [3572].

Colourless oil [3572]; ^{1}H NMR [3572], ^{13}C NMR [3572], IR [3572], MS [3572].

1-[4-(Benzoyloxy)-2-hydroxy-3,5-dinitrophenyl]ethanone



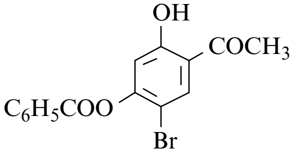
$C_{15}H_{10}N_2O_8$ mol.wt. 346.25

Synthesis

- Preparation (by-product) by reaction of concentrated nitric acid on 4-(benzoyloxy)-2-hydroxyacetophenone in acetic acid at r.t. [1808].

m.p. 171–172° [1808].

1-[4-(Benzoyloxy)-5-bromo-2-hydroxyphenyl]ethanone



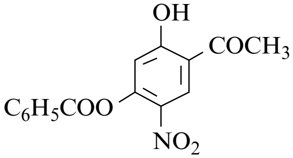
$C_{15}H_{11}BrO_4$ mol.wt. 335.15

Synthesis

- Preparation by bromination of 4-(benzoyloxy)-2-hydroxy-acetophenone [1808,2707].

m.p. 176° [2707], 175–177° [1808].

1-[4-(Benzoyloxy)-2-hydroxy-5-nitrophenyl]ethanone



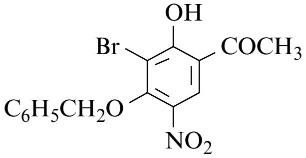
$C_{15}H_{11}NO_6$ mol.wt. 301.26

Synthesis

- Preparation by reaction of concentrated nitric acid on 4-(benzoyloxy)-2-hydroxyacetophenone in acetic acid at r.t. [1808].

m.p. 125–126° [1808].

1-[3-Bromo-2-hydroxy-5-nitro-4-(phenylmethoxy)phenyl]ethanone



$C_{15}H_{12}BrNO_5$ mol.wt. 366.17

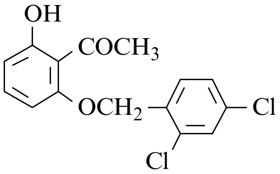
Synthesis

- Preparation by bromination of 4-(benzyloxy)-2-hydroxy-5-nitroacetophenone in chloroform solution in the presence of iron wire [1808].

m.p. 150–151° [1808].

1-[2-[(2,4-Dichlorophenyl)methoxy]-6-hydroxyphenyl]ethanone

[63411-86-9]



$C_{15}H_{12}Cl_2O_3$ mol.wt. 311.16

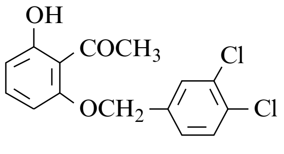
Synthesis

- Obtained by reaction of 2,4-dichlorobenzyl chloride with 2,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (62%) [3242].

m.p. 126–127° [3242].

1-[2-[(3,4-Dichlorophenyl)methoxy]-6-hydroxyphenyl]ethanone

[63411-85-8] $C_{15}H_{12}Cl_2O_3$ mol.wt. 311.16



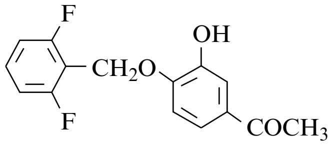
Synthesis

- Obtained by reaction of 3,4-dichlorobenzyl bromide or chloride with 2,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (52%) [3242].

m.p. 175–176° [3242].

1-[4-(2,6-Difluorophenyl)methoxy-3-hydroxyphenyl]ethanone

$C_{15}H_{12}F_2O_3$ mol.wt. 278.26



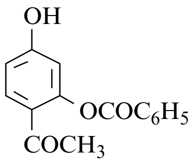
Synthesis

- Obtained by stirring a mixture of 3,4-dihydroxy-acetophenone, lithium carbonate and α-bromo-2,6-di-fluorotoluene in DMF for 2 days at r.t. (38%) [3573].

Solid [3573]; ^{1}H NMR [3573].

1-[2-(Benzoyloxy)-4-hydroxyphenyl]ethanone

[144406-93-9] $C_{15}H_{12}O_4$ mol.wt. 256.26

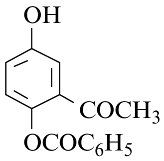


Synthesis

- Obtained by conventional acylation of resacetophenone with benzoyl chloride [3574].

1-[2-(Benzoyloxy)-5-hydroxyphenyl]ethanone

[88087-03-0] $C_{15}H_{12}O_4$ mol.wt. 256.26



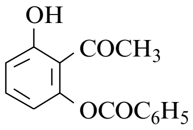
Synthesis

- Preparation by hydrogenolysis of 5-(benzyloxy)-2-(benzoyloxy)acetophenone in ethyl acetate using 10% Pd/C as catalyst, at r.t. for 5 h (78%) [2842].

m.p. 168–169° [2842]; ^{1}H NMR [2842].

1-[2-(Benzoyloxy)-6-hydroxyphenyl]ethanone

[50634-01-0] $C_{15}H_{12}O_4$ mol.wt. 256.26



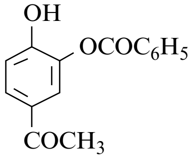
Synthesis

- Preparation by reaction of benzoyl chloride on 2,6-dihydroxyacetophenone in aqueous sodium hydroxide solution (60%) [2433].

m.p. 99–100° [2433].

1-[3-(Benzoyloxy)-4-hydroxyphenyl]ethanone

[101140-07-2] $C_{15}H_{12}O_4$ mol.wt. 256.26



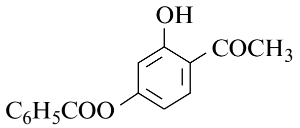
Synthesis

- Preparation by reaction of benzoyl chloride with 3,4-dihydroxyacetophenone in the presence of pyridine, first at 0° for 5 min, then at r.t. for 24 h under nitrogen atmosphere (77%) [3410].

m.p. 143–156° [3410]. This gap of 13° appears in the publication.
^{1}H NMR [3410], IR [3410], MS [3410].

1-[4-(Benzoyloxy)-2-hydroxyphenyl]ethanone

[109311-05-9] $C_{15}H_{12}O_4$ mol.wt. 256.26



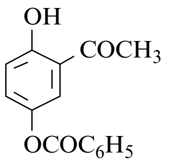
Syntheses

- Preparation by reaction of benzoyl chloride on resacetophenone [2293,2613,2707,2792],
  - with aqueous sodium hydroxide (68%) [2613];
  - with aqueous potassium hydroxide [2293];
  - with potassium carbonate in toluene by heating on a steam bath (52%) [2613];
  - with aluminium chloride in nitrobenzene by heating in a water bath (21%) [2707].
- Also refer to: [3575].

m.p. 110° [2707], 107–108° [2792], 106–107° [2613], 105–106° [2293].

1-[5-(Benzoyloxy)-2-hydroxyphenyl]ethanone

$C_{15}H_{12}O_4$ mol.wt. 256.26



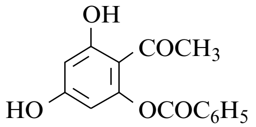
Synthesis

- Obtained by reaction of aluminium chloride on 4-(benzoyloxy)phenyl acetate without solvent between 125° and 155° (13%) [2344].

m.p. 77–78° [2344].

1-[2-(Benzoyloxy)-4,6-dihydroxyphenyl]ethanone

[83332-29-0] $C_{15}H_{12}O_5$ mol.wt. 272.26



Synthesis

- Obtained by reaction of benzoyl chloride on phloroacetophenone with 2% aqueous potassium hydroxide solution at 0° (6%) [3077] or with dilute aqueous sodium hydroxide solution [2827].

m.p. 186° [3077], 177–178° [2827].

1-[4-(Benzoyloxy)-2,6-dihydroxyphenyl]ethanone

[130471-75-9] $C_{15}H_{12}O_5$ mol.wt. 272.26

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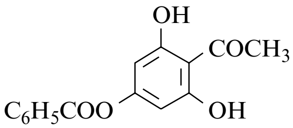


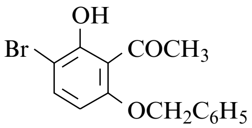
Synthesis

- Obtained by reaction of benzoyl chloride on phloroacetophenone with 2% aqueous potassium hydroxide solution at 0° (19%) [3077].

m.p. 210–211° [3077].

1-[3-Bromo-2-hydroxy-6-(phenylmethoxy)phenyl]ethanone

$C_{15}H_{13}BrO_3$ mol.wt. 321.17



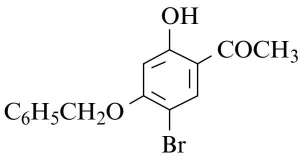
Synthesis

- Preparation by reaction of bromine on 2-(benzyloxy)-6-hydroxyacetophenone in methylene chloride (84%) [2524] or in acetic acid at r.t. (58%) [2523].

m.p. 125–126° [2523], 124° [2524]; IR [2524].

1-[5-Bromo-2-hydroxy-4-(phenylmethoxy)phenyl]ethanone

$C_{15}H_{13}BrO_3$ mol.wt. 321.17



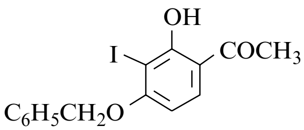
Syntheses

- Obtained from 3,3′-diacetyl-4,4′-dihydroxy-6,6′-dibenzyl-oxydiphenyl thioether by treatment with bromine in the presence of a crystal of iodine in boiling acetic acid for 8 h then at r.t. overnight [3576].
- Also obtained by bromination of 4-(benzyloxy)-2-hydroxy-acetophenone with bromine in carbon disulfide in the presence of a trace of iodine [3577].

m.p. 154–155° [3576,3577].

1-[2-Hydroxy-3-iodo-4-(phenylmethoxy)phenyl]ethanone

$C_{15}H_{13}IO_3$ mol.wt. 368.17

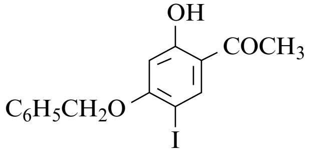


Synthesis

- Preparation by iodination of 4-(benzyloxy)-2-hydroxy-acetophenone [1910].

m.p. 165° [1910].

1-[2-Hydroxy-5-iodo-4-(phenylmethoxy)phenyl]ethanone

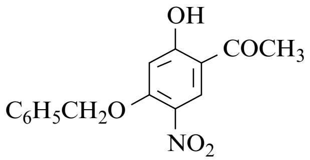


$C_{15}H_{13}IO_3$ mol.wt. 368.17

Synthesis

- Obtained (by-product) during the rearrangement of 2-(benzoyloxy)-4-(benzyloxy)-3-iodoacetophenone to give 7-(benzyloxy)-8-iodoflavone [1910].

1-[2-Hydroxy-5-nitro-4-(phenylmethoxy)phenyl]ethanone



$C_{15}H_{13}NO_5$ mol.wt. 287.27

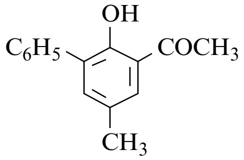
Synthesis

- Preparation by nitration of 4-(benzyloxy)-2-hydroxy-acetophenone in acetic acid at r.t. [1808].

m.p. 140–141° [1808].

1-(2-Hydroxy-5-methyl[1,1′-biphenyl]-3-yl)ethanone

[360791-68-0]



$C_{15}H_{14}O_2$ mol.wt. 226.27

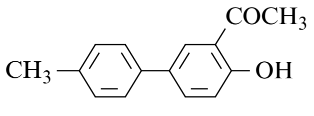
Synthesis

- Obtained by successively adding an aqueous solution of sodium bicarbonate (6 mmol), then tetrakis(triphenyl-phosphine)palladium (0) (0.1 mmol) to a solution of 3-bromo-2-hydroxy-5-methylacetophenone (2 mmol) and phenylboronic acid (2 mmol) in ethylene glycol dimethyl ether (DME). After, the reaction mixture was refluxed for 10 min and then heated at 75° overnight (71%) (compound **26**) [1804].

Yellow solid [1804]; ^{1}H NMR [1804], MS [1804].

1-(4-Hydroxy-4′-methyl[1,1′-biphenyl]-3-yl)ethanone

[229007-00-5]



$C_{15}H_{14}O_2$ mol.wt. 226.27

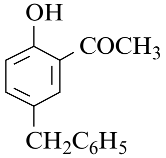
Synthesis

- Obtained by Fries rearrangement of 4′-methyl-4-biphenylyl acetate (1 mol) with aluminium chloride (1.1 mol) in refluxing o-dichlorobenzene for 3 h (67%) [3578].

m.p. 82°5–83°3 [3578]; ^{1}H NMR [3578], IR [3578], GC-MS [3578].

1-[2-Hydroxy-5-(phenylmethyl)phenyl]ethanone

[61300-15-0] $C_{15}H_{14}O_2$ mol.wt. 226.27



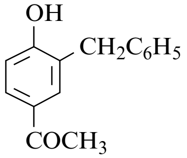
Synthesis

- Preparation by Fries rearrangement of 4-hydroxydiphenylmethane acetate with aluminium chloride in chlorobenzene at 130° [2270,3579], (85%) [3579].

m.p. 56° [3579], 55–56° [2270].

1-[4-Hydroxy-3-(phenylmethyl)phenyl]ethanone

[95102-24-2] $C_{15}H_{14}O_2$ mol.wt. 226.27



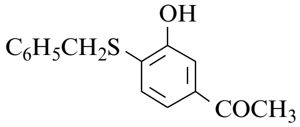
Syntheses

- Preparation by Fries rearrangement of 2-hydroxy-diphenylmethane acetate in the presence of aluminium chloride in nitrobenzene at 50–60° (70%) [3579].
- Also refer to: [3580,3581].

m.p. 137–138° [3579].

1-[3-Hydroxy-4-(phenylmethyl)thiophenyl]ethanone

$C_{15}H_{14}O_2S$ mol.wt. 258.34



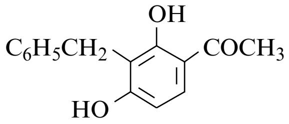
Synthesis

- Obtained by reaction of benzyl bromide with 3-hydroxy-4-mercaptoacetophenone (SM) in the presence of potassium carbonate in refluxing acetone for 2 h (20%). SM was prepared by demethylation of 3-methoxy-4-mercapto-acetophenone with boron tribromide in methylene chloride for 2 h at −78° [3573].

^{1}H NMR [3573].

1-[2,4-Dihydroxy-3-(phenylmethyl)phenyl]ethanone

[67088-16-8] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

- Preparation by reaction of boron tribromide with 3-benzyl-2,4-dimethoxyacetophenone in methylene chloride, first at −50°, then at r.t. (90%) [2464].
- Preparation by reaction of concentrated hydrochloric acid with 3-benzyl-4-(benzyloxy)-2-hydroxyacetophenone in refluxing acetic acid (69%) [3582].

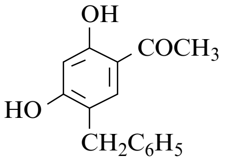
- Also obtained by reacting 4-(benzyloxy)-2-hydroxyacetophenone with trifluoroacetic acid at r.t. for 70 h (17%) [2324].
- Also obtained by reaction of benzyl alcohol with resacetophenone in the presence of boron trifluoride etherate and dioxane at 60–70° (16%) [2324].
- Also obtained by reaction of acetonitrile on 2-benzylresorcinol (Hoesch reaction) (11%) [3582].
- Also obtained by reaction of benzyl bromide with resacetophenone in the presence of methanolic potassium hydroxide at r.t. (18%) [3583].
- Also obtained by catalytic reduction of 2′,4′-dihydroxy-3′-[phenyl-(1-piperidyl) methyl]-acetophenone in the presence of Pd/C [2673,2674], (68%) [2674]. An alternative route of reduction consisted in heating the starting material and Pd/C in tetralin between 140° and 150° for 8 h (57%) [2674]. The starting material was obtained by treatment of resacetophenone in ethanol or benzene with benzylidene-bis-piperidine.
- Also refer to: [3584].

m.p. 207–208° [2673,2674], 196–198° [3583], 195–197° [3582], 195–196° [2324];

^{1}H NMR [2324,2464,2674], IR [2324,2674], UV [2324].

1-[2,4-Dihydroxy-5-(phenylmethyl)phenyl]ethanone

[93898-99-8] $C_{15}H_{14}O_3$ mol.wt. 242.27



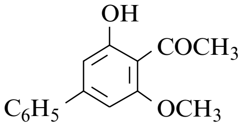
Syntheses

- Preparation by Fries rearrangement of 4-benzylresorcinol diacetate with aluminium chloride in the presence of 4-benzylresorcinol in nitrobenzene at 50° (85%) [3579].
- Also obtained by reacting 2-hydroxy-4-(benzyloxy)acetophenone with trifluoroacetic acid at r.t. for 70 h (29%) [2324].
- Also obtained by reaction of benzyl alcohol with resacetophenone in the presence of boron trifluoride etherate and dioxane at 60–70° (24%) [2324].
- Also obtained (poor yield) by reaction of benzyl bromide with resacetophenone in the presence of methanolic potassium hydroxide at r.t. (<2%) [3583].

m.p. 149° [3579], 140–142° [3583], 140–141° [2324]; ^{1}H NMR [2324], IR [2324], UV [2324].

1-(3-Hydroxy-5-methoxy[1,1′-biphenyl]-4-yl)ethanone

[32101-40-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



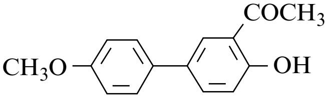
Synthesis

- Preparation by dehydrogenation of 2-acetyl-3-methoxy-5-phenyl-2-cyclohexenone in the presence of Pd black powder in refluxing cyclohexene for 6 h (70%) [3527].

m.p. 96–97° [3527]; IR [3527], UV [3527].

1-(4-Hydroxy-4′-methoxy[1,1′-biphenyl]-3-yl)ethanone

[114412-47-4] $C_{15}H_{14}O_3$ mol.wt. 242.27

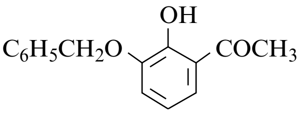


Synthesis

- Obtained by Fries rearrangement of 4-acetoxy-4′-methoxydiphenyl with aluminium chloride in tetrachloroethane at 140° for 30 min [3585].

1-[2-Hydroxy-3-(phenylmethoxy)phenyl]ethanone

[30992-64-4] $C_{15}H_{14}O_3$ mol.wt. 242.27



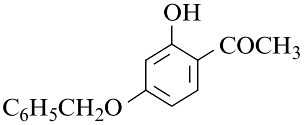
Synthesis

- Preparation by reaction of benzyl chloride with 2,3-di-hydroxyacetophenone in the presence of potassium iodide and potassium carbonate in refluxing acetone for 4 h (43%) [3586] or for 18 h (40%) [2287].

m.p. 55–56° [2287]; b.p.$_{0.05}$ 122–130° [3586]; ^{1}H NMR [2287], IR [2287].

1-[2-Hydroxy-4-(phenylmethoxy)phenyl]ethanone

[29682-12-0] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

- Preparation by reaction of resacetophenone, with benzyl chloride,
  - in the presence of potassium carbonate in refluxing acetone [2417,3493], (85%) [3204], (50–52%) [2423,2529,2823].

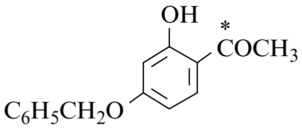
  N.B.: The butanone can also be used instead of acetone [3204]. The addition of potassium iodide improved yields [2417],

  - in the presence of potassium carbonate in DMF at 150–153° (62%) [3587];
  - in the presence of potassium carbonate and potassium iodide in refluxing acetone for 4 h [3586] or for 18 h (67%) [2287];
  - in the presence of potassium hydroxide in refluxing methanol [3582].
- with benzyl bromide,
  - in the presence of potassium carbonate in a methyl ethyl ketone and DMSO mixture (53%) [3588];
  - in the presence of methanolic potassium hydroxide at r.t. (10%) [3583].
- Also obtained by hydrolysis of 2-(acetyloxy)-4-(benzyloxy)acetophenone (m.p.111–112°) with 10% aqueous sodium hydroxide in boiling methanol for 5 min (76%) [2763].
- Also obtained (trace) by heating 4-acetyloxy-2-hydroxyacetophenone with benzyl chloride (<1%) [2904].
- Also refer to: [2777,3589–3596].

m.p. 111° [2423], 110° [2324], 109–110° [3583], 106–107° [3582], 105–106° [3586], 104–104°5 [2763], 104–105° [3204], 103–105° [3493], 103–104° [2823], 102–103° [2904], 101–102° [2287], 94–95° [3587];
^{1}H NMR [2287,2904,3587], ^{13}C NMR [2328], UV [2763,2904].

1-[2-Hydroxy-4-(phenylmethoxy)phenyl]ethanone-*1*-^{14}C

[71815-42-4] $C_{15}H_{14}O_3$ mol.wt. 244.27



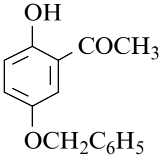
Syntheses

- Preparation by reaction of benzyl chloride with [carbonyl-^{14}C]resacetophenone in the presence of potassium carbonate and potassium iodide in DMF at 80° for 2 h [3597], (69%) [3598] or at 50–55° for 1 h (63%) [3599].

m.p. 105°5–106°5 [3599], 103–105° [3598];
sp. act. 6.09 × 10^7 dpm/mM [3598]; 0.316 mCi/mM [3597].
sp. act. 6.09 × 10^7 dpm/mM [3598]; 0.316 mCi/mM [3597].

1-[2-Hydroxy-5-(phenylmethoxy)phenyl]ethanone

[30992-63-3] $C_{15}H_{14}O_3$ mol.wt. 242.27



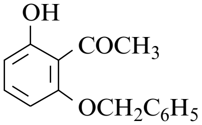
Syntheses

- Preparation by reaction of benzyl chloride with quinacetophenone [2226,2358,2529] in refluxing acetone in the presence of potassium carbonate,
  - with potassium iodide (85%) [2226], (71%) [3586], (55%) [2287];
  - without potassium iodide (31%) [2358].

m.p. 100–102° [2226], 69–70° [2358,3586], 67–68° [2287]. One of the reported melting points is obviously wrong.
^{1}H NMR [2226,2287], IR [2226], MS [2226].

1-[2-Hydroxy-6-(phenylmethoxy)phenyl]ethanone

[4047-24-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

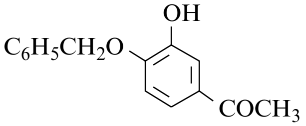
- Preparation by reaction of benzyl chloride with 2,6-dihydroxyacetophenone,
  - in the presence of potassium carbonate in refluxing acetone [1984,2408], (58%) [2408];
  - in the presence of potassium carbonate and potassium iodide in refluxing acetone for 18 h (40%) [2287];
  - in the presence of 15% aqueous sodium hydroxide, by heating in a water bath (67%) [2364].

- Preparation by reaction of benzyl bromide with 2,6-dihydroxyacetophenone monosodium salt (SM) in DMSO at r.t. for 1 h (76%). SM was prepared by reaction of sodium hydride (1 mol) with 2,6-dihydroxyacetophenone (1 mol) in DMSO at r.t. for 10 min [3600].
- Preparation by reaction of benzyl bromide with 2,6-dihydroxyacetophenone,
  - in the presence of potassium carbonate in refluxing acetone (60%) [2524];
  - in the presence of potassium carbonate and potassium iodide in refluxing acetone (about 80°) for 12 h (72%) [3601].

Monohydrate [3601];
m.p. 110–111° [3601], 109–110° [2408,3600,3602], 109° [2524], 108–109° [2287]
106–109° [1984], 106–107° [2364];
^{1}H NMR [2287,3600,3601], ^{13}C NMR [3601], MS [3601].

1-[3-Hydroxy-4-(phenylmethoxy)phenyl]ethanone

[21092-94-4] $C_{15}H_{14}O_3$ mol.wt. 242.27



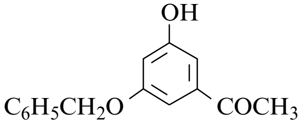
Syntheses

- Preparation by reaction of benzyl chloride with 3,4-di-hydroxyacetophenone in the presence of potassium hydroxide in a refluxing mixture of ethanol, methanol and water (53%) [2248].
- Also refer to: [1828].

m.p. 118° [2248].

1-[3-Hydroxy-5-(phenylmethoxy)phenyl]ethanone

[81732-54-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



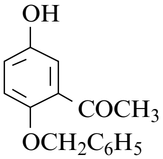
Synthesis

- Preparation by partial catalytic hydrogenolysis of 3,5-bis (benzyloxy)acetophenone in acetone in the presence of Pd/C (48%) [3603].

m.p. 126–128° [3603]; ^{1}H NMR [3603].

1-[5-Hydroxy-2-(phenylmethoxy)phenyl]ethanone

[83069-04-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



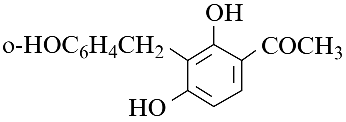
Synthesis

- Preparation by hydrolysis of 5-acetoxy-2-(benzyloxy)-acetophenone (m.p. 111–112°) with 5% sodium hydroxide in boiling aqueous methanol (69%) [2763].

m.p. 117° [2763]; UV [2763].

1-[2,4-Dihydroxy-3-[(2-hydroxyphenyl)methyl]phenyl]ethanone

[103633-38-1] $C_{15}H_{14}O_4$ mol.wt. 258.27



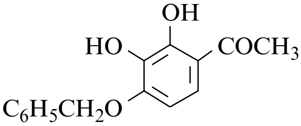
Synthesis

– Preparation by catalytic hydrogenolysis of 3-(o-benzyl-oxybenzyl)-2,4-dihydroxy-acetophenone at r.t. in the presence of 10% Pd/C in ethyl acetate (67%) [2325].

m.p. 204–205° [2325]; ^{1}H NMR [2325], IR [2325], UV [2325].

1-[2,3-Dihydroxy-4-(phenylmethoxy)phenyl]ethanone

[69114-99-4] $C_{15}H_{14}O_4$ mol.wt. 258.27



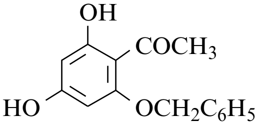
Synthesis

– Preparation by reaction of benzyl chloride on gallacetophenone with sodium bicarbonate and sodium iodide in refluxing acetone–ethanol mixture (33–37%) [1989,2817].

m.p. 137–138° [2817], 133–133°5 [1989]; ^{1}H NMR [1989].

1-[2,4-Dihydroxy-6-(phenylmethoxy)phenyl]ethanone

[39548-86-2] $C_{15}H_{14}O_4$ mol.wt. 258.27



Syntheses

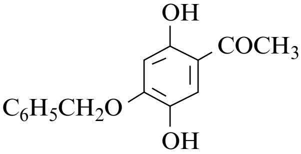
– Preparation from 2-(benzyloxy)-4,6-dihydroxyacetophenone ditosilate (SM) by hydrolysis with 5% sodium hydroxide in ethanol (72%). The starting ketone (SM) was prepared by a two-step procedure from phloroacetophenone [3604].

– Also obtained (poor yield) by reaction of benzyl chloride on phloroacetophenone with potassium carbonate in refluxing acetone (4%) [3605].

m.p. 239–240° [3605], 233°5 [3604]; ^{1}H NMR [3605], UV [3605].

1-[2,5-Dihydroxy-4-(phenylmethoxy)phenyl]ethanone

[34176-17-5] $C_{15}H_{14}O_4$ mol.wt. 258.27



Syntheses

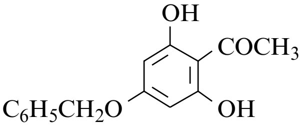
– Preparation by reaction of benzyl halide with 2,4,5-tri-hydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (62%) [2418].

– Preparation from 4-(benzyloxy)-2-hydroxyacetophenone by persulfate oxidation (Elbs reaction) (25%) [2417,2823,2906].

m.p. 160–162° [2417], 159–160° [2823], 157–159° [2418].

1-[2,6-Dihydroxy-4-(phenylmethoxy)phenyl]ethanone

[35028-02-5] $C_{15}H_{14}O_4$ mol.wt. 258.27



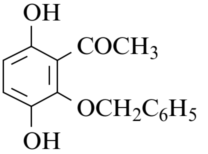
Syntheses

- Preparation from 2,4-diacetyl-5-(benzyloxy) resorcinol by selective deacetylation by refluxing in 1 N sodium hydroxide for 1 h [2856].
- Also obtained by reaction of benzyl chloride on phloroacetophenone with potassium carbonate in refluxing acetone (13%) [2838].

m.p. 188–189° (compound **VIII**) [2838],
139–140° (compound **II**, R=H; R′=$PhCH_2$-) [2856]; ^{1}H NMR [2856].

1-[3,6-Dihydroxy-2-(phenylmethoxy)phenyl]ethanone

[33537-81-4] $C_{15}H_{14}O_4$ mol.wt. 258.27



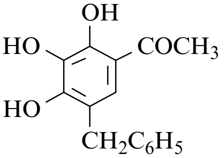
Syntheses

- Preparation from 2-(benzyloxy)-6-hydroxyacetophenone by persulfate oxidation (Elbs reaction) (49%) [2408].
- Easy preparation by reduction of 2-acetyl-3-(benzyloxy)-1,4-benzoquinone using conventional methods [2869].
- Also obtained in low yield by reaction of 2-acetyl-1,4-benzo-quinone with an excess of benzyl alcohol at r.t., with exclusion of light [2869].

m.p. 94° [2408], 87°5–89° [2869]; ^{1}H NMR [2869], IR [2869].

1-[2,3,4-Trihydroxy-5-(phenylmethyl)phenyl]ethanone

[105485-44-7] $C_{15}H_{14}O_4$ mol.wt. 258.27



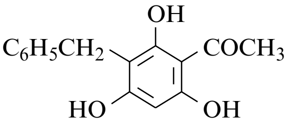
Syntheses

- Preparation by reaction of benzyl alcohol with gallacetophenone in the presence of boron trifluoride etherate in dioxane at 60–70° (49%) [3583].
- Preparation by Claisen rearrangement of 3,4-bis-(benzyloxy)-2-hydroxyacetophenone in the presence of trifluoroacetic acid at r.t. (54%) [3583].

m.p. 127–128° [3583]; ^{1}H NMR [3583], IR [3583], UV [3583].

1-[2,4,6-Trihydroxy-3-(phenylmethyl)phenyl]ethanone

$C_{15}H_{14}O_4$ mol.wt. 258.27



Syntheses

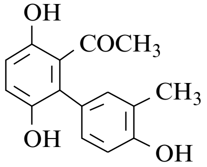
- Obtained (poor yield) by reaction of a benzyl halogenide with phloroacetophenone in the presence of sodium methoxide in methanol at r.t. (8%) [2878].

- Also obtained from 3-benzyl-4,6-bis(benzyloxy)-2-hydroxyacetophenone by hydrogenolysis in the presence of 5% Pd/C in ethanol [3606].

m.p. 208° [2878], 207–208° [3606]; IR [3606], UV [3606].

1-(3,4′,6-Trihydroxy-3′-methyl[1,1′-biphenyl]-2-yl)ethanone

[32541-10-9] $C_{15}H_{14}O_4$ mol.wt. 258.27



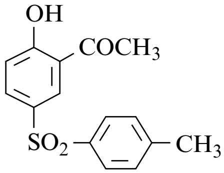
Synthesis

- Obtained by catalytic reduction of 2-acetyl-3-(4-hydroxy-3-methylphenyl)-1,4-benzoquinone (SM). SM was obtained by condensation of o-cresol with 2-acetyl-1,4-benzoquinone in ethyl ether in the presence of trifluoroacetic acid at 0° (12%) [3607].

m.p. 194° [3607].

1-[2-Hydroxy-5-[(4-methylphenyl)sulfonyl]phenyl]ethanone

[147816-51-1] $C_{15}H_{14}O_4S$ mol.wt. 290.34



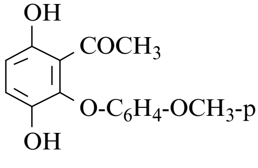
Syntheses

- Preparation by Fries rearrangement of 4-(p-tolylsulfonyl)-phenyl acetate with aluminium chloride (5 mol) at 160° for 1 h (58%) [3541].
- Also obtained by photo-Fries rearrangement of the same ester in acetonitrile (26%) or (40%) based on consumed starting material [3541].

m.p. 206–208° [3541]; ^{1}H NMR [3541], UV [3541].

1-[3,6-Dihydroxy-2-(4-methoxyphenoxy)phenyl]ethanone

[52095-11-1] $C_{15}H_{14}O_5$ mol.wt. 274.27



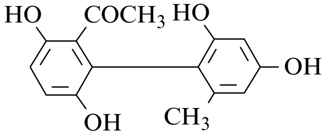
Synthesis

- Preparation by reaction of hydroquinone monomethyl ether on 2-acetylquinone with pyridine in benzene (43%) [2020].

m.p. 74–77° [2020]; ^{1}H NMR [2020], IR [2020].

1-(2′,3,4′,6-Tetrahydroxy-6′-methyl[1,1′-biphenyl]-2-yl)ethanone

[32546-66-0] $C_{15}H_{14}O_5$ mol.wt. 274.27



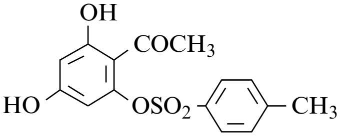
Synthesis

- Obtained by condensation of 2-acetyl-1,4-benzoquinone with orcinol in a acetic acid/ethyl ether solution at r.t. for 1 h (50%) [3607].

m.p. 185° [3607]; IR [3607].

1-[2,4-Dihydroxy-6-[[(4-methylphenyl)sulfonyl]oxy]phenyl]ethanone

[225088-72-2] $C_{15}H_{14}O_6S$ mol.wt. 322.05



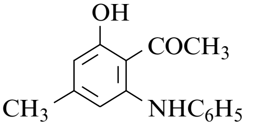
Synthesis

- Preparation by hydrogenolysis of 2-toluenesulfonyloxy-4,6-bis(benzyloxy)acetophenone (m.p. 122–123°) in methanol with hydrogen in the presence of 10% Pd/C at r.t. for 20 h (94%) [3608].

m.p. 150–152° [3608]; ^{1}H NMR [3608], IR [3608], MS [3608].

1-[2-Hydroxy-4-methyl-6-(phenylamino)phenyl]ethanone

[97066-04-1] $C_{15}H_{15}NO_2$ mol.wt. 241.29



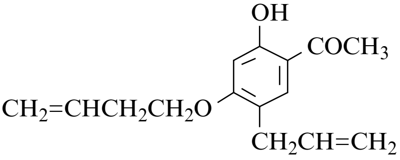
Synthesis

- Obtained by reaction of aniline with 2-acetyl-3-dimethyl-amino-5-hydroxy-5-methyl-2-cyclohexenone in refluxing ethanol (17%) [2712].

m.p. 115–117° [2712]; ^{1}H NMR [2712], IR [2712], UV [2712], MS [2712].

1-[4-(3-Butenyloxy)-2-hydroxy-5-(2-propenyl)phenyl]ethanone

[117690-53-6] $C_{15}H_{18}O_3$ mol.wt. 246.31



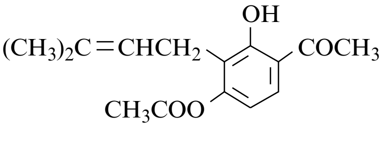
Synthesis

- Preparation by reaction of 3-butenyl bromide with 5-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide at reflux (26%) [2678,2679].

m.p. <25° [2678,2679].

1-[4-(Acetyloxy)-2-hydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[95604-05-0] $C_{15}H_{18}O_4$ mol.wt. 262.31



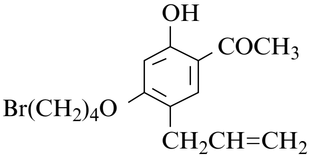
Synthesis

- Preparation by acetylation of 3-prenylresaceto-phenone with acetic anhydride in the presence of boric acid, first under reflux for 5 min, then at r.t. for 24 h (60%) [3609].

m.p. 66° [3609]; ^{1}H NMR [3609].

1-[4-(4-Bromobutoxy)-2-hydroxy-5-(2-propenyl)phenyl]ethanone

[117706-42-0] $C_{15}H_{19}BrO_3$ mol.wt. 327.22



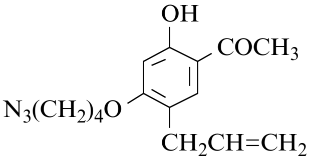
Synthesis

– Preparation by reaction of 4-bromobutyl bromide with 5-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide at reflux (56%) [2678,2679].

m.p. <25° [2678,2679].

1-[4-(4-Azidobutoxy)-2-hydroxy-5-(2-propenyl)phenyl]ethanone

[140660-37-3] $C_{15}H_{19}N_3O_3$ mol.wt. 289.33

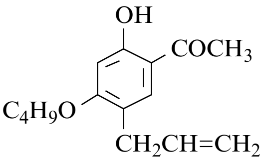


Synthesis

– Preparation by reaction of sodium azide on 5-allyl-4-(4-bromobutoxy)-2-hydroxyacetophenone in DMF at r.t. [2671,2679].

1-[4-Butoxy-2-hydroxy-5-(2-propenyl)phenyl]ethanone

[117690-48-9] $C_{15}H_{20}O_3$ mol.wt. 248.32



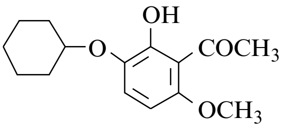
Synthesis

– Preparation by reaction of n-butyl bromide with 5-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing methyl ethyl ketone (60%) [2671,2678,2679].

Oil [2671,2678,2679].

1-[3-(Cyclohexyloxy)-2-hydroxy-6-methoxyphenyl]ethanone

[126405-80-9] $C_{15}H_{20}O_4$ mol.wt. 264.32



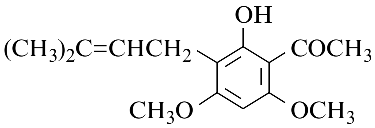
Synthesis

– Preparation by adding 2-hydroxy-3-iodo-6-methoxy-acetophenone and cuprous iodide to a solution of sodium cyclohexanolate, previously prepared from cyclohexanol and sodium hydride in DMF [2524].

Oil [2524]; ^{1}H NMR [2524], IR [2524].

1-[2-Hydroxy-4,6-dimethoxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[33523-62-5] $C_{15}H_{20}O_4$ mol.wt. 264.32



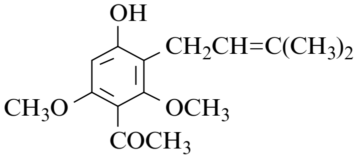
Synthesis

– Obtained by partial methylation of 2,4-dihydroxy-6-methoxy-3-prenylacetophenone [2834].

m.p. 113–114° [2834].

1-[4-Hydroxy-2,6-dimethoxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[18780-96-6] $C_{15}H_{20}O_4$ mol.wt. 264.32



Syntheses

– Preparation: γ,γ-dimethylallyl bromide was added to the lithium salt of 4-acetyl-3,5-dimethoxyphenol, which is easily prepared in benzene by reaction with butyl lithium (6%) [3610].

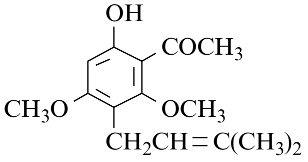
– Preparation by thermal Claisen rearrangement of 4-(γ,γ-dimethylallyoxy)-2,6-dimethoxyacetophenone in refluxing diethylaniline [3611,3612], (> 90%) [3611].

m.p. 66–68° [3610]; ^{1}H NMR [3610], UV [3610], MS [3610].

1-[6-Hydroxy-2,4-dimethoxy-3-(3-methyl-2-butenyl)phenyl]ethanone

(Acronylin methyl ether)

[4683-33-4] $C_{15}H_{20}O_4$ mol.wt. 264.32



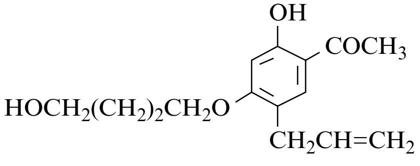
Synthesis

– Obtained by reaction of dimethyl sulfate with 4,6-di-hydroxy-2-methoxy-3-prenylacetophenone (m.p. 127–128°) in the presence of potassium carbonate in refluxing acetone for 3 h (38%) [2835].

m.p. 78–79° [2835]; TLC [2835].

1-[2-Hydroxy-4-(4-hydroxybutoxy)-5-(2-propenyl)phenyl]ethanone

[117690-52-5] $C_{15}H_{20}O_4$ mol.wt. 264.32



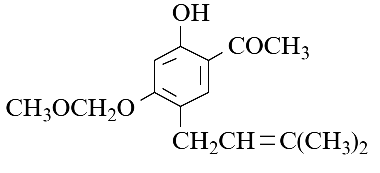
Synthesis

– Obtained by reaction of 4-hydroxybutyl bromide on 5-allyl-2,4-dihydroxyacetophenone with potassium carbonate and potassium iodide in refluxing methyl ethyl ketone (5–12%) [2671,2678,2679].

m.p. 117–119° [2671,2678,2679].

1-[2-Hydroxy-4-(methoxymethoxy)-5-(3-methyl-2-butenyl)phenyl]ethanone

[99217-72-8] $C_{15}H_{20}O_4$ mol.wt. 264.32



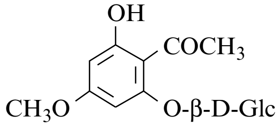
Synthesis

- Preparation by treatment of 2,4-dihydroxy-5-prenyl-acetophenone with methoxymethyl chloride in the presence of potassium carbonate in acetone [3613], (good yield) [3614].

^{1}H NMR [3613], IR [3613], UV [3613].

1-[2-(β-D-Glucopyranosyloxy)-6-hydroxy-4-methoxyphenyl]ethanone

[24587-97-1] $C_{15}H_{20}O_9$ mol.wt. 344.32



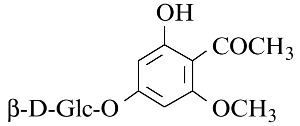
Isolation from natural sources

- From the leaves of *Ribes alpinum* L. (alpine currant) (Grossulariaceae) [3615].

spectral data [3616]; TLC [3615].

1-[4-(β-D-Glucopyranosyloxy)-2-hydroxy-6-methoxyphenyl]ethanone
(Annphenone)

[61775-18-6] $C_{15}H_{20}O_9$ mol.wt. 344.32



Synthesis

- Preparation by reaction, first, of a saturated aqueous barium hydroxide solution on 2-hydroxy-6-methoxy-4-(tetraacetyl-β-D-glucoside) acetophenone, and then carbon dioxide (38%) [2828].

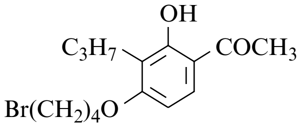
Isolation from natural sources

- From the aerial parts of *Artemisia iwayomogi* (Compositae) [3617].
- From the aerial parts of *Artemisia sacrorum* [3618] and from *Artemisia sacrorum* Ledeb. (Compositae) [3619].
- From the aerial parts of *Artemisia stolonifera* (Max.) Kom (Compositae) [3565].
- From the rhizomes of *Rhodiola linearifolia* Boriss [3620].
- From the aerial parts of *Artemisia annua* [3621].
- From the leaves of *Monochaetum multiflorum* (Melastomataceae) [3622].

m.p. 215° [2828], 185–188° [3619], 160–162° [3621], 156–158° [3565,3617];
$(\alpha)_D^{20}$ = –56°4 (pyridine) [2828];
^{1}H NMR [3565,3617,3619,3621], ^{13}C NMR [3565,3617,3619,3621],
IR [3565,3617,3619,3621], UV [3565,3617,3621],
MS [3617,3619], EIMS [3621].

1-[4-(4-Bromobutoxy)-2-hydroxy-3-propylphenyl]ethanone

[92518-06-4] $C_{15}H_{21}BrO_3$ mol.wt. 329.23



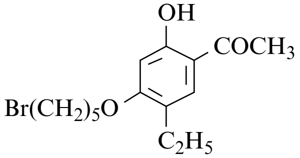
Synthesis

- Preparation by reaction of 1,4-dibromobutane with 2,4-dihydroxy-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (78%) [3181].

b.p.$_{0.25}$ 180° [3181].

1-[4-[(5-Bromopentyl)oxy]-5-ethyl-2-hydroxyphenyl]ethanone

[117690-80-9] $C_{15}H_{21}BrO_3$ mol.wt. 329.23



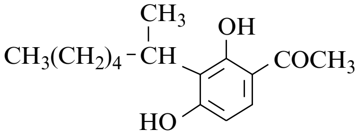
Synthesis

- Preparation by reaction of 5-bromopentyl bromide with 2,4-dihydroxy-5-ethylacetophenone in the presence of potassium carbonate and potassium iodide at reflux (60%) [2678,2679].

m.p. 60–62° [2678,2679]; ^{1}H NMR [2678,2679].

1-[2,4-Dihydroxy-3-(1-methylhexyl)phenyl]ethanone

[79557-94-1] $C_{15}H_{22}O_3$ mol.wt. 250.34



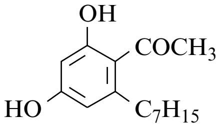
Synthesis

- Preparation by hydrogenation of 2,4-dihydroxy-3-(1-methyl-2-hexenyl) acetophenone in ethanol using 10% PdO/C as catalyst (54%) [3184].

m.p. 78–81° [3184].

1-(2-Heptyl-4,6-dihydroxyphenyl)ethanone

[83375-18-2] $C_{15}H_{22}O_3$ mol.wt. 250.34



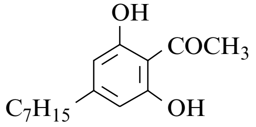
Syntheses

- Preparation by reaction of acetonitrile with 5-heptylresorcinol according to Hoesch reaction (57%) [3623].
- Also refer to: [3624].

m.p. 61–62° [3623]; ^{1}H NMR [3623].

1-(4-Heptyl-2,6-dihydroxyphenyl)ethanone

[83375-19-3] $C_{15}H_{22}O_3$ mol.wt. 250.34



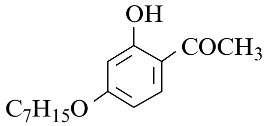
Synthesis

- Obtained (poor yield) by reaction of acetonitrile with 5-heptylresorcinol according to Hoesch reaction (5%) [3623].

m.p. 46–47° [3623]; ^{1}H NMR [3623].

1-[4-(Heptyloxy)-2-hydroxyphenyl]ethanone

[219696-56-7] $C_{15}H_{22}O_3$ mol.wt. 250.34



Synthesis

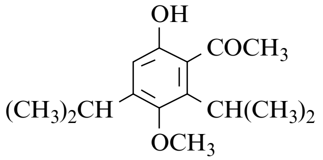
- Preparation by reaction of 1-heptanol with resacetophenone using polymer-bound triphenylphosphine* and diethyl azodicarboxylate (DEAD) in methylene chloride at 25° for 5 h (72%) [3625].

* Polystyryl-diphenylphosphine–2% divinylbenzene.

N.B.: The polymer-bound triphenylphosphines – commercially available – are easily removed by filtration from the reaction products.
Oil [3625].

1-[6-Hydroxy-3-methoxy-2,4-bis(1-methylethyl)phenyl]ethanone

[188903-79-9] $C_{15}H_{22}O_3$ mol.wt. 250.34



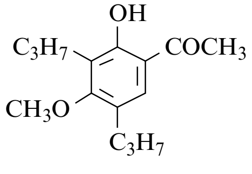
Synthesis

- Refer to: [3626].

N.B.: this compound, that has never been prepared, is however mistakenly mentioned in Chem. Abstr., **126**, 277303r (1997). However, it is not mentioned in the original paper [3626]. In this paper, the compound is the 2-hydroxy-4,6-diisopropyloxy-5-methoxyacetophenone, a ketone already obtained by [3506].

1-(2-Hydroxy-4-methoxy-3,5-dipropylphenyl)ethanone

[72018-37-2] $C_{15}H_{22}O_3$ mol.wt. 250.34



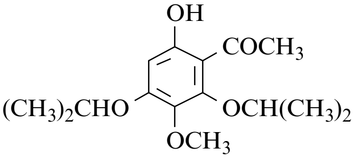
Synthesis not yet described

- Refer to: [2326,2327].

N.B.: Pr indicates the *propyl* group -C_3H_7 in Chem. Abstr., **92**, 6368x (1980) and **98**, 54239b (1983), an usual abbreviation. However, in the two references [2326,2327], Pr represented the *prenyl* group –$CH_2CH=C(CH_3)_2$. Therefore they concern the 2-hydroxy-4-methoxy-3,5-diprenylacetophenone and not the above mentioned 2-hydroxy-4-methoxy-3,5-dipropylacetophenone.

1-[6-Hydroxy-3-methoxy-2,4-bis(1-methylethoxy)phenyl]ethanone

[93344-50-4] $C_{15}H_{22}O_5$ mol.wt. 282.34



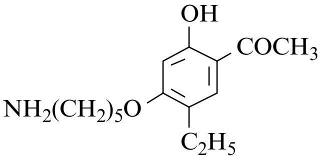
Syntheses

- Obtained by partial methylation of 2,5-dihydroxy-4,6-diisopropyloxyacetophenone (m.p. 37°) according to [3505], (67%) (compound **14**) [3506].
- Also refer to: [3503,3627].

Pale yellow oil [3506]; ^{1}H NMR [3506].

1-[4-[(5-Aminopentyl)oxy]-5-ethyl-2-hydroxyphenyl]ethanone

[117705-90-5] $C_{15}H_{23}NO_3$ mol.wt. 265.35



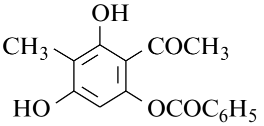
Synthesis

- Preparation by hydrogenation of 5-(4-acetyl-2-ethyl-5-hydroxyphenoxy)pentane nitrile in the presence of 10% Pd/C in acetic acid (>98%) [2678,2679].

m.p. 75–76° [2678,2679]; ^{1}H NMR [2678,2679].

1-[6-(Benzoyloxy)-2,4-dihydroxy-3-methylphenyl]ethanone

$C_{16}H_{14}O_5$ mol.wt. 286.28



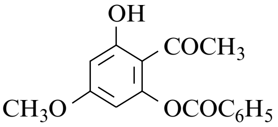
Synthesis

- Obtained by reaction of benzoyl chloride on 2,4,6-tri-hydroxy-3-methylacetophenone with 2% sodium hydroxide solution at 0° (13%) [3051].

m.p. 189° [3051].

1-[2-(Benzoyloxy)-6-hydroxy-4-methoxyphenyl]ethanone

[49602-08-6] $C_{16}H_{14}O_5$ mol.wt. 286.28

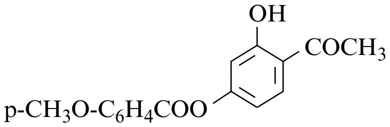


Synthesis

- Preparation by reaction of benzoyl chloride with 2,6-di-hydroxy-4-methoxyacetophenone in the presence of 1.5 N sodium hydroxide at r.t. [2854].

Oil [2854].

1-[2-Hydroxy-4-(4-methoxybenzoyloxy)phenyl]ethanone



$C_{16}H_{14}O_5$ mol.wt. 286.28

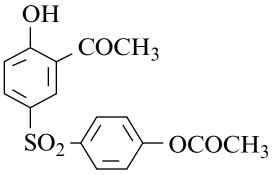
Synthesis

- Preparation by reaction of p-anisoyl chloride on resacetophenone with potassium carbonate in toluene (70%) [2613] or in aqueous sodium hydroxide solution (the best way) [2613].

m.p. 151° [2613].

1-[5-[[4-(Acetyloxy)phenyl]sulfonyl]-2-hydroxyphenyl]ethanone

[147816-49-7]



$C_{16}H_{14}O_6S$ mol.wt. 334.35

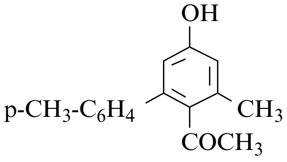
Syntheses

- Obtained by UV light irradiation (254 nm) of 0.02 M bisacetate of bisphenol-S in acetonitrile for 3.5 h (20%) [3541].
- Also obtained by Fries rearrangement of bisacetate of bisphenol-S with aluminium chloride (3 equiv.) at 160° (17%) [3541].

m.p. 166°6 [3541]; ^{1}H NMR [3541], UV [3541].

1-(5-Hydroxy-3,4′-dimethyl[1,1′-biphenyl]-2-yl)ethanone

[108909-47-3]



$C_{16}H_{16}O_2$ mol.wt. 240.30

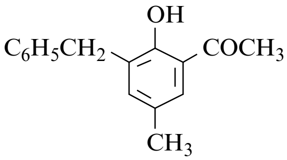
Syntheses

- Obtained by aromatization of 4-acetyl-3-methyl-5-(4-methylphenyl)-2-cyclohexen-1-one (m.p. 130°) with bromine in chloroform (60%) or by heating at 170° for 3 h [3628].
- Also obtained by deacylation of 1,1′-(3-hydroxy-4′,5-di-methyl[1,1′-biphenyl]-2,6-diyl)bis-ethanone (m.p. 165°) with sodium hydroxide in refluxing dilute ethanol for 3 h (60%) [3628].
- Also refer to: [3629].

m.p. 180° [3628]; IR [3628].

1-[2-Hydroxy-5-methyl-3-(phenylmethyl)phenyl]ethanone

[350981-92-9]



$C_{16}H_{16}O_2$ mol.wt. 240.30

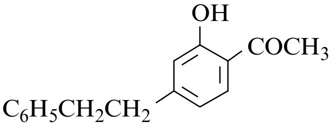
Synthesis

- Obtained from 3-acetyl-2-hydroxy-5-methylbenzyl O,O-di-methylphosphorothionothiolate (m.p. 99°5) on treatment with aluminium chloride in refluxing benzene for 5–10 min (93%) [3630].

m.p. 84° [3630]; ^{1}H NMR [3630], IR [3630], MS [3630].

1-[2-Hydroxy-4-(2-phenylethyl)phenyl]ethanone

[122379-44-6] $C_{16}H_{16}O_2$ mol.wt. 240.30

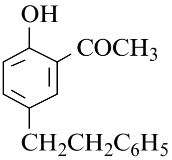


Synthesis

- Refer to: [3631].

1-[2-Hydroxy-5-(2-phenylethyl)phenyl]ethanone

[136608-20-3] $C_{16}H_{16}O_2$ mol.wt. 240.30



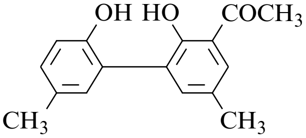
Syntheses

- Preparation by Fries rearrangement of 4-acetoxy-diphenylmethane with aluminium chloride in boiling chlorobenzene (70%) [3579].
- Also refer to: [3632].

m.p. 52° [3579]; b.p.$_{18}$ 250° [3579].

1-(2,2′-Dihydroxy-5,5′-dimethyl[1,1′-biphenyl]-3-yl)ethanone

[24046-00-2] $C_{16}H_{16}O_3$ mol.wt. 256.30



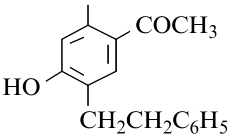
Syntheses

- Obtained by Fries rearrangement of 2,2′-diacetoxy-5,5′-di-methylbiphenyl with aluminium chloride in nitrobenzene at 120° for 2 h (53%) [3633].
- Also refer to: [3634].

m.p. 129–130° [3633]; TLC [3633];
^{1}H NMR [3633,3634], IR [3633,3634].

1-[2,4-Dihydroxy-5-(2-phenylethyl)phenyl]ethanone

[60640-95-1] $C_{16}H_{16}O_3$ mol.wt. 256.30



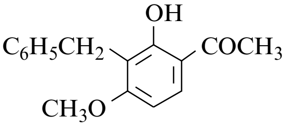
Syntheses

- Preparation by Fries rearrangement of 2,4-dihydroxy-diphenylethane diacetate with aluminium chloride in the presence of 2,4-dihydroxydiphenylethane in nitrobenzene at 50° [2508].
- Also refer to: [3635].

m.p. 136° [2508].

1-[2-Hydroxy-4-methoxy-3-(phenylmethyl)phenyl]ethanone

[95832-45-4] $C_{16}H_{16}O_3$ mol.wt. 256.30



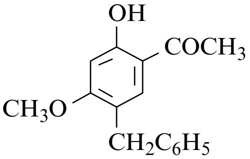
Synthesis

- Preparation by partial methylation of 3-benzyl-2,4-di-hydroxyacetophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone (87%) [2324].

m.p. 119–120° [2324]; ^{1}H NMR [2324].

1-[2-Hydroxy-4-methoxy-5-(phenylmethyl)phenyl]ethanone

[93434-27-6] $C_{16}H_{16}O_3$ mol.wt. 256.30



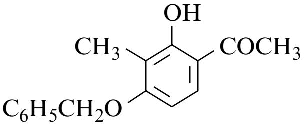
Synthesis

- Preparation by partial methylation of 5-benzyl-2,4-di-hydroxyacetophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone (82%) [2324].

m.p. 95–96° [2324]; ^{1}H NMR [2324].

1-[2-Hydroxy-3-methyl-4-(phenylmethoxy)phenyl]ethanone

[73640-74-1] $C_{16}H_{16}O_3$ mol.wt. 256.30



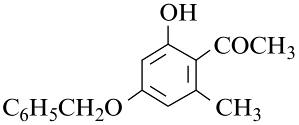
Syntheses

- Preparation by reaction of benzyl chloride on 2,4-di-hydroxy-3-methylacetophenone with potassium carbonate and potassium iodide in refluxing acetone (81%) [2582].
- Also refer to: [2025,2675].

m.p. 87–88° [2582]; ^{1}H NMR [2582], IR [2582], MS [2582].

1-[2-Hydroxy-6-methyl-4-(phenylmethoxy)phenyl]ethanone

[72545-51-8] $C_{16}H_{16}O_3$ mol.wt. 256.30



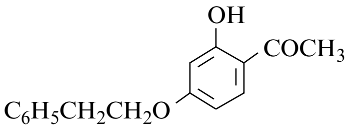
Synthesis

- Preparation by partial benzylation of 2,4-dihydroxy-6-methylacetophenone [3636].

m.p. 83–84° [3636].

1-[2-Hydroxy-4-(2-phenylethoxy)phenyl]ethanone

[63359-84-2] $C_{16}H_{16}O_3$ mol.wt. 256.30



Synthesis

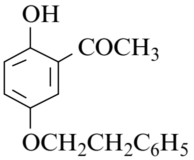
– Obtained by reaction of 1-bromo-2-phenylethane with resacetophenone in the presence of potassium carbonate in refluxing acetone for 24 h (45%) [3204].

N.B.: the 1-chloro derivative and butanone can also be used instead of the mentioned starting materials.

m.p. 69° [3204].

1-[2-Hydroxy-5-(2-phenylethoxy)phenyl]ethanone

[63359-85-3] $C_{16}H_{16}O_3$ mol.wt. 256.30



Synthesis

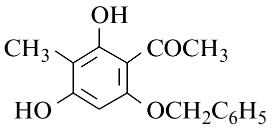
– Obtained by reaction of 1-bromo-2-phenylethane with quinacetophenone in the presence of potassium carbonate in refluxing acetone for 24 h (21%) [3204].

N.B.: the 1-chloro derivative and the butanone can also be used instead of the mentioned starting materials.

m.p. 36° [3204].

1-[2,4-Dihydroxy-3-methyl-6-(phenylmethoxy)phenyl]ethanone

[39548-93-1] $C_{16}H_{16}O_4$ mol.wt. 272.30



Synthesis

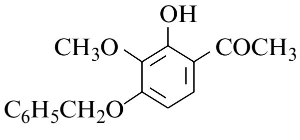
– Obtained by reaction of benzyl chloride on the 2,4,6-tri-hydroxy-3-methylacetophenone with potassium carbonate in boiling acetone (20%) [3605], (<2%) [3043].

m.p. 212° [3043], 187–188° [3605]. One of the reported melting points is obviously wrong.

^{1}H NMR [3605], UV [3605].

1-[2-Hydroxy-3-methoxy-4-(phenylmethoxy)phenyl]ethanone

[52249-85-1] $C_{16}H_{16}O_4$ mol.wt. 272.30



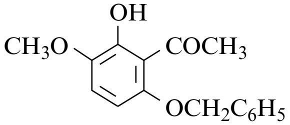
Synthesis

– Preparation by reaction of dimethyl sulfate on 4-(benzyloxy)-2,3-dihydroxyacetophenone with potassium carbonate in refluxing acetone [1989,2817,3637], (82%) [2817].

m.p. 146° [2817], 143–145° [1989]; ^{1}H NMR [1989].

1-[2-Hydroxy-3-methoxy-6-(phenylmethoxy)phenyl]ethanone

[126405-75-2] $C_{16}H_{16}O_4$ mol.wt. 272.30



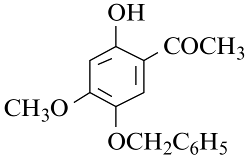
Synthesis

– Preparation by adding 6-(benzyloxy)-2-hydroxy-3-iodo-acetophenone and cuprous iodide to a solution of sodium methoxide, previously prepared from methyl alcohol and sodium hydride in DMF [2524].

m.p. 103° [2524]; IR [2524].

1-[2-Hydroxy-4-methoxy-5-(phenylmethoxy)phenyl]ethanone

[52249-88-4] $C_{16}H_{16}O_4$ mol.wt. 272.30



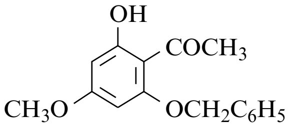
Synthesis

– Preparation by reaction of benzyl bromide on 2,5-di-hydroxy-4-methoxyacetophenone with potassium carbonate in boiling acetone (67%) [2849].

m.p. 151° [2849].

1-[2-Hydroxy-4-methoxy-6-(phenylmethoxy)phenyl]ethanone

[10299-59-9] $C_{16}H_{16}O_4$ mol.wt. 272.30



Syntheses

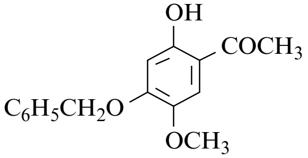
– Preparation by reaction of dimethyl sulfate on 2-(benzyloxy)-4,6-dihydroxyacetophenone with potassium carbonate in refluxing acetone (95%) [3605], (85%) [3604].

– Preparation by reaction of benzyl bromide with phloroacetophenone 4-methyl ether in the presence of potassium carbonate in acetone at r.t. under nitrogen (82%) [2853].

m.p. 120–121° [3605], 110–113° [3604], 110–111°5 [2853]; ^{1}H NMR [2853].

1-[2-Hydroxy-5-methoxy-4-(phenylmethoxy)phenyl]ethanone

[34176-18-6] $C_{16}H_{16}O_4$ mol.wt. 272.30



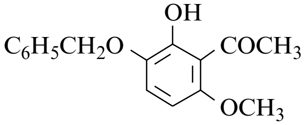
Synthesis

– Preparation by reaction of dimethyl sulfate on 4-(benzyloxy)-2,5-dihydroxyacetophenone with potassium carbonate in refluxing acetone [2417,2823,2824], (85–86%) [2823,2824].

m.p. 130° [2824], 128–129° [2823], 126° [2417].

1-[2-Hydroxy-6-methoxy-3-(phenylmethoxy)phenyl]ethanone

[126405-79-6] $C_{16}H_{16}O_4$ mol.wt. 272.30



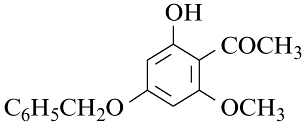
Synthesis

- Preparation by adding 2-hydroxy-3-iodo-6-methoxy-acetophenone and cuprous iodide to a solution of sodium benzylate, previously prepared from benzyl alcohol and sodium hydride in DMF [2524].

Oil [2524]; ^{1}H NMR [2524], IR [2524].

1-[2-Hydroxy-6-methoxy-4-(phenylmethoxy)phenyl]ethanone

[39548-89-5] $C_{16}H_{16}O_4$ mol.wt. 272.30



Syntheses

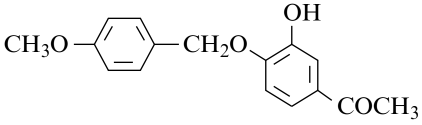
- Preparation by reaction of benzyl chloride on 2,4-dihydroxy-6-methoxyacetophenone with potassium carbonate in boiling acetone [2831,2838,3605], (55%) [3605].
- Preparation by reaction of dimethyl sulfate on 4-(benzyloxy)-2,6-dihydroxyacetophenone with potassium carbonate in refluxing acetone (73%) [2838].

m.p. 90–91° [3605], 73–74° [2838], 72° [2831]. A melting points is obviously wrong.

^{1}H NMR [3605], UV [3605]; TLC [2885].

1-[3-Hydroxy-4-(4-methoxybenzyloxy)phenyl]ethanone

[187966-38-7] $C_{16}H_{16}O_4$ mol.wt. 272.30



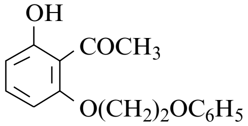
Syntheses

- Preparation by reaction of p-methoxybenzyl bromide with the sodium salt of 3,4-di-hydroxyacetophenone (SM) in DMF at r.t. for 24 h (75%). SM was obtained by adding a solution of 3,4-dihydroxy-acetophenone in DMF to a suspension of sodium hydride (2 mol) in the same solvent [3638].
- Also refer to: [3639].

Crystals [3638] (m.p. not mentioned); ^{1}H NMR [3638].

1-[2-Hydroxy-6-(2-phenoxyethoxy)phenyl]ethanone

[61270-14-2] $C_{16}H_{16}O_4$ mol.wt. 272.30



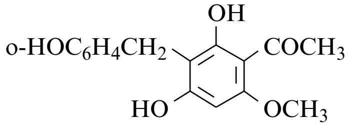
Synthesis

- Preparation by reaction of 2-bromoethoxybenzene with 2,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (34%) [2270].

m.p. 79–80° [2270].

1-[2,4-Dihydroxy-3-[(2-hydroxyphenyl)methyl]-6-methoxyphenyl]ethanone

[102056-82-6] $C_{16}H_{16}O_5$ mol.wt. 288.30



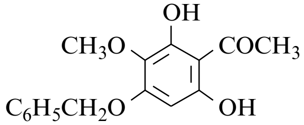
Syntheses

- Preparation by catalytic hydrogenolysis of 3-(o-benzyloxybenzyl)-2,4-dihydroxy-6-methoxy-acetophenone at r.t. and atmospheric pressure in the presence of 10% Pd/C in ethyl acetate (66%) [2325].
- Preparation from the 2′,4′-dihydroxy-3′-[(2-hydroxyphenyl)-(1-piperidino) methyl]-6′-methoxy-acetophenone, the piperidine moiety was removed by catalytic hydrogenation using 10% Pd/C as catalyst (50–60%) [2832].

m.p. 184–185° [2325]; ^{1}H NMR [2325], IR [2325], UV [2325].

1-[2,6-Dihydroxy-3-methoxy-4-(phenylmethoxy)phenyl]ethanone

[204590-48-7] $C_{16}H_{16}O_5$ mol.wt. 288.30



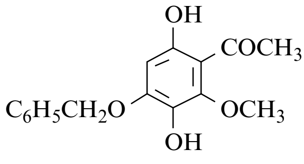
Synthesis

- Preparation by hydrolysis of 4-benzyloxy-6-hydroxy-3-methoxy-2-tosyloxyacetophenone with potassium carbonate in refluxing methanol for 2 h (94%) [3640].

m.p. 149–150° [3640].

1-[3,6-Dihydroxy-2-methoxy-4-(phenylmethoxy)phenyl]ethanone

[25892-94-8] $C_{16}H_{16}O_5$ mol.wt. 288.30



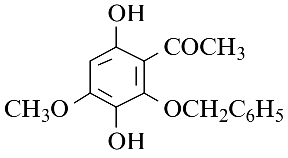
Syntheses

- Obtained from 4-(benzyloxy)-2-hydroxy-6-methoxy-acetophenone by persulfate oxidation (Elbs reaction) [3641], (23%) [2838].
- Also refer to: [3642,3643].

m.p. 161–162° [2838], 109–110° [3641]. One of the reported melting points is obviously wrong. ^{1}H NMR [3641].

1-[3,6-Dihydroxy-4-methoxy-2-(phenylmethoxy)phenyl]ethanone

[41997-38-0] $C_{16}H_{16}O_5$ mol.wt. 288.30



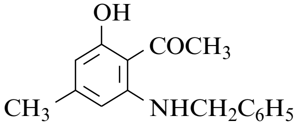
Synthesis

– Obtained from 6-(benzyloxy)-2-hydroxy-4-methoxy-acetophenone by persulfate oxidation (Elbs reaction) (17%) [3604].

m.p. 119–120° [3604]; TLC [2885].

1-[2-Hydroxy-4-methyl-6-[(phenylmethyl)amino]phenyl]ethanone

[97066-16-5] $C_{16}H_{17}NO_2$ mol.wt. 255.32



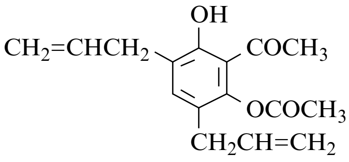
Synthesis

– Preparation by reaction of potassium hydroxide with 2-acetyl-3-benzylamino-5-hydroxy-5-methyl-2-cyclo-hexene-1-one in ethanol at 40° (73%) [2712].

m.p. 160° [2712]; ^{1}H NMR [2712], IR [2712], UV [2712], MS [2712].

1-[2-(Acetyloxy)-6-hydroxy-3,5-di-2-propenylphenyl]ethanone

[117156-76-0] $C_{16}H_{18}O_4$ mol.wt. 274.32



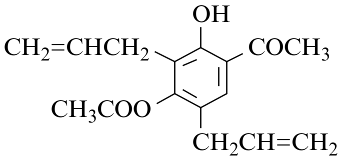
Synthesis

– Obtained (by-product) by thermal rearrangement of 3-acetyl-4,6-bis(allyloxy) acetophenone in refluxing diphenyl ether (3%) [3331].

^{1}H NMR [3331], IR [3331].

1-[4-(Acetyloxy)-2-hydroxy-3,5-di-2-propenylphenyl]ethanone

[106987-29-5] $C_{16}H_{18}O_4$ mol.wt. 274.32



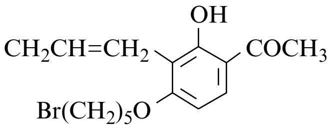
Synthesis

– Obtained (by-product) by thermal Claisen rearrangement of 3-acetyl-4,6-bis(allyloxy) acetophenone or of 3-acetyl-2,4-bis(allyloxy)acetophenone in refluxing N,N-dimethyl-aniline (5–6%) [3331].

Pale greenish yellow oil [3331]; ^{1}H NMR [3331], IR [3331], UV [3331], MS [3331].

1-[4-[(5-Bromopentyl)oxy]-2-hydroxy-3-(2-propenyl)phenyl]ethanone

[61270-23-3]



Oil [2270].

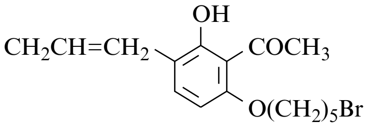
$C_{16}H_{21}BrO_3$ mol.wt. 341.24

Synthesis

- Preparation by reaction of 1,5-dibromopentane with 3-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone [2270].

1-[6-[(5-Bromopentyl)oxy]-2-hydroxy-3-(2-propenyl)phenyl]ethanone

[61270-18-6]



b.p.$_{0.1}$ 180–200° [2270].

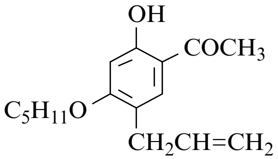
$C_{16}H_{21}BrO_3$ mol.wt. 341.24

Synthesis

- Preparation by reaction of 1,5-dibromopentane with 3-allyl-2,6-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone [2270].

1-[2-Hydroxy-4-(pentyloxy)-5-(2-propenyl)phenyl]ethanone

[117690-49-0]



Oil [2671,2678,2679].

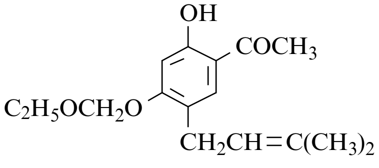
$C_{16}H_{22}O_3$ mol.wt. 262.35

Synthesis

- Preparation by reaction of n-pentyl bromide with 5-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing methyl ethyl ketone (43–44%) [2671,2678,2679].

1-[4-(Ethoxymethoxy)-2-hydroxy-5-(3-methyl-2-butenyl)phenyl]ethanone

[175546-56-2]



^{1}H NMR [3644].

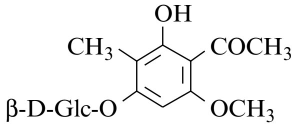
$C_{16}H_{22}O_4$ mol.wt. 278.35

Synthesis

- Preparation by reaction of ethoxymethyl chloride with 2,4-dihydroxy-5-(3-methyl-2-butenyl)acetophenone in acetone for 10 min at 30° (66%) [3644].

1-[4-(β-D-Glucopyranosyloxy)-2-hydroxy-6-methoxy-3-methylphenyl] ethanone

[145194-40-7] $C_{16}H_{22}O_9$ mol.wt. 358.35

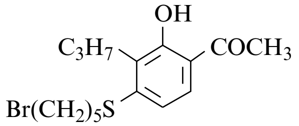


Isolation from natural sources

- From the roots of *Euphorbia ebracteolata* Hayata (Euphorbiaceae) [3040].

1-[4-[(5-Bromopentyl)thio]-2-hydroxy-3-propylphenyl]ethanone

[125617-44-9] $C_{16}H_{23}BrO_2S$ mol.wt. 359.33



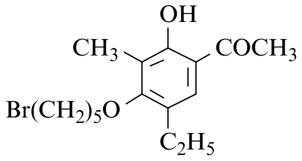
Synthesis

- Preparation by reaction of 1,5-dibromopentane with 2-hydroxy-4-mercapto-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing methyl ethyl ketone [2271].

Pale yellow oil [2271].

1-[4-[(5-Bromopentyl)oxy]-5-ethyl-2-hydroxy-3-methylphenyl]ethanone

[140660-35-1] $C_{16}H_{23}BrO_3$ mol.wt. 343.26



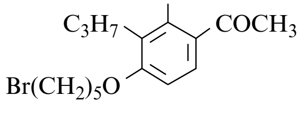
Synthesis

- Preparation by reaction of 1,5-dibromopentane with 5-ethyl-2,4-dihydroxy-3-methylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (76%) [2671].

oil [2671].

1-[4-[(5-Bromopentyl)oxy]-2-hydroxy-3-propylphenyl]ethanone

[99453-85-7] $C_{16}H_{23}BrO_3$ mol.wt. 343.26



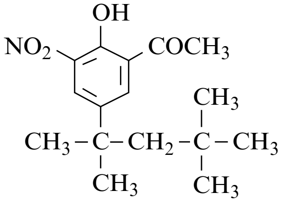
Synthesis

- Preparation by reaction of 1,5-dibromopentane with 2,4-dihydroxy-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone [2270,3181], (22%) [3181].

b.p.$_{0.02}$ 172–180° [2270]; MS [3181].

1-[2-Hydroxy-3-nitro-5-(1,1,3,3-tetramethylbutyl)phenyl]ethanone

[30299-56-0]



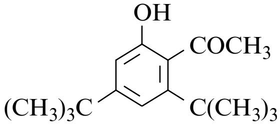
$C_{16}H_{23}NO_4$ mol.wt. 293.36

Synthesis

- Preparation by reaction of 65% nitric acid with 2-hydroxy-5-tert-octylacetophenone in acetic acid, first at 0°, then at 20° [3645].

m.p. 86°5 [3645]; IR [3645], UV [3645].

1-[2,4-Bis(1,1-dimethylethyl)-6-hydroxyphenyl]ethanone



$C_{16}H_{24}O_2$ mol.wt. 248.37

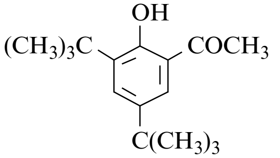
Synthesis

- Obtained by UV light irradiation of 3,5-di-tert-butylphenyl acetate in benzene at r.t. (photo-Fries rearrangement) [3646].

m.p. 198°5 [3646].

1-[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone

[37456-29-4]



$C_{16}H_{24}O_2$ mol.wt. 248.37

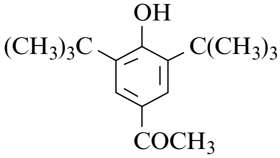
Syntheses

- Preparation by reaction of methylmagnesium iodide on 3,5-di-tert-butyl-2-hydroxybenzonitrile in ethyl ether, first at 0° and then at r.t. (58%) [3647].
- Also obtained by reaction of sec-butyllithium on 2-bromo-4,6-di-tert-butylphenyl acetate in ethyl ether at −95° and −78°, followed by hydrolysis of mixture with saturated ammonium chloride (metal-promoted Fries rearrangement) (43–52%) [3648].
- Preparation by reaction of acetic anhydride with 2,4-di-tert-butylphenol in the presence of boron trifluoride–acetic acid complex at 100° [2997].

m.p. 45–46° [2997], 43–44°5 [3647]; b.p.$_{0.45}$ 100° [2997];
^{1}H NMR [3647], IR [3647].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]ethanone

[14035-33-7]



$C_{16}H_{24}O_2$ mol.wt. 248.37

Syntheses

- Preparation by reaction of acetic acid on 2,6-di-tert-butylphenol with trifluoroacetic anhydride at r.t. [1952,3649–3651], (78–87%) [1952,3651].

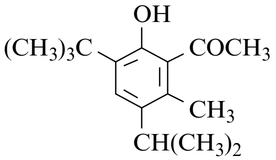
- Preparation by reaction of acetic anhydride on 2,6-di-tert-butylphenol with sulfodifluoroacetic acid in acetic acid at 20° (81%) [3652] or with boron trifluoride etherate at 5° (30%) [3653].
- Preparation by reaction of acetyl chloride on 2,6-di-tert-butylphenol with aluminium chloride at −10° (95%) [3654] and at 0° (70%) [3655].
- Also obtained by reaction of potassium ferricyanide on 2,6-di-tert-butyl-4-(1-methoxyethyl)-phenol with aqueous sodium hydroxide in benzene (20%) [3655].
- Also obtained (poor yield) by bubbling air into a cumene solution of 2,6-di-tert-butyl-4-ethylphenol in the presence of cumene hydroperoxide and cobalt phthalate between 80° and 100° (2%) [3486].

m.p. 150–151°5 [1952,3653], 148° [3486], 147–148° [3650,3651,3655], 146–147° [3654], 141–143° [3652];

^{1}H NMR [3651,3653], ^{13}C NMR [3653], IR [3653], MS [3656].

1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methyl-5-(1-methylethyl)phenyl] ethanone

[129375-13-9] $C_{16}H_{24}O_2$ mol.wt. 248.37



Synthesis

- Preparation by Fries rearrangement of 2-tert-butyl-5-methyl-4-isopropylphenyl acetate with titanium tetrachloride in chlorobenzene at 100° (23%) [3360].

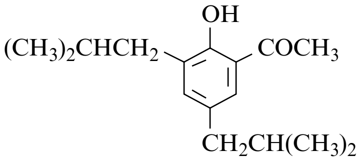
m.p. 44° [3360];

^{1}H NMR [3360] (Sadtler: standard n° 52742 M);

IR [3360] (Sadtler: standard n° 79801 K); UV [3360], MS [3360].

1-[2-Hydroxy-3,5-bis(2-methylpropyl)phenyl]ethanone

[35158-27-1] $C_{16}H_{24}O_2$ mol.wt. 248.37



Synthesis

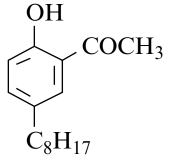
- Preparation by reaction of ethyl acetoacetate with 2-isobutyl-6-methyl-2-heptenal in the presence of pyridine and piperidine as catalysts in refluxing benzene (29%). The 2-isobutyl-6-methyl-2-heptenal was obtained by self-condensation of iso-capro-aldehyde in the presence of 15% potassium hydroxide solution (Aldol condensation) [2958–2960].

b.p.$_{1-2}$ 116–118° [2958,2959,2960];

IR [2958–2960], UV [2958–2960], MS [2958–2960].

1-(2-Hydroxy-5-octylphenyl)ethanone

[74604-19-6] $C_{16}H_{24}O_2$ mol.wt. 248.37



Synthesis

- Preparation by reaction of acetyl chloride on 4-octylphenol with aluminium chloride in ethylene dichloride at 110–120° (58%) [2625].

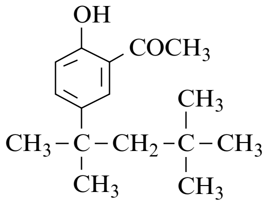
b.p.$_4$ 168–170° [2625].

1-[2-Hydroxy-5-(1,1,3,3-tetramethylbutyl)phenyl]ethanone

[57373-80-5] $C_{16}H_{24}O_2$ mol.wt. 248.37

and

[30299-53-7] (2′-Hydroxy-5′-(1,1,3,3-tetramethylbutyl)acetophenone)



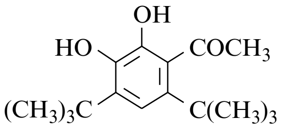
Syntheses

- Obtained by Fries rearrangement of 4-(1,1,3,3-tetramethyl-butyl)phenyl acetate also called 4-tert-octylphenyl acetate with aluminium chloride [3355,3357], in 1,2,3-trichloro-propane or in tetrachloroethane at 120° under nitrogen (71–77%) [3357]; (high yield) [3355].
- Preparation by demethylation of 2-methoxy-5-tert-octyl-acetophenone with 4% hydrobromic acid in refluxing acetic acid (47–52%) [3355].
- Preparation by UV light irradiation of 4-tert-octylphenyl acetate in benzene or in ethanol solution (37%) (photo-Fries rearrangement) [3645].

^{1}H NMR [3645], IR [3645], UV [3645].

1-[4,6-Bis(1,1-dimethylethyl)-2,3-dihydroxyphenyl]ethanone

[84296-99-1] $C_{16}H_{24}O_3$ mol.wt. 264.37



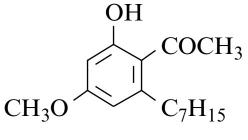
Synthesis

- Obtained by irradiation of a benzene solution of 3,5-di-tert-butyl-o-benzoquinone in the presence of a large excess of acetaldehyde (6%) [3386].

m.p. 169–170° [3386]; ^{1}H NMR [3386], IR [3386].

1-(2-Heptyl-6-hydroxy-4-methoxyphenyl)ethanone

[4670-13-7] $C_{16}H_{24}O_3$ mol.wt. 264.37



Synthesis

- Preparation by partial methylation of 2,4-dihydroxy-6-heptylacetophenone in acetone with dimethyl sulfate in the presence of 10% sodium hydroxide at 45° for 4 h (70%) [3623].

Isolation from natural sources

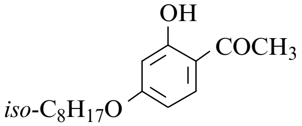
- Obtained by alkaline degradation of various *siphulin* derivatives* with potassium hydroxide in refluxing methanol [3657]. The siphulin (an homoflavone) 7-hydroxy-5-heptyl-2-[3′,5′-dihydroxy-2-carboxybenzyl]chroman-4-one (m.p. 180°) [3658] is a constituent of the North Scandinavican lichen *siphula ceratites* (Wahlenberg) Fr.

* siphulin methyl ester trimethyl ether [3658], decarboxysiphulin trimethyl ether or a lactol.

Oil [3623,3657], liquid compound [3658];
b.p.$_{0.01}$ 110° [3658], b.p.$_{0.01}$ 120° [3657]; n_D^{18} = 1.5372 [3657], n_D^{19} = 1.5339 [3658];
^{1}H NMR [3623,3657], IR [3657,3658], UV [3657,3658].

1-[2-Hydroxy-4-(isooctyloxy)phenyl]ethanone

[127313-67-1] $C_{16}H_{24}O_3$ mol.wt. 264.37

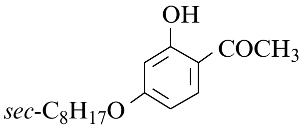


Synthesis

- Refer to: [3659] (Chinese paper).

1-[2-Hydroxy-4-(sec-octyloxy)phenyl]ethanone

[127313-63-7] $C_{16}H_{24}O_3$ mol.wt. 264.37

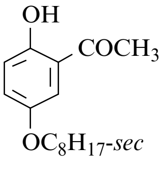


Synthesis

- Refer to: [3659] (Chinese paper).

1-[2-Hydroxy-5-(sec-octyloxy)phenyl]ethanone

[127313-62-6] $C_{16}H_{24}O_3$ mol.wt. 264.37

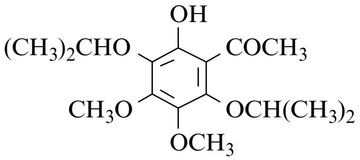


Synthesis

- Refer to: [3659] (Chinese paper).

1-[2-Hydroxy-4,5-dimethoxy-3,6-bis(1-methylethoxy)phenyl]ethanone

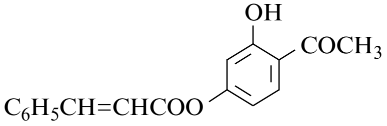
[169130-25-0] $C_{16}H_{24}O_6$ mol.wt. 312.36



Synthesis

- Preparation by smooth demethylation of 2,5-diiso-propyloxy-3,4,6-trimethoxyacetophenone with aluminium bromide in acetonitrile (high yield) [3320].

1-[4-(Cinnamoyloxy)-2-hydroxyphenyl]ethanone



$C_{17}H_{14}O_4$ mol.wt. 282.30

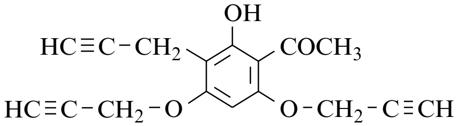
Synthesis

- Obtained by reaction of cinnamoyl chloride on resacetophenone with potassium carbonate in toluene (17%) [2613].

m.p. 131° [2613].

1-[2-Hydroxy-3-(2-propynyl)-4,6-bis(2-propynyloxy)phenyl]ethanone

[53771-25-8]



$C_{17}H_{14}O_4$ mol.wt. 282.30

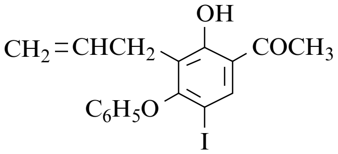
Synthesis

- Obtained (poor yield) by reaction of 2-propynyl bromide with phloroacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (4%) [3160].

m.p. 150–151° [3160]; IR [3160], UV [3160].

1-[2-Hydroxy-5-iodo-4-phenoxy-3-(2-propenyl)phenyl]ethanone

[144691-36-1]



$C_{17}H_{15}IO_3$ mol.wt. 394.21

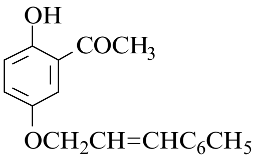
Synthesis

- Obtained by reaction of iodosobenzene diacetate (DAIB) with 3-allyl-2,4-dihydroxyacetophenone in refluxing methanol (38%) [2624].

m.p. 160° [2624]; ^{1}H NMR [2624], IR [2624].

1-[2-Hydroxy-5-[(3-phenyl-2-propenyl)oxy]phenyl]ethanone

[79950-56-4]



$C_{17}H_{16}O_3$ mol.wt. 268.31

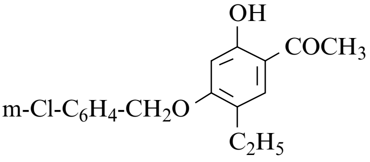
Synthesis

- Preparation by reaction of cinnamyl bromide with quinacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone under nitrogen (90%) [2357].

m.p. 105–107° [2357]; ^{1}H NMR [2357], IR [2357], MS [2357].

1-[4-[(3-Chlorophenyl)methoxy]-5-ethyl-2-hydroxyphenyl]ethanone

[117706-49-7] $C_{17}H_{17}ClO_3$ mol.wt. 304.77

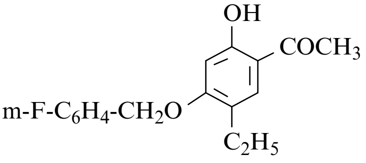


Synthesis

- Preparation by reaction of 3-chlorobenzyl bromide with 5-ethyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide (47%) [2678,2679].

1-[5-Ethyl-4-[(3-fluorophenyl)methoxy]-2-hydroxyphenyl]ethanone

[117706-48-6] $C_{17}H_{17}FO_3$ mol.wt. 288.32



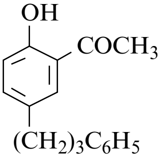
Synthesis

- Preparation by reaction of 3-fluorobenzyl bromide with 5-ethyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide (18%) [2678,2679].

m.p. 104–105° [2678,2679].

1-[2-Hydroxy-5-(3-phenylpropyl)phenyl]ethanone

[61270-17-5] $C_{17}H_{18}O_2$ mol.wt. 254.33



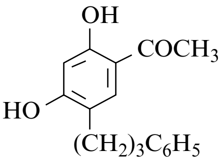
Syntheses

- Preparation by Fries rearrangement of 4′-acetoxy-1,3-diphenylpropane with aluminium chloride in boiling chlorobenzene [3579].
- Preparation by reaction of acetic anhydride with 4-(3-phenylpropyl)phenol in the presence of boron trifluoride–acetic acid complex at 100° [2270].

Oil [2270]; b.p.$_{18.5}$ 232° [3579]; ^{1}H NMR [2270], MS [2270].

1-[2,4-Dihydroxy-5-(3-phenylpropyl)phenyl]ethanone

$C_{17}H_{18}O_3$ mol.wt. 270.33



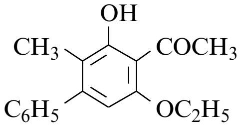
Synthesis

- Preparation by Fries rearrangement of 2′,4′-diacetoxy-1,3-diphenylpropane with aluminium chloride in the presence of 2′,4′-dihydroxy-1,3-diphenylpropane in nitrobenzene at 50° [3579].

m.p. 106° [3579].

1-(5-Ethoxy-3-hydroxy-2-methyl[1,1′-biphenyl]-4-yl)ethanone

[138151-67-4] $C_{17}H_{18}O_3$ mol.wt. 270.33



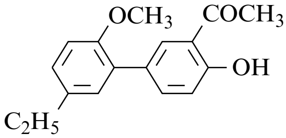
Synthesis

- Preparation by heating at 100° for 18 h a dioxane solution of 4-chloro-2-methyl-3-phenyl-2-cyclobutenone and 4-ethoxy-4-(tri-n-butylstannyl)-3-buten-2-one (**I**) with Pd(benzonitrile)$_2$Cl$_2$ and tris(2-furyl)phosphine (50%).
- The compound (**I**) was obtained by adding a tetrahydrofuran solution of tetrabutylammonium cyanide (Bu$_4$NCN) to a tetrahydrofuran solution of 3-ethoxy-2-cyclobutenone and n-(C$_4$H$_9$)$_3$ SnSi(CH$_3$)$_3$ cooled to −22° and then the mixture was warmed at r.t. [3660].

m.p. 78–80° [3660]; ^{1}H NMR [3660], ^{13}C NMR [3660], IR [3660].

1-(5′-Ethyl-4-hydroxy-2′-methoxy[1,1′-biphenyl]-3-yl)ethanone

[131845-25-5] $C_{17}H_{18}O_3$ mol.wt. 270.33

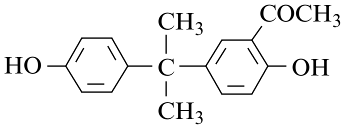


Synthesis

- Refer to: [3661].

1-[2-Hydroxy-5-[1-(4-hydroxyphenyl)-1-methylethyl]phenyl]ethanone

[104676-26-8] $C_{17}H_{18}O_3$ mol.wt. 270.33



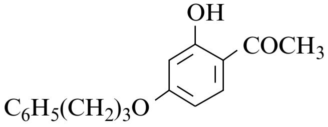
Synthesis

- Obtained by Fries rearrangement of 2,2-bis (acetoxy-phenyl)propane (bisphenol-A diacetate) (1 mol) with titanium tetrachloride (4 mol) in nitrobenzene, first for 24 h at r.t., then for 6 h at 55° (23%) [3662].

m.p. 139–140° [3662]; ^{1}H NMR [3662], ^{13}C NMR [3662], IR [3662].

1-[2-Hydroxy-4-(3-phenylpropoxy)phenyl]ethanone

[63359-86-4] $C_{17}H_{18}O_3$ mol.wt. 270.33



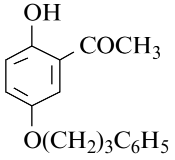
Syntheses

- Obtained by reaction of 1-bromo-3-phenylpropane with resacetophenone in the presence of potassium carbonate in refluxing acetone for 24 h. The 1-chloro derivative and butanone can also be used instead of the mentioned starting materials (83%) [3204].
- Also refer to: [3663].

m.p. 75–77° [3204].

1-[2-Hydroxy-5-(3-phenylpropoxy)phenyl]ethanone

[63359-87-5] $C_{17}H_{18}O_3$ mol.wt. 270.33



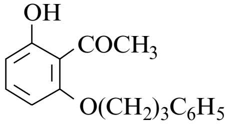
Syntheses

- Obtained by reaction of 1-bromo-3-phenylpropane with quinacetophenone in the presence of potassium carbonate in refluxing acetone for 24 h. The 1-chloro derivative and butanone can also be used instead of the mentioned starting material and solvent (54%) [3204].
- Also refer to: [3663].

m.p. 34–35° [3204].

1-[2-Hydroxy-6-(3-phenylpropoxy)phenyl]ethanone

[69079-93-2] $C_{17}H_{18}O_3$ mol.wt. 270.33



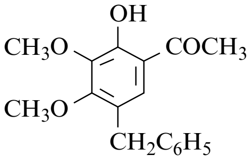
Synthesis

- Obtained by reaction of 1-bromo-3-phenylpropane with 2,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 24 h. The 1-chloro derivative and butanone can also be used instead of the mentioned starting material and solvent (75%) [3204].

m.p. 95–96° [3204].

1-[2-Hydroxy-3,4-dimethoxy-5-(phenylmethyl)phenyl]ethanone

[105485-57-2] $C_{17}H_{18}O_4$ mol.wt. 286.33



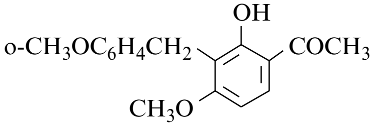
Synthesis

- Preparation by reaction of dimethyl sulfate with 5-benzyl-2,3,4-trihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (68%) [3583].

m.p. 42–43° [3583]; ^{1}H NMR [3583], IR [3583], UV [3583].

1-[2-Hydroxy-4-methoxy-3-[(2-methoxyphenyl)methyl]phenyl]ethanone

[103633-39-2] $C_{17}H_{18}O_4$ mol.wt. 286.33

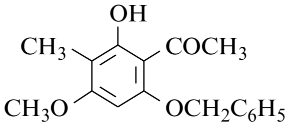


Synthesis

- Obtained by reaction of dimethyl sulfate with 2,4-di-hydroxy-3-(o-hydroxybenzyl)acetophenone in the presence of potassium carbonate in refluxing acetone (9%) [2325].

Oil [2325]; ^{1}H NMR [2325].

1-[2-Hydroxy-4-methoxy-3-methyl-6-(phenylmethoxy)phenyl]ethanone



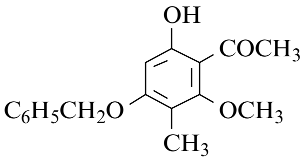
$C_{17}H_{18}O_4$ mol.wt. 286.33

Synthesis

- Preparation by reaction of methyl iodide with 6-(benzyloxy)-2,4-dihydroxy-3-methylacetophenone in the presence of potassium carbonate in refluxing acetone (88%) [3043].

m.p. 127° [3043].

1-[6-Hydroxy-2-methoxy-3-methyl-4-(phenylmethoxy)phenyl]ethanone



$C_{17}H_{18}O_4$ mol.wt. 286.33

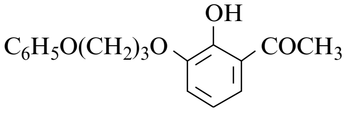
Syntheses

- Preparation by reaction of benzyl bromide on 4,6-di-hydroxy-2-methoxy-3-methylacetophenone with potassium carbonate in boiling acetone (48%) [3051].
- Preparation by partial catalytic hydrogenolysis of 4,6-bis-(benzyloxy)-2-methoxy-3-methylacetophenone with $PdCl_2$/C in methanol (86%) [3051] or by reaction of 10% ethanolic hydrochloric acid on the same starting material in refluxing dioxane (42%) [3043].

m.p. 105° [3051], 103° [3043].

1-[2-Hydroxy-3-(3-phenoxypropoxy)phenyl]ethanone

[69079-92-1]



$C_{17}H_{18}O_4$ mol.wt. 286.33

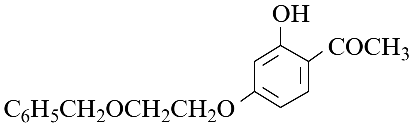
Synthesis

- Obtained by reaction of 1-bromo-3-phenoxypropane with 2,3-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 24 h. The 1-chloro derivative and butanone can also be used instead of the mentioned starting material and solvent (32%) [3204].

m.p. 56–57° [3204].

1-[2-Hydroxy-4-[2-(phenylmethoxy)ethoxy]phenyl]ethanone

[307520-94-1]



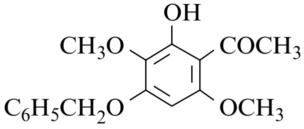
$C_{17}H_{18}O_4$ mol.wt. 286.33

Synthesis

- Refer to: [3596] (compound **1d**).

1-[2-Hydroxy-3,6-dimethoxy-4-(phenylmethoxy)phenyl]ethanone

[3162-52-5] $C_{17}H_{18}O_5$ mol.wt. 302.33



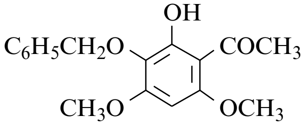
Syntheses

- Preparation by reaction of benzyl chloride with 2,4-di-hydroxy-3,6-dimethoxyacetophenone in the presence of potassium carbonate [3135,3472] or potassium carbonate and potassium iodide [3130] in refluxing acetone (72%) [3472], (63%) [3130], (53%) [3135]. The same reaction using benzyl bromide instead of benzyl chloride led to an inseparable mixture (68%) of 2-benzyl- and 4-benzyl ethers (m.p. 105–107°) [3137].
- Preparation by reaction of acetyl chloride on 2,5-dimethoxyresorcinol dibenzyl ether with aluminium chloride in benzene at 0° (35%) [3472].
- Obtained (by-product) by reaction of acetonitrile with 2,6-bis(benzyloxy)-1,4-dimethoxybenzene (Hoesch reaction) [3135].

m.p. 113–115° [3130], 111–111°5 [3135], 109°5–110° [3472].

1-[2-Hydroxy-4,6-dimethoxy-3-(phenylmethoxy)phenyl]ethanone

[54299-57-9] $C_{17}H_{18}O_5$ mol.wt. 302.33



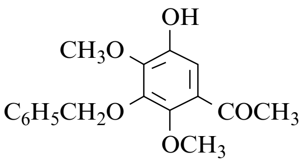
Syntheses

- Preparation by reaction of benzyl chloride with 2,3-di-hydroxy-4,6-dimethoxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (56%) [3130].
- Preparation from 2,3-bis(benzyloxy)-4,6-dimethoxy-acetophenone. The 2-benzyloxy group was selectively split with concentrated hydrochloric acid in acetic acid at r.t. (80%) [3132].

m.p. 95–97° [3132], 90–92° [3130].

1-[5-Hydroxy-2,4-dimethoxy-3-(phenylmethoxy)phenyl]ethanone

[65039-99-8] $C_{17}H_{18}O_5$ mol.wt. 302.33

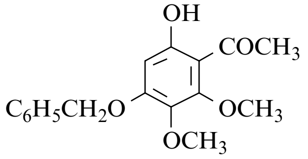


Synthesis

- Obtained [3664] according to the procedure [3665].

1-[6-Hydroxy-2,3-dimethoxy-4-(phenylmethoxy)phenyl]ethanone

[25892-95-9] $C_{17}H_{18}O_5$ mol.wt. 302.33



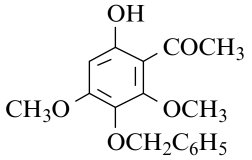
Syntheses

- Obtained by partial methylation of 4-(benzyloxy)-3,6-di-hydroxy-2-methoxyacetophenone [3130,3148,3507,3641,3666–3669], with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone [3643], for 5 h (57%) [3642].
- Also refer to: [3670,3671].

m.p. 86–87° [3642], 84–85° [3641]; IR [3642].

1-[6-Hydroxy-2,4-dimethoxy-3-(phenylmethoxy)phenyl]ethanone

[52249-87-3] $C_{17}H_{18}O_5$ mol.wt. 302.33



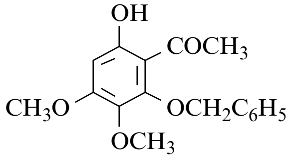
Syntheses

- Preparation by reaction of benzyl chloride with 3,6-di-hydroxy-2,4-dimethoxyacetophenone,
  - in the presence of potassium carbonate and sodium iodide in refluxing acetone for 10 h, then at r.t. for 6 h [3669,3672], (57%) [3665];
  - in the presence of potassium carbonate in DMF [3673].
- Also refer to: [3664,3674–3678].

Oil [3665,3673]; ^{1}H NMR [3673].

1-[6-Hydroxy-3,4-dimethoxy-2-(phenylmethoxy)phenyl]ethanone

[41997-39-1] $C_{17}H_{18}O_5$ mol.wt. 302.33

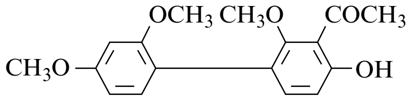


Synthesis

- Preparation by reaction of dimethyl sulfate on 6-(benzyloxy)-2,5-dihydroxy-4-methoxyacetophenone with potassium carbonate in refluxing acetone (57%) [3604].

m.p. 87–89° [3604].

1-(4-Hydroxy-2,2′,4′-trimethoxy[1,1′-biphenyl]-3-yl)ethanone



$C_{17}H_{18}O_5$ mol.wt. 302.33

Syntheses

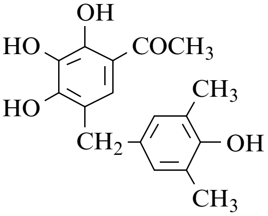
- Obtained by alkaline degradation of 2-(2,4-di-methoxyphenyl)-1,9-di-O-methylhemiergo-flavinone ($C_{25}H_{24}O_9$) with 10% sodium hydroxide on a steam bath for 1.75 h [3679].

- Also obtained by alkaline degradation of 6-(2,4-dimethoxyphenyl)-5-methoxy-2-methylchromone (m.p. 141–142°) with 25%(w/v) aqueous sodium hydroxide during 2 h on a steam bath (72%) [2569].

m.p. 94° [3679], 93° [2569]; IR [2569].

1-[2,3,4-Trihydroxy-5-[(4-hydroxy-3,5-dimethylphenyl)methyl]phenyl] ethanone

[142045-74-7] $C_{17}H_{18}O_5$ mol.wt. 302.33

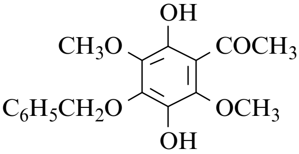


Synthesis

- Refer to: [3680] (Japanese patent).

1-[2,5-Dihydroxy-3,6-dimethoxy-4-(phenylmethoxy)phenyl]ethanone

[3162-50-3] $C_{17}H_{18}O_6$ mol.wt. 318.33



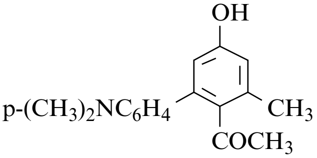
Syntheses

- Obtained by oxidation of 4-(benzyloxy)-2-hydroxy-3,6-di-methoxyacetophenone with potassium persulfate (Elbs reaction), (46%) [3428], (14%) [3681].
- Also refer to: [3150,3682–3684].

m.p. 60–62° [3681], 59–61° [3428];
IR [3428]; TLC [3681].

1-[4′-(Dimethylamino)-5-hydroxy-3-methyl[1,1′-biphenyl]-2-yl]ethanone

[108909-48-4] $C_{17}H_{19}NO_2$ mol.wt. 269.34



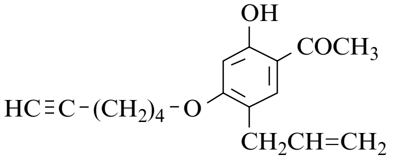
Syntheses

- Obtained by aromatization of 4-acetyl-5-[4-(dimethyl-amino)phenyl]-3-methyl-2-cyclohexen-1-one (m.p. 162°) with bromine in chloroform (70%) or by heating at 170° for 3 h [3628].
- Also obtained by deacylation of 1,1′-[4′-(dimethylamino)-3-hydroxy-5-methyl[1,1′-biphenyl]-2,6-diyl]bis-ethanone (m.p. 153°) with sodium hydroxide in refluxing dilute ethanol for 3 h (70%) [3628].
- Also refer to: [3629].

m.p. 192° [3628].

1-[4-(5-Hexynyloxy)-2-hydroxy-5-(2-propenyl)phenyl]ethanone

[117706-50-0]



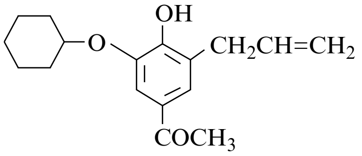
$C_{17}H_{20}O_3$ mol.wt. 272.34

Synthesis

- Preparation by reaction of 6-bromo-1-hexyne with 5-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing methyl ethyl ketone (15–20%) [2671,2678,2679].

Oil [2671]; m.p. <25° [2678,2679]; ^{1}H NMR [2678,2679].

1-[3-(Cyclohexyloxy)-4-hydroxy-5-(2-propenyl)phenyl]ethanone



$C_{17}H_{22}O_3$ mol.wt. 274.36

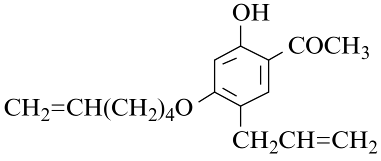
Synthesis

- Preparation by thermal Claisen rearrangement of 4-(allyloxy)-3-cyclohexyloxyacetophenone in boiling N,N-diethylaniline (69%) [2946].

m.p. 58° [2946]; b.p.$_{0.6}$ 170–180° [2946].

1-[4-(5-Hexenyloxy)-2-hydroxy-5-(2-propenyl)phenyl]ethanone

[117690-54-7]



$C_{17}H_{22}O_3$ mol.wt. 274.36

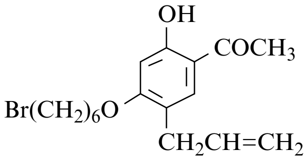
Synthesis

- Preparation by reaction of 6-bromo-1-hexene with 5-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing methyl ethyl ketone (30%) [2671,2678,2679].

Oil [2671]; m.p. <25° [2678,2679].

1-[4-[(6-Bromohexyl)oxy]-2-hydroxy-5-(2-propenyl)phenyl]ethanone

[117706-41-9]

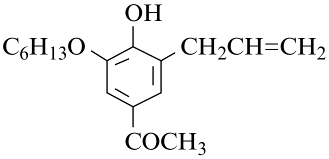


$C_{17}H_{23}BrO_3$ mol.wt. 355.27

Synthesis

- Preparation by reaction of 1,6-dibromohexane with 5-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide at reflux (42%) [2678,2679].

1-[3-(Hexyloxy)-4-hydroxy-5-(2-propenyl)phenyl]ethanone



$C_{17}H_{24}O_3$ mol.wt. 276.38

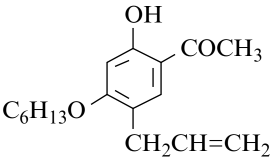
Synthesis

- Preparation by thermal Claisen rearrangement of 4-(allyloxy)-3-(hexyloxy)acetophenone without solvent at 200° (47%) [2946].

m.p. 83° [2946]; b.p.$_{0.9}$ 175–180° [2946].

1-[4-(Hexyloxy)-2-hydroxy-5-(2-propenyl)phenyl]ethanone

[117690-47-8]



$C_{17}H_{24}O_3$ mol.wt. 276.38

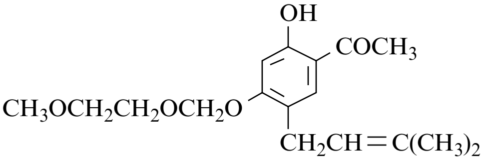
Synthesis

- Preparation by reaction of hexyl bromide with 5-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing methyl ethyl ketone (85%) [2671], (36%) [2678,2679].

m.p. 42–44° [2671,2678,2679].

1-[2-Hydroxy-4-[(2-methoxyethoxy)methoxy]-5-(3-methyl-2-butenyl)phenyl] ethanone

[181047-51-8]



$C_{17}H_{24}O_5$ mol.wt. 308.37

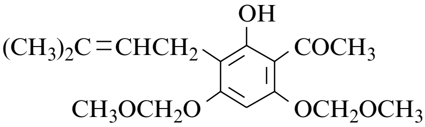
Synthesis

- Preparation by treatment of 4-(2-methoxy-ethoxymethoxy)-2-(3,3-dimethylallyloxy)-acetophenone in refluxingN,N-diethyl-aniline at 220° for 4 h under argon atmosphere (77%) (Claisen rearrangement) [3408].

^1H NMR [3408], ^{13}C NMR [3408], MS [3408].

1-[2-Hydroxy-4,6-bis(methoxymethoxy)-3-(3-methyl-2-butenyl)phenyl] ethanone

[84092-45-5]



$C_{17}H_{24}O_6$ mol.wt. 324.37

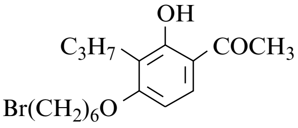
Syntheses

- Obtained by reaction of prenyl bromide with 2-hydroxy-4,6-di-(methoxymethoxy)aceto-phenone in methanolic potassium hydroxide solution, first at 0°, then at r.t. for 24 h (74%) [3413].
- Also refer to: [3685].

liquid [3413]; ^{1}H NMR [3413], IR [3413], UV [3413].

1-[4-[(6-Bromohexyl)oxy]-2-hydroxy-3-propylphenyl]ethanone

[92518-46-2]

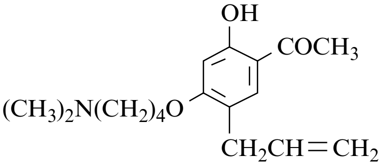


$C_{17}H_{25}BrO_3$ mol.wt. 357.29

Synthesis

- Preparation by reaction of 1,6-dibromohexane with 2,4-di-hydroxy-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (37%) [3181].

1-[4-[4-(Dimethylamino)butoxy]-2-hydroxy-5-(2-propenyl)phenyl]ethanone



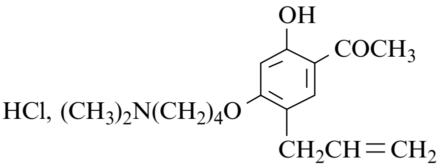
$C_{17}H_{25}NO_3$ mol.wt. 291.39

Synthesis

- Preparation by reaction of dimethylamine with 5-allyl-4-(4-bromobutoxy)-2-hydroxyacetophenone during 16 h [2671].

1-[4-[4-(Dimethylamino)butoxy]-2-hydroxy-5-(2-propenyl)phenyl]ethanone *(Hydrochloride)*

[117706-32-8]



$C_{17}H_{25}NO_3$, HCl mol.wt. 327.85

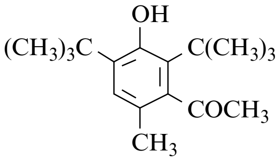
Synthesis

- Obtained by reaction of hydrogen chloride with the corresponding base in ethanol, then adding ethyl ether to the mixture [2671].

m.p. 88–90° [2671].

1-[2,4-Bis(1,1-dimethylethyl)-3-hydroxy-6-methylphenyl]ethanone

[175438-44-5] $C_{17}H_{26}O_2$ mol.wt. 262.39



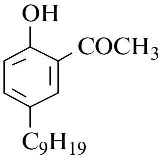
Synthesis

- Refer to: Chem. Abstr., **124**, 260501q (1995).

N.B.: this ketone is erroneously referenced in the Chemical Abstracts (Vol. **124**, 1996, Formula Index, 2675F). The compound actually obtained by reaction between acetic anhydride and 2,6-di-tert-butyl-6-methylphenol in the presence of various metal bis(trifluoromethylsulfonyl)-amides such as a titanium and ytterbium bistrifylamides in methylene chloride or acetonitrile at r.t. is the phenolic ester, i.e. the 2,6-di-tert-butyl-6-methylphenyl acetate (90–99%), which has been unambiguously characterized [3686] (personal communication from professor Koichi Mikami).

1-(2-Hydroxy-5-nonylphenyl)ethanone

[115851-77-9] $C_{17}H_{26}O_2$ mol.wt. 262.39



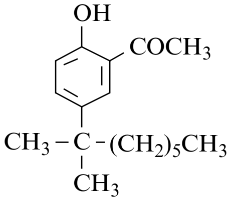
Synthesis

- Preparation by reaction of acetyl chloride on 4-nonylphenol with aluminium chloride in ethylene dichloride at 110–120° (63%) [2625].

b.p.$_4$ 178–182° [2625].

1-(2-Hydroxy-5-tert-nonylphenyl)ethanone

[57375-45-8] $C_{17}H_{26}O_2$ mol.wt. 262.39

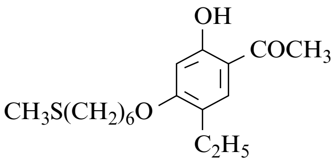


Synthesis

- Preparation by Fries rearrangement of 4-tert-nonylphenyl acetate with aluminium chloride under nitrogen in tetra-chloroethane or in tetrachloroethylene at 120–125° (77–79%) [3357] or in refluxing chlorobenzene (46%) [3357].

1-[5-Ethyl-2-hydroxy-4-[[6-(methylthio)hexyl]oxy]phenyl]ethanone

[117706-37-3] $C_{17}H_{26}O_3S$ mol.wt. 310.46



Synthesis

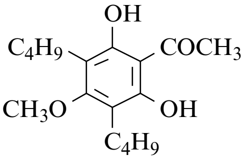
- Preparation by adding a DMF solution of 4-(6-bromo-hexyloxy)-5-ethyl-2-hydroxy acetophenone to a DMF solution of methanethiol previously treated with sodium hydride (78%) [2671,2678,2679].

m.p. 52–53° [2678,2679], 47–48° [2671];

^{1}H NMR [2671,2678,2679], MS [2671].

1-(3,5-Dibutyl-2,6-dihydroxy-4-methoxyphenyl)ethanone

[175785-88-3] $C_{17}H_{26}O_4$ mol.wt. 294.39

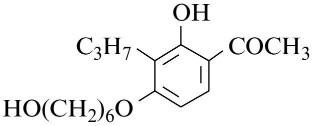


Synthesis

– Refer to: [3495] (Japanese patent).

1-[2-Hydroxy-4-[(6-hydroxyhexyl)oxy]-3-propylphenyl]ethanone

[106627-20-7] $C_{17}H_{26}O_4$ mol.wt. 294.39



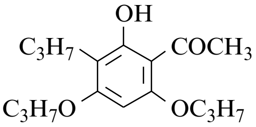
Synthesis

– Preparation by reaction of 6-chlorohexanol with 2,4-di-hydroxy-3-propylacetophenone in the presence of potassium carbonate in refluxing methyl ethyl ketone (20%) [3181].

Oily solid [3181]; MS [3181].

1-[2-Hydroxy-3-propyl-4,6-bis(propyloxy)phenyl]ethanone

$C_{17}H_{26}O_4$ mol.wt. 294.39



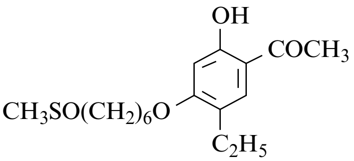
Synthesis

– Preparation by catalytic hydrogenation of 2-hydroxy-3-(2-propynyl)-4,6-bis(2-propynyloxy)acetophenone in the presence of 10% Pd/C in ethanol (77%) [3160].

m.p. 78–80° [3160].

1-[5-Ethyl-2-hydroxy-4-[[6-(methylsulfinyl)hexyl]oxy]phenyl]ethanone

[117706-38-4] $C_{17}H_{26}O_4S$ mol.wt. 326.46



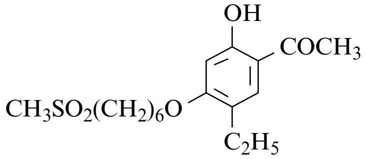
Synthesis

– Obtained (by-product) by reaction of m-chloro-perbenzoic acid on 2-hydroxy-4-[6-(methylthio)-hexyloxy]-5-ethylacetophenone in methylene chloride, first at 0°, then at r.t. (17%) [2671,2678,2679].

m.p. 87–90° [2678,2679], 87–89° [2671].

1-[5-Ethyl-2-hydroxy-4-[[6-(methylsulfonyl)hexyl]oxy]phenyl]ethanone

[117690-76-3] $C_{17}H_{26}O_5S$ mol.wt. 342.46



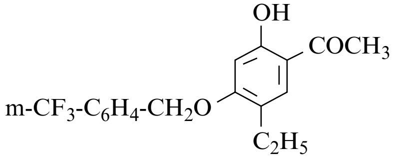
Synthesis

- Preparation by reaction of m-chloroperbenzoic acid on 5-ethyl-2-hydroxy-4-[6-(methylthio)hexyloxy]-acetophenone in methylene chloride, first at 0°, then at r.t. (70–76%) [2671,2678,2679].

m.p. 124–126° [2671,2678,2679].

1-[5-Ethyl-2-hydroxy-4-[[3-(trifluoromethyl)phenyl]methoxy]phenyl] ethanone

[117706-51-1] $C_{18}H_{17}F_3O_3$ mol.wt. 338.33

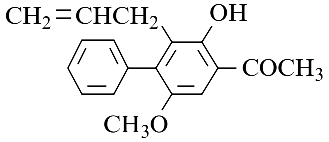


Synthesis

- Preparation by reaction of 3-(trifluoromethyl)benzyl bromide with 5-ethyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide (11%) [2678,2679].

1-[3-Hydroxy-6-methoxy-2-(2-propenyl)[1,1′-biphenyl]-4-yl]ethanone

[43037-65-6] $C_{18}H_{18}O_3$ mol.wt. 282.34

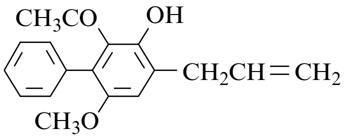


Synthesis

- Obtained by thermal Claisen rearrangement of 1-[2-methoxy-5-(2-propenyloxy)[1, 1′-biphenyl]-4-yl]ethanone in N,N-dimethylaniline at 170° (52%) [3467].

1-[3-Hydroxy-6-methoxy-4-(2-propenyl)[1,1′-biphenyl]-2-yl]ethanone

[43037-67-8] $C_{18}H_{18}O_3$ mol.wt. 282.34

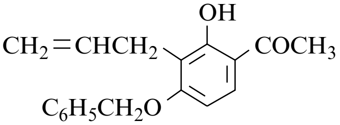


Synthesis

- Obtained by thermal Claisen rearrangement of 1-[2-methoxy-5-(2-propenyloxy)[1, 1′-biphenyl]-4-yl]ethanone in N,N-dimethylaniline at 170° (33%) [3467]. The formation of this ketone is rationalised as involving a [2368,,3466] acetyl shift.

1-[2-Hydroxy-4-(phenylmethoxy)-3-(2-propenyl)phenyl]ethanone

[137170-49-1] $C_{18}H_{18}O_3$ mol.wt. 282.34

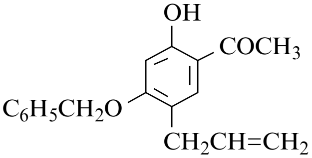


Syntheses

- Refer to: [3268,3687] (patents).

1-[2-Hydroxy-4-(phenylmethoxy)-5-(2-propenyl)phenyl]ethanone

[117690-55-8] $C_{18}H_{18}O_3$ mol.wt. 282.34



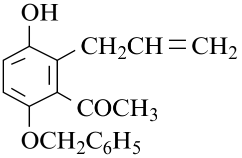
Synthesis

- Preparation by reaction of benzyl bromide on 5-allyl-2,4-dihydroxyacetophenone with potassium carbonate and potassium iodide (60%) [2678,2679].

m.p. 86° [2678,2679].

1-[3-Hydroxy-6-(phenylmethoxy)-2-(2-propenyl)phenyl]ethanone

$C_{18}H_{18}O_3$ mol.wt. 282.34

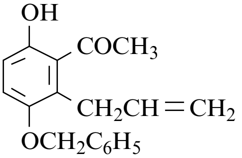


Synthesis

- Obtained (by-product) by reaction of benzyl bromide with 2-allyl-3,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 5.5 h under nitrogen atmosphere (3%) [3471].

1-[6-Hydroxy-3-(phenylmethoxy)-2-(2-propenyl)phenyl]ethanone

[263138-72-3] $C_{18}H_{18}O_3$ mol.wt. 282.34



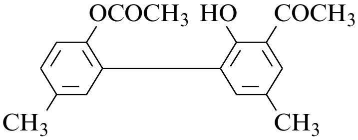
Synthesis

- Obtained by reaction of benzyl bromide with 2-allyl-3,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 5.5 h under nitrogen atmosphere (54%) [3471].

m.p. 92–93° [3471]; ^{1}H NMR [3471], ^{13}C NMR [3471], IR [3471], MS [3471].

1-(2′-Acetoxy-2-hydroxy-5,5′-dimethyl[1,1′-biphenyl]-3-yl)ethanone

[24046-01-3] $C_{18}H_{18}O_4$ mol.wt. 298.34



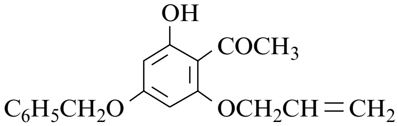
Synthesis

- Obtained by stirring a solution of 1-(2,2′-dihydroxy-5,5′-dimethyl[1,1′-biphenyl]-3-yl)ethanone in pyridine with freshly fused sodium acetate for 1 h (95%) [3633].

Pale yellow oil [3633]; b.p.$_{10^{-5}}$ 120° [3633].

1-[2-Hydroxy-4-(phenylmethoxy)-6-(2-propenyloxy)phenyl]ethanone

[76609-36-4] $C_{18}H_{18}O_4$ mol.wt. 298.34



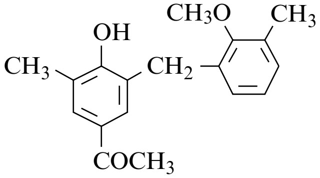
m.p. 64–65° [3190].

Synthesis

– Obtained by partial benzylation of 6-(allyloxy)-2,4-dihydroxyacetophenone [3190].

1-[4-Hydroxy-3-[(2-methoxy-3-methylphenyl)methyl]-5-methylphenyl] ethanone

[38778-41-5] $C_{18}H_{20}O_3$ mol.wt. 284.36



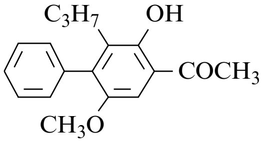
m.p. 124–125° [3688];
^1H NMR [3688], IR [3688], UV [3688].

Synthesis

– Preparation by Fries rearrangement of 2-acetoxy-2′-methoxy-3,3′-dimethyldiphenylmethane with aluminium chloride in nitrobenzene at 40° for 3 h (50%) [3688].

1-(3-Hydroxy-6-methoxy-2-propyl[1,1′-biphenyl]-4-yl)ethanone

[43037-69-0] $C_{18}H_{20}O_3$ mol.wt. 284.36

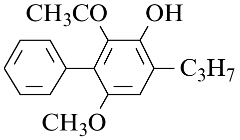


Synthesis

– Obtained by catalytic hydrogenation of 1-[3-hydroxy-6-methoxy-2-(2-propenyl)[1,1′-biphenyl]-4-yl]ethanone or by thermal Fries rearrangement of 3-(acetyloxy)-6-methoxy-2-propyl [1,1′-biphenyl] with aluminium chloride [3467].

1-(3-Hydroxy-6-methoxy-4-propyl[1,1′-biphenyl]-2-yl)ethanone

[43037-70-3] $C_{18}H_{20}O_3$ mol.wt. 284.36

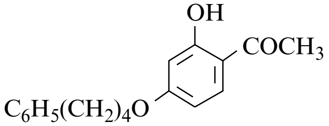


Synthesis

– Obtained by catalytic hydrogenation of 1-[3-hydroxy-6-methoxy-4-(2-propenyl)[1,1′-biphenyl]-2-yl]ethanone or by thermal Fries rearrangement of 5-(acetyloxy)-2-methoxy-4-propyl[1,1′-biphenyl] with aluminium chloride [3467].

1-[2-Hydroxy-4-(4-phenylbutoxy)phenyl]ethanone

[63359-88-6] $C_{18}H_{20}O_3$ mol.wt. 284.36



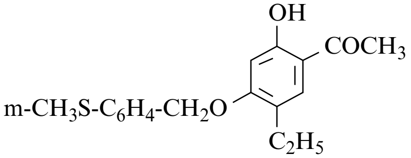
Synthesis

- Obtained by reaction of 1-bromo-4-phenylbutane with resacetophenone in the presence of potassium carbonate in refluxing acetone for 24 h. The 1-chloro derivative and the butanone can also be used instead of the mentioned starting material and solvent (59%) [3204].
- Also refer to: [3663].

m.p. 55° [3204].

1-[5-Ethyl-2-hydroxy-4-[[3-(methylthio)phenyl]methoxy]phenyl]ethanone

[117706-52-2] $C_{18}H_{20}O_3S$ mol.wt. 316.42



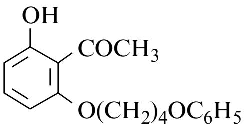
Synthesis

- Preparation by reaction of 3-methylmercapto- benzyl bromide with 2,4-dihydroxy-5-ethyl-acetophenone in the presence of potassium carbonate and potassium iodide (18%) [2678,2679].

m.p. 89° [2678,2679]; ^{1}H NMR [2678,2679].

1-[2-Hydroxy-6-(4-phenoxybutoxy)phenyl]ethanone

[69079-91-0] $C_{18}H_{20}O_4$ mol.wt. 300.35



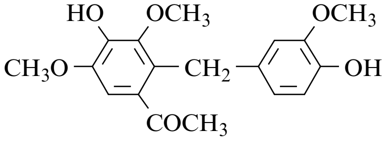
Synthesis

- Obtained by reaction of 1-bromo-4-phenoxybutane with 2,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 24 h (91%). The 1-chloro derivative and butanone can also be used instead of the mentioned starting material and solvent [3204].

m.p. 81° [3204].

1-[4-Hydroxy-2-[(4-hydroxy-3-methoxyphenyl)methyl]-3,5-dimethoxyphenyl] ethanone

[147904-71-0] $C_{18}H_{20}O_6$ mol.wt. 332.35



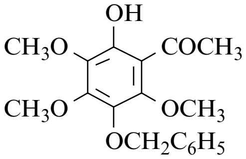
Synthesis

- Obtained by alkaline CuO oxidation of lignin (compound Vm2Sn) named 2-vanillylacetosyringone [3689].

GC [3689], GC-MS [3689].

1-[2-Hydroxy-3,4,6-trimethoxy-5-(phenylmethoxy)phenyl]ethanone

[76844-54-7] $C_{18}H_{20}O_6$ mol.wt. 332.35

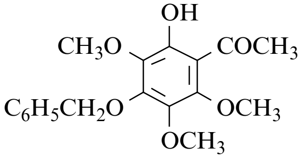


Syntheses

- Preparation by [3320] according to [3321] (Japanese paper).
- Also obtained by benzylation of 2,5-dihydroxy-3,4,6-tri-methoxyacetophenone with benzyl chloride [3322].

1-[2-Hydroxy-3,5,6-trimethoxy-4-(phenylmethoxy)phenyl]ethanone

[3162-49-0] $C_{18}H_{20}O_6$ mol.wt. 332.35



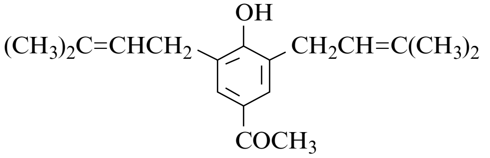
Syntheses

- Preparation by partial methylation of 4-benzyloxy-2,5-di-hydroxy-3,6-dimethoxyacetophenone with dimethyl sulfate in the presence of potassium carbonate in petroleum ether (b.p. 40–60°) (70%) [3428].
- Also refer to: [3690,3691].

m.p. 35–36° [3428]; IR [3428], UV [3428].

1-[4-Hydroxy-3,5-bis(3-methyl-2-butenyl)phenyl]ethanone

[41607-43-6] $C_{18}H_{24}O_2$ mol.wt. 272.39



Synthesis

- Preparation by thermal Claisen rearrangement of 3-(3,3-dimethylallyl)-4-(3,3-dimethylallyloxy)acetophenone in N,N-diethylaniline at 170–175° (84%) [2060].

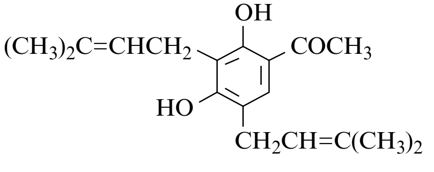
Isolation from natural sources

- From the roots of several *Gerbera* species (Tribus *Arctotideae*, Fam. Compositae): *Gerbera asplenifolia* (1.5%) [3692], *Gerbera crocea* (1%) [3692] and as a trace in *Gerbera cordata* Less. (0.008%) [3693].
- From the roots of several *Ageratina* species (Compositae): *Ageratina aschenbornia* (0.017%) [3450] and *Ageratina altissima* (0.005%) [3450].

m.p. 93°6 [3692], 92°3 [2060]; ^{1}H NMR [3692], IR [3692], UV [3692].

1-[2,4-Dihydroxy-3,5-bis-(3-methyl-2-butenyl)phenyl]ethanone

[24672-82-0] $C_{18}H_{24}O_3$ mol.wt. 288.39



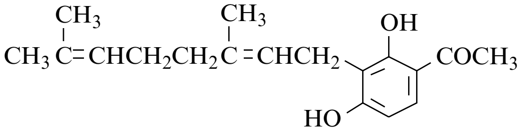
Syntheses

- Obtained (poor yield) by reaction of 2-methyl-but-3-en-2-ol on resacetophenone with boron trifluoride etherate [2326,3453] in dioxane at r.t. (6%) [3453].
- Also obtained [3457] (poor yield) [3456] by reaction of prenyl bromide with resacetophenone in potassium hydroxide solution at r.t. (3%) [3456].

m.p. 117° [3457], 109–114° [3456], 109–110° [3453];
^{1}H NMR [2326,2327,3456,3457], IR [3456,3457], UV [2326,3456].

1-[3-(3,7-Dimethyl-2,6-octadienyl)-2,4-dihydroxyphenyl]ethanone

[88661-97-6] $C_{18}H_{24}O_3$ mol.wt. 288.39



Synthesis

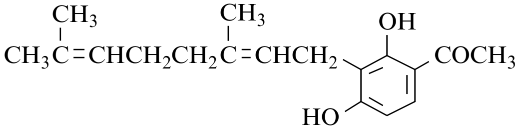
- Preparation by condensation of resacetophenone with 3,7-dimethyl-3-hydroxy-1,6-octadiene in the presence of boron trifluoride etherate in dioxane at r.t. (compound **14**) [3458].

^{1}H NMR [3458], IR [3458].

1-[3-(3,7-Dimethyl-2,6-octadienyl)-2,4-dihydroxyphenyl]ethanone

[18296-19-0] *(Z)* $C_{18}H_{24}O_3$ mol.wt. 288.39

[20212-67-3] *(E)*



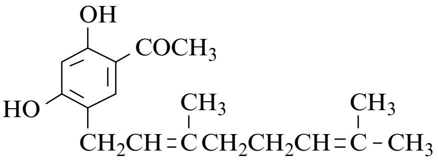
Syntheses

- Preparation by alkylation of 2,4-di-hydroxyacetophenone lithium salt with geranyl bromide in refluxing benzene (23%) [3694] (no specification).
- Also obtained by treatment of 6-acetyl-2-methyl-2-(4-methylpent-3-enyl)-3-phenylthiochroman-5-ol with potassium naphthalenide in tetrahydrofuran at −78°; then, the mixture was allowed to warm to −30° (48%) (2*E*, 6*Z*) [3459].

m.p. 120–121° [3694]; ^{1}H NMR [3694], UV [3694] (compound **XX**); (no specification) (*E* isomer ?).
m.p. 93–96° [3459]; ^{1}H NMR [3459], MS [3459] (compound **29**);
(as a 1:1 mixture of *E* and *Z* isomers by ^{13}C NMR) [3459].

1-[5-(3,7-Dimethyl-2,6-octadienyl)-2,4-dihydroxyphenyl]ethanone

[146954-92-9]



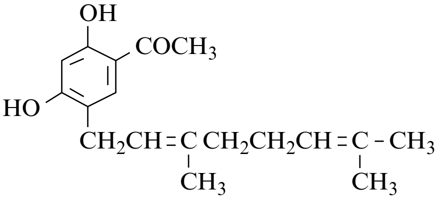
$C_{18}H_{24}O_3$ mol.wt. 288.39

Synthesis

- Preparation by condensation of resaceto-phenone with 3,7-dimethyl-3-hydroxy-1,6-octadiene in the presence of boron trifluoride etherate in dioxane at r.t. (compound **15**) [3458].

1-[5-(3,7-Dimethyl-2,6-octadienyl)-2,4-dihydroxyphenyl]ethanone *(E)*

[20212-68-4]



$C_{18}H_{24}O_3$ mol.wt. 288.39

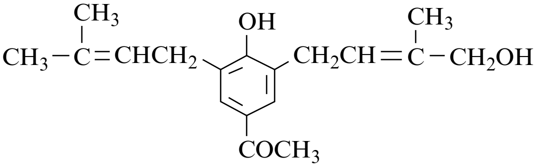
Synthesis

- Obtained (poor yield) by alkylation of 2,4-di-hydroxyacetophenone lithium salt with geranyl bromide in benzene (<2%) [3694].

m.p. 88° [3694];
^{1}H NMR [3694], UV [3694].

1-[4-Hydroxy-3-(4-hydroxy-3-methyl-2-butenyl)-5-(3-methyl-2-butenyl) phenyl]ethanone *(E)*

[81053-02-3]



$C_{18}H_{24}O_3$ mol.wt. 288.39

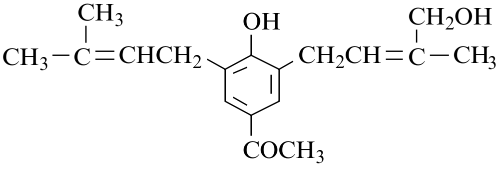
Isolation from natural sources

- From the aerial parts of *Artemisia campestris* L. subsp. glutinosa (Gay ex Besser) Batt. (Compositae) (1.8%) [3441].

m.p. 83° [3441]; ^{1}H NMR [3441], IR [3441], UV [3441], MS [3441].

1-[4-Hydroxy-3-(4-hydroxy-3-methyl-2-butenyl)-5-(3-methyl-2-butenyl) phenyl]ethanone *(Z)*

[77370-28-6]



$C_{18}H_{24}O_3$ mol.wt. 288.39

Isolation from natural sources

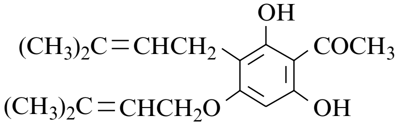
- From *Artemisia campestris* L. ssp. glutinosa (Gay ex Besser) Batt,

- compound (7) (12%) (from the aerial parts) [3441];
- compound (2) [3465].

m.p. 109–110° [3465]; ^{1}H NMR [3441,3465], IR [3465], UV [3465], MS [3441,3465].

1-[2,6-Dihydroxy-3-(3-methyl-2-butenyl)-4-[(3-methyl-2-butenyl)oxy]phenyl] ethanone

[158499-98-0] $C_{18}H_{24}O_4$ mol.wt. 304.39



Synthesis

– Obtained by reaction of 4,6-dihydroxy-3-prenyl-2-tosyloxyacetophenone with prenyl bromide in the presence of potassium carbonate in acetone at 20° for 2 h, followed by hydrolysis of the resulting 6-hydroxy-3-prenyl-4-prenyloxy-2-tosyloxyacetophenone with 30% potassium hydroxide in refluxing ethanol under nitrogen atmosphere for 1 h [3695].

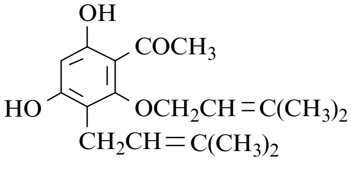
Isolation from natural sources

– From the fruit of *Evodia merrillii* [3695].

m.p. 108–110° [3695]; ^{1}H NMR [3695], IR [3695].

1-[4,6-Dihydroxy-3-(3-methyl-2-butenyl)-2-[(3-methyl-2-butenyl)oxy]phenyl] ethanone

[153399-38-3] $C_{18}H_{24}O_4$ mol.wt. 304.39



Syntheses

– Obtained by hydrolysis of 4′,6′-bis(benzoyloxy)-2′-(3-methyl-2-butenyloxy)-3′-(3-methyl-2-butenyl)-acetophenone with dilute sodium hydroxide under nitrogen atmosphere at 50° [3571].

– Also refer to: [3695].

Isolation from natural sources

– From the root bark of *Euodia lunu-ankenda* (Rutaceae) [3696].

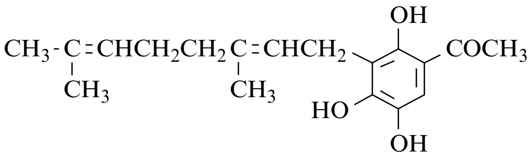
m.p. 73–75° [3696], 71–72° [3571]; TLC [3696];
^{1}H NMR [3571,3696], IR [3571,3696], UV [3696],
MS [3696], HRMS [3696].

N.B.: The synthetic works [3571,3695] have suggested that the prenylphenol of natural products isolated by [3696] had an incorrectly assigned structure. This compound [3696] is identical with 1-[2,6-dihydroxy-3-(3-methyl-2-butenyl)-4-[(3-methyl-2-butenyl)oxy]phenyl]ethanone [158499-98-0].

1-[3-(3,7-Dimethyl-2,6-octadienyl)-2,4,5-trihydroxyphenyl]ethanone

[18296-18-9] *(Z)*
[20212-66-2] *(E)*

$C_{18}H_{24}O_4$ mol.wt. 304.39



Syntheses

- Preparation by reaction of geraniol of 2,4,5-trihydroxyacetophenone in refluxing decalin [3694] (no specification).
- Also obtained by reaction of potassium naphthalenide with 6-acetyl-2-methyl-2-(4-methylpent-3-enyl)-3-phenylthiochroman-5,8-diol in tetrahydrofuran at r.t. (11%) (*E*, *Z*) [3459].

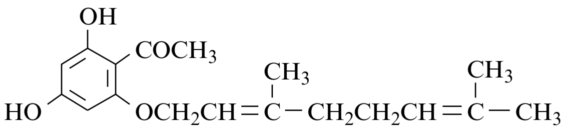
m.p. 127–130° [3694]; UV [3694]; (compound **XVIII**); (no specification) (*E* isomer ?).

Unstable oil [3459]; ^{1}H NMR [3459], MS [3459]; (compound **30**); (as a 1:1 mixture of *E* and *Z* isomers by ^{13}C NMR) [3459].

1-[2-[(3,7-Dimethyl-2,6-octadienyl)oxy]-4,6-dihydroxyphenyl]ethanone *(E)*

[142905-39-3]

$C_{18}H_{24}O_4$ mol.wt. 304.39



Isolation from natural sources

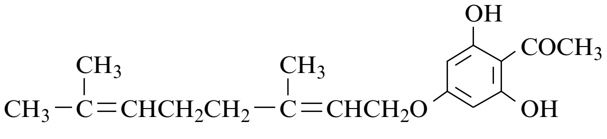
- From the fruit of *Evodia Merrillii* Kanehira & Sasaki ex Kanehira (Rutaceae) [3697].

m.p. 147–150° [3697]; column chromatography [3697]; ^{1}H NMR [3697], ^{13}C NMR [3697], IR [3697], UV [3697], MS [3697].

1-[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2,6-dihydroxyphenyl]ethanone *(E)*

[142905-40-6]

$C_{18}H_{24}O_4$ mol.wt. 304.39



Synthesis

- Obtained by hydrolysis of 2-toluenesulfonyloxy-6-hydroxy-4-(1′-geranyloxy)-acetophenone with 30% potassium hydroxide in refluxing ethanol for 1.5 h (75%) [3608].

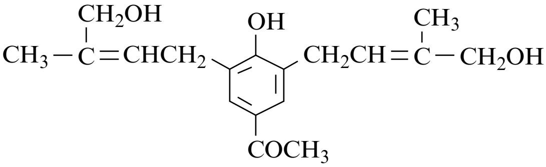
Isolation from natural sources

- From the fruit of *Evodia Merrillii* Kanehira & Sasaki ex Kanehira (Rutaceae) [3697].

Waxy substance [3697]; m.p. 147–148° [3608];
^{1}H NMR [3608,3697], ^{13}C NMR [3697], IR [3608,3697], UV [3697],
MS [3697], EIMS [3608], HREIMS [3608];
column chromatography [3697].

1-[4-Hydroxy-3,5-bis(4-hydroxy-3-methyl-2-butenyl)phenyl]ethanone *(E,Z)*

[81053-03-4] $C_{18}H_{24}O_4$ mol.wt. 304.39



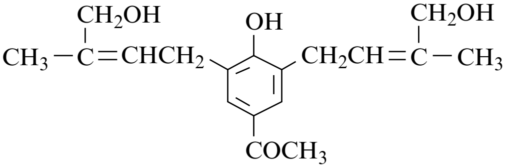
Isolation from natural sources

– From the aerial parts of *Artemisia campestris* L. subsp. glutinosa (Gay ex Besser) Batt. (Compositae) (2%) [3441].

m.p. 97° [3441]; ^{1}H NMR [3441], IR [3441], UV [3441], MS [3441].

1-[4-Hydroxy-3,5-bis(4-hydroxy-3-methyl-2-butenyl)phenyl]ethanone *(Z,Z)*

[77370-30-0] $C_{18}H_{24}O_4$ mol.wt. 304.39



Isolation from natural sources

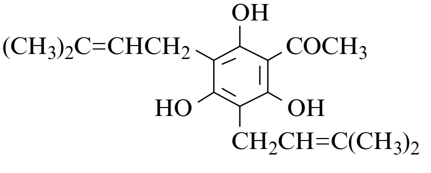
– From *Artemisia camprstris* L. ssp. glutinosa (Gay ex Besser) Batt,

- compound (10) (0.6%) (from the aerial parts) [3441];
- compound (3) (12%) [3465].

m.p. 120° [3465]; ^{1}H NMR [3441,3465], IR [3465], UV [3465], MS [3441,3465].

1-[2,4,6-Trihydroxy-3,5-bis(3-methyl-2-butenyl)phenyl]ethanone

[35458-19-6] $C_{18}H_{24}O_4$ mol.wt. 304.39



Syntheses

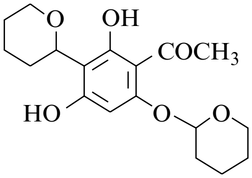
– Preparation by reaction of 2-methylbut-3-en-2-ol with phloroacetophenone in the presence of boron trifluoride etherate in dioxane at 20° [3477,3500], (21%) [3477] or at 50° [3401].

– Also obtained by reaction of prenyl bromide with phloroacetophenone in the presence of potassium hydroxide in 80% aqueous methanol (15%) [3117].

m.p. 78–79° [3117,3401,3477,3500], 68–72° [2879]; b.p.$_1$ 135–140° [3117];
^{1}H NMR [2879]; UV [3117,3477].

1-[2,4-Dihydroxy-3-(tetrahydro-2*H*-pyran-2-yl)-6-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone

[136257-83-5] $C_{18}H_{24}O_6$ mol.wt. 336.38



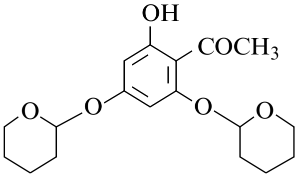
Synthesis

– Obtained (by-product) by reaction of 3,4-dihydro-2*H*-pyran on phloroacetophenone with p-toluenesulfonic acid in dioxane at r.t. (8%) [2368].

m.p. 118–121° [2368]; ^{1}H NMR [2368], ^{13}C NMR [2368].

1-[2-Hydroxy-4,6-bis[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone

[130600-90-7] $C_{18}H_{24}O_6$ mol.wt. 336.38



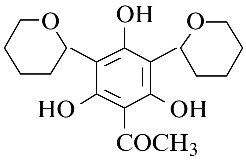
Synthesis

– Obtained (by-product) by reaction of 3,4-dihydro-2*H*-pyran on phloroacetophenone with p-toluenesulfonic acid in dioxane at r.t. (7%) [2368].

^{1}H NMR [2368], ^{13}C NMR [2368].

1-[2,4,6-Trihydroxy-3,5-bis(tetrahydro-2*H*-pyran-2-yl)phenyl]ethanone

[136257-82-4] $C_{18}H_{24}O_6$ mol.wt. 336.38



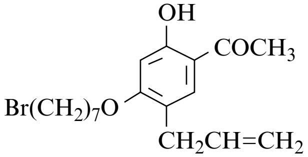
Synthesis

– Obtained (by-product) by reaction of 3,4-dihydro-2*H*-pyran on phloroacetophenone with p-toluenesulfonic acid in dioxane at r.t. (1%) [2368].

^{1}H NMR [2368], ^{13}C NMR [2368].

1-[4-[(7-Bromoheptyl)oxy]-2-hydroxy-5-(2-propenyl)phenyl]ethanone

[117706-40-8] $C_{18}H_{25}BrO_3$ mol.wt. 369.30



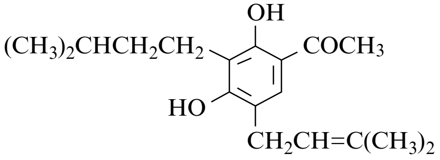
Synthesis

– Preparation by reaction of 1,7-dibromoheptane with 5-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (70%) [2671,2678,2679].

oil [2671,2678,2679].

1-[2,4-Dihydroxy-5-(3-methyl-2-butenyl)-3-(3-methylbutyl)phenyl]ethanone

[50773-38-1] $C_{18}H_{26}O_3$ mol.wt. 290.40



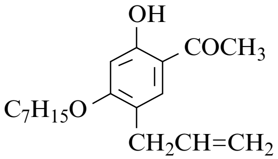
Synthesis

– Preparation by reaction of prenyl bromide with 2,4-dihydroxy-3-isopentylacetophenone in aqueous potassium hydroxide solution at r.t. [3457].

m.p. 113°5 [3457];
^{1}H NMR [3457], IR [3457].

1-[4-(Heptyloxy)-2-hydroxy-5-(2-propenyl)phenyl]ethanone

[117706-56-6] $C_{18}H_{26}O_3$ mol.wt. 290.40



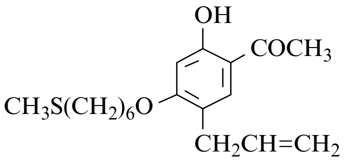
Synthesis

– Preparation by reaction of heptyl bromide with 5-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing methyl ethyl ketone (77%) [2671], (40%) [2678,2679].

Oil [2671,2678,2679].

1-[2-Hydroxy-4-[[6-(methylthio)hexyl]oxy]-5-(2-propenyl)phenyl]ethanone

[117706-34-0] $C_{18}H_{26}O_3S$ mol.wt. 322.47



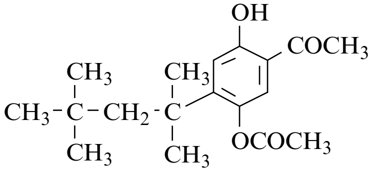
Synthesis

– Preparation by adding a DMF solution of 5-allyl-4-(6-bromohexyloxy)-2-hydroxyacetophenone to a DMF solution of methanethiol previously treated with sodium hydride (54%) [2678,2679].

m.p. 42° [2678,2679].

1-[5-(Acetyloxy)-2-hydroxy-4-(1,1,3,3-tetramethylbutyl)phenyl]ethanone

[107188-54-5] $C_{18}H_{26}O_4$ mol.wt. 306.40

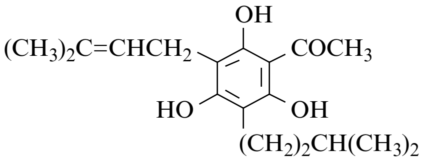


Synthesis

– Refer to: [3251].

1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)-5-(3-methylbutyl)phenyl] ethanone

[57744-70-4]



$C_{18}H_{26}O_4$ mol.wt. 306.40

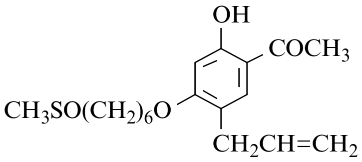
Synthesis

- Obtained by reaction of 2-methyl-3-buten-2-ol with isopentylphloroacetophenone in the presence of boron trifluoride etherate in dioxane at 20° (13%) [3500].

m.p. 92°5–93°5 [2879], 92–93°5 [3500];
^{1}H NMR [2879].

1-[2-Hydroxy-4-[[6-(methylsulfinyl)hexyl]oxy]-5-(2-propenyl)phenyl] ethanone

[117706-35-1]



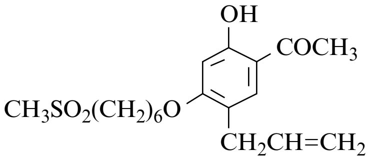
$C_{18}H_{26}O_4S$ mol.wt. 338.47

Synthesis

- Obtained (by-product) by reaction of m-chloro-perbenzoic acid with 5-allyl-2-hydroxy-4-[6-(methyl-thio)hexyloxy] acetophenone in methylene chloride, first at 0°, then at r.t. (9%) [2678,2679].

1-[2-Hydroxy-4-[[6-(methylsulfonyl)hexyl]oxy]-5-(2-propenyl)phenyl] ethanone

[117706-36-2]



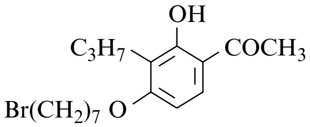
$C_{18}H_{26}O_5S$ mol.wt. 354.47

Synthesis

- Obtained by reaction of m-chloroperbenzoic acid with 5-allyl-2-hydroxy-4-[6-(methylthio)hexyloxy]-acetophenone in methylene chloride, first at 0°, then at r.t. (20%) [2678,2679].

1-[4-[(7-Bromoheptyl)oxy]-2-hydroxy-3-propylphenyl]ethanone

[106627-33-2]



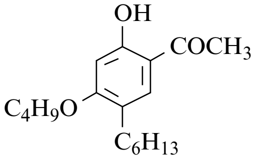
$C_{18}H_{27}BrO_3$ mol.wt. 371.31

Synthesis

- Preparation by reaction of 1,7-dibromoheptane with 2,4-di-hydroxy-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (21%) [3181].

1-(4-Butoxy-5-hexyl-2-hydroxyphenyl)ethanone

[101002-31-7] $C_{18}H_{28}O_3$ mol.wt. 292.42



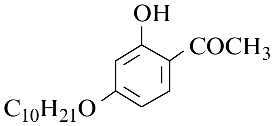
Synthesis

- Obtained by partial alkylation of 2,4-dihydroxy-5-hexyl-acetophenone with butyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [3493].

m.p. 37° [3493].

1-[4-(Decyloxy)-2-hydroxyphenyl]ethanone

[143286-86-6] $C_{18}H_{28}O_3$ mol.wt. 292.42



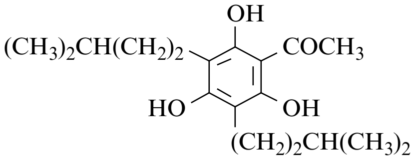
Syntheses

- Preparation by partial alkylation of resacetophenone with decyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [3493].
- Also refer to: [3698].

m.p. 35° [3493,3698].

1-[2,4,6-Trihydroxy-3,5-bis(3-methylbutyl)phenyl]ethanone

[55380-57-9] $C_{18}H_{28}O_4$ mol.wt. 308.42



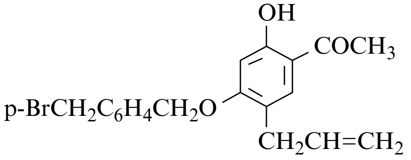
Synthesis

- Preparation by catalytic hydrogenation of deoxyacetohumulone [2,4,6-trihydroxy-3,5-(dimethylallyl) acetophenone] in the presence of 10% Pd/C in ethanol [3401].

^{1}H NMR [3401], UV [3401], MS [3401].

1-[4-[[4-(Bromomethyl)phenyl]methoxy]-2-hydroxy-5-(2-propenyl)phenyl] ethanone

[117706-46-4] $C_{19}H_{19}BrO_3$ mol.wt. 375.26



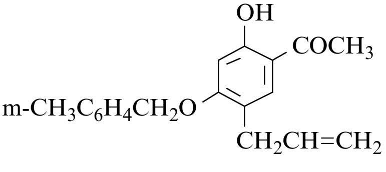
Synthesis

- Preparation by reaction of 4-(bromomethyl)benzyl bromide with 5-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide (18%) [2678,2679].

Oil [2678,2679].

1-[2-Hydroxy-4-[(3-methylphenyl)methoxy]-5-(2-propenyl)phenyl]ethanone

[117706-45-3]



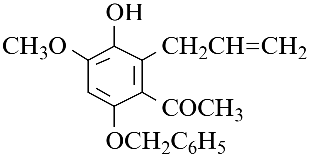
$C_{19}H_{20}O_3$ mol.wt. 296.37

Synthesis

- Preparation by reaction of 3-methylbenzyl bromide on 5-allyl-2,4-dihydroxyacetophenone with potassium carbonate and potassium iodide (50%) [2678,2679].

m.p. 87–88° [2678,2679].

1-[3-Hydroxy-4-methoxy-6-(phenylmethoxy)-2-(2-propenyl)phenyl]ethanone



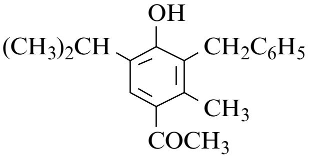
$C_{19}H_{20}O_4$ mol.wt. 312.37

Synthesis

- Preparation by thermal Claisen rearrangement of 3-(allyloxy)-6-(benzyloxy)-4-methoxyacetophenone in boiling carbitol (diethylene glycol monoethyl ether) (89%) [2849].

m.p. 116° [2849].

1-[4-Hydroxy-2-methyl-5-(1-methylethyl)-3-(phenylmethyl)phenyl]ethanone



$C_{19}H_{22}O_2$ mol.wt. 282.38

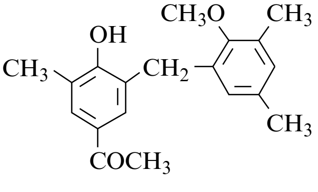
Synthesis

- Obtained (poor yield) by reaction of benzyl chloride with 4-hydroxy-2-methyl-5-isopropylacetophenone in the presence of zinc chloride in boiling chloroform (4%) [3699].

m.p. 88° [3699]; b.p.$_{14}$ 243–245° [3699].

1-[4-Hydroxy-3-[(2-methoxy-3,5-dimethylphenyl)methyl]-5-methylphenyl] ethanone

[38778-48-2]



$C_{19}H_{22}O_3$ mol.wt. 298.38

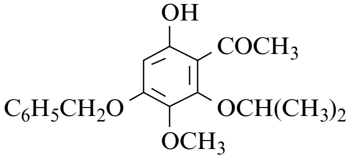
Synthesis

- Obtained by Fries rearrangement of 2-acetoxy-2′-methoxy-3,3′,5-trimethyldiphenylmethane with aluminium chloride in nitrobenzene at 55° for 3 h (23%) [3688].

m.p. 128° [3688];
^{1}H NMR [3688], IR [3688], UV [3688].

1-[6-Hydroxy-3-methoxy-2-(1-methylethoxy)-4-(phenylmethoxy)phenyl] ethanone

[188927-31-3]



$C_{19}H_{22}O_5$ mol.wt. 330.38

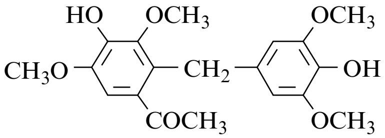
Synthesis

- Preparation by treatment of 4-(benzyloxy)-3,6-di-methoxy-2-isopropoxyacetophenone (m.p. 74–75°) with aluminium bromide in acetonitrile at 0° for 10–15 min (75%) [3507].

m.p. 69–70° [3507].

1-[4-Hydroxy-2-[(4-hydroxy-3,5-dimethoxyphenyl)methyl]-3,5-dimethoxyphenyl]ethanone

[147904-74-3]



$C_{19}H_{22}O_7$ mol.wt. 362.38

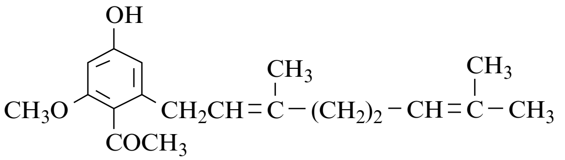
Synthesis

- Obtained by upon alkaline CuO oxidation of lignin (compound Sm2Sn) named 2-syringylaceto-syringone [3689].

GC [3689], GC-MS [3689].

1-[2-(3,7-Dimethyl-2,6-octadienyl)-4-hydroxy-6-methoxyphenyl]ethanone

[121379-44-0]

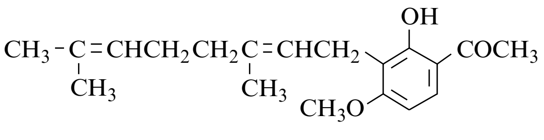


$C_{19}H_{26}O_3$ mol.wt. 302.41

Isolation from natural sources

- From the bulbs of *Dioscorea bulbifera* [3404].

1-[2-Hydroxy-4-methoxy-3-(3,7-dimethyl-2,6-octadienyl)phenyl]ethanone *(E)*



$C_{19}H_{26}O_3$ mol.wt. 302.41

Synthesis

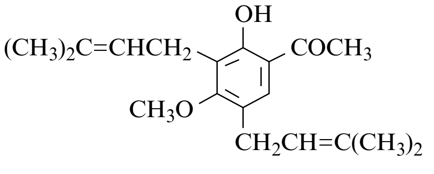
- Preparation by reaction of dimethyl sulfate on 3-geranyl-2,4-di-hydroxyacetophenone with potassium carbonate in refluxing acetone [3694].

^{1}H NMR [3694]; UV [3694].

1-[2-Hydroxy-4-methoxy-3,5-bis(3-methyl-2-butenyl)phenyl]ethanone

[50773-40-5]

$C_{19}H_{26}O_3$ mol.wt. 302.41



Syntheses

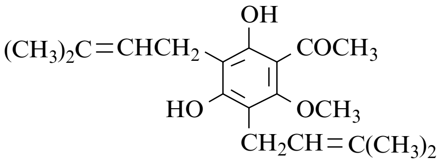
- Preparation by reaction of 2-methylbut-3-en-2-ol with 2-hydroxy-4-methoxyacetophenone in the presence of boron trifluoride etherate [2326].
- Preparation by reaction of dimethyl sulfate with 2,4-dihydroxy-3,5-(dimethylallyl) acetophenone in the presence of potassium carbonate in refluxing acetone [3457].

^{1}H NMR [2326,2327,3457], UV [2326].

1-[2,4-Dihydroxy-6-methoxy-3,5-bis(3-methyl-2-butenyl)phenyl]ethanone

[123999-38-2]

$C_{19}H_{26}O_4$ mol.wt. 318.41



Isolation from natural sources

- From *Euodia lunu-ankenda* root bark (Rutaceae) [3696].
- From *Acronychia pedunculata* root bark (Rutaceae) [3700].

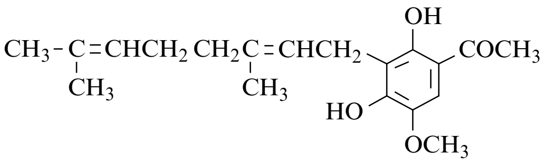
Yellow oil [3696,3700];

TLC [3700]; HPLC [3696]; ^{1}H NMR [3700], IR [3700], HRMS [3700], MS [3700].

1-[2,4-Dihydroxy-5-methoxy-3-(3,7-dimethyl-2,6-octadienyl)phenyl]ethanone *(E)*

[20212-64-0]

$C_{19}H_{26}O_4$ mol.wt. 318.41



Synthesis

- Obtained by alkylation of 2,4-di-hydroxy-5-methoxyacetophenone lithium salt with geranyl bromide in benzene (7%) [3610,3694].

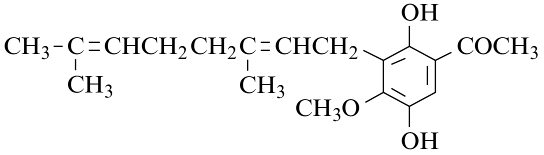
Isolation from natural sources

- Also obtained on barium hydroxide degradation of homoflemingin, a chalkone isolated from the seed pods of *Flemingia rhodocarpa* Baker (Leguminosae) [3610,3694].

m.p. 107° [3694]; ^{1}H NMR [3694], UV [3694], MS [3694].

1-[2,5-Dihydroxy-4-methoxy-3-(3,7-dimethyl-2,6-octadienyl)phenyl]ethanone *(E)*

[20180-88-5]



$C_{19}H_{26}O_4$ mol.wt. 318.41

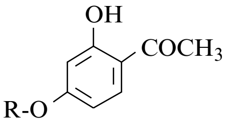
Synthesis

- Obtained by Elbs persulfate oxidation of 3-geranyl-2-hydroxy-4-methoxyacetophenone (10%) [3694].

m.p. 69–70° [3694]; ^{1}H NMR [3694], UV [3694].

1-[2-Hydroxy-4-[(6-O-β-D-xylopyranosyl-β-D-glucopyranosyl)oxy]phenyl] ethanone *(Bungeiside D)*

[149475-54-7]



R= β-D-xyl (1→ 6) β-D-glc

$C_{19}H_{26}O_{12}$ mol.wt. 446.41

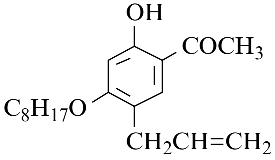
Isolation from natural sources

- From the roots of *Cynanchum bungei* DECNE (Asclepiadaceae) [3554].

Colourless needles [3554]; m.p. 238–240° [3554]; $(\alpha)_D$ = −68°3 (c = 0.5, methanol) [3554]; MS [3554], ^{1}H NMR [3554], ^{13}C NMR [3554], IR [3554].

1-[2-Hydroxy-4-(octyloxy)-5-(2-propenyl)phenyl]ethanone

[117690-46-7]



$C_{19}H_{28}O_3$ mol.wt. 304.43

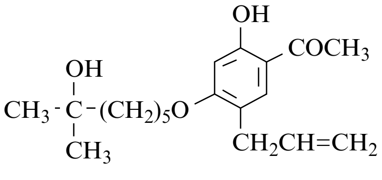
Synthesis

- Preparation by reaction of n-octyl bromide with 5-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide (21%) [2678,2679].

Oil [2678,2679].

1-[2-Hydroxy-4-[(6-hydroxy-6-methylheptyl)oxy]-5-(2-propenyl)phenyl] ethanone

[117706-02-2]



$C_{19}H_{28}O_4$ mol.wt. 320.43

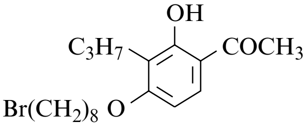
Synthesis

- Obtained by reaction of 7-(4-acetyl-2-allyl-5-hydroxy-phenoxy)heptanoyl chloride on methyl lithium in ether at −98°; the reaction mixture was allowed to warm up to −50° and then poured into dilute hydrochloric acid (8%) [2671].

Oil [2671].

1-[4-[(8-Bromooctyl)oxy]-2-hydroxy-3-propylphenyl]ethanone

[106627-34-3] $C_{19}H_{29}BrO_3$ mol.wt. 343.26



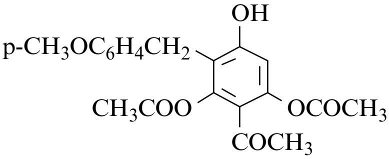
Synthesis

– Preparation by reaction of 1,8-dibromooctane with 2,4-di-hydroxy-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (63%) [3181].

pale green oil [3181]; ^{1}H NMR [3181].

1-[2,6-Bis(acetyloxy)-4-hydroxy-3-[(4-methoxyphenyl)methyl]phenyl] ethanone

[145747-40-6] $C_{20}H_{20}O_7$ mol.wt. 372.37

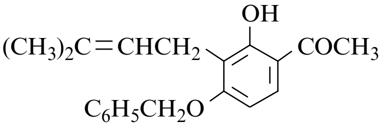


Synthesis

– Obtained by enzymatic hydrolysis of 2,4,6-tri-acetoxy-3-(4-methoxy)benzylacetophenone in the presence of porcine pancreas lipase in tetrahydrofuran at 42–45° (65%) [2388].

1-[2-Hydroxy-3-(3-methyl-2-butenyl)-4-(phenylmethoxy)phenyl]ethanone

$C_{20}H_{22}O_3$ mol.wt. 310.39



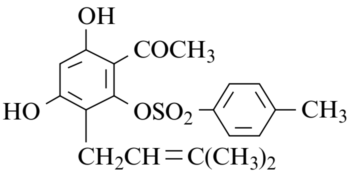
Synthesis

– Obtained by reaction of benzyl bromide with 3-prenylresacetophenone in the presence of potassium carbonate in refluxing acetone for 8 h (64%) [3701].

m.p. 70–71° [3701]; ^{1}H NMR [3701], IR [3701].

1-[4,6-Dihydroxy-3-(3-methyl-2-butenyl)-2-[[(4-methylphenyl)sulfonyl]oxy] phenyl]ethanone

[158499-95-7] $C_{20}H_{22}O_6S$ mol.wt. 390.46



Synthesis

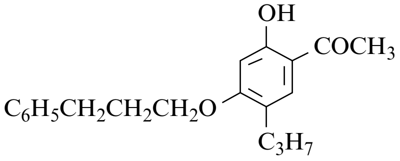
– Obtained by hydrolysis of 4,6-bis(benzoyloxy)-3-prenyl-2-(tosyloxy)acetophenone with aqueous methanolic sodium hydroxide solution under nitrogen at 50° [3695].

Paste [3695].

1-[2-Hydroxy-4-(3-phenylpropoxy)-5-propylphenyl]ethanone

[117706-47-5]

$C_{20}H_{24}O_3$ mol.wt. 312.41



Synthesis

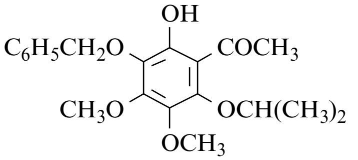
- Preparation by reaction of 3-phenylpropyl bromide on 2,4-dihydroxy-5-propylacetophenone with potassium carbonate and potassium iodide (26%) [2678,2679].

m.p. 60° [2678,2679].

1-[2-Hydroxy-4,5-dimethoxy-6-(1-methylethoxy)-3-(phenylmethoxy)phenyl] ethanone

[169130-27-2]

$C_{20}H_{24}O_6$ mol.wt. 360.41



Synthesis

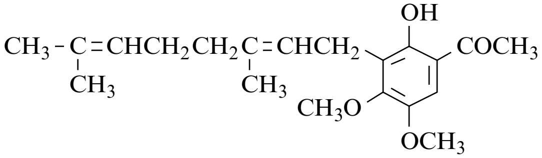
- Obtained by selective demethylation of 3-benzyloxy-6-isopropoxy-2,4,5-trimethoxyacetophenone with aluminium bromide in acetonitrile, first at 0° for 15 min, then at 50–60° for 15–20 min after dilution with ca. 3% hydrochloric acid (68%) [3320].

m.p. 76–77° [3320]; ^{1}H NMR [3320].

1-[2-Hydroxy-4,5-dimethoxy-3-(3,7-dimethyl-2,6-octadienyl)phenyl]ethanone *(E)*

[20212-65-1]

$C_{20}H_{28}O_4$ mol.wt. 332.44



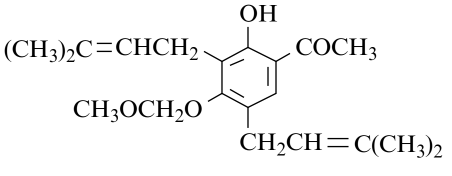
Synthesis

- Preparation by methylation of 3-geranyl-2,4-dihydroxy-5-methoxyacetophenone or of 3-geranyl-2,4,5-trihydroxyaceto-phenone with diazomethane [3694].

Oil [3694]; ^{1}H NMR [3694], IR [3694], UV [3694].

1-[2-Hydroxy-4-(methoxymethoxy)-3,5-bis(3-methyl-2-butenyl)phenyl] ethanone

[217442-59-6]



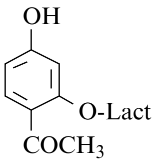
$C_{20}H_{28}O_4$ mol.wt. 332.44

Synthesis

- Obtained by reaction of chloromethyl methyl ether with 3,5-diprenylresacetophenone in acetone in the presence of potassium carbonate [3614] at r.t. for 3 h (83%) [3702].

m.p. 67–68° [3702]; ^{1}H NMR [3702].

1-[2-[(4-O-β-D-Galactopyranosyl-β-D-glucopyranosyl)oxy]-4-hydroxyphenyl] ethanone



Lact = β-D-Lactosid rest

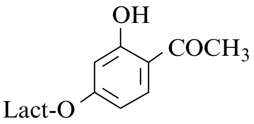
$C_{20}H_{28}O_{13}$ mol.wt. 476.41

Synthesis

- Preparation by reaction of 0.2 M sodium methoxide with 4-acetylresacetophenone-2-heptaacetyl-β-D-lactosid in boiling methanol for 3 min (40%) [3552].

Monohydrate [3552]; m.p. 165–168° [3552];
$(\alpha)_D^{21} = -45°$ (c = 1, water) [3552].

1-[4-[(4-O-β-D-Galactopyranosyl-β-D-glucopyranosyl)oxy]-2-hydroxyphenyl] ethanone



Lact = β-D-Lactosid rest

$C_{20}H_{28}O_{13}$ mol.wt. 476.43

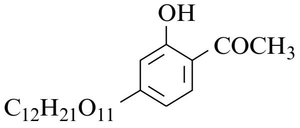
Synthesis

- Preparation by reaction of 0.2 M sodium methoxide with resacetophenone-4-heptaacetyl-β-D-lactosid in boiling methanol for 3 min (50%) [3552].

m.p. 255–258° [3552];
$(\alpha)_D^{21} = -62°6$ (c = 1, water) [3552].

1-[4-[(4-O-β-D-Glucopyranosyl-β-D-glucopyranosyl)oxy]-2-hydroxyphenyl] ethanone

[54918-30-8] $C_{20}H_{28}O_{13}$ mol.wt. 476.43



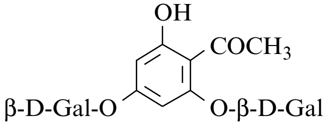
Synthesis

– Preparation by reaction of sodium on 2′,4′-dihydroxy-acetophenone-4-β-hepta-O-acetyl-D-cellobioside in methanol (67%) [1936].

m.p. 212° [1936];
$(\alpha)_D^{17} = -60°$ (pyridine) [1936].

1-[2,4-Bis-(β–D-galactopyranosyloxy)-6-hydroxyphenyl]ethanone

[88087-01-8] $C_{20}H_{28}O_{14}$ mol.wt. 492.43



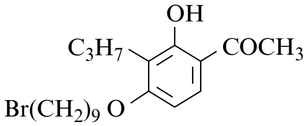
Synthesis

– Preparation by deacetylation of phloracetophenone 2,4-di-O-(2,3,4,6-tetra-O-acetyl)-β-D-galactopyranoside with 0.1 N methanolic sodium methoxide (87%) [2842].

m.p. 183–185° [2842]; ^{1}H NMR [2842].

1-[4-[(9-Bromononyl)oxy]-2-hydroxy-3-propylphenyl]ethanone

[79557-82-7] $C_{20}H_{31}BrO_3$ mol.wt. 399.37

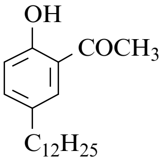


Synthesis

– Preparation by reaction of 1,9-dibromononane with 2,4-dihydroxy-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (63%) [3181].

1-(5-Dodecyl-2-hydroxyphenyl)ethanone

[84744-37-6] $C_{20}H_{32}O_2$ mol.wt. 304.47



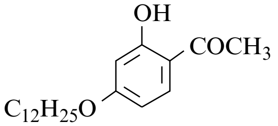
Synthesis

– Preparation by reaction of acetyl chloride on 4-dodecylphenol with aluminium chloride in ethylene dichloride at 110–120° (46%) [2625].

b.p.$_4$ 198–203° [2625].

1-[4-(Dodecyloxy)-2-hydroxyphenyl]ethanone

[52122-72-2] $C_{20}H_{32}O_3$ mol.wt. 318.37



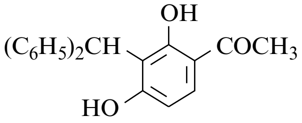
Syntheses

- Preparation by partial alkylation of resacetophenone with dodecyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [3493].
- Also refer to: [3698].

m.p. 51° [3493,3698].

1-[3-(Diphenylmethyl)-2,4-dihydroxyphenyl]ethanone

[107114-32-9] $C_{21}H_{18}O_3$ mol.wt. 318.37



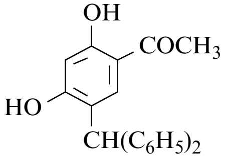
Synthesis

- Obtained by reaction of resacetophenone with diphenylcarbinol in the presence of boron trifluoride etherate in dioxane at r.t. (21%) [3703].

m.p. 203–204° [3703];
^{1}H NMR [3703], IR [3703], UV [3703].

1-[5-(Diphenylmethyl)-2,4-dihydroxyphenyl]ethanone

[107114-35-2] $C_{21}H_{18}O_3$ mol.wt. 318.37



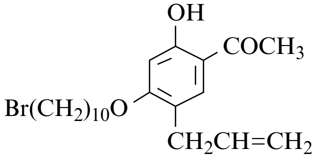
Synthesis

- Preparation by reaction of resacetophenone with diphenylcarbinol in the presence of boron trifluoride etherate in dioxane at r.t. (39%) [3703].

m.p. 179–180° [3703];
^{1}H NMR [3703], IR [3703], UV [3703].

1-[4-[(10-Bromodecyl)oxy]-2-hydroxy-5-(2-propenyl)phenyl]ethanone

[117706-39-5] $C_{21}H_{31}BrO_3$ mol.wt. 411.38



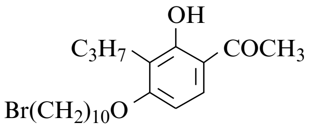
Synthesis

- Preparation by reaction of 1,10-dibromodecane with 5-allyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide (18%) [2678,2679].

m.p. < 25° [2678,2679]; ^{1}H NMR [2678,2679].

1-[4-[(10-Bromodecyl)oxy]-2-hydroxy-3-propylphenyl]ethanone

[106627-35-4] $C_{21}H_{33}BrO_3$ mol.wt. 413.40

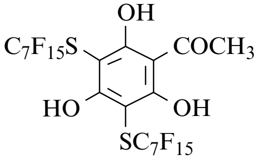


Synthesis

– Preparation by reaction of 1,10-dibromodecane with 2,4-di-hydroxy-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (60%) [3181].

1-[2,4,6-Trihydroxy-3,5-bis[(pentadecafluoroheptyl)thio]phenyl]ethanone

$C_{22}H_6F_{30}O_4S_2$ mol.wt. 968.37



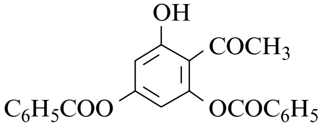
Synthesis

– Preparation by reaction of perfluoroheptanesulfenyl chloride with phloroacetophenone in chloroform in the presence of a slight excess of pyridine and a little quantity of iron powder, first at –40°, then at 60° for 3 h (39%) [2480].

m.p. 106–108° [2480]; ^{1}H NMR [2480], IR [2480].

1-[2,4-Bis(benzoyloxy)-6-hydroxyphenyl]ethanone

$C_{22}H_{16}O_6$ mol.wt. 376.37



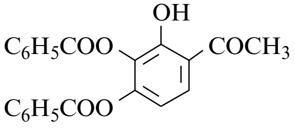
Synthesis

– Preparation by reaction of benzoyl chloride on phloroacetophenone in dilute aqueous sodium hydroxide [2827].

m.p. 109–110° [2827].

1-[3,4-Bis(benzoyloxy)-2-hydroxyphenyl]ethanone

[27865-59-4] $C_{22}H_{16}O_6$ mol.wt. 367.37



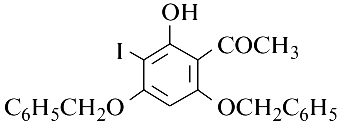
Synthesis

– Preparation by partial esterification of gallacetophenone with benzoyl chloride in pyridine at 100° (21%) [2403].

m.p. 108–109° [2403]; ^{1}H NMR [2403], IR [2403].

1-[2-Hydroxy-3-iodo-4,6-bis(phenylmethoxy)phenyl]ethanone

[95165-66-5] $C_{22}H_{19}IO_4$ mol.wt. 474.29



Syntheses

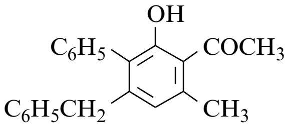
– Obtained by reaction of 2-hydroxy-4,6-bis(benzyloxy)-acetophenone with iodine in the presence of silver trifluoroacetate in chloroform at r.t. (83%) [3571].

– Also refer to: [3695].

m.p. 204–206° [3571]; ^{1}H NMR [3571].

1-[2-Hydroxy-6-methyl-3-phenyl-4-(phenylmethyl)phenyl]ethanone

[64648-09-5] $C_{22}H_{20}O_2$ mol.wt. 316.40



Synthesis

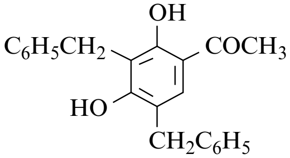
– Obtained by a potassium fluoride catalyzed self-condensation of 1-phenyl-2,4-pentanedione in DMF solution [2648,2976], (52%) [2976]; also refer to: “erratum” [2647].

m.p. 124° [2647,2976];

^{1}H NMR [2647,2648,2976], ^{13}C NMR [2647,2976], IR [2647,2976], MS [2647,2976].

1-[2,4-Dihydroxy-3,5-bis(phenylmethyl)phenyl]ethanone

[95832-44-3] $C_{22}H_{20}O_3$ mol.wt. 332.40



Syntheses

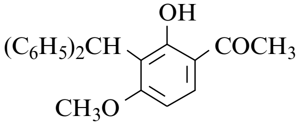
– Obtained by reaction of benzyl alcohol with resacetophenone in the presence of boron trifluoride etherate and dioxane at 60–70° (9%) [2324].

– Also obtained (poor yield) by reaction of benzyl bromide with resacetophenone in the presence of potassium hydroxide in methanol at r.t. (<2%) [3583].

m.p. 159–160° [3583], 157–158° [2324]; ^{1}H NMR [2324], IR [2324], UV [2324].

1-[3-(Diphenylmethyl)-2-hydroxy-4-methoxyphenyl]ethanone

[107114-34-1] $C_{22}H_{20}O_3$ mol.wt. 332.40



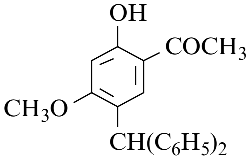
Synthesis

– Preparation by partial methylation of 3-(diphenyl-methyl)resacetophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone (90%) [3703].

m.p. 161–162° [3703]; ^{1}H NMR [3703].

1-[5-(Diphenylmethyl)-2-hydroxy-4-methoxyphenyl]ethanone

[107114-37-4] $C_{22}H_{20}O_3$ mol.wt. 332.40



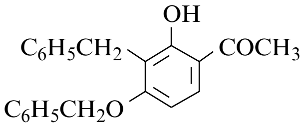
Synthesis

– Preparation by partial methylation of 5-(diphenylmethyl)resacetophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone (90%) [3703].

m.p. 145–146° [3703]; ^{1}H NMR [3703].

1-[2-Hydroxy-4-(phenylmethoxy)-3-(phenylmethyl)phenyl]ethanone

[105485-45-8] $C_{22}H_{20}O_3$ mol.wt. 332.40



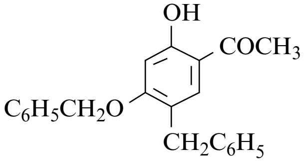
Synthesis

– Obtained by reaction of benzyl chloride [3582] or benzyl bromide [3583] with resacetophenone in the presence of potassium hydroxide in refluxing methanol (10%) [3582], (<1%) [3583].

m.p. 120–121° [3582], 118–120° [3583]; ^{1}H NMR [3583], IR [3583], UV [3583].

1-[2-Hydroxy-4-(phenylmethoxy)-5-(phenylmethyl)phenyl]ethanone

[105485-48-1] $C_{22}H_{20}O_3$ mol.wt. 332.40



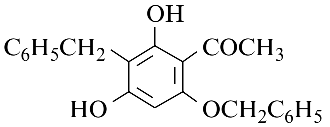
Synthesis

– Preparation by reaction of benzyl chloride with 5-benzyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone [3583].

m.p. 100–102° [3583]; ^{1}H NMR [3583], UV [3583].

1-[2,4-Dihydroxy-6-(phenylmethoxy)-3-(phenylmethyl)phenyl]ethanone

[39548-85-1] $C_{22}H_{20}O_4$ mol.wt. 348.40



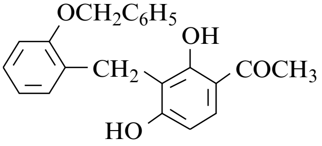
Synthesis

– Obtained (poor yield) by reaction of benzyl chloride with phloroacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (6%) [3605].

m.p. 166–167° [3605]; UV [3605].

1-[2,4-Dihydroxy-3-[[2-(phenylmethoxy)phenyl]methyl]phenyl]ethanone

[103633-36-9] $C_{22}H_{20}O_4$ mol.wt. 348.40



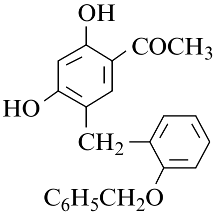
Syntheses

- Obtained by reaction of o-benzyloxybenzyl alcohol with resacetophenone in dioxane in the presence of boron trifluoride etherate at 60–70° for 3 h (10%) [2325].
- Also obtained by reaction of o-benzyloxybenzyl bromide with resacetophenone in methanol in the presence of potassium hydroxide at r.t. for 24 h (14%) [2325].

m.p. 155–156° [2325]; ^{1}H NMR [2325], IR [2325], UV [2325].

1-[2,4-Dihydroxy-5-[[2-(phenylmethoxy)phenyl]methyl]phenyl]ethanone

[103633-37-0] $C_{22}H_{20}O_4$ mol.wt. 348.40



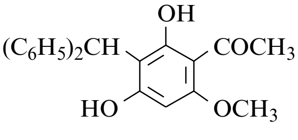
Syntheses

- Obtained by reaction of o-benzyloxybenzyl alcohol with resacetophenone in dioxane in the presence of boron trifluoride etherate at 60–70° for 3 h (18%) [2325].
- Also obtained by reaction of o-benzyloxybenzyl bromide with resacetophenone in methanol in the presence of potassium hydroxide at r.t. for 24 h (7%) [2325].

m.p. 127–128° [2325]; ^{1}H NMR [2325], IR [2325], UV [2325].

1-[3-(Diphenylmethyl)-2,4-dihydroxy-6-methoxyphenyl]ethanone

[101161-94-8] $C_{22}H_{20}O_4$ mol.wt. 348.40



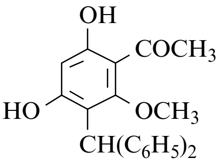
Synthesis

- Obtained by reaction of diphenylcarbinol with 2,4-di-hydroxy-6-methoxyacetophenone in the presence of boron trifluoride etherate in dioxane at r.t. (15%) [2432].

m.p. 150–151° [2432]; ^{1}H NMR [2432], IR [2432], UV [2432].

1-[3-(Diphenylmethyl)-4,6-dihydroxy-2-methoxyphenyl]ethanone

[101161-95-9] $C_{22}H_{20}O_4$ mol.wt. 348.40



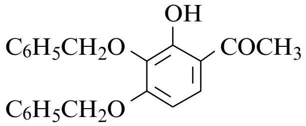
Synthesis

- Obtained by reaction of diphenylcarbinol with 2,4-dihydroxy-6-methoxyacetophenone in the presence of boron trifluoride etherate in dioxane at r.t. (20%) [2432].

m.p. 148–149° [2432]; ^{1}H NMR [2432], IR [2432], UV [2432].

1-[2-Hydroxy-3,4-bis(phenylmethoxy)phenyl]ethanone

[2652-27-9] $C_{22}H_{20}O_4$ mol.wt. 348.40



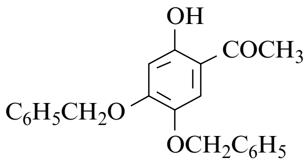
Syntheses

- Obtained by reaction of benzyl chloride with gallacetophenone in the presence of sodium bicarbonate and sodium iodide in refluxing mixture of acetone and ethanol [2403,2817,3637], (48%) [2403], (<2%) [2817].
- Also refer to: [3704].

m.p. 114–115° [2403], 113–114° [2817]; ^{1}H NMR [2403], IR [2403].

1-[2-Hydroxy-4,5-bis(phenylmethoxy)phenyl]ethanone

[7298-39-7] $C_{22}H_{20}O_4$ mol.wt. 348.40



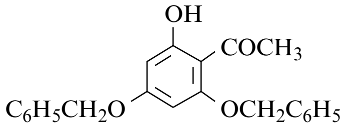
Syntheses

- Preparation by reaction of benzyl halide with 2,4,5-tri-hydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (69%) [2418].
- Preparation by reaction of benzyl chloride with 5-acetoxy-2,4-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (52%) [2906].

m.p. 96–97° [2906], 94–95° [2418]; ^{1}H NMR [2906], IR [2906].

1-[2-Hydroxy-4,6-bis(phenylmethoxy)phenyl]ethanone

[18065-05-9] $C_{22}H_{20}O_4$ mol.wt. 348.40



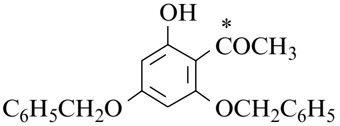
Synthesis

- Preparation by reaction of benzyl chloride on phloroacetophenone with potassium carbonate in refluxing acetone (29%) [2838], (20%) [3605], in DMF at 100° (54%) [2830] and at 150–153° (26%) [3705] or in HMPA at 90–93° (80%) [2829].

m.p. 119–120°5 [2830], 101–102° [3605], 100–102° [2829], 98–100° [2838], 96–98° [3705]. One of the reported melting points is obviously wrong.
^{1}H NMR [2830], IR [2830], UV [3605], MS [2830].

1-[2-Hydroxy-4,6-bis(phenylmethoxy)phenyl]ethanone-*1*-^{13}*C*

[332900-03-5]



$C_{22}H_{20}O_4$ mol.wt. 349.40

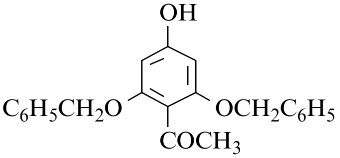
Syntheses

- Preparation by selective deprotection of tri-O-benzyl-phloroacetophenone labelled at the carbonyl group with titanium tetrachloride in methylene chloride for 160 min at 0° (80%) [3706].
- Also refer to: [3707,3708].

m.p. 104° [3706], 103° [3707];
^{1}H NMR [3706], ^{13}C NMR [3706], IR [3706], UV [3706], MS [3706].

1-[4-Hydroxy-2,6-bis(phenylmethoxy)phenyl]ethanone

[76799-38-7]

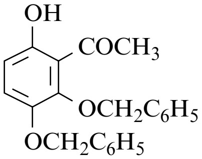


$C_{22}H_{20}O_4$ mol.wt. 348.40

Synthesis

- Refer to: [3198] (compound **7**) (45%).

1-[6-Hydroxy-2,3-bis(phenylmethoxy)phenyl]ethanone



$C_{22}H_{20}O_4$ mol.wt. 348.40

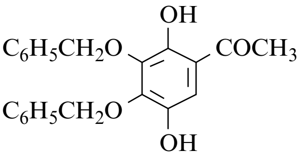
Synthesis

- Preparation by reaction of benzyl chloride 2-(benzyloxy)-3,6-dihydroxyacetophenone with potassium carbonate in refluxing acetone (10%) [2358].

m.p. 57°5 [2358].

1-[2,5-Dihydroxy-3,4-bis(phenylmethoxy)phenyl]ethanone

[151148-87-9]



$C_{22}H_{20}O_5$ mol.wt. 364.40

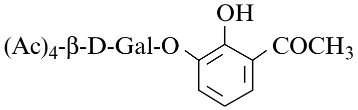
Synthesis

- Obtained by persulfate oxidation of 3,4-bis(benzyloxy)-2-hydroxyacetophenone (Elbs reaction) (10%) [3709].

m.p. 121° [3709];
^{1}H NMR [3709], UV [3709]; TLC [3709].

1-[2-Hydroxy-3-[(2,3,4,6-tetra-O-acetyl-β-D-galactopyranosyl)oxy]phenyl] ethanone

[88086-96-8]



$C_{22}H_{26}O_{12}$ mol.wt. 482.44

Syntheses

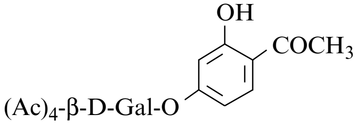
– Obtained by reaction of acetobromo-α-D-galactose with 2,3-dihydroxyacetophenone,

- in the presence of silver carbonate in quinoline at r.t., according to the Koenigs–Knorr method [2842];
- in the presence of 10% aqueous potassium hydroxide in acetone at r.t. for 24 h, according to the Fischer method (14%) [2842].

m.p. 152–153° [2842];
$(\alpha)^{20}_{\mathrm{D}} = -47°$ (c = 1 in chloroform) [2842];
^{1}H NMR [2842].

1-[2-Hydroxy-4-[(2,3,4,6-tetra-O-acetyl-β-D-galactopyranosyl)oxy]phenyl] ethanone

[54918-27-3]



$C_{22}H_{26}O_{12}$ mol.wt. 482.44

Syntheses

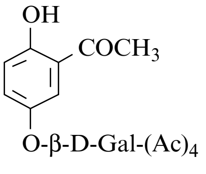
– Preparation by reaction of acetobromo-α-D-galactose (m.p. 79–81°) with resacetophenone,

- in the presence of silver oxide in quinoline at r.t. for 2 h (30%) [3552];
- in the presence of 10% aqueous potassium hydroxide in acetone at r.t. for 24 h (12%) [2842].

m.p. 115–117° [3552], 115° [2842];
$(\alpha)^{22}_{\mathrm{D}} = -2°6$ (c = 4 in chloroform) [3552].

1-[2-Hydroxy-5-[(2,3,4,6-tetra-O-acetyl-β-D-galactopyranosyl)oxy]phenyl] ethanone

[88086-98-0]



$C_{22}H_{26}O_{12}$ mol.wt. 482.44

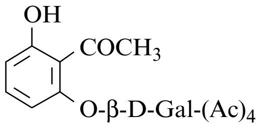
Syntheses

– Preparation by reaction of acetobromo-α-D-galactose,

- with 2-benzoyloxy-5-hydroxyacetophenone in the presence of silver carbonate in dry quinoline at r.t. for 3 h according to the Koenigs–Knorr method (quantitative yield) [2842];
- with quinacetophenone in the presence of 10% aqueous sodium hydroxide in acetone at r.t. for 24 h according to the Fischer method (14%) [2842].

m.p. 69–73° [2842]; ^{1}H NMR [2842].

1-[2-Hydroxy-6-[(2,3,4,6-tetra-O-acetyl-β-D-galactopyranosyl)oxy]phenyl] ethanone

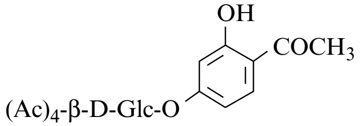


$C_{22}H_{26}O_{12}$ mol.wt. 482.44

Synthesis

- Preparation by reaction of acetobromo-α-D-galactose with 2,6-dihydroxyacetophenone in the presence of silver carbonate in quinoline at r.t. for 3 h [2842], according to Koenigs–Knorr method [3710].

1-[2-Hydroxy-4-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy]phenyl] ethanone



$C_{22}H_{26}O_{12}$ mol.wt. 482.44

Syntheses

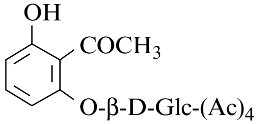
- Obtained by reaction of acetobromo-α-D-glucose with resacetophenone,

- in acetone in the presence of 10% sodium hydroxide at r.t. for 4 days (30%) [3563];
- in acetone in the presence of aqueous potassium hydroxide solution at r.t. for 24–40 h (20–45%) [2528];
- in quinoline in the presence of silver oxide during 15 min (11%) [2736].

m.p. 131–132° [2736], 130–131° [3563];
$(\alpha)_D^{20} = -29°7$ (in acetone) [3563].

1-[2-Hydroxy-6-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy]phenyl] ethanone

[23141-00-6]



$C_{22}H_{26}O_{12}$ mol.wt. 482.44

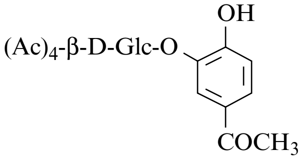
Syntheses

- Preparation by glycosation of 2,6-dihydroxyacetophenone with acetobromo-α-D-glucose,
  - in the presence of cadmium carbonate in refluxing toluene for 20 h, with removal of generated water (71%) [3711], according to Dick's method [3712];
  - in the presence of benzyltributylammonium chloride and potassium carbonate in chloroform at r.t. for 24 h (93%) [3713];
  - in the presence of potassium hydroxide in aqueous acetone (38%) [3711], (32%) [3714].

m.p. 201–203° [3714], 200°5–201°5 [3711], 197–200° [3713];
^1H NMR [3711,3713], IR [3711,3713], MS [3711], FAB-MS [3713].

1-[4-Hydroxy-3-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy]phenyl]ethanone *(Tetraacetylpungenin)*

[101140-11-8] $C_{22}H_{26}O_{12}$ mol.wt. 482.44



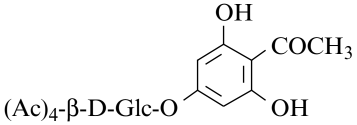
Synthesis

– Preparation by treatment of 3-(β-tetraacetylglucopyranosyl-oxy)-4-(2-methoxyethoxy)methoxyacetophenone (SM) with zinc bromide in methylene chloride at r.t. under nitrogen atmosphere (76%). SM was obtained by action of acetobromo-α-D-glucose with 3-hydroxy-4-(2-methoxy-ethoxy)-methoxyacetophenone in the presence of silver oxide in dry quinoline at r.t. under nitrogen atmosphere [3410].

m.p. 106–112° [3410]; ^{1}H NMR [3410], IR [3410], MS [3410].

1-[2,6-Dihydroxy-4-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy]phenyl] ethanone

$C_{22}H_{26}O_{13}$ mol.wt. 498.42



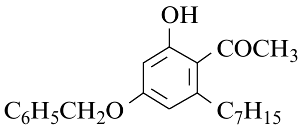
Synthesis

– Obtained by reaction of acetobromo-α-D-glucose with phloracetophenone in the presence of 2.25 N aqueous sodium hydroxide in acetone at 0° (9–12%) [3715].

m.p. 215–216° [3715];
$(\alpha)_D^{20} = -52°7$ (pyridine) [3715].

1-[2-Heptyl-6-hydroxy-4-(phenylmethoxy)phenyl]ethanone

[96864-14-1] $C_{22}H_{28}O_3$ mol.wt. 340.46



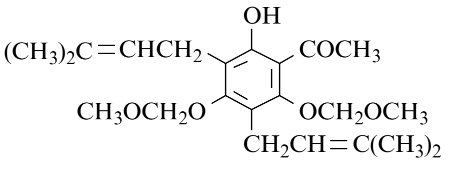
Synthesis

– Preparation by reaction of benzyl bromide with 2,4-dihydroxy-6-heptylacetophenone in the presence of potassium carbonate in refluxing acetone for 1.5 h (42%) [3624].

m.p. 45° [3624]; ^{1}H NMR [3624], MS [3624].

1-[2-Hydroxy-4,6-bis(methoxymethoxy)-3,5-bis(3-methyl-2-butenyl)phenyl] ethanone

[131303-37-2] $C_{22}H_{32}O_6$ mol.wt. 392.49



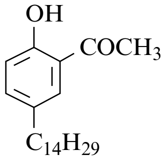
Syntheses

– Obtained by reaction of methoxymethyl chloride with 3,5-diprenyl-2,4,6-trihydroxyacetophenone (57%) [3716].

– Also refer to: [3419,3717,3718].

1-(2-Hydroxy-5-tetradecylphenyl)ethanone

[118469-76-4] $C_{22}H_{36}O_2$ mol.wt. 332.53



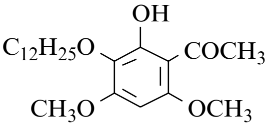
Synthesis

– Preparation by Fries rearrangement of 4-tetradecylphenyl acetate with aluminium chloride without solvent at 120° (94%) [3719].

m.p. 39–40° [3719]; ^{1}H NMR [3719], IR [3719].

1-[3-(Dodecyloxy)-2-hydroxy-4,6-dimethoxyphenyl]ethanone

[103777-47-5] $C_{22}H_{36}O_5$ mol.wt. 380.52



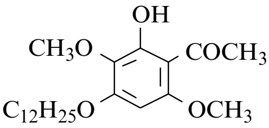
Synthesis

– Preparation by partial demethylation of 3-(dodecyloxy)-2,4,6-trimethoxyacetophenone (SM) with aluminium chloride in acetonitrile at 60° for 1.5 h (83%). SM was obtained by alkylation of 3-hydroxy-2,4,6-trimethoxy-acetophenone with dodecyl iodide in the presence of potassium carbonate in refluxing acetone for 12 h [3148].

m.p. 53–54° [3148].

1-[4-(Dodecyloxy)-2-hydroxy-3,6-dimethoxyphenyl]ethanone

[103777-44-2] $C_{22}H_{36}O_5$ mol.wt. 380.52



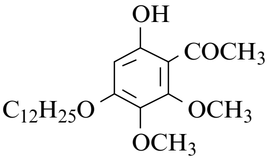
Synthesis

– Preparation by partial alkylation of 4,6-dihydroxy-2,5-di-methoxyacetophenone with dodecyl iodide (92%) [3148].

m.p. 55–56° [3148].

1-[4-(Dodecyloxy)-6-hydroxy-2,3-dimethoxyphenyl]ethanone

[103777-43-1] $C_{22}H_{36}O_5$ mol.wt. 380.52

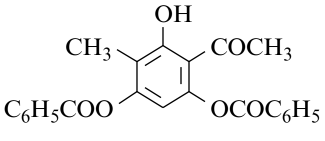


Synthesis

– Preparation by partial alkylation of 4,6-dihydroxy-2,3-di-methoxyacetophenone with dodecyl iodide [3148].

Oil [3148].

1-[4,6-Bis(benzoyloxy)-2-hydroxy-3-methylphenyl]ethanone



$C_{23}H_{18}O_6$ mol.wt. 390.39

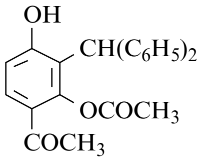
Synthesis

- Obtained by reaction of benzoyl chloride on 2,4,6-tri-hydroxy-3-methylacetophenone with 2% sodium hydroxide solution at 0° (9–12%) [3051].

m.p. 149° [3051].

1-[2-(Acetyloxy)-3-(diphenylmethyl)-4-hydroxyphenyl]ethanone

[145747-37-1] $C_{23}H_{20}O_4$ mol.wt. 360.41

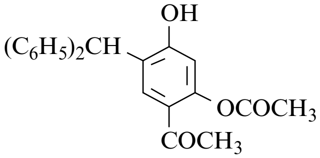


Synthesis

- Obtained by enzymatic hydrolysis of 2,4-diacetoxy-3-(diphenylmethyl)acetophenone in the presence of porcine pancreas lipase in tetrahydrofuran at 42–45° (70%) [2388].

1-[2-(Acetyloxy)-5-(diphenylmethyl)-4-hydroxyphenyl]ethanone

[145747-38-2]



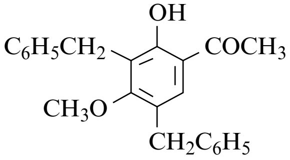
$C_{23}H_{20}O_4$ mol.wt. 360.41

Synthesis

- Obtained by enzymatic hydrolysis of 2,4-diacetoxy-5-(diphenylmethyl)acetophenone in the presence of porcine pancreas lipase in tetrahydrofuran at 42–45° (68%) [2388].

1-[2-Hydroxy-4-methoxy-3,5-bis(phenylmethyl)phenyl]ethanone

[95832-46-5]



$C_{23}H_{22}O_3$ mol.wt. 346.43

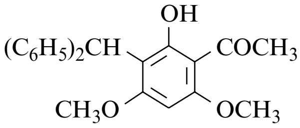
Synthesis

- Preparation by reaction of dimethyl sulfate with 3,5-di-benzyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (90%) [3703].

Oil [3703]; ^{1}H NMR [3703].

1-[3-(Diphenylmethyl)-2-hydroxy-4,6-dimethoxyphenyl]ethanone

[101161-96-0]



$C_{23}H_{22}O_4$ mol.wt. 362.42

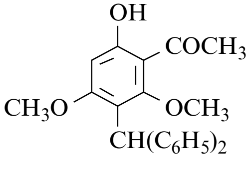
Synthesis

- Preparation by reaction of dimethyl sulfate with 2,4-di-hydroxy-6-methoxy-3-(diphenylmethyl) acetophenone in the presence of potassium carbonate in refluxing acetone (82%) [2432].

m.p. 173–174° [2432]; ^{1}H NMR [2432], UV [2432].

1-[3-(Diphenylmethyl)-6-hydroxy-2,4-dimethoxyphenyl]ethanone

[101161-97-1] $C_{23}H_{22}O_4$ mol.wt. 362.42



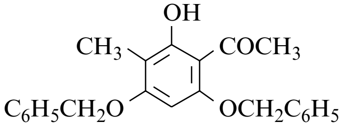
Synthesis

- Preparation by reaction of dimethyl sulfate with 2,4-di-hydroxy-6-methoxy-5-(diphenylmethyl)acetophenone in the presence of potassium carbonate in refluxing acetone (69%) [2432].

m.p. 167–168° [2432]; ^{1}H NMR [2432], UV [2432].

1-[2-Hydroxy-3-methyl-4,6-bis(phenylmethoxy)phenyl]ethanone

[39548-92-0] $C_{23}H_{22}O_4$ mol.wt. 362.42



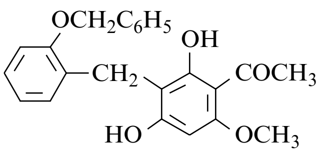
Syntheses

- Preparation by reaction of benzyl chloride on 3-methylphloracetophenone with potassium carbonate in refluxing acetone (27–31%) [3043,3051], (18%) [3605].
- Also refer to: [3054].

m.p. 145° [3051,3605,3720], 142° [3043].

1-[2,4-Dihydroxy-6-methoxy-3-[[2-(phenylmethoxy)phenyl]methyl]phenyl] ethanone

[103633-31-4] $C_{23}H_{22}O_5$ mol.wt. 378.42



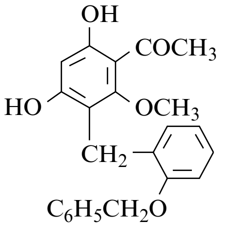
Syntheses

- Obtained by reaction of o-(benzyloxy)benzyl bromide with 2,4-dihydroxy-6-methoxyacetophenone in the presence of potassium hydroxide in methanol at r.t. for 24 h (18%) [2325].
- Also obtained by reaction of o-(benzyloxy)benzyl alcohol with 2,4-dihydroxy-6-methoxyacetophenone in the presence of boron trifluoride etherate in dioxane (7%) [2325].

m.p. 152–153° [2325]; TLC [2325];
^{1}H NMR [2325], IR [2325], UV [2325].

1-[4,6-Dihydroxy-2-methoxy-3-[[2-(phenylmethoxy)phenyl]methyl]phenyl] ethanone

[103633-32-5] $C_{23}H_{22}O_5$ mol.wt. 378.42



Syntheses

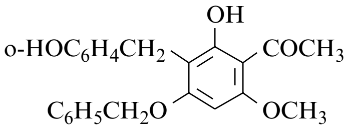
- Obtained by reaction of o-(benzyloxy)benzyl alcohol with 2,4-dihydroxy-6-methoxyacetophenone in the presence of boron trifluoride etherate in dioxane at 60–70° for 3 h (<15%) [2325].
- Also obtained by reaction of o-(benzyloxy)benzyl bromide with 2,4-dihydroxy-6-methoxyacetophenone in the presence of potassium hydroxide in methanol at r.t. for 24 h (7%) [2325].

m.p. 145–146° [2325]; TLC [2325]; ^{1}H NMR [2325], IR [2325], UV [2325].

1-[2-Hydroxy-3-[(2-hydroxyphenyl)methyl]-6-methoxy-4-(phenylmethoxy)-phenyl]ethanone

1-[2,4-Dihydroxy-3-[(2-hydroxyphenyl)methyl]-6-methoxyphenyl]ethanone, monoether with benzenemethanol

[102056-83-7] $C_{23}H_{22}O_5$ mol.wt. 378.42

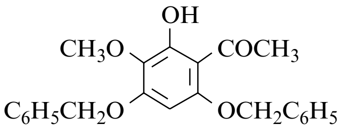


Synthesis

- Obtained by reaction of benzyl chloride with 2′,4′-di-hydroxy-3′-(2-hydroxybenzyl)-6′-methoxy-acetophenone in the presence of potassium carbonate in refluxing acetone (10%) [2832].

1-[2-Hydroxy-3-methoxy-4,6-bis(phenylmethoxy)phenyl]ethanone

[24126-73-6] $C_{23}H_{22}O_5$ mol.wt. 378.42



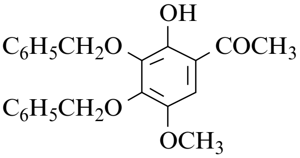
Syntheses

- Preparation by selective cleavage of 2-benzyloxy group of 2,4,6-tris(benzyloxy)-3-methoxyacetophenone (SM) with concentrated hydrochloric acid in acetic acid at r.t. for 50 min (82%). SM (oily material) was obtained by reaction of benzyl chloride with 4-benzyloxy-2,6-di-hydroxy-3-methoxyacetophenone in the presence of potassium carbonate in DMF at 150–160°for 10 min [3640].
- Also obtained by partial benzylation of 3-methoxy-2,4,6-trihydroxyacetophenone [3721], with benzyl chloride in the presence of potassium carbonate in refluxing acetone for 16 h (6%) [3722].

m.p. 140–141° [3721,3722], 137–138° [3640]; ^{1}H NMR [3721]; UV [3721,3722].

1-[2-Hydroxy-5-methoxy-3,4-bis(phenylmethoxy)phenyl]ethanone

[158148-88-0] $C_{23}H_{22}O_5$ mol.wt. 378.42



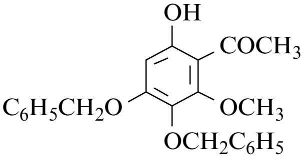
Synthesis

- Preparation by reaction of dimethyl sulfate with 3,4-bis(benzyloxy)-2,5-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 8 h (79%) [3709].

m.p. 131° [3709]; TLC [3709];
^{1}H NMR [3709], IR [3709], UV [3709].

1-[6-Hydroxy-2-methoxy-3,4-bis(phenylmethoxy)phenyl]ethanone

[73239-53-9] $C_{23}H_{22}O_5$ mol.wt. 378.42



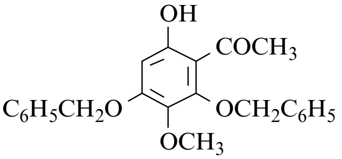
Synthesis not yet described

- Refer to: [2885].

TLC [2885].

1-[6-Hydroxy-3-methoxy-2,4-bis(phenylmethoxy)phenyl]ethanone

$C_{23}H_{22}O_5$ mol.wt. 378.42



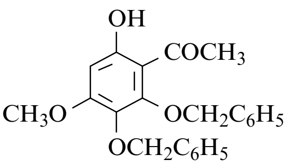
Syntheses

- Obtained by partial benzylation of 3-methoxy-2,4,6-tri-hydroxyacetophenone [3721], with benzyl chloride in the presence of potassium carbonate in refluxing acetone for 16 h (31%) [3722].
- Also refer to: [3507,3723].

Oil [3722]; m.p. 70–71° [3721]; ^{1}H NMR [3721].

1-[6-Hydroxy-4-methoxy-2,3-bis(phenylmethoxy)phenyl]ethanone

[54299-56-8] $C_{23}H_{22}O_5$ mol.wt. 378.42



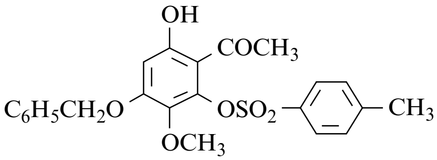
Synthesis

- Preparation by reaction of benzyl chloride with 6-(benzyloxy)-2,5-dihydroxy-4-methoxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (71%) [3130].

m.p. 95–97° [3130].

1-[6-Hydroxy-3-methoxy-2-[[(4-methylphenyl)sulfonyl]oxy]-4-(phenylmethoxy)phenyl]ethanone

[188927-30-2] $C_{23}H_{22}O_7S$ mol.wt. 442.49



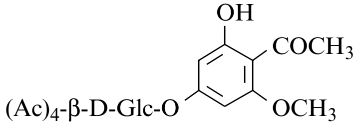
Synthesis

- Preparation by treatment of 4-(benzyloxy)-3,6-dimethoxy-2-(tosyloxy)acetophenone (m.p. 106–108°) with aluminium bromide in acetonitrile at 0° for 1 h (quantitative yield) [3507].

m.p. 94–95° [3507].

1-[2-Hydroxy-6-methoxy-4-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy] phenyl]ethanone

[139545-92-9] $C_{23}H_{28}O_{13}$ mol.wt. 512.47



Syntheses

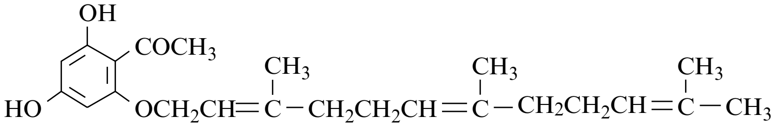
- Obtained by treatment of 2,4-dihydroxy-6-methoxy-acetophenone with aceto-bromo-α-D-glucose,

- in acetone in the presence of 10% aqueous sodium hydroxide at r.t. for 4.5 h (48%) [3619];
- in quinoline or in acetone in the presence of silver oxide at r.t. for 20 min (22% and 30% yields, respectively) [3724].

m.p. 169–171° [3619], 168° [3724];
$(\alpha)_D^{25} = -39°7$ to $-42°3$ (pyridine) [3724];
^{1}H NMR [3619], IR [3619], MS [3619]; TLC [3619].

1-[2,4-Dihydroxy-6-[(3,7,11-trimethyl-2,6,10-dodecatrienyl)oxy]phenyl]ethanone *(E,E)*

[200129-18-6] $C_{23}H_{32}O_4$ mol.wt. 372.50

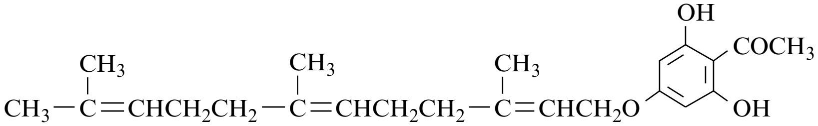


Synthesis

- Refer to: [3725] (Chinese paper).

1-[2,6-Dihydroxy-4-[(3,7,11-trimethyl-2,6,10-dodecatrienyl)oxy]phenyl] ethanone

[156499-52-4] $C_{23}H_{32}O_4$ mol.wt. 372.50

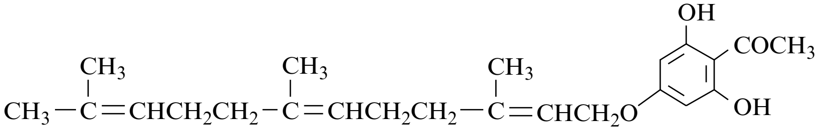


Isolation from natural sources

- From the aerial parts of *Boronia Ramosa* (Rutaceae) [3726].
  Gum [3726];
  ^{1}H NMR [3726], ^{13}C NMR [3726], IR [3726], UV [3726], MS [3726].

1-[2,6-Dihydroxy-4-[(3,7,11-trimethyl-2,6,10-dodecatrienyl)oxy]phenyl] ethanone *(E,E)*

[183143-91-1] $C_{23}H_{32}O_4$ mol.wt. 372.50



Syntheses

- Preparation by hydrolysis of 2-p-toluenesulfonyloxy-6-hydroxy-4-(1′-farnesyloxy)acetophenone with 30% potassium hydroxide in refluxing ethanol for 1.5 h (89%) [3608].
- Obtained (poor yield) by reaction of farnesyl bromide with phloroacetophenone in the presence of potassium carbonate in refluxing acetone for 4 h (7%) [3725,3727].

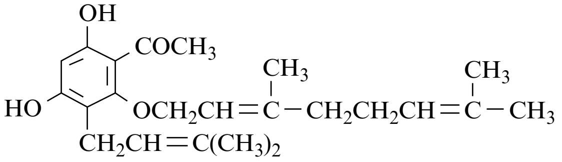
Isolation from natural sources

- From the aerial parts of *Boronia ramosa* in Australian genus *Boronia* (Rutaceae) [3725,3726].

  White gum [3608,3726];
  ^{1}H NMR [3608,3726], ^{13}C NMR [3726], IR [3608,3726], UV [3726], EIMS [3608,3726].

1-[2-[(3,7-Dimethyl-2,6-octadienyl)oxy]-4,6-dihydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone *(E)*

[126259-76-5] $C_{23}H_{32}O_4$ mol.wt. 372.50



Synthesis

- Obtained by hydrolysis of 4′,6′-bis(benzoyloxy)-2′-geranyloxy-3′-prenylacetophenone with dilute sodium hydroxide under nitrogen atmosphere at 50° (good yield) [3571].

Isolation from natural sources

- From the *Euodia Lunu-Ankenda* root bark (Rutaceae) [3696].

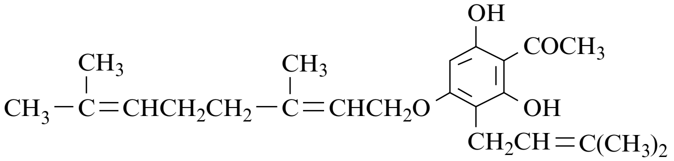
N.B.: The synthetic works [3571,3695] have suggested that the natural product ketone isolated by [3696] had an incorrectly assigned structure. This compound [3696] will be identical with 1-[4-[(3,7-dimethyl-2,6-octadienyl)oxy]-2,6-dihydro-3-(3-methyl-2-butenyl)phenyl]ethanone [142905-38-2].

m.p. 88–90° [3696], 71–73° [3571]. One of the reported melting points is obviously wrong.

^{1}H NMR [3571,3696], IR [3696][3571], UV [3696], MS [3696],
HRMS [3696]; TLC [3696];

1-[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2,6-dihydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone *(E)*

[142905-38-2] $C_{23}H_{32}O_4$ mol.wt. 372.50



Synthesis

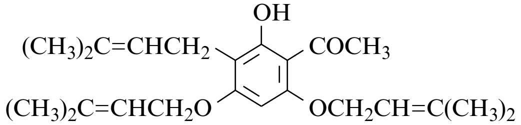
- Obtained via a nine-step synthesis from the 4′,6′-bis(benzyloxy)-2′-hydroxyacetophenone [3695].

Isolation from natural sources

- From the fruit of *Evodia Merrillii* Kanehira and Sasaki ex Kanehira (Rutaceae) [3697,3728].

m.p. 98–101° [3697,3728], 98–100° [3695]; column chromatography [3697];
^{1}H NMR [3695,3697,3728], ^{13}C NMR [3697],
IR [3695,3697,3728], UV [3697], MS [3697].

1-[2-Hydroxy-3-(3-methyl-2-butenyl)-4,6-bis[(3-methyl-2-butenyl)oxy]phenyl] ethanone



$C_{23}H_{32}O_4$ mol.wt. 372.50

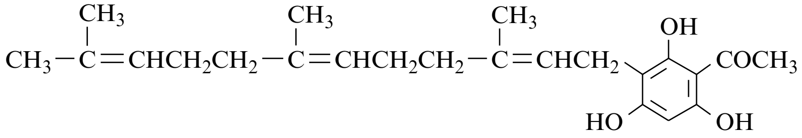
Synthesis

- Obtained (poor yield) by reaction of prenyl bromide with phloroaceto-phenone in the presence of aqueous potassium hydroxide solution (3%) [3117].

b.p.$_{0.2}$ 135–150° [3117].

1-[2,4,6-Trihydroxy-3-(3,7,11-trimethyl-2,6,10-dodecatrienyl)phenyl]ethanone

[156499-51-3] $C_{23}H_{32}O_4$ mol.wt. 372.50



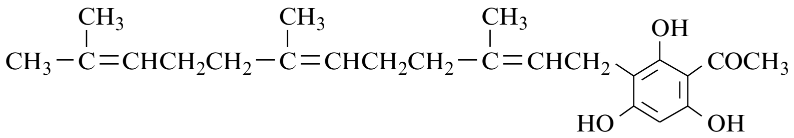
Isolation from natural sources

- From the aerial parts of *Boronia Ramosa* (Rutaceae) [3726].

m.p. 112–115° [3726];
^{1}H NMR [3726], ^{13}C NMR [3726], IR [3726], UV [3726], MS [3726].

1-[2,4,6-Trihydroxy-3-(3,7,11-trimethyl-2,6,10-dodecatrienyl)phenyl]ethanone *(E,E)*

[183143-90-0] $C_{23}H_{32}O_4$ mol.wt. 372.50

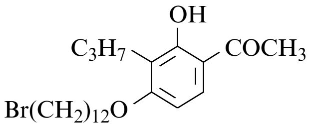


Synthesis

- Obtained by reaction of farnesyl bromide with phloroacetophenone in the presence of potassium carbonate in refluxing acetone for 4 h (52%) [3725,3727].Isolation from natural sources
- From the aerial parts of *Boronia ramosa* in Australia [3725].

1-[4-[(12-Bromododecyl)oxy]-2-hydroxy-3-propylphenyl]ethanone

[106627-36-5] $C_{23}H_{37}BrO_3$ mol.wt. 441.45

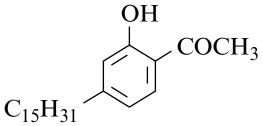


Synthesis

- Preparation by reaction of 1,12-dibromododecane with 2,4-dihydroxy-3-propylacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (45%) [3181].

1-(2-Hydroxy-4-pentadecylphenyl)ethanone

[52122-69-7] $C_{23}H_{38}O_2$ mol.wt. 346.55



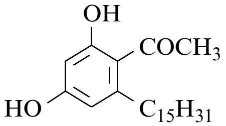
Synthesis

- Preparation by Fries rearrangement of 3-pentadecylphenyl acetate with aluminium chloride without solvent at 140–150° [2601].

m.p. 50° [2601]; b.p.$_2$ 212–214° [2601].

1-(2,4-Dihydroxy-6-pentadecylphenyl)ethanone

$C_{23}H_{38}O_3$ mol.wt. 362.55



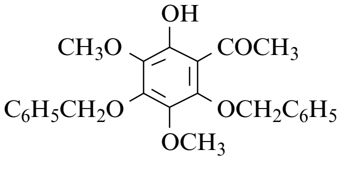
Synthesis

- Preparation by reaction of acetic acid on 5-pentadecyl-resorcinol with boron trifluoride and hydrofluoric acid in xylene at 50–60° (75%) [3729].

m.p. 63° [3729].

1-[2-Hydroxy-3,5-dimethoxy-4,6-bis(phenylmethoxy)phenyl]ethanone

[3162-54-7] $C_{24}H_{24}O_6$ mol.wt. 408.45



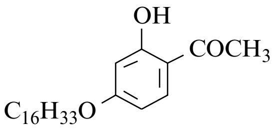
Synthesis

- Obtained by alkaline degradation of *Lucidin dibenzyl ether* by refluxing with 10% aqueous ethanolic potassium hydroxide under nitrogen atmosphere for 17 h [3428].

b.p.$_{0.2}$ 160–180° [3428]; m.p. 86–87° [3428].

1-[4-(Hexadecyloxy)-2-hydroxyphenyl]ethanone

[143286-87-7] $C_{24}H_{40}O_3$ mol.wt. 376.58



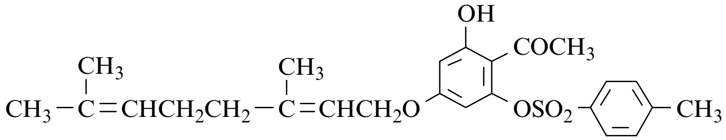
Synthesis

- Preparation by partial alkylation of resacetophenone with hexadecyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [3493].

m.p. 56° [3493].

1-[4-[[(2*E*)-3,7-Dimethyl-2,6-octadienyl]oxy]-2-hydroxy-6-[[(4-methylphenyl) sulfonyl]oxy]phenyl]ethanone

[225088-73-3] $C_{25}H_{30}O_6S$ mol.wt. 458.58



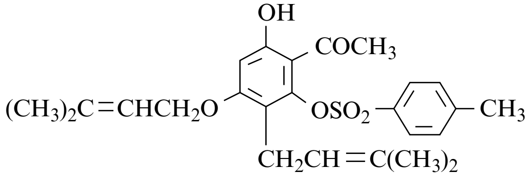
Synthesis

- Preparation by treatment of 2-toluenesulfonyl-oxy-4,6-dihydroxy-acetophenone with geranyl bromide in acetone in the presence of potassium carbonate at r.t. for 2 h (84%) [3608].

  Colourless gum [3608];
  ^{1}H NMR [3608], IR [3608], EIMS [3608], HREIMS [3608].

1-[6-Hydroxy-3-(3-methyl-2-butenyl)-4-[(3-methyl-2-butenyl)oxy]-2-[[(4-methylphenyl)sulfonyl]oxy]phenyl]ethanone

[158499-97-9] $C_{25}H_{30}O_6S$ mol.wt. 458.58

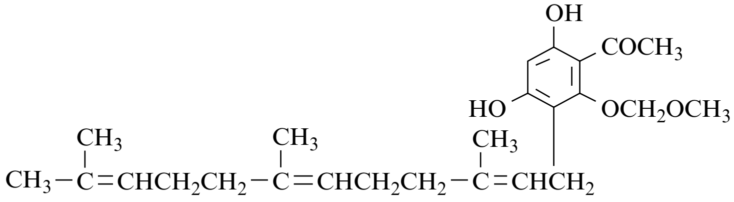


Synthesis

- Obtained by reaction of prenyl bromide with 4′,6′-dihydroxy-3′-prenyl-2′-tosyloxyacetophenone in the presence of potassium carbonate in acetone at 20° for 2 h [3695].
  ^{1}H NMR [3695].

1-[4,6-Dihydroxy-3-(methoxymethoxy)-3-(3,7,11-trimethyl-2,6,10-dodecatrienyl)phenyl]ethanone

$C_{25}H_{36}O_5$ mol.wt. 416.56

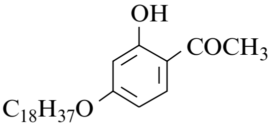


Synthesis

- Obtained by reaction of chloromethyl methyl ether with 3-farnesyl-2,4,6-trihydroxyacetophe-none in the presence of potassium carbonate in refluxing acetone for 5 h (82%) [3727].

1-[2-Hydroxy-4-(octadecyloxy)phenyl]ethanone

[99283-88-2] $C_{26}H_{44}O_3$ mol.wt. 404.63



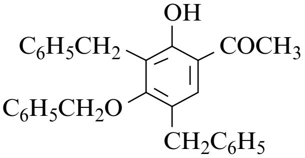
Syntheses

- Preparation by partial alkylation of resacetophenone with octadecyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [3493].
- Also refer to: [3698].

m.p. 61° [3493,3698]; GC [3730].

1-[2-Hydroxy-4-(phenylmethoxy)-3,5-bis(phenylmethyl)phenyl]ethanone

[105485-47-0] $C_{29}H_{26}O_3$ mol.wt. 422.52



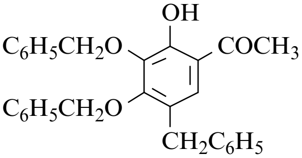
Synthesis

- Obtained by reaction of benzyl chloride with 3,5-dibenzyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone [3583].

m.p. 87–89° [3583]; ^{1}H NMR [3583], IR [3583], UV [3583].

1-[2-Hydroxy-3,4-bis(phenylmethoxy)-5-(phenylmethyl)phenyl]ethanone

[105485-46-9] $C_{29}H_{26}O_4$ mol.wt. 438.52



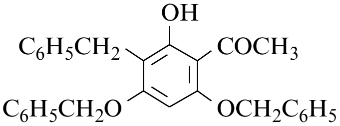
Synthesis

- Preparation by reaction of benzyl chloride with 5-benzyl-2,3,4-trihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone (64%) [3583].

m.p. 56–58° [3583]; ^{1}H NMR [3583], IR [3583], UV [3583].

1-[2-Hydroxy-4,6-bis(phenylmethoxy)-3-(phenylmethyl)phenyl]ethanone

[18065-06-0] $C_{29}H_{26}O_4$ mol.wt. 438.52



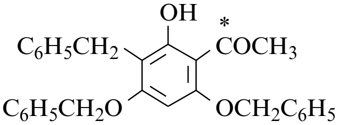
Synthesis

- Obtained by reaction of benzyl chloride with phloroacetophenone in the presence of potassium carbonate in DMF at 150–153° (31%) [3705] or in refluxing acetone [3605,3606], (2%) [3605].

m.p. 123–124° [3605,3606], 111–112° [3705];
^{1}H NMR [3605], IR [3606], UV [3605,3606].

1-[2-Hydroxy-4,6-bis(phenylmethoxy)-3-(phenylmethyl)phenyl]ethanone-*1*-^{13}*C*

[357409-15-5]



$C_{29}H_{26}O_4$ mol.wt. 439.52

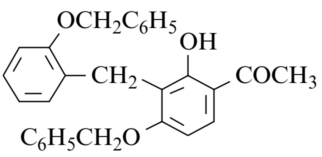
Syntheses

- Obtained (by-product) by selective deprotection of tri-O-benzylphloroacetophenone labelled at the carbonyl group with titanium tetrachloride in methylene chloride for 160 min at 0° (7%) [3706].
- Also refer to: [3708].

m.p. 117° [3706];
^{1}H NMR [3706], ^{13}C NMR [3706], IR [3706], UV [3706], MS [3706].

1-[2-Hydroxy-4-(phenylmethoxy)-3-[[2-(phenylmethoxy)phenyl]methyl]phenyl]ethanone

[103633-40-5]



$C_{29}H_{26}O_4$ mol.wt. 438.52

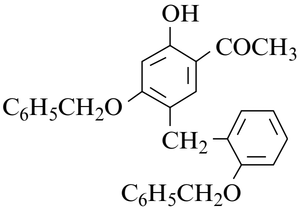
Synthesis

- Preparation by reaction of benzyl chloride with 3-(o-benzyloxybenzyl)-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone for 6 h (73%) [2325].

m.p. 135–136° [2325]; TLC [2325]; ^{1}H NMR [2325], IR [2325], UV [2325].

1-[2-Hydroxy-4-(phenylmethoxy)-5-[[2-(phenylmethoxy)phenyl]methyl]phenyl]ethanone

[103633-43-8]



$C_{29}H_{26}O_4$ mol.wt. 438.52

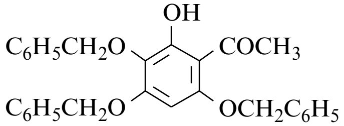
Synthesis

- Preparation by reaction of benzyl chloride with 5-(o-benzyl-oxybenzyl)-2,4-dihydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone for 6 h (75%) [2325].

m.p. 113–114° [2325]; TLC [2325];
^{1}H NMR [2325], IR [2325], UV [2325].

1-[2-Hydroxy-3,4,6-tris(phenylmethoxy)phenyl]ethanone

[60840-18-8] $C_{29}H_{26}O_5$ mol.wt. 454.52



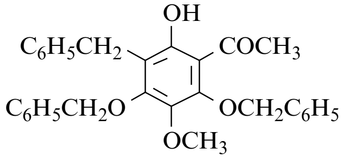
Synthesis

- Preparation from 2,3,4,6-tetrakis(benzyloxy) acetophenone on refluxing with 90% aqueous acetic acid (91%) [3731], (78%) [2442,3149]. There is a selective hydrolysis of the 2-(benzyloxy) group [3731].

m.p. 141–142° [2442,3149];
^{1}H NMR [2442,3149,3731], IR [2442,3149,3731], MS [3731].

1-[2-Hydroxy-5-methoxy-4,6-bis(phenylmethoxy)-3-(phenylmethyl)phenyl] ethanone

$C_{30}H_{28}O_5$ mol.wt. 468.55



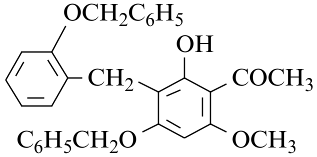
Synthesis

- Obtained (by-product) by partial benzylation of 3-methoxy-2,4,6-trihydroxyacetophenone [3721].

Oil [3721]; ^{1}H NMR [3721].

1-[2-Hydroxy-6-methoxy-4-(phenylmethoxy)-3-[[2-(phenylmethoxy)phenyl] methyl]phenyl]ethanone

[102104-05-2] $C_{30}H_{28}O_5$ mol.wt. 468.55



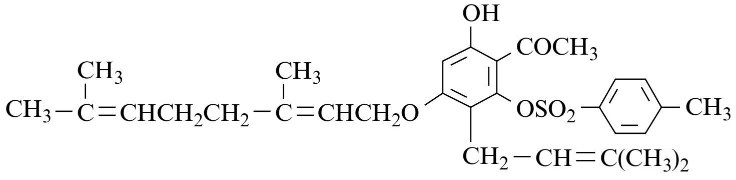
Synthesis

- Preparation by reaction of benzyl chloride with 3-(o-benzyloxybenzyl)-2,4-dihydroxy-6-methoxy-acetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone for 6 h (73%) [2325].

m.p. 167–168° [2325]; TLC [2325];
^{1}H NMR [2325], IR [2325], UV [2325].

1-[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-6-hydroxy-3-(3-methyl-2-butenyl)-2-[[(4-methylphenyl)sulfonyl]oxy]phenyl]ethanone *(E)*

[158499-96-8] $C_{30}H_{38}O_6S$ mol.wt. 526.69

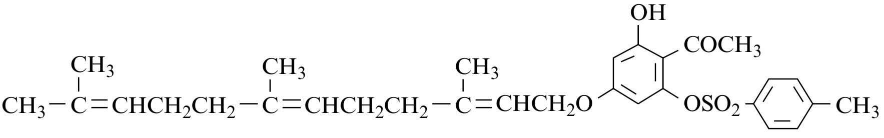


Synthesis

- Obtained by reaction of geranyl bromide with 4′,6′-dihydroxy-3′-prenyl-2′-tosyloxyacetophenone in the presence of potassium carbonate in acetone at 20° for 2 h [3695].
  ^{1}H NMR [3695].

1-[2-Hydroxy-6-[[(4-methylphenyl)sulfonyl]oxy]-4-[[*(2E,6E)*-3,7,11-trimethyl-2,6,10-dodecatrienyl]oxy]phenyl]ethanone

[225088-74-4] $C_{30}H_{38}O_6S$ mol.wt. 526.69

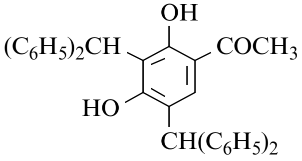


Synthesis

- Obtained by reaction of 2-toluenesulfonyloxy-4,6-dihydroxyacetophenone with farnesyl bromide in acetone in the presence of potassium carbonate at r.t. for 2 h (60%) [3608].
  Colourless gum [3608];
  ^{1}H NMR [3608], IR [3608], EIMS [3608].

1-[3,5-Bis(diphenylmethyl)-2,4-dihydroxyphenyl]ethanone

[107114-29-4] $C_{34}H_{28}O_3$ mol.wt. 484.59



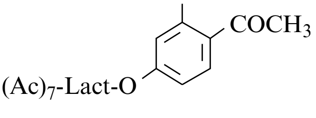
Synthesis

- Obtained by reaction of resacetophenone with diphenyl-carbinol in the presence of boron trifluoride etherate in dioxane at r.t. (14%) [3703].

m.p. 144–145° [3703];
^{1}H NMR [3703], IR [3703], UV [3703].

1-[2-Hydroxy-4-[(per-O-acetyl-β-D-galactopyranosyl-β-D-glucopyranosyl)oxy]phenyl]ethanone

$C_{34}H_{42}O_{20}$ mol.wt. 770.70



(Ac)$_7$-Lact = heptaacetyllactosid rest

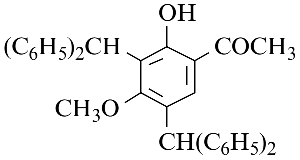
Synthesis

- Preparation by reaction of α-acetobromolactose with resacetophenone in the presence of silver oxide in quinoline at r.t. for 2 h (40%) [3552].

m.p. 195–197° [3552];
$(\alpha)_D^{21}$ = −32°3 (c = 2 in chloroform) [3552].

1-[3,5-Bis(diphenylmethyl)-2-hydroxy-4-methoxyphenyl]ethanone

[107114-31-8] $C_{35}H_{30}O_3$ mol.wt. 498.62



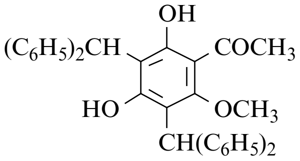
Synthesis

– Preparation by reaction of dimethyl sulfate with 3,5-bis-(diphenylmethyl)-2,4-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone (81%) [3703].

m.p. 152–153° [3703]; ^{1}H NMR [3703].

1-[3,5-Bis(diphenylmethyl)-2,4-dihydroxy-6-methoxyphenyl]ethanone

[101161-93-7] $C_{35}H_{30}O_4$ mol.wt. 514.62



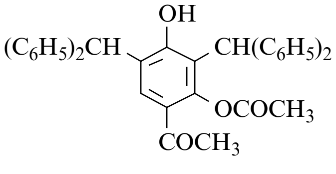
Synthesis

– Obtained (poor yield) by reaction of diphenylcarbinol with 2,4-dihydroxy-6-methoxyacetophenone in dioxane at r.t. in the presence of boron trifluoride etherate (7%) [2432].

m.p. 122–123° [2432]; ^{1}H NMR [2432], IR [2432], UV [2432].

1-[2-(Acetyloxy)-3,5-bis(diphenylmethyl)-4-hydroxyphenyl]ethanone

[145747-39-3] $C_{36}H_{30}O_4$ mol.wt. 526.63

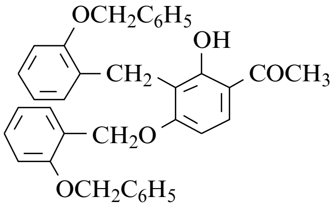


Synthesis

– Obtained by partial deacylation of 2,4-diacetoxy-3,5-bis-(diphenylmethyl)acetophenone by porcine pancreatic lipase in tetrahydrofuran at 42–45° (63%) [2388,2389].

1-[2-Hydroxy-4-[[2-(phenylmethoxy)phenyl]methoxy]-3-[[2-(phenylmethoxy)phenyl]methyl]phenyl]ethanone

[103633-46-1] $C_{36}H_{32}O_5$ mol.wt. 544.65



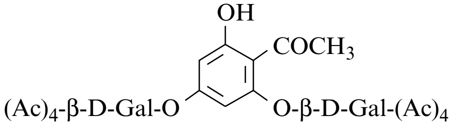
Synthesis

– Obtained (poor yield) by reaction of o-benzyloxy-benzyl bromide with resacetophenone in methanol in the presence of potassium hydroxide at r.t. for 24 h (<2%) [2325].

m.p. 135–136° [2325]; TLC [2325];
^{1}H NMR [2325], IR [2325], UV [2325].

1-[2-Hydroxy-4,6-bis[(2,3,4,6-tetra-O-acetyl-β-D-galactopyranosyl)oxy]phenyl]ethanone

[88087-00-7] $C_{36}H_{44}O_{22}$ mol.wt. 828.73



Synthesis

– Obtained by reaction of 2,3,4,6-tetra-O-acetyl-α-D-galactopyranosyl bromide with phloracetophenone in the presence of 30% aqueous sodium hydroxide in acetone (17%) [2842], according to Zemplen's procedure [3715].

m.p. 184–186° [2842]; ^{1}H NMR [2842].

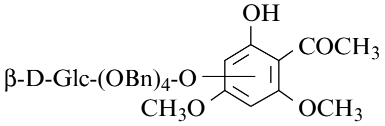
1-[2-Hydroxy-4,6-dimethoxy-3-[2,3,4,6-tetrakis-O-(phenylmethyl)-β-D-glucopyranosyl]phenyl]ethanone

[115130-46-6] $C_{44}H_{46}O_9$ mol.wt. 718.84

and

1-[6-Hydroxy-2,4-dimethoxy-3-[2,3,4,6-tetrakis-O-(phenylmethyl)-β-D-glucopyranosyl]phenyl]ethanone

[169566-54-5] $C_{44}H_{46}O_9$ mol.wt. 718.84



Synthesis

– Obtained (via O C glycoside rearrangement) by adding boron trifluoride etherate to a mixture of 2-hydroxy-4,6-dimethoxyacetophenone, 2,3,4,6-tetra-O-benzyl-α-D-glucopyranosyl fluoride (R) and powdered molecular sieves 4 Å in methylene chloride and stirring at −78° for 2 h under an argon atmosphere; after the disappearance of R on TLC, the reaction temperature was raised from −78° to r.t. and the mixture stirred for 45 min (92%) [3198].

N.B.: The *3-isomer* was obtained by selective glycosylation of 2-hydroxy-4,6-dimethoxy-acetophenone with O-(2,3,4,6-tetra-O-benzyl-α-D-glucopyranosyl) trichloroacetimidate and trimethylsilyl triflate as promoter in methylene chloride first at −25°, then heating up to r.t. [3732,3733].

^{1}H NMR [3198], IR [3198], MS [3198].

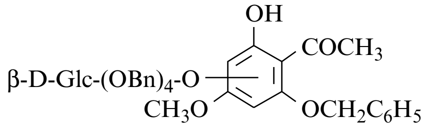
1-[2-Hydroxy-4-methoxy-6-(phenylmethoxy)-3-[2,3,4,6-tetrakis-O-(phenylmethyl)-β-D-glucopyranosyl]phenyl]ethanone

[169566-44-3] $C_{50}H_{50}O_9$ mol.wt. 794.94

and

1-[6-Hydroxy-4-methoxy-2-(phenylmethoxy)-3-[2,3,4,6-tetrakis-O-(phenylmethyl)-β-D-glucopyranosyl]phenyl]ethanone

[169566-55-6] $C_{50}H_{50}O_9$ mol.wt. 794.94



Synthesis

– Obtained (via O fi C glycoside rearrangement) by adding boron trifluoride etherate to a mixture of 2-(benzyloxy)-6-hydroxy-4-methoxy-acetophenone, 2,3,4,6-tetra-O-benzyl-α-D-glucopyranosyl fluoride (R) and powdered molecular sieves 4 Å in methylene chloride and stirring at −78° for 2 h under an argon atmosphere; after the disappearance of (R) on TLC, the reaction temperature was raised from −78° to r.t. and the mixture stirred for 45 min (78%) [3198].

^{1}H NMR [3198], IR [3198], MS [3198].

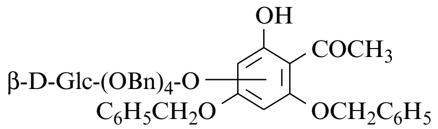
1-[2-Hydroxy-4,6-bis(phenylmethoxy)-3-[2,3,4,6-tetrakis-O-(phenylmethyl)-β-D-glucopyranosyl]phenyl]ethanone

[169566-46-5] $C_{56}H_{54}O_9$ mol.wt. 871.04

and

1-[6-Hydroxy-2,4-bis(phenylmethoxy)-3-[2,3,4,6-tetrakis-O-(phenylmethyl)-β-D-glucopyranosyl]phenyl]ethanone

[169566-56-7] $C_{56}H_{54}O_9$ mol.wt. 871.04



Synthesis

– Obtained (via O fi C glycoside rearrangement) by adding boron trifluoride etherate to a mixture of 2-acetylphloroglucinol 3,5-bis-(benzyl ether), 2,3,4,6-tetra-O-benzyl-α-D-glucopyranosyl fluoride (R) and powdered molecular sieves 4 Å in methylene chloride and stirring at −20° for 2 h under an argon atmosphere; after the disappearance of (R) on TLC, the reaction temperature was raised from −20° to r.t. and the mixture stirred for 45 min (92%) [3198].

Colourless syrup [3198]; ^{1}H NMR [3198], IR [3198], MS [3198].

Part VI
Addendum to Volume 2

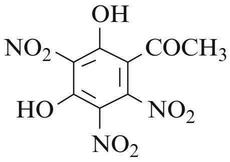
Chapter 10
Addendum 2005–2008

Monoketones Unsubstituted on the Acetyl Groups

Chapter 9. Compounds Derived from Acetic Acid [3734] p. 659–1092

1-(2,4-Dihydroxy-3,5,6-trinitrophenyl)ethanone

$C_8H_5N_3O_9$ mol.wt. 287.14



New compound

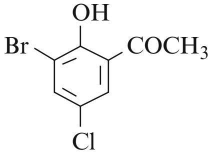
Syntheses

- Obtained by reaction of nitric acid with 3,5-bis(5-acetyl-2-benzoyloxy-4-hydroxyphenyl)sulfide in acetic acid [3735,3736].

m.p. 174–175° [3735,3736].

1-(3-Bromo-5-chloro-2-hydroxyphenyl)ethanone

[59443-15-1] $C_8H_6BrClO_2$ mol.wt. 249.49



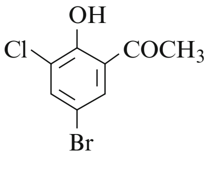
Described [3734] **p. 662**

Syntheses

- Also refer to: [3737–3741].

1-(5-Bromo-3-chloro-2-hydroxyphenyl)ethanone

[331821-10-4] $C_8H_6BrClO_2$ mol.wt. 249.49



New compound

Synthesis

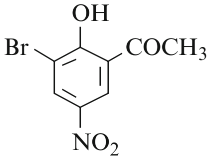
- Refer to: [3742].

^{1}H NMR [3742], IR [3742], UV [3742].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_10, © Springer Science+Business Media B.V. 2011

1-(3-Bromo-2-hydroxy-5-nitrophenyl)ethanone

[90004-97-0] $C_8H_6BrNO_4$ mol.wt. 260.04



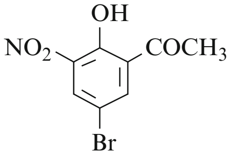
Described [3734] **p. 663**

Synthesis

- Also refer to: [3739].

1-(5-Bromo-2-hydroxy-3-nitrophenyl)ethanone

[70978-54-0] $C_8H_6BrNO_4$ mol.wt. 260.04



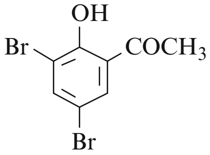
Described [3734] **p. 663**

Syntheses

- Also refer to: [3743,3744].

1-(3,5-Dibromo-2-hydroxyphenyl)ethanone

[22362-66-9] $C_8H_6Br_2O_2$ mol.wt. 293.94



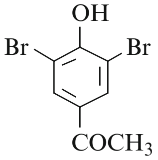
Described [3734] **p. 665**

Syntheses

- Also refer to: [3740,3742].

1-(3,5-Dibromo-4-hydroxyphenyl)ethanone

[2887-72-1] $C_8H_6Br_2O_2$ mol.wt. 293.94



Described [3734] **p. 665**

Syntheses

- Also obtained by adding HMTAB (hexamethylene tetramine–bromine complex) to 4-hydroxyacetophenone in methylene chloride at r.t. for 5 min (44%) [3745].
- Also refer to: [3737,3738,3746–3748].

m.p. 180–183° [3745].

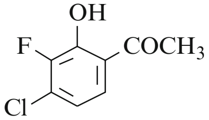
Methyl ether [79324-79-1] $C_9H_8Br_2O_2$ mol.wt. 307.97

- Preparation by reaction of dimethyl sulfate with 3,5-dibromo-4-hydroxyacetophenone in the presence of sodium hydroxide [3749,3750].
- Also refer to: [3751–3758].

m.p. 78° [3750]; ^{1}H NMR [3753,3756], ^{13}C NMR [3751,3754], UV [3757].

1-(4-Chloro-3-fluoro-2-hydroxyphenyl)ethanone

[949900-54-3] $C_8H_6ClFO_2$ mol.wt. 188.59



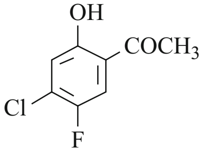
New compound

Syntheses

- Refer to: [3759,3760].

1-(4-Chloro-5-fluoro-2-hydroxyphenyl)ethanone

[105533-69-5] $C_8H_6ClFO_2$ mol.wt. 188.59



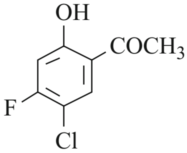
Described [3734] **p. 668**

Syntheses

- Also refer to: [3761,3762].

1-(5-Chloro-4-fluoro-2-hydroxyphenyl)ethanone

[865451-01-0] $C_8H_6ClFO_2$ mol.wt. 188.59



New compound

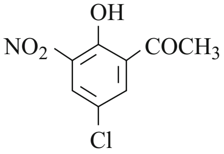
Synthesis

- Obtained by reaction of acetyl chloride with 4-chloro-3-fluorophenol in the presence of aluminium chloride in toluene, first at 40° for 1 h, then at 80° for 3 h (61%) [3762].

^{1}H NMR [3762].

1-(5-Chloro-2-hydroxy-3-nitrophenyl)ethanone

[84942-40-5] $C_8H_6ClNO_4$ mol.wt. 215.59



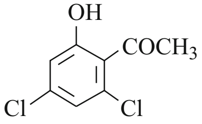
Described [3734] **p. 670**

Syntheses

- Also refer to: [3763,3764].

1-(2,4-Dichloro-6-hydroxyphenyl)ethanone

[57051-50-0] $C_8H_6Cl_2O_2$ mol.wt. 205.04



Described [3734] **p. 671**

Syntheses

- Also obtained by heating its methyl ether with pyridinium chloride for 30 min (70%) [3765].
- Also obtained by Fries rearrangement of 3,5-dichlorophenyl acetate [3766] according to [3767].

Methyl ether [41068-37-5] $C_9H_8Cl_2O_2$ mol.wt. 219.07 [3765]

Benzyl ether [1023279-01-7] $C_{15}H_{12}Cl_2O_2$ mol.wt. 295.16

- Obtained by reaction of benzyl chloride with 2,4-dichloro-6-hydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone for 16 h (92%) [3766].

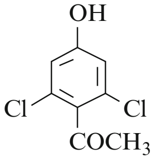
m.p. 87–88° [3766]; ^{1}H NMR [3766], ^{13}C NMR [3766], MS [3766].

2,4-Dichlorobenzoate [1023278-90-1] $C_{15}H_8Cl_4O_3$ mol.wt. 378.04

- Obtained by reaction of 2,4-dichloro-6-hydroxyacetophenone with 2,4-dichlorobenzoic acid in the presence of N,N-dicyclohexylcarbodiimide and 4-pyrrolidinopyridine in methylene chloride under nitrogen at r.t. for 24 h (98%) [3766].

m.p. 112–113° [3766]; ^{1}H NMR [3766], ^{13}C NMR [3766], MS [3766].

1-(2,6-Dichloro-4-hydroxyphenyl)ethanone



$C_8H_6Cl_2O_2$ mol.wt. 205.04

Described [3734] **p. 671**

Synthesis

- Also obtained by reaction of acetyl chloride with 3,5-dichloroanisole in the presence of aluminium chloride in carbon disulfide [3768].

m.p. 117–119° [3768].

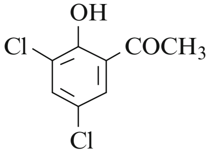
Methyl ether [157487-30-4] $C_9H_8Cl_2O_2$ mol.wt. 219.07

- Obtained by heating a mixture of 1-(2,6-dichloro-4-methoxyphenyl)ethan-1-ol and manganese (IV) oxide in benzene for 15 h (87%) [3769].
- Also refer to: [3770].

b.p.$_{0.6}$ 125–128° [3769]; ^{1}H NMR [3769], IR [3769].

1-(3,5-Dichloro-2-hydroxyphenyl)ethanone

[3321-92-4] $C_8H_6Cl_2O_2$ mol.wt. 205.04



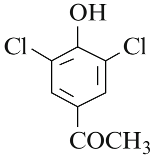
Described [3734] **p. 672**

Syntheses

- Also refer to: [3737,3738,3740,3742,3771–3776].

1-(3,5-Dichloro-4-hydroxyphenyl)ethanone

[17044-70-1] $C_8H_6Cl_2O_2$ mol.wt. 205.04



Described [3734] **p. 672**

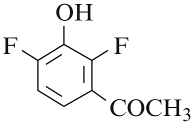
Methyl ether [41727-59-7] $C_9H_8Cl_2O_2$ mol.wt. 219.07

- Obtained by oxychlorination of 4-methoxyacetophenone (2 mol) with ammonium chloride (2.2 mol) and oxone (2.2 mol) in acetonitrile at r.t. for 24 h (14%) [3777].
- Also refer to: [3751–3757,3778–3780].

^{1}H NMR [3753,3756,3777],
^{13}C NMR [3751,3754], IR [3779,3780], UV [3757], MS [3777].

1-(2,4-Difluoro-3-hydroxyphenyl)ethanone

[951163-65-8] $C_8H_6F_2O_2$ mol.wt. 172.13



New compound

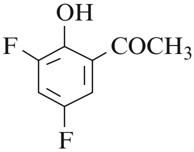
Syntheses

- Refer to: [3781–3786].

Methyl ether [373603-19-1] $C_9H_8F_2O_2$ mol.wt. 186.16 [3782,3784,3785]

1-(3,5-Difluoro-2-hydroxyphenyl)ethanone

[140675-42-9] $C_8H_6F_2O_2$ mol.wt. 172.13



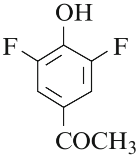
Described [3734] **p. 674**

Syntheses

- Also refer to: [3771,3787].

1-(3,5-Difluoro-4-hydroxyphenyl)ethanone

[133186-55-7] $C_8H_6F_2O_2$ mol.wt. 172.13



Described [3734] **p. 675**

Syntheses

- Also refer to: [3771,3788].

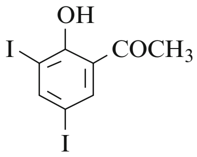
Methyl ether [170570-79-3] $C_9H_8F_2O_2$ mol.wt. 186.16

- Obtained by fluorination of 4-methoxyacetophenone with manganese (IV) tetrafluoride formed in situ using manganese (IV) dioxide and pyridinium polyhydrogenofluoride under very mild conditions [3789].
- Also refer to: [3778,3788].

MS [3789].

1-(2-Hydroxy-3,5-diiodophenyl)ethanone

[7191-46-0] $C_8H_6I_2O_2$ mol.wt. 387.94



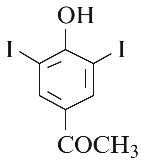
Described [3734] **p. 676**

Syntheses

- Also refer to: [3740,3742,3747].

1-(4-Hydroxy-3,5-diiodophenyl)ethanone

[7191-55-1] $C_8H_6I_2O_2$ mol.wt. 387.94



Described [3734] **p. 676**

Synthesis

- Also refer to: [3747].

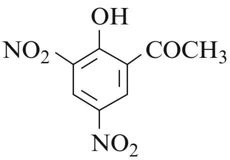
Methyl ether [31827-84-6] $C_9H_8I_2O_2$ mol.wt. 401.97

- Preparation by reaction of methyl iodide with 4-hydroxy-3,5-diiodoacetophenone in the presence of sodium hydroxide [3790].
- Obtained from the potassium salt [3791,3792].
- Also refer to: [3752–3755,3757,3793].

m.p. 107° [3790], 97–98° [3791,3792]; ^{1}H NMR [3753], ^{13}C NMR [3754], UV [3757].

1-(2-Hydroxy-3,5-dinitrophenyl)ethanone

[69027-37-8] $C_8H_6N_2O_6$ mol.wt. 226.15



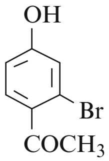
Described [3734] **p. 677**

Synthesis

- Also refer to: [3739].

1-(2-Bromo-4-hydroxyphenyl)ethanone

[61791-99-9] $C_8H_7BrO_2$ mol.wt. 215.05



Described [3734] **p. 679**

Methyl ether [89691-67-8] $C_9H_9BrO_2$ mol.wt. 229.07

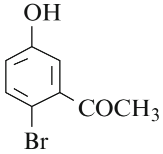
- Obtained by reaction of acetyl chloride with 3-bromoanisole in the presence of aluminium chloride [3794], (59%) [3795].
- Obtained by reaction of acetic anhydride with 3-bromoanisole in the presence of aluminium chloride in carbon disulfide at r.t., then at 30–35° until no more hydrochloric acid is evolved [3796,3797].
- Also obtained by reaction of acetic anhydride with m-bromoanisole in the presence of lithium perchlorate at 100° for 5 h (60%) [3798].
- Also refer to: [3770,3799,3800].

b.p.$_8$ 149–151° [3795], b.p.$_{15}$ 163–166° [3795];
m.p. 69–71° [3800], 23° [3795]. One of the reported melting points is obviously wrong.
^{1}H NMR [3796,3797,3799], ^{13}C NMR [3799], IR [3796,3797,3799].

2,4-Dinitrophenylhydrazone (of the methyl ether) $C_{15}H_{13}BrN_4O_5$ mol.wt. 409.20

m.p. 193–194° [3795].

1-(2-Bromo-5-hydroxyphenyl)ethanone



$C_8H_7BrO_2$ mol.wt. 215.05

New compound

Syntheses

- Refer to: [3801,3802].

Methyl ether [6342-63-8] $C_9H_9BrO_2$ mol.wt. 229.07

- Obtained by action of pyridinium chlorochromate with 1-(2-bromo-5-methoxy) ethanol [3803].
- Also obtained by bromination of 3-methoxyacetophenone with N-bromosuccinimide in dilute sulfuric acid at 60° for 5 h (94%) [3804].
- Also obtained from 3-methoxyacetophenone by reaction of hypobromous acid, generated in situ, in aqueous acetic acid containing perchloric acid as a catalyst at r.t. [3805].
- Also refer to: [3801,3806].

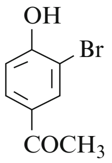
b.p.$_{0.4}$ 102° [3803], b.p.$_{0.7}$ 105° [3801], b.p.$_{1.5}$ 115–125° [3805], b.p.$_{13}$ 157–158° [3807];
^{1}H NMR [3807], IR [3807], UV [3801].

Oxime (of the methyl ether) $C_9H_{10}BrNO_2$ mol.wt. 244.09

m.p. 131–132° [3801], 125–126° [3805].

1-(3-Bromo-4-hydroxyphenyl)ethanone

[1836-06-2] $C_8H_7BrO_2$ mol.wt. 215.05



Described [3734] **p. 680**

Synthesis

- Also obtained by adding HMTAB (hexamethylene tetramine–bromine complex) to 4-hydroxyacetophenone in methylene chloride at r.t. for 5 min (56%) or at −5° for 150 min (88%) [3745].

m.p. 108–110° [3745].

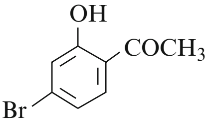
Methyl ether [35310-75-9] $C_9H_9BrO_2$ mol.wt. 229.07

- Obtained by reaction of acetic anhydride with o-bromoanisole in the presence of lithium perchlorate at 100° for 5 h (99%) [3744,3798,3808].

^{13}C NMR [3751,3754].

1-(4-Bromo-2-hydroxyphenyl)ethanone

[30186-18-6] $C_8H_7BrO_2$ mol.wt. 215.05



Described [3734] **p. 680**

Syntheses

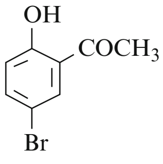
- Also refer to: [3809–3814].

Methyl ether [89368-12-7] $C_9H_9BrO_2$ mol.wt. 229.07

- Obtained by reaction of acetic anhydride with m-bromoanisole in the presence of lithium perchlorate at 100° for 5 h (40%) [3798].
- Also refer to: [3815,3816].

1-(5-Bromo-2-hydroxyphenyl)ethanone

[1450-75-5] $C_8H_7BrO_2$ mol.wt. 215.05



Described [3734] **p. 681**

Syntheses

- Also refer to: [3738,3744,3773,3809,3817–3826].

UV [3801].

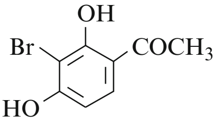
Methyl ether [16740-73-1] $C_9H_9BrO_2$ mol.wt. 229.07

- Preparation by reaction of methyl iodide with 5-bromo-2-hydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 3 h (86%) [3820].
- Obtained from 2-methoxyacetophenone by reaction of hypobromous acid, generated in situ, in aqueous acetic acid containing perchloric acid as a catalyst at r.t. (34%) [3805].
- Also refer to: [3827].

b.p.$_{1.5}$ 120° [3805]; white solid [3820];
^1H NMR [3805,3820], ^{13}C NMR [3820], UV [3801], MS [3820].

1-(3-Bromo-2,4-dihydroxyphenyl)ethanone

[60990-39-8] $C_8H_7BrO_3$ mol.wt. 231.05



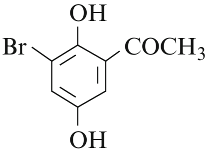
Described [3734] **p. 682**

Synthesis

- Also refer to: [3739].

1-(3-Bromo-2,5-dihydroxyphenyl)ethanone

[33857-20-4] $C_8H_7BrO_3$ mol.wt. 231.05



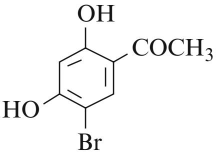
Described [3734] **p. 682**

Synthesis

- Also refer to: [3739].

1-(5-Bromo-2,4-dihydroxyphenyl)ethanone

[60965-25-5] $C_8H_7BrO_3$ mol.wt. 231.05



Described [3734] **p. 683**

Synthesis

- Also refer to: [3828].

Dibenzyl ether [747413-68-9] $C_{22}H_{19}BrO_3$ mol.wt. 411.30

- Obtained by reaction of N-bromosuccinimide with 2,4-Bis(benzyloxy)acetophenone in DMF at r.t. for 3 h (97%) [3829].
  White powder [3829]; ^{1}H NMR [3829], MS [3829].

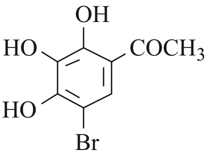
Dimethyl ether [182056-48-0] $C_{10}H_{11}BrO_3$ mol.wt. 259.10

- Obtained by reaction of acetic anhydride with 1-bromo-2,4-dimethoxybenzene in the presence of boron trifluoride etherate in methylene chloride first at 0°, then at r.t. for 24 h (85%) [3830].

  m.p. 149–150° [3830];
  ^{1}H NMR [3830], ^{13}C NMR [3830], MS [3830].

1-(5-Bromo-2,3,4-trihydroxyphenyl)ethanone

[870652-37-2] $C_8H_7BrO_4$ mol.wt. 247.05



Described [3734] **p. 684**

Syntheses

- Also refer to: [3831,3832].

Trimethyl ether [23030-56-0] $C_{11}H_{13}BrO_4$ mol.wt. 289.13

- Obtained by treatment of 5-bromogallacetophenone with dimethyl sulfate in the presence of sodium hydroxide [3833].
- Also obtained by Friedel–Crafts acetylation of 4-bromopyrogallol trimethyl ether with acetyl chloride in the presence of aluminium chloride in carbon disulfide, then treatment of the compound obtained (partly demethylated) with dimethyl sulfate in the presence of sodium hydroxide [3833].
- Also obtained by reaction of N-bromosuccinimide (NBS) with 2,3,4-trimethoxyacetophenone in acetic acid/acetic anhydride mixture under reflux for 1 h (54%) [3834].
- Also refer to: [3835].

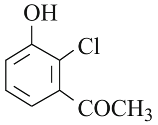
b.p.$_{0.3}$ 112–115° [3834], b.p.$_{0.3}$ 134–135° [3833], b.p.$_{13}$ 174–176° [3835];
m.p. 52.5–53° [3834];
^{1}H NMR [3834], IR [3833].

2,4-Dinitrophenylhydrazone (of the trimethyl ether) $C_{17}H_{17}BrN_4O_7$ mol. wt. 469.25

m.p. 137–139° [3833,3835]; IR [3833,3835].

1-(2-Chloro-3-hydroxyphenyl)ethanone

[69240-96-6] $C_8H_7ClO_2$ mol.wt. 170.60



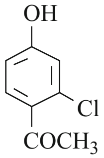
Described [3734] **p. 684**

Syntheses

- Also refer to: [3836,3837].

1-(2-Chloro-4-hydroxyphenyl)ethanone

[68301-59-7] $C_8H_7ClO_2$ mol.wt. 170.60



Described [3734] **p. 684**

Syntheses

- Also obtained by heating its methyl ether with pyridinium chloride for 15 min (36%) [3765].
- Also refer to: [3838].

Methyl ether [41068-36-4] $C_9H_9ClO_2$ mol.wt. 184.62

- Obtained by reaction of acetyl chloride with 3-chloroanisole in the presence of aluminium chloride (55%) [3839], in carbon disulfide (62%) [3795] or in 1,2-dichloroethane (81%) [3840].
- Also obtained by reaction of acetic anhydride with 3-chloroanisole,
  - in the presence of aluminium chloride [3796,3797], (58%) [3841];
  - in the presence of ferric chloride (50%) [3839].
- Also refer to: [3765,3770,3819,3842–3846].

b.p.$_{10}$ 139–141° [3795], b.p.$_{11}$ 142° [3841], b.p.$_{12}$ 151° [3839];
m.p. 26–27° [3795];
^{1}H NMR [3796,3797,3844,3845], ^{13}C NMR [3845], IR [3796,3797,3844].

2,4-Dinitrophenylhydrazone (of the methyl ether) $C_{15}H_{13}ClN_4O_5$ mol.wt. 364.78

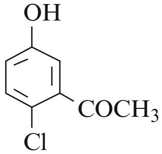
m.p. 181–183° [3795].

Oxime (of the methyl ether) $C_9H_{10}ClNO_2$ mol.wt. 199.64

m.p. 97–98° [3770].

1-(2-Chloro-5-hydroxyphenyl)ethanone

[58020-38-5] $C_8H_7ClO_2$ mol.wt. 170.60



Described [3734] **p. 684**

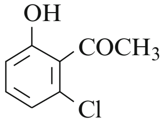
Methyl ether [77344-69-5] $C_9H_9ClO_2$ mol.wt. 184.52

- Obtained by reaction of dimethylcadmium with 2-chloro-5-methoxybenzoyl chloride [3847].
- Also obtained by reduction of 1-(2-chloro-5-methoxyphenyl)-2-diazoethanone with hydrogen iodide [3847].
- Also refer to: [3801,3848–3850].

b.p.$_3$ 116° [3801], b.p.$_5$ 122° [3847];
UV [3801,3850].

1-(2-Chloro-6-hydroxyphenyl)ethanone

[55736-04-4] $C_8H_7ClO_2$ mol.wt. 170.60



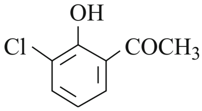
Described [3734] **p. 685**

Synthesis

- Also refer to: [3851].

1-(3-Chloro-2-hydroxyphenyl)ethanone

[3226-34-4] $C_8H_7ClO_2$ mol.wt. 170.60



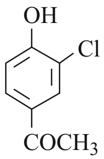
Described [3734] **p. 685**

Syntheses

- Also refer to: [3852,3853].

1-(3-Chloro-4-hydroxyphenyl)ethanone

[2892-29-7] $C_8H_7ClO_2$ mol.wt. 170.60



Described [3734] **p. 685**

Syntheses

- Also obtained by heating its methyl ether with pyridinium chloride for 30 min (54%) [3765].
- Also refer to: [3852,3854].

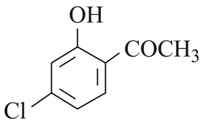
Methyl ether [37612-52-5] $C_9H_9ClO_2$ mol.wt. 184.62

- Obtained by oxychlorination of 4-methoxyacetophenone (2 mol) with ammonium chloride (2 mol) and oxone (2 mol) in acetonitrile at r.t. for 24 h (68%) [3777].
- Also obtained by heating for 4 h a mixture of 2-chloroanisole,
  - and acetic anhydride in the presence of ferric chloride (40%) [3839];
  - and acetyl chloride in the presence of aluminium chloride (52%) [3839].
- Also refer to: [3744,3765,3808,3855].

b.p.$_{12}$ 148° [3839]; ^{1}H NMR [3777], ^{13}C NMR [3751,3754], MS [3777].

1-(4-Chloro-2-hydroxyphenyl)ethanone

[6921-66-0] $C_8H_7ClO_2$ mol.wt. 170.60



Described [3734] **p. 686**

Syntheses

- Also obtained by Fries rearrangement of 3-chlorophenyl acetate [3766] according to [3767].
- Also refer to: [3762,3766,3856,3857].

Methyl ether [60207-19-4] $C_9H_9ClO_2$ mol.wt. 184.62

- Refer to: [3858,3859]; ^{13}C NMR [3858].

Benzyl ether $C_{15}H_{13}ClO_2$ mol.wt. 260.72

- Obtained by reaction of benzyl chloride with 4-chloro-2-hydroxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone for 16 h (59%) [3766].

m.p. 58–59° [3766]; ^{1}H NMR [3766], ^{13}C NMR [3766], MS [3766].

2,4-Dichlorobenzoate [1023278-89-8] $C_{15}H_9Cl_3O_3$ mol.wt. 343.59

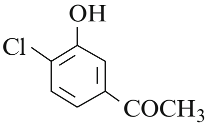
- Obtained by reaction of 4-chloro-2-hydroxyacetophenone with 2,4-dichlorobenzoic acid in the presence of N,N-dicyclohexylcarbodiimide and 4-pyrrolidinopyridine in methylene chloride under nitrogen at r.t. for 24 h (81%) [3766].

m.p. 81–82° [3766]; ^{1}H NMR [3766], ^{13}C NMR [3766], MS [3766].

Oxime [56484-63-0] $C_8H_8ClNO_2$ mol.wt. 185.62 [3856].

1-(4-Chloro-3-hydroxyphenyl)ethanone

[61124-56-9] $C_8H_7ClO_2$ mol.wt. 170.60

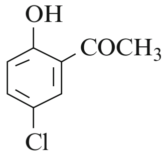


Described [3734] **p. 687**

Methyl ether [78898-63-2] $C_9H_9ClO_2$ mol.wt. 184.62 [3860]

1-(5-Chloro-2-hydroxyphenyl)ethanone

[1450-74-4] $C_8H_7ClO_2$ mol.wt. 170.60



Described [3734] **p. 687**

Syntheses

- Also refer to: [3738,3761,3762,3771,3773,3774,3817,3823,3861], UV [3801].

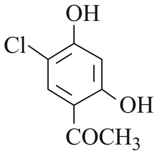
Methyl ether [6342-64-9] $C_9H_9ClO_2$ mol.wt. 184.62 [3862]

- Obtained by reaction of methylmagnesium bromide with 5-chloro-2-methoxybenzoyl chloride in the presence of bis[2-(N,N-dimethylamino)ethyl] ether in THF at –5° to 0° (69%) [3863].
- Also obtained by heating for 4 h a mixture of acetic anhydride and 4-chloroanisole in the presence of ferric chloride (40%) [3839].
- Also obtained by heating for 4 h a mixture of acetyl chloride and 4-chloroanisole in the presence of aluminium chloride (52%) [3839].
- Also obtained by reaction of methyl iodide with 5-chloro-2-hydroxyacetophenone in the presence of potassium carbonate in acetone at r.t. for 15 h under nitrogen (97%) [3864].
- Also refer to: [3801,3858,3864–3868].

b.p.$_2$ 108° [3867], b.p.$_6$ 135° [3865], b.p.$_{12}$ 149° [3839], b.p.$_{25}$ 162° [3801];
m.p. 30° [3839], 29–30° [3868];
^{1}H NMR [3864], ^{13}C NMR [3858,3864], IR [3866], UV [3801], MS [3864].

1-(5-Chloro-2,4-dihydroxyphenyl)ethanone

[90110-32-0] $C_8H_7ClO_3$ mol.wt. 186.59



Described [3734] **p. 690**

Syntheses

- Also obtained by reaction of acetic acid with 4-chlororesorcinol in the presence of boron trifluoride etherate at 90° for 3.5 h under nitrogen (58%) [3829].
- Also refer to: [3869].

^{1}H NMR [3829], ^{13}C NMR [3829], MS [3829]; TLC [3829].

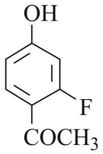
Dibenzyl ether [705963-54-8] $C_{22}H_{19}ClO_3$ mol.wt. 366.84

- Obtained by reaction of benzyl bromide with 5-chloro-2,4-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetonitrile for 6 h (90%) [3829].
- Also refer to: [3870–3874].

Off-white solid [3829];
^{1}H NMR [3829], MS [3829]; TLC [3829].

1-(2-Fluoro-4-hydroxyphenyl)ethanone

[98619-07-9] $C_8H_7FO_2$ mol.wt. 154.14



Described [3734] **p. 691**

Syntheses

- Also refer to: [3788,3862,3875,3876].

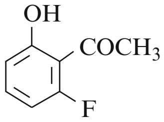
Methyl ether [74457-86-6] $C_9H_9FO_2$ mol.wt. 168.17

- Obtained by reaction of acetic anhydride with 3-fluoroanisole in the presence of aluminium chloride in carbon disulfide at r.t., then at 30–35° until no more hydrochloric acid is evolved [3797], (61%) [3796].
- Also obtained by reaction of acetyl chloride with 3-fluoroanisole in the presence of aluminium chloride in 1,2-dichloroethane at 0–5°, then at r.t. for 2 h [3840].
- Also obtained by reaction of acetic anhydride with m-fluoroanisole in the presence of lithium perchlorate at 100° for 1 h (70%) [3798].
- Also refer to: [3770,3862,3877–3881].

Colourless crystals [3796]; m.p. 53–54° [3796];
^{1}H NMR [3796,3797], IR [3796,3797].

1-(2-Fluoro-6-hydroxyphenyl)ethanone

[93339-98-1] $C_8H_7FO_2$ mol.wt. 154.14



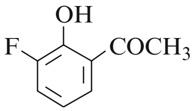
Described [3734] **p. 691**

Syntheses

- Also refer to: [3787,3851,3882–3884].

1-(3-Fluoro-2-hydroxyphenyl)ethanone

[699-92-3] $C_8H_7FO_2$ mol.wt. 154.14



Described [3734] **p. 691**

Synthesis

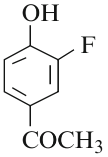
- Also refer to: [3851].

Methyl ether [295779-86-1] $C_9H_9FO_2$ mol.wt. 168.17

- Obtained by reaction of 1-fluoro-4-hydroxy-1,4-diazoniabicyclo[2.2.2]octane bis(tetrafluoro-borate) with 2-methoxyacetophenone in refluxing acetonitrile for 0.5–4 h [3885].

1-(3-Fluoro-4-hydroxyphenyl)ethanone

[403-14-5] $C_8H_7FO_2$ mol.wt. 154.14



Described [3734] **p. 692**

Synthesis

- Also obtained by reaction of 1-fluoro-4-hydroxy-1,4-diazoniabicyclo[2.2.2]octane bis(tetrafluoroborate) with 4-hydroxyacetophenone in refluxing acetonitrile for 0.5–4 h [3885].

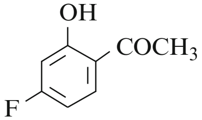
Methyl ether [455-91-4] $C_9H_9FO_2$ mol.wt. 168.17

- Obtained by fluorination of 4-methoxyacetophenone with manganese (IV) tetrafluoride formed in situ using manganese (IV) dioxide and pyridinium polyhydrogenofluoride under very mild conditions [3789].
- Also obtained by reaction of 1-fluoro-4-hydroxy-1,4-diazoniabicyclo[2.2.2] octane bis(tetrafluoroborate) with 4-methoxyacetophenone in refluxing acetonitrile for 0.5–4 h [3885].
- Also refer to: [3744,3886–3888].

^{13}C NMR [3754], MS [3789].

1-(4-Fluoro-2-hydroxyphenyl)ethanone

[1481-27-2] $C_8H_7FO_2$ mol.wt. 154.14



Described [3734] **p. 692**

Syntheses

- Also obtained by Friedel–Crafts acylation of m-fluorophenol with acetic acid in a mixture of graphite and methanesulfonic acid at 120° for 4 h (60%) [3889].
- Also obtained by Fries rearrangement of 3-fluorophenyl acetate in the presence of aluminium chloride (88%) [3890], at 140° for 2 h (48%) [3891].
- Also refer to: [3741,3892–3897].

^{1}H NMR [3889,3891,3898], ^{13}C NMR [3889], ^{19}F NMR [3899], IR [3889,3891,3900].

BIOLOGICAL DATA: Cytotoxicity [3891]; anticoagulant [3891]; antithrombotic [3901].

Methyl ether [51788-80-8] $C_9H_9FO_2$ mol.wt. 168.17

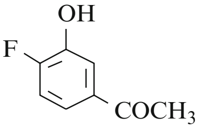
- Preparation by reaction of methyl iodide with 4-fluoro-2-hydroxyacetophenone in the presence of potassium carbonate,
  - in DMSO at r.t. for 1 h (100%) [3902];
  - in acetone (97%) [3890].

- Preparation by reaction of methyl iodide on the Na salt of 4-fluoro-2-hydroxyacetophenone obtained by treatment with sodium hydride in DMF at 0° for 1 h (92%) [3903].
- Preparation by reaction of dimethyl sulfate with 4-fluoro-2-hydroxyacetophenone in the presence of aqueous sodium hydroxide [3899].
- Also obtained by reaction of acetic anhydride with m-fluoroanisole in the presence of lithium perchlorate at 100° for 1 h (30%) [3798].
- Also refer to: [3904].

White solid; m.p. 50–51.5° [3902];
^{1}H NMR [3902], ^{19}F NMR [3899], MS [3902].

1-(4-Fluoro-3-hydroxyphenyl)ethanone

[949159-95-9] $C_8H_7FO_2$ mol.wt. 154.14



New compound

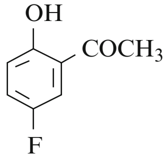
Synthesis

- Refer to: [3905].

Methyl ether [64287-19-0] $C_9H_9FO_2$ mol.wt. 168.17 [3905,3906]

1-(5-Fluoro-2-hydroxyphenyl)ethanone

[394-32-1] $C_8H_7FO_2$ mol.wt. 154.14



Described [3734] **p. 692**

Syntheses

- Also obtained by reaction of acetyl chloride with 4-fluorophenol in the presence of aluminium chloride, first at r.t. for 30 min, then at 130° for 2 h (89%) [3907].
- Also refer to: [3741,3773,3861,3908–3910].

m.p. 53–54° [3907]; UV [3801].

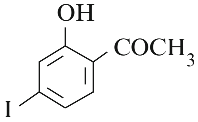
Methyl ether [455-82-9] $C_9H_9FO_2$ mol.wt. 168.17

- Also obtained by reaction of 1-fluoro-4-hydroxy-1,4-diazoniabicyclo[2.2.2]octane bis(tetra-fluoroborate) with 2-methoxyacetophenone in refluxing acetonitrile for 0.5–4 h [3885].

UV [3801].

1-(2-Hydroxy-4-iodophenyl)ethanone

[39730-66-0] $C_8H_7IO_2$ mol.wt. 262.05



Described [3734] **p. 694**

Syntheses

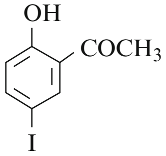
- Also obtained by Fries rearrangement of 3-iodophenyl acetate (m.p. 32.4–34.6°) with aluminium chloride in chlorobenzene at 140° for 90 h (69%) [3911].

- Also refer to: [3912].

brown solid [3911]; m.p. 51.5–52° [3911];
^{1}H NMR [3911], ^{13}C NMR [3911], IR [3911], MS [3911].

1-(2-Hydroxy-5-iodophenyl)ethanone

[7191-41-5] $C_8H_7IO_2$ mol.wt. 262.05



Described [3734] **p. 694**

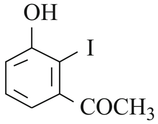
Synthesis

- Also obtained by diazotization of 5-amino-2-hydroxyacetophenone and replacement of the diazonium salt by iodine (39%) [3911].

Purple oil [3911].

1-(3-Hydroxy-2-iodophenyl)ethanone

[348616-32-0] $C_8H_7IO_2$ mol.wt. 262.05



Described [3734] **p. 694**

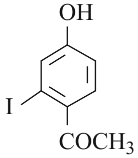
Methyl ether [110718-83-7] $C_9H_9IO_2$ mol.wt. 276.07

- Refer to: [3913–3916], (61%) [3917].

Pale yellow oil [3917];
^{1}H NMR [3914,3917], ^{13}C NMR [3914,3917], IR [3917], MS [3917].

1-(4-Hydroxy-2-iodophenyl)ethanone

[89942-32-5] $C_8H_7IO_2$ mol.wt. 262.05



Described [3734] **p. 695**

Synthesis

- Obtained by Fries rearrangement of 3-iodophenyl acetate in the presence of aluminium chloride in nitrobenzene [3918].

m.p. 128–136° [3918].

Methyl ether [90347-63-0] $C_9H_9IO_2$ mol.wt. 276.07

- Obtained by reaction of acetyl chloride with 3-iodoanisole in the presence of aluminium chloride in carbon disulfide [3919], (55%) [3795].
- Also refer to: [3807,3915], (37%) [3917].

b.p.$_{0.01}$ 107–109° [3807], b.p.$_3$ 139–140° [3919], b.p.$_6$ 158–159° [3795], b.p.$_{9.5}$ 168–170° [3795];

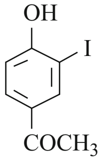
m.p. 68–70° [3795], 45–47° [3919]. One of the reported melting points is obviously wrong.
^{1}H NMR [3807,3917], ^{13}C NMR [3917], IR [3807,3917], MS [3917].

2,4-Dinitrophenylhydrazone (of the methyl ether) $C_{15}H_{13}IN_4O_5$ mol.wt. 456.20

m.p. 137–140° [3919], 108.5–109° [3795].

1-(4-Hydroxy-3-iodophenyl)ethanone

[62615-24-1] $C_8H_7IO_2$ mol.wt. 262.05



Described [3734] **p. 695**

Syntheses

- Also obtained by adding iodine to a solution of 4-hydroxyacetophenone in dilute THF in the presence of sodium bicarbonate and stirring at r.t. for 3 h (47%) [3920].
- Also obtained by adding rapidly a solution of potassium iodide and iodine in water to a solution of 4-hydroxyacetophenone in concentrated ammonium hydroxide. After overnight stirring at r.t., the mixture was filtered through Celite and the filtrate acidified to pH 1 (56%) [3921].
- Also refer to: [3922,3923].

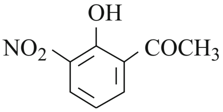
m.p. 154–156° [3921], 152–154° [3920];
^{1}H NMR [3920], MS [3920].

Methyl ether [79324-77-9] $C_9H_9IO_2$ mol.wt. 276.07 [3924]

^{13}C NMR [3754].

1-(2-Hydroxy-3-nitrophenyl)ethanone

[28177-69-7] $C_8H_7NO_4$ mol.wt. 181.15



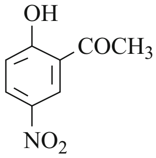
Described [3734] **p. 697**

Synthesis

- Also refer to: [3744].

1-(2-Hydroxy-5-nitrophenyl)ethanone

[1450-76-6] $C_8H_7NO_4$ mol.wt. 181.15



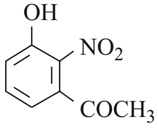
Described [3734] **p. 698**

Syntheses

- Also refer to: [3892,3908].

1-(3-Hydroxy-2-nitrophenyl)ethanone

[53967-72-9] $C_8H_7NO_4$ mol.wt. 181.15



Described [3734] **p. 698**

Synthesis

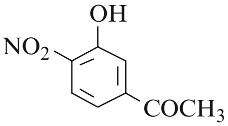
- Obtained by nitration of 3-hydroxyacetophenone with cupric nitrate in acetic acid–acetic anhydride mixture, at 10–15° for 7–8 h (25%) [3925].

Yellow solid; m.p. 134–136° [3925];
^{1}H NMR [3925], ^{13}C NMR [3925], IR [3925].

Methyl ether [33852-43-6] $C_9H_9NO_4$ mol.wt. 195.17 [3926]

1-(3-Hydroxy-4-nitrophenyl)ethanone

[89942-63-2] $C_8H_7NO_4$ mol.wt. 181.15



Described [3734] **p. 699**

Synthesis

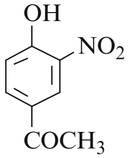
- Also refer to: [3927].

Methyl ether [22106-39-4] $C_9H_9NO_4$ mol.wt. 195.17

- Refer to: [3928].

1-(4-Hydroxy-3-nitrophenyl)ethanone

[6322-56-1] $C_8H_7NO_4$ mol.wt. 181.15



Described [3734] **p. 699**

Syntheses

- Also obtained by nitration of 4-hydroxyacetophenone with zirconyl nitrate in acetone at 50° for 5 h (84%) [3929].
- Also refer to: [3930–3932].

Methyl ether [6277-38-9] $C_9H_9NO_4$ mol.wt. 195.17

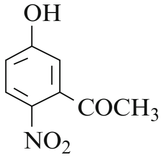
- Preparation by reaction of acetyl chloride with 2-nitroanisole in the presence of aluminium chloride,
  - in nitrobenzene at 0° [3933,3934];
  - in carbon disulfide [3935].
- Also obtained by reaction of dimethyl sulfate with 4-hydroxy-3-nitroacetophenone in the presence of lithium hydroxide hydrate in THF at 70° for 1.5 h (72%) [3936].
- Also obtained by treatment of 4-chloro-3-nitroacetophenone with sodium methoxide in methanol [3937].
- Also obtained by reaction of diazomethane with 4-hydroxy-3-nitroacetophenone in ethyl ether [3938].

- Also obtained by treatment of 4-methoxyacetophenone,
  - with nitric acid in sulfuric acid at 0° [3939];
  - with lithium nitrate in the presence of scandium triflate in acetic anhydride and acetonitrile mixture at 20° for 5 h (67%) [3940].
- Also refer to: [3754,3941–3947], (14%) [3948].

m.p. 99.5° [3935], 98° [3943,3945], 97–99° [3937], 92–94° [3940].
^{1}H NMR [3940], ^{13}C NMR [3754,3938], IR [3940,3942], UV [3757].

1-(5-Hydroxy-2-nitrophenyl)ethanone

[30879-49-3] $C_8H_7NO_4$ mol.wt. 181.15



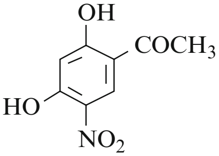
Described [3734] **p. 700**

Methyl ether [42887-67-2] $C_9H_9NO_4$ mol.wt. 195.17

- Refer to: [3949].

1-(2,4-Dihydroxy-5-nitrophenyl)ethanone

[3328-77-6] $C_8H_7NO_5$ mol.wt. 197.15



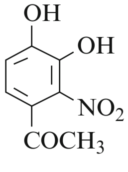
Described [3734] **p. 701**

Synthesis

- Also obtained by nitration of resacetophenone with zirconyl nitrate in acetone at 50° for 5 h (98%) [3929].

1-(3,4-Dihydroxy-2-nitrophenyl)ethanone

[383382-42-1] $C_8H_7NO_5$ mol.wt. 197.15



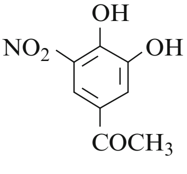
New compound

Synthesis

- Obtained from 4-hydroxy-3-methoxy-2-nitroacetophenone by ether cleavage with aluminium chloride in a pyridine/1, 2-dichloroethane mixture [3950].

1-(3,4-Dihydroxy-5-nitrophenyl)ethanone

[116313-84-9] $C_8H_7NO_5$ mol.wt. 197.15



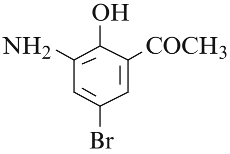
Described [3734] **p. 703**

Synthesis

- Also refer to: [3951].

1-(3-Amino-5-bromo-2-hydroxyphenyl)ethanone

[70977-85-4] $C_8H_8BrNO_2$ mol.wt. 230.06



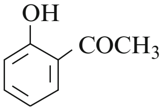
Described [3734] **p. 703**

Synthesis

- Also refer to: [3952].

1-(2-Hydroxyphenyl)ethanone

[118-93-4] $C_8H_8O_2$ mol.wt. 136.15



Described [3734] **p. 706**

Syntheses

- Also obtained by heating its methyl ether with pyridinium chloride for 15 min (52%) [3765].
- Also obtained by reaction of acetyl chloride with phenol in the presence of aluminium chloride at 120° [3953].

Oil [3953]; IR [3954], UV [3801,3955,3956].

Methyl ether [579-74-8] $C_9H_{10}O_2$ mol.wt. 150.18

- Preparation by reaction of dimethyl sulfate with o-hydroxyacetophenone in the presence of sodium hydroxide [3953].
- Also refer to: [3859,3957], (2%) [3765,3917].

Oil; b.p.$_{10}$ 117–119° [3953].
^{1}H NMR [3917], ^{13}C NMR [3858,3917], IR [3917,3954], UV [3801,3955,3956].

Oxime (of the methyl ether) [22233-79-0] $C_9H_{11}NO_2$ mol.wt. 165.19 [3958]

Acetate $C_{10}H_{10}O_3$ mol.wt. 178.19

- Preparation by reaction of acetic anhydride with o-hydroxyacetophenone [3953].

m.p. 89° [3953]; IR [3954].

Benzoate $C_{15}H_{12}O_3$ mol.wt. 240.26

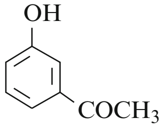
- Obtained by reaction of benzoyl chloride with o-hydroxyacetophenone in suspension of 2 N sodium hydroxide solution at r.t. for 20 min (80%) [3959].

m.p. 87–88° [3959]; IR [3954].

Benzyl ether [31165-67-0] $C_{15}H_{14}O_2$ mol.wt. 226.27 [3960]

1-(3-Hydroxyphenyl)ethanone

[121-71-1] $C_8H_8O_2$ mol.wt. 136.15



Described [3734] **p. 709**

Syntheses

- Obtained by bioconversion of acetophenone through the living cells of *Escherichia coli* carrying plasmid pKF6256 expressing *todCI-bphA2A3A4* (6.1%) [3961].
- Also obtained by heating its methyl ether with pyridinium chloride for 30 min (19%) [3765].
- Also obtained by diazotization of 3-aminoacetophenone, followed by hydrolysis of the diazonium salt obtained [3953].
- Also refer to: [3957,3962].

m.p. 96° [3953];
^{1}H NMR [3961], ^{13}C NMR [3961], UV [3801,3956,3963], MS [3961].

Methyl ether [586-37-8] $C_9H_{10}O_2$ mol.wt. 150.18

- Refer to: [3808,3957], (84%) [3765,3917].
  Colourless oil [3917];
  ^{1}H NMR [3917], ^{13}C NMR [3917], IR [3917], UV [3801,3955,3956].

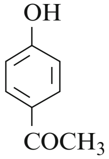
Oxime (of the methyl ether) [122806-25-1] $C_9H_{11}NO_2$ mol.wt. 165.19 [3958]

Acetate $C_{10}H_{10}O_3$ mol.wt. 178.19

- Obtained by reaction of acetic anhydride with 3-hydroxyacetophenone in the presence of pyridine [3953].

1-(4-Hydroxyphenyl)ethanone

[99-93-4] $C_8H_8O_2$ mol.wt. 136.15



Described [3734] **p. 710**

Syntheses

- Also obtained by heating its methyl ether with pyridinium chloride for 60 min (54%) [3765].
- Also obtained by reaction of acetyl chloride with phenol in the presence of aluminium chloride at 120° [3953].
- Also refer to: [3954,3957,3964].

m.p. 107° [3953]; IR [3954], UV [3801,3956].

Methyl ether [100-06-1] $C_9H_{10}O_2$ mol.wt. 150.18

- Preparation by reaction of acetic acid with anisole in the presence of trifluoromethyl sulfonic anhydride (Tf_2O) for <1 min at r.t. (91%) [3965].
- Also obtained by reaction of acetic anhydride with anisole in the presence of aluminium chloride in carbon disulfide (94–96%) [3966], at r.t., then at 30–35° until no more hydrochloric acid [3796,3797].

- Preparation by reaction of dimethyl sulfate with p-hydroxyacetophenone in the presence of sodium hydroxide [3953].
- Also refer to: [3808,3957,3967,3968], (37%) [3765,3917].

b.p.$_{15}$ 139° [3966]; m.p. 36–37.5° [3966], 35° [3953];
^{1}H NMR [3796,3797,3917], ^{13}C NMR [3858,3917], IR [3796,3797,3917,3954], UV [3801,3956].

Oxime (of the methyl ether) [2475-92-5] $C_9H_{11}NO_2$ mol.wt. 165.19 [3958,3969]

Acetate [13031-43-1] $C_{10}H_{10}O_3$ mol.wt. 178.19

- Preparation by acetylation of 4-hydroxyacetophenone with acetic anhydride,
  - in the presence of aluminium triflate for 10 min at r.t. (98%) [3964];
  - in the presence of sodium acetate [3953].
- Also refer to: [3954,3962].

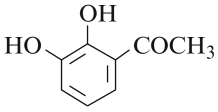
b.p.$_{19}$ 165–170° [3970];
m.p. 54° [3953], 52–54° [3971], 52° [3972];
^{1}H NMR [3973,3974], ^{13}C NMR [3973], IR [3954,3973], UV [3975], MS [3976].
Ecotoxicology: [3977].

Benzyl ether [54696-05-8] $C_{15}H_{14}O_2$ mol.wt. 226.27 [3960]

- Obtained by reaction of benzyl chloride with 4-hydroxyacetophenone in the presence of KOH in acetonitrile. Then, the mixture was refluxed for 3 h [3978].
- Also refer to: [3960].

1-(2,3-Dihydroxyphenyl)ethanone

[13494-10-5] $C_8H_8O_3$ mol.wt. 152.15



Described [3734] **p. 713**

Syntheses

- Also obtained by bioconversion of acetophenone through the living cells of *Escherichia coli* carrying plasmid pUC6256B expressing *todCI-bphA2A3A4* and *bphB* (39.2%) [3961].
- Also refer to: [3895].

^{1}H NMR [3961], ^{13}C NMR [3961], UV [3956], MS [3961].

Diacetate $C_{12}H_{12}O_5$ mol.wt. 236.22

- Obtained by heating 2,3-dihydroxyacetophenone with acetic anhydride and sodium acetate at reflux for 5 min (50%) [3979].

m.p. 109° [3979].

Dimethyl ether [38480-94-3] $C_{10}H_{12}O_3$ mol.wt. 180.20

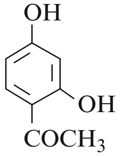
- Obtained by heating its oxime with pyridinium chloride containing 5% of water (35%) [3980].
- Also obtained by reaction of methylmagnesium iodide with 2,3-dimethoxybenzonitrile [3981], (57%) [3982].
- Also obtained from 2,3-dimethoxybenzaldehyde (multi-step reaction) [3983].
- Also obtained from diethyl malonate and 2,3-dimethoxybenzoyl chloride [3984].
- Also obtained by treatment of ethyl 3-(2,3-dimethoxyphenyl)-3-oxopropionate with sulfuric acid [3985].
- Also obtained by oxidation of 1-hydroxy-1-(2,3-dimethoxyphenyl)ethane with Jones' reagent in acetone (61%) [3986].
- Also obtained from acetyl chloride and veratrole by the procedure [3987], (71%) [3988].
- Also obtained by adding an acidic dichromate solution to 2,3-dimethoxyphenylmethylcarbinol in 67% aqueous acetone below 10° (65%) [3989].
- Also refer to: [3990,3991].

b.p.$_{14}$ 146–147° [3955], b.p.$_{20}$ 150° [3980], b.p.$_{20}$ 150–153° [3984];
^{1}H NMR [3982,3983], IR [3982,3986], UV [3955,3956,3963].

Oxime [870-70-1] $C_8H_9NO_3$ mol.wt. 167.16 [3737]

1-(2,4-Dihydroxyphenyl)ethanone

[89-84-9] $C_8H_8O_3$ mol.wt. 152.15



Described [3734] **p. 714**

Syntheses

- Also obtained by heating its dimethyl ether with pyridinium chloride for 30 min (80%) [3765].
- Also refer to: [3954].

IR [3954], UV [3956,3963].

Dimethyl ether [829-20-9] $C_{10}H_{12}O_3$ mol.wt. 180.20

- Obtained by reaction of dimethyl sulfate with resacetophenone in the presence of 10% aqueous sodium hydroxide (61%) [3953].
- Also obtained by reaction of acetic anhydride with resorcinol dimethyl ether in the presence of aluminium chloride in carbon disulfide (77–80%) [3966], at r.t., then at 30–35° until no more hydrochloric acid [3796,3797].
- Also refer to: [3765,3859,3962,3992–3995].

b.p.$_7$ 157.5° [3966];
m.p. 40° [3953], 39–40° [3966], 37–41.5° [3984];
^{1}H NMR [3796,3797], ^{13}C NMR [3858], IR [3796,3797,3954], UV [3801,3956,3963].

Diacetate $C_{12}H_{12}O_5$ mol.wt. 236.22 IR [3954].

Dibenzyl ether [22877-01-6] $C_{22}H_{20}O_3$ mol.wt. 332.40

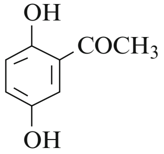
- Obtained by reaction of benzyl bromide with resacetophenone in the presence of potassium carbonate in refluxing acetonitrile for 18 h (97%) [3829].
- Also refer to: [3870–3874].

White powder [3829]; ^{1}H NMR [3829], MS [3829].

Oxime [6134-79-8] $C_8H_9NO_3$ mol.wt. 167.16 [3737]

1-(2,5-Dihydroxyphenyl)ethanone

[490-78-8] $C_8H_8O_3$ mol.wt. 152.15



Described [3734] **p. 716**

Syntheses

- Also obtained by action of aluminium chloride with a mixture of hydroquinone dimethyl ether and 3,3-dimethylacryloyl chloride in boiling benzene for 3 h [3996].
- Also obtained by treatment of 2′,5′-dihydroxy-3,3-dimethylacrylophenone with aluminium chloride in boiling benzene for 2 h (56%) [3996].
- Also obtained by heating its methyl ether with pyridinium chloride for 30 min (96%) [3765].
- Also obtained by reaction of acetic acid with hydroquinone in the presence of zinc chloride at 140–150° [3953].
- Also refer to: [3895,3997–3999].

m.p. 202–204° [3996]; UV [3801,3956,3963].

Dibenzoate $C_{22}H_{16}O_5$ mol.wt. 360.37

m.p. 114° [3996].

Dimethyl ether [1201-38-3] $C_{10}H_{12}O_3$ mol.wt. 180.20

- Obtained by reaction of dimethyl sulfate with quinacetophenone in the presence of 10% aqueous sodium hydroxide [3953].
- Also refer to: [3765,3993,3995,4000–4002].

b.p.$_8$ 149–154° [3984], b.p.$_{13}$ 159–162° [3953];
UV [3955,3956].

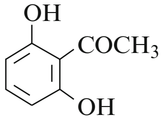
Oxime [24558-42-7] $C_8H_9NO_3$ mol.wt. 167.16

- Refer to: [3737,3996].

m.p. 152° [3996].

1-(2,6-Dihydroxyphenyl)ethanone

[699-83-2] $C_8H_8O_3$ mol.wt. 152.15



Described [3734] **p. 718**

Syntheses

– Also refer to: [3822,3931,3997,4003–4013].

UV [3956,3963].

Dimethyl ether [2040-04-2] $C_{10}H_{12}O_3$ mol.wt. 180.20 [3992,3993]

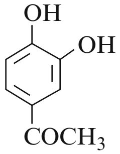
UV [3956,3963].

Dibenzyl ether $C_{22}H_{20}O_3$ mol.wt. 332.40

- Preparation by reaction of benzyl bromide with 2,6-dihydroxyacetophenone in the presence of potassium carbonate and sodium iodide in refluxing acetone for 18 h (46%) [4006].

1-(3,4-Dihydroxyphenyl)ethanone

[1197-09-7] $C_8H_8O_3$ mol.wt. 152.15



Described [3734] **p. 718**

Synthesis

– Also refer to: [3738]; UV [3956].

Dimethyl ether [1131-62-0] $C_{10}H_{12}O_3$ mol.wt. 180.20
(Acetoveratrone)

- Obtained by reaction of dimethyl sulfate with acetoguaiacone [4014] in the presence of sodium hydroxide in ethanol, first at 50°, then at reflux for 1 h (78%) [4015].
- Using tungstophosphoric acid supported over zirconia in mesoporous channels of MCM-41 as catalyst in veratrole acetylation [4016].
- Preparation by reaction of acetic acid with veratrole in the presence of trifluoromethyl sulfonic anhydride (Tf_2O) for 3 min at r.t. (95%) [3965].
- Also refer to: [3738,3808,3967,4017–4024].

Isolation from natural sources

- Recovery of residual plant components after distillation of essential oils *(Mentha longifolia)* [4025].
- From the fresh Iris rhizomes (0.47%) [4026].

b.p.$_{2\text{-}3}$ 135–136° [4014], b.p.$_{7.5}$ 160–164° [3984];
m.p. 50–51° [4015]; ^{1}H NMR [4014], UV [3801,3956,4015].

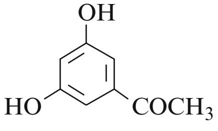
Diacetate $C_{12}H_{12}O_5$ mol.wt. 236.22

- Obtained by reaction of acetic anhydride with pyrocatechol (40%) [4027].

m.p. 87.8–88.2° [4027].

1-(3,5-Dihydroxyphenyl)ethanone

[51863-60-6] $C_8H_8O_3$ mol.wt. 152.15



Described [3734] **p. 720**

Syntheses

- Also refer to: [3738,3964,3991,4011,4028–4032].

UV [3956].

Hydrate (1:1) [957864-27-6] $C_8H_8O_3, H_2O$ mol.wt. 170.17

- Refer to: [4033].

Diacetate [35086-59-0] $C_{12}H_{12}O_5$ mol.wt. 236.22

- Preparation by acetylation of 3,5-dihydroxyacetophenone with acetic anhydride in the presence of aluminium triflate for 20 min at r.t. (96%) [3964].
- Also obtained by reaction of dimethylcadmium with 3,5-diacetoxybenzoyl chloride in benzene [4034].
- Also refer to: [3954,4035–4038].

b.p.$_{0.6}$ 165–168° [4034]; m.p. 93° [4034], 91–92° [4038].

^{14}C-3,5-(Diacetoxyphenyl)ethanone [62492-84-6] $C_{11}{}^{(14)}CH_{12}O_5$ mol.wt. 238.21

- Obtained by reaction of acetyl chloride with ^{14}C-3,5-dihydroxyacetophenone [4039].

m.p. 91–93° [4039].

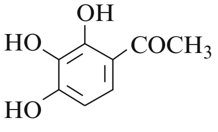
Dimethyl ether [39151-19-4] $C_{10}H_{12}O_3$ mol.wt. 180.20

- Obtained by reaction of acetyl chloride with 3,5-dimethoxybenzene according to [3987], (84%) [3988].
- Also refer to: [3991,4040,4041].

UV [3956].

1-(2,3,4-Trihydroxyphenyl)ethanone *(Gallacetophenone)*

[528-21-2] $C_8H_8O_4$ mol.wt. 168.15



Described [3734] **p. 720**

Syntheses

- Obtained by reaction of acetic acid with pyrogallol in the presence of zinc chloride at 145–150° [3953].
- Also refer to: [3814,4042].

Trimethyl ether [13909-73-4] $C_{11}H_{14}O_4$ mol.wt. 210.23

- Obtained by reaction of dimethyl sulfate with gallacetophenone in methanol in the presence of potassium hydroxide [3953].
- Also refer to: [4043].

Isolation from natural sources

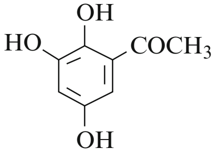
- From the essential oil in buds of *Syringa oblata* Lindl (lilac) [4044].

Oil [3953];

b.p.$_{14}$ 172–172.5° [3953], b.p.$_{17}$ 170–178° [3984]; GC-MS [4044].

1-(2,3,5-Trihydroxyphenyl)ethanone

[316819-88-2] $C_8H_8O_4$ mol.wt. 168.15



New compound

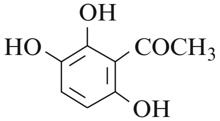
Syntheses

- Refer to: in 2006 [3737,3738].

N.B.: The compound previously obtained in 1983 actually was 2,4,5-trihydroxyacetophenone [4045,4046].

1-(2,3,6-Trihydroxyphenyl)ethanone

[85918-30-5] $C_8H_8O_4$ mol.wt. 168.15



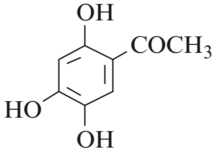
Described [3734] **p. 721**

Syntheses

- Also refer to: [3737,3738].

1-(2,4,5-Trihydroxyphenyl)ethanone

[1818-27-5] $C_8H_8O_4$ mol.wt. 168.15



Described [3734] **p. 721**

Synthesis

- Also refer to: [3738].

UV [3963].

Trimethyl ether [1818-28-6] $C_{11}H_{14}O_4$ mol.wt. 210.23

- Preparation by reaction of acetyl chloride with hydroxyhydroquinone trimethyl ether in the presence of aluminium chloride [4047], (quantitative yield) [3953].
- Also obtained by reaction of acetic anhydride with 1,2,4-trimethoxybenzene in the presence of iodine on heating for 18 h (88%) [4048].

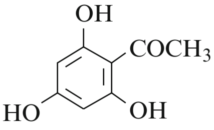
- Also obtained by reaction of dimethyl sulfate,
  - with 2,4,5-triacetoxyacetophenone in the presence of sodium methoxide in methanol [4047,4049];
  - with 2,4,5-trihydroxyacetophenone in the presence of sodium hydroxide in dilute ethanol [4046,4050] or sodium methoxide in methanol [4047,4049];
  - with 2,5-dihydroxy-4-methoxyacetophenone in the presence of sodium methoxide in methanol [4051];
  - with 2-hydroxy-4,5-dimethoxyacetophenone in the presence of sodium methoxide in methanol [4051] or in the presence of potassium hydroxide [4052].
- Also obtained by treatment of 2′,4′,5,5′,6,7,8-heptamethoxyflavone with 50% potassium hydroxide in methanol [4053].
- Also obtained by irradiation of 2,3-bis(2,4,5-trimethoxyphenyl)-2,3-butanediol in the presence of carbon tetrachloride in acetonitrile [4054].
- Also refer to: [4055–4057].

b.p.$_{33}$ 285–290° [4047,4049];
m.p. 102–103° [4046,4056], 102° [3953,4047,4055], 101° [4050], 99° [4052], 98–98.5° [4048], 96–100° [3984], 95–96° [4057];
^{1}H NMR [4048,4053,4058], ^{13}C NMR [4048,4058], IR [4053,4057,4058], UV [3801],
MS [4053,4058].

BIOLOGICAL ACTIVITY: Toxicity [4058,4059]; hypolipaemic [4058]; activity on second instar larvae of the cotton bug, *Dysdercus cingulatus* [4059].

1-(2,4,6-Trihydroxyphenyl)ethanone

[480-66-0] $C_8H_8O_4$ mol.wt. 168.15



Described [3734] **p. 722**

Syntheses

- Also obtained by heating its trimethyl ether with pyridinium chloride for 15 min (53%) [3765].
- Also obtained by reaction of acetyl chloride with phloroglucinol in the presence of aluminium chloride at 50° (70%) [4060].

UV [3963].

Tribenzoate $C_{29}H_{20}O_7$ mol.wt. 480.47

^{1}H NMR [4008], ^{13}C NMR [4008], MS [4008].

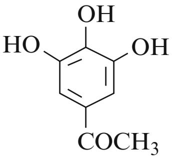
Trimethyl ether [832-58-6] $C_{11}H_{14}O_4$ mol.wt. 210.23

- Preparation by reaction of dimethyl sulfate with phloroacetophenone [3953].
- Also obtained by reaction of acetic acid with phloroglucinol trimethyl ether in the presence of PPA on a boiling water bath for 5–20 min (76%) [4061].
- Also refer to: [3765,3825,3931].

m.p. 102° [4061], 93.5–104° [3984], 91° [3953]; UV [3963].

1-(3,4,5-Trihydroxyphenyl)ethanone

[33709-29-4] $C_8H_8O_4$ mol.wt. 168.15



Described [3734] **p. 723**

Synthesis

- Also refer to: [3738].

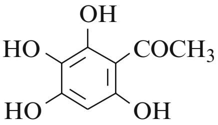
Trimethyl ether [1136-86-3] $C_{11}H_{14}O_4$ mol.wt. 210.23

- Obtained by treatment of a mixture of methyl 3,4,5-trimethoxybenzoate (m.p. 82°) and ethyl acetate with sodium on a water boiler for 8 h, then with 25% sulfuric acid [3953].
- Also refer to: [3825,3931,3967,4021,4022,4062].

b.p.$_5$ 160° [3953];
m.p. 78–81.5° [3984], 72° [3953]; GC-MS [3967].

1-(2,3,4,6-Tetrahydroxyphenyl)ethanone

[63635-39-2] $C_8H_8O_5$ mol.wt. 184.15



Described [3734] **p. 724**

Synthesis

- Also refer to: [4063].

Tetramethyl ether [7508-05-6] $C_{12}H_{16}O_5$ mol.wt. 240.26

- Preparation by reaction of acetyl chloride with 1,2,3,5-tetramethoxybenzene (m.p. 47°) [3953], in the presence of aluminium chloride [4064–4066], (quantitative yield) [3953].
- Also obtained by treatment of 1,2,3,5-tetramethoxybenzene with acetic anhydride in the presence of $In(CF_3SO_3)_3$ on microwave irradiation at 85–95° for 3 min (87%) [4067] or 6 min at 75–90° (53%) [4068].
- Also obtained by treatment of 2-hydroxy-3,4,6-trimethoxyacetophenone with dimethyl sulfate in ethyl acetate/chloroform mixture [4069] or in the presence of potassium carbonate in boiling acetone for 10 h [4070].
- Also obtained by reaction of excess dimethyl sulfate with 3,6-dihydroxy-2,4-dimethoxy-acetophenone in the presence of potassium carbonate in refluxing acetone for 24 h [4071].
- Also obtained by treatment of (*E*)-1-(2,3,4,6-tetramethoxyphenyl)-2-buten-1-one with 15% potassium hydroxide in boiling dilute ethanol for 7 h [4070].
- Also obtained by reaction of methyl acetate with 1,2,3,5-tetramethoxybenzene in the presence of PPA [4072].
- Also obtained by treatment of sinensetin with potassium hydroxide in dilute ethanol, followed by methylation of the product so obtained [4073].

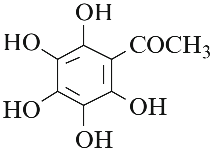
Isolation from natural sources

- From liverwort *Adelanthus decipiens* [4074].

b.p.$_{0.2}$ 116–118° [4075], 310° [4064,4076];
crystals [4071];
m.p. 55–56° [4077], 54° [3953], 53–54° [4065,4066], 53° [4073], 49.5–51° [4067], 48–50° [4076];
^{1}H NMR [4070,4074,4075], IR [4070,4075].

1-(Pentahydroxyphenyl)ethanone

[881672-75-9] $C_8H_8O_6$ mol.wt. 200.15



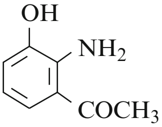
New compound

Synthesis

- Refer to: [3737].

1-(2-Amino-3-hydroxyphenyl)ethanone

[4502-10-7] $C_8H_9NO_2$ mol.wt. 151.17



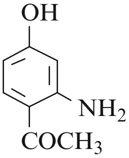
Described [3734] **p. 724**

Synthesis

- Also obtained by bioconversion of nitro precursor using recombinant *Escherichia coli* strains [4078].

1-(2-Amino-4-hydroxyphenyl)ethanone

[90033-64-0] $C_8H_9NO_2$ mol.wt. 151.17



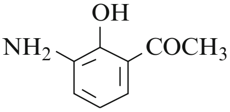
Described [3734] **p. 725**

Methyl ether *(Hydrochloride)* [335104-63-7] $C_9H_{11}NO_2$, HCl mol.wt. 201.65

- Refer to: [4079].

1-(3-Amino-2-hydroxyphenyl)ethanone

[70977-72-9] $C_8H_9NO_2$ mol.wt. 151.17



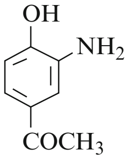
Described [3734] **p. 726**

Syntheses

- Also obtained by bioconversion of nitro precursor using recombinant *Escherichia coli* strains [4078].
- Also refer to: [4080].

1-(3-Amino-4-hydroxyphenyl)ethanone

[54255-50-4] $C_8H_9NO_2$ mol.wt. 151.17



Described [3734] **p. 727**

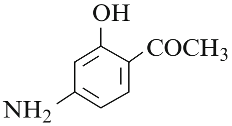
Syntheses

- Also obtained by reduction of 4-hydroxy-3-nitro-acetophenone by catalytic hydrogenation with hydrogen in the presence of,
  - Pd/C in ethanol [3932];
  - Pt on carbon disulfide in ethanol [3932].

- Also refer to: [4081,4082].

1-(4-Amino-2-hydroxyphenyl)ethanone

[2476-29-1] $C_8H_9NO_2$ mol.wt. 151.17



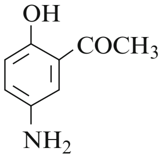
Described [3734] **p. 728**

Synthesis

- Also refer to: [3809].

1-(5-Amino-2-hydroxyphenyl)ethanone

[50-80-6] $C_8H_9NO_2$ mol.wt. 151.17



Described [3734] **p. 728**

Syntheses

- Also obtained by refluxing a suspension of N-(3-acetyl-4-hydroxyphenyl)acetamide in 15% hydrochloric acid for 40 min (84%) [3911].
- Also refer to: [3809].

Green solid [3911]; ^{1}H NMR [3911], ^{13}C NMR [3911], MS [3911].

Methyl ether [85276-70-6] $C_9H_{11}NO_2$ mol.wt. 201.65

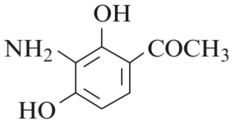
- Obtained by treatment of 2-methoxy-5-nitroacetophenone in ethanol and granulated tin with hydrochloric acid (d = 1.18) at 25° for 16 h (65%) [4085].
- Also obtained by Fries rearrangement of 4-(N-acetylamino)anisole in the presence of lithium perchlorate and ytterbium triflate in nitromethane at 100° for 8 h [4086].
- Also refer to: [3858,4087].

b.p.$_{0.25}$ 125–127° [4085]; m.p. 91° [4086];
^{1}H NMR [4086], ^{13}C NMR [3858], IR [4086].

Hydrochloride (of the methyl ether) $C_9H_{11}NO_2$, HCl mol.wt. 201.65 m.p. 186° (d) [4085]

1-(3-Amino-2,4-dihydroxyphenyl)ethanone

[909255-13-6] $C_8H_9NO_3$ mol.wt. 167.16



New compound

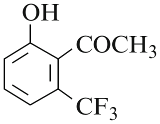
Syntheses

– Refer to: [4082,4088].

m.p. 216–218° [4088]; ^{1}H NMR [4088], MS [4088].

1-[2-Hydroxy-6-(trifluoromethyl)phenyl]ethanone

[1024605-96-6] $C_9H_7F_3O_2$ mol.wt. 204.15



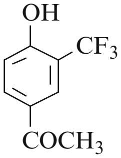
New compound

Synthesis

– Refer to: [3851].

1-[4-Hydroxy-3-(trifluoromethyl)phenyl]ethanone

[149105-11-3] $C_9H_7F_3O_2$ mol.wt. 204.15



Described [3734] **p. 730**

Synthesis

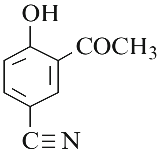
– Also refer to: [4089].

Methyl ether [149105-10-2] $C_{10}H_9F_3O_2$ mol.wt. 218.18

– Refer to: [4089].

3-Acetyl-4-hydroxybenzonitrile

[35794-84-4] $C_9H_7NO_2$ mol.wt. 161.16



New compound

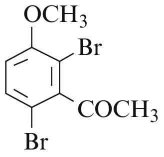
Syntheses

– Obtained by heating a mixture of 4-cyanophenyl acetate and aluminium chloride at 180–185° for 2 h (58%) [4090].

– Also refer to: [4091].

m.p. 103° [4091], 100–101° [4090];
^{1}H NMR [4090], IR [4090], UV [4090].

1-(2,6-Dibromo-3-methoxyphenyl)ethanone



$C_9H_8Br_2O_2$ mol.wt. 307.97

New compound

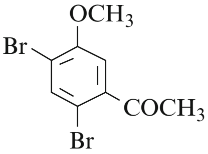
Synthesis

- Obtained from 3-methoxyacetophenone by reaction of hypobromous acid, generated in situ, in aqueous acetic acid containing perchloric acid catalyst at r.t. (14–15%) [3805].

^{1}H NMR [3805].

Oxime $C_9H_9Br_2NO_2$ mol.wt. 322.98 [3805]

1-(4,6-Dibromo-3-methoxyphenyl)ethanone



$C_9H_8Br_2O_2$ mol.wt. 307.97

New compound

Synthesis

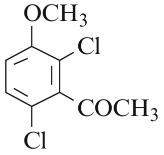
- Obtained from 3-methoxyacetophenone by reaction of hypobromous acid, generated in situ, in aqueous acetic acid containing perchloric acid catalyst at r.t. (2–3%) [3805].

^{1}H NMR [3805].

Oxime $C_9H_9Br_2NO_2$ mol.wt. 322.98 [3805]

1-(2,6-Dichloro-3-methoxyphenyl)ethanone

[157487-31-5] $C_9H_8Cl_2O_2$ mol.wt. 219.07



New compound

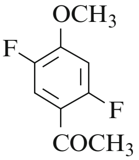
Syntheses

- Obtained by heating a mixture of 1-(2,6-dichloro-3-methoxyphenyl)ethan-1-ol and manganese (IV) oxide in benzene for 15 h (74%) [3769].
- Also refer to: [3770].

b.p.$_{0.5}$ 121–122° [3769]; m.p. 38–40° [3769];
^{1}H NMR [3769], IR [3769].

1-(2,5-Difluoro-4-methoxyphenyl)ethanone

[1010800-85-7] $C_9H_8F_2O_2$ mol.wt. 186.16



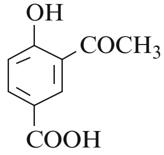
New compound

Synthesis

- Refer to: [3887].

3-Acetyl-4-hydroxybenzoic acid

[16357-40-7] $C_9H_8O_4$ mol.wt. 180.16



New compound

Synthesis

- Obtained by Fries rearrangement of 4-acetoxybenzoic acid (1 mol) with aluminium chloride (3.3 mol) at 150–155° for 1 h (24%) [4092].

m.p. 244–246° [4093], 240–241° [4092], 237–239° [4094]; IR [4093].

Methyl ether [103203-97-0] $C_{10}H_{10}O_4$ mol.wt. 194.19

- Obtained by treatment of 4-(2-methoxy-5-methylphenyl)-4-methyl-2,5-cyclohexadienone with $Zn(MnO_4)_2$ in acetone [3941].
- Also obtained by reaction of dimethyl sulfate with 3-acetyl-4-hydroxybenzoic acid [3941].
- Also refer to: [4095].

m.p. 225–226° [3941].

Methyl ester [57009-12-8] $C_{10}H_{10}O_4$ mol.wt. 194.19

- Refer to: [4096].

m.p. 91–92° [4096].

Ethyl ester [57009-53-7] $C_{11}H_{12}O_4$ mol.wt. 208.21

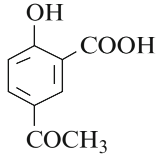
- Prepared by refluxing the acid and ethyl alcohol in the presence of a few drops of concentrated sulfuric acid [4092].
- Also refer to: [4090].

m.p. 90–92° [4097], 71° [4092]. One of the reported melting points is obviously wrong.

^{1}H NMR [4097,4098], UV [4098].

5-Acetyl-2-hydroxybenzoic acid

[13110-96-8] $C_9H_8O_4$ mol.wt. 180.16



New compound

Syntheses

- Obtained by Fries rearrangement of 2-(acetyloxy)-benzoic acid (Aspirin) (1 mol) in the presence of aluminium chloride (3.3 mol),
  - in nitrobenzene at r.t. for 1 h (36%) [4092];
  - without solvent at 120–125° for 1 h (19%) [4092].
- Also obtained by treatment of 2-(acetyloxy)benzoic acid with aluminium chloride in nitrobenzene at 60° [4099].

- Also obtained by reaction of acetyl chloride with 2-hydroxybenzoic acid in the presence of ferric chloride at 110–115° [4100].
- Also obtained by treatment of phenyl o-acetoxybenzoate with aluminium chloride in carbon disulfide [4101].
- Also obtained by treament of methyl o-(acetyloxy)salicylate with aluminium chloride in nitrobenzene [4102].
- Also obtained by reaction of acetyl chloride with 2-hydroxybenzoic acid in the presence of aluminium chloride in nitrobenzene [4103].

m.p. 217° [4103], 216–217° [4092], 209–210° [4104].

BIOLOGICAL ACTIVITY: Inhibition of cyclooxygenase [4105].

Methyl ether $C_{10}H_{10}O_4$ mol.wt. 194.19

- Obtained by reaction of dimethyl sulfate with 5-Acetyl-2-hydroxybenzoic acid in the presence of aqueous sodium hydroxide solution [4100].

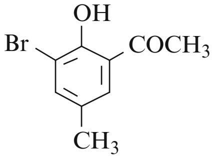
Methyl ester [16475-90-4] $C_{10}H_{10}O_4$ mol.wt. 194.19

- Obtained by heating a solution of 5-acetyl-2-hydroxybenzoic acid and sulfuric acid in methanol for 24 h (82%) [4106].
- Also obtained by reaction of acetyl chloride with methyl 2-methoxybenzoate in the presence of aluminium chloride at r.t. for 12 h (60%) [3934].
- Also refer to: [4107–4109].

b.p.$_{15}$ 162° [4109];
m.p. 62° [3934], 60–62° [4108], 58–61° [4106];
^{1}H NMR [4107], ^{13}C NMR [4107], IR [4107].

1-(3-Bromo-2-hydroxy-5-methylphenyl)ethanone

[56609-15-5] $C_9H_9BrO_2$ mol.wt. 229.07



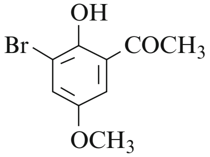
Described [3734] **p. 737**

Syntheses

- Also refer to: [4110,4111].

1-(3-Bromo-2-hydroxy-5-methoxyphenyl)ethanone

[37113-61-4] $C_9H_9BrO_3$ mol.wt. 245.07



Described [3734] **p. 739**

Synthesis

- Also obtained by reaction of bromine with 2-hydroxy-5-methoxyacetophenone in acetic acid in the presence of sodium acetate at r.t. for 48 h (87%) [4112].

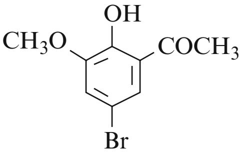
Green/yellow needles; m.p. 74–77° [4112];
^{1}H NMR [4112].

Methyl ether [286931-60-0] $C_{10}H_{11}BrO_3$ mol.wt. 259.10

- Obtained by reaction of dimethyl sulfate with 3-bromo-2-hydroxy-5-methoxyacetophenone in the presence of potassium carbonate in refluxing acetone for 24 h (82%) [4112].

b.p.$_{0.5}$ 110–114° [4112]; m.p. 41–42° [4112];
^{1}H NMR [4112], MS [4112].

1-(5-Bromo-2-hydroxy-3-methoxyphenyl)ethanone



$C_9H_9BrO_3$ mol.wt. 245.07

Described [3734] **p. 740**

Syntheses

- Also obtained by reaction of bromine with 2-hydroxy-3-methoxyphenylacetophenone in the presence of sodium bromide in dilute ethanol at 25° [3955].
- Also obtained by treatment of its methyl ether with hydrogen bromide in acetic acid for 4.5 h (36%) [3955].

Yellow crystals; m.p. 108–109° [3955]; UV [3955].

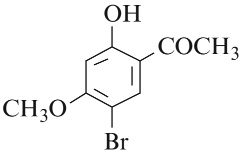
Methyl ether [7507-91-7] $C_{10}H_{11}BrO_3$ mol.wt. 250.10

- Obtained from 5-bromo-2,3-dimethoxybenzoic acid (m.p. 121–124°) (49.5%) [3955].
- Also obtained by treatment of 1-(5-bromo-2,3-dimethoxyphenyl)ethanol with chromium trioxide in the presence of pyridine in methylene chloride for 0.25 h (86%) [4113].

m.p. 65–66° [4113], 63.7–64.9° [3955];
^{1}H NMR [4113], IR [4113], UV [3955,4113].

1-(5-Bromo-2-hydroxy-4-methoxyphenyl)ethanone

[39503-61-2]



$C_9H_9BrO_3$ mol.wt. 245.07

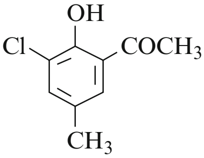
Described [3734] **p. 740**

Synthesis

- Also refer to: [4114].

1-(3-Chloro-2-hydroxy-5-methylphenyl)ethanone

[7507-88-2]



$C_9H_9ClO_2$ mol.wt. 184.62

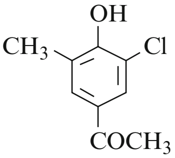
Described [3734] **p. 741**

Syntheses

- Also refer to: [4111,4115].

1-(3-Chloro-4-hydroxy-5-methylphenyl)ethanone

[54556-95-5] $C_9H_9ClO_2$ mol.wt. 184.62



Described [3734] **p. 742**

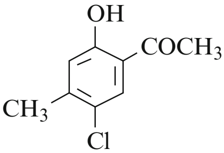
Methyl ether [56755-88-5] $C_{10}H_{11}ClO_2$ mol.wt. 198.65

- Obtained by reaction of dimethyl sulfate with 3-chloro-4-hydroxy-5-methylacetophenone in the presence of sodium ethoxide and sodium iodide in boiling ethanol [4116].
- Also refer to: [3778].

b.p.$_{0.05}$ 95–100° [4116];
$n_D^{20} = 1.5502$ [4116].

1-(5-Chloro-2-hydroxy-4-methylphenyl)ethanone

[28480-70-8] $C_9H_9ClO_2$ mol.wt. 184.62



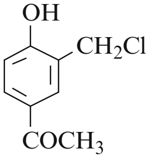
Described [3734] **p. 743**

Syntheses

- Also refer to: [3773,3775,3857,4117–4119].

1-[3-(Chloromethyl)-4-hydroxyphenyl]ethanone

[24085-05-0] $C_9H_9ClO_2$ mol.wt. 184.62



Described [3734] **p. 744**

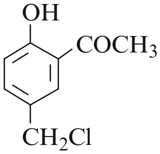
Synthesis

- Also obtained by chloromethylation of 4-hydroxy-acetophenone by the methode of Trave [4120,4121].

m.p. 160° (d) [4121];
^{1}H NMR [4121], IR [4121], MS [4121].

1-[5-(Chloromethyl)-2-hydroxyphenyl]ethanone

[30787-43-0] $C_9H_9ClO_2$ mol.wt. 184.62



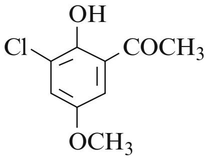
Described [3734] **p. 744**

Synthesis

- Also refer to: [3744].

1-(3-Chloro-2-hydroxy-5-methoxyphenyl)ethanone

[286931-53-1] $C_9H_9ClO_3$ mol.wt. 200.62



Described [3734] **p. 746**

Synthesis

- Also obtained by reaction of N-chlorosuccinimide with 2-hydroxy-5-methoxyacetophenone in acetic acid in the presence of magnesium acetate at r.t. for 24 h (80%) [4112].

Green-yellow needles; m.p. 78–79° [4112];
^{1}H NMR [4112], MS [4112].

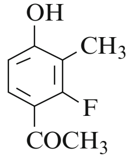
Methyl ether [286931-54-2] $C_{10}H_{11}ClO_3$ mol.wt. 214.65

- Obtained by reaction of dimethyl sulfate with 3-chloro-2-hydroxy-5-methoxyacetophenone in the presence of potassium carbonate in refluxing acetone for 24 h [4112].

Clear oil; b.p.$_{0.1}$ 91° [4112];
^{1}H NMR [4112], MS [4112].

1-(2-Fluoro-4-hydroxy-3-methylphenyl)ethanone

[872415-45-7] $C_9H_9FO_2$ mol.wt. 168.17



New compound

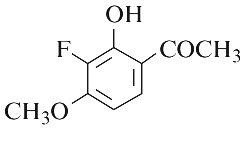
Synthesis

- Refer to: [3862].

Methyl ether [872415-44-6] $C_{10}H_{11}FO_2$ mol.wt. 182.19 [3862]

1-(3-Fluoro-2-hydroxy-4-methoxyphenyl)ethanone

[1018451-08-5] $C_9H_9FO_3$ mol.wt. 184.17



New compound

Synthesis

- Preparation by treatment of 2-fluoro-1,3-dimethoxybenzene with boron trifluoride–acetic acid complex at 80° for 4 h (98%) [4122].

Light yellow solid; m.p. 103° [4122];
^{1}H NMR [4122], ^{19}F NMR [4122], MS [4122].

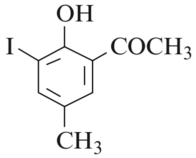
Methyl ether [1018451-09-6] $C_{10}H_{11}FO_3$ mol.wt. 198.07

- Obtained by treatment of 3-fluoro-2-hydroxy-4-methoxyacetophenone with methyl iodide in the presence of potassium carbonate in refluxing acetone for 1.5 h (93%) [4122].

m.p. 65° [4122];
^{1}H NMR [4122], ^{19}F NMR [4122], IR [4122], MS [4122].

1-(2-Hydroxy-3-iodo-5-methylphenyl)ethanone

[175655-10-4] $C_9H_9IO_2$ mol.wt. 276.07



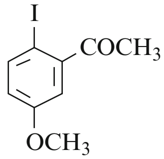
Described [3734] **p. 749**

Syntheses

- Also refer to: [3740,4111,4115].

1-(2-Iodo-5-methoxyphenyl)ethanone

[110718-87-1] $C_9H_9IO_2$ mol.wt. 276.07



New compound

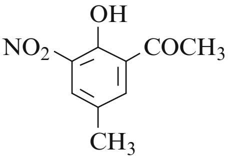
Syntheses

- Obtained from 4-bromoanisole (multi-step reaction) (37%) [3917].
- Also obtained from 2-bromoanisole (multi-step reaction) (73%) [3913].
- Also obtained from 2-amino-5-methoxyacetophenone [3915,4123].
- Also obtained by reaction of equimolar amounts of 3-methoxyacetophenone, iodine and silver trifluoroacetate [4124].
- Also refer to: [4125,4126].

Golden oil [3913];
^{1}H NMR [3917], ^{13}C NMR [3917], IR [3917], MS [3917].

1-(2-Hydroxy-5-methyl-3-nitrophenyl)ethanone

[66108-30-3] $C_9H_9NO_4$ mol.wt. 195.17



Described [3734] **p. 752**

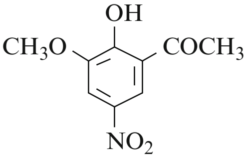
Syntheses

- Also refer to: [3764,4110,4127–4129].

IR [4127].

1-(2-Hydroxy-3-methoxy-5-nitrophenyl)ethanone

$C_9H_9NO_5$ mol.wt. 211.17



New compound

Synthesis

- Preparation by reaction of 70% nitric acid with 2-hydroxy-3-methoxyacetophenone in acetic acid at 25° (95%) [3955].

m.p. 148.1–149.5° [3955]; UV [3955].

Methyl ether [102652-91-5] $C_{10}H_{11}NO_5$ mol.wt. 225.20

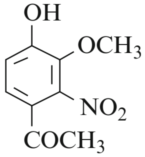
- Obtained by methylation of 2-hydroxy-3-methoxy-5-nitroacetophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 4 h (53%) [3955].

m.p. 81.2–83.2° [3955]; UV [3955].

Acetate $C_{11}H_{11}NO_6$ mol.wt. 253.21

m.p. 125.2–126.8° [3955].

1-(4-Hydroxy-3-methoxy-2-nitrophenyl)ethanone



$C_9H_9NO_5$ mol.wt. 211.17

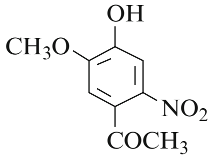
New compound

Synthesis

- Obtained by hydrolysis of 4-acetoxy-3-methoxy-2-nitroacetophenone with 3 N sodium hydroxide in dilute methanol at r.t. [3950].

1-(4-Hydroxy-5-methoxy-2-nitrophenyl)ethanone

[418759-58-7] $C_9H_9NO_5$ mol.wt. 211.17



Described [3734] **p. 756**

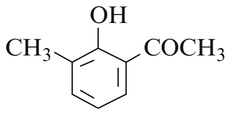
Syntheses

- Also refer to: [4130,4131].

Benzyl ether [75665-88-2] $C_{16}H_{15}NO_5$ mol.wt. 301.30 [4131]

1-(2-Hydroxy-3-methylphenyl)ethanone

[699-91-2] $C_9H_{10}O_2$ mol.wt. 150.18



Described [3734] **p. 757**

Syntheses

- Also refer to: [3773,3853,3892].

UV [3955].

Methyl ether [6342-75-2] $C_{10}H_{12}O_2$ mol.wt. 164.20

- Preparation by reaction of dimethyl sulfate with 2-hydroxy-3-methylacetophenone in the presence of sodium hydroxide solution [4132].
- Also obtained by reaction of methyl iodide with 2-hydroxy-3-methylacetophenone in the presence of sodium ethoxide in ethanol on a water bath for 3 h [3865].
- Also refer to: [3955,4133].

colourless oil [3865];

b.p.$_{12}$ 115° [4132], b.p.$_{14}$ 116–117° [3955], b.p.$_{3}$ 120° [3865],
b.p.$_{15}$ 125–128° [4133], b.p.$_{760}$ 240° [4132];
UV [3955].

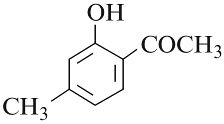
Benzoate $C_{16}H_{14}O_3$ mol.wt. 254.29

- Obtained by reaction of benzoyl chloride with 2-hydroxy-3-methylacetophenone in suspension of 2 N sodium hydroxide solution at r.t. for 20 min (75%) [3959].

m.p. 102–103° [3959].

1-(2-Hydroxy-4-methylphenyl)ethanone

[6921-64-8] $C_9H_{10}O_2$ mol.wt. 150.18



Described [3734] **p. 758**

Syntheses

- Also obtained by Fries rearrangement of 3-methylphenyl acetate in the presence of zirconium chloride with ultrasound irradiation in DCM at r.t. for 24 h (93%) [4134].
- Also obtained by heating its methyl ether with pyridinium chloride for 15 min (66%) [3765].
- Also obtained by Friedel–Crafts acylation of m-cresol with acetic acid in a mixture of graphite and methanesulfonic acid at 120° for 2 h (80%) [3889].
- Also refer to: [3773,3959,4018,4135,4136].

^{1}H NMR [3889], ^{13}C NMR [3889].

Methyl ether [35633-35-3] $C_{10}H_{12}O_2$ mol.wt. 164.20

- Obtained by reaction of dimethyl sulfate with 2-hydroxy-4-methylacetophenone in the presence of potassium hydroxide in acetone at r.t. for 15 h (97%) [4134].
- Also obtained by reaction of acetic anhydride with 3-methylanisole in the presence of lithium perchlorate at 60° for 2 h (30%) [3798].
- Also refer to: [3765,4137].

^{13}C NMR [3858].

Benzyl ether [58110-89-7] $C_{16}H_{16}O_2$ mol.wt. 240.30 Refer to: [4136].

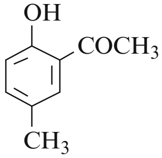
Benzoate [5177-98-0] $C_{16}H_{14}O_3$ mol.wt. 254.29

- Obtained by reaction of benzoyl chloride with 2-hydroxy-4-methylacetophenone in suspension in 2 N sodium hydroxide solution at r.t. for 20 min (78%) [3959].
- Also refer to: [4138–4140].

b.p.$_{0.02}$ 150–165° [4138]; m.p. 98–99° [3959].

1-(2-Hydroxy-5-methylphenyl)ethanone

[1450-72-2] $C_9H_{10}O_2$ mol.wt. 150.18



Described [3734] **p. 760**

Syntheses

- Also obtained by treatment of 2′-hydroxy-5′-methyl-3,3-dimethylacrylophenone with aluminium chloride in boiling benzene for 7 h [3996].
- Also obtained by treatment of 2-hydroxy-5-methyl-α-chloroacetophenone with zinc powder and hydrochloric acid [4141].
- Also obtained by treatment of its methyl ether with hydrogen bromide in acetic acid (6.6%) [3955].
- Also refer to: [3773,3959,3967,3991,4018,4110,4142].

m.p. 50–50.5° [4141], 50° [3996], 47–48° [4142], 46–47.5° [3955];
UV [3801,3955].

Methyl ether [20628-07-3] $C_{10}H_{12}O_2$ mol.wt. 164.20

- Preparation by reaction of acetic acid with 4-methylanisole in the presence of trifluoromethyl-sulfonic anhydride (Tf_2O) for 3 min at r.t. (97%) [3965].
- Also obtained by reaction of acetic acid with p-cresyl methyl ether in the presence of PPA (74%) [3955].
- Also obtained by reaction of acetic anhydride with 4-methylanisole,
  - in the presence of aluminium chloride in carbon disulfide (64–70%) [3966];
  - in the presence of lithium perchlorate at 100° for 3 h (99%) [3798].
- Also refer to: [4142], (42%) [3917].

b.p.$_7$ 120.5° [3966], b.p.$_7$ 121–123° [3955];
^{1}H NMR [3917], ^{13}C NMR [3917], UV [3801,3955].

Benzyl ether [36808-17-0] $C_{16}H_{16}O_2$ mol.wt. 240.30

- Obtained by reaction of benzyl chloride with 2-hydroxy-5-methylacetophenone in the presence of KOH in acetonitrile. Then, the mixture was refluxed for 3 h (89%) [3978].

m.p. 81° [3978];
^{1}H NMR [3978], ^{13}C NMR [3978], IR [3978], MS [3978].

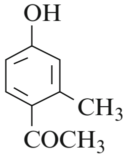
Benzoate $C_{16}H_{14}O_3$ mol.wt. 254.29

- Obtained by reaction of benzoyl chloride with 2-hydroxy-5-methylacetophenone in suspension in 2 N sodium hydroxide solution at r.t. for 20 min (76%) [3959].

m.p. 96–97° [3959].

1-(4-Hydroxy-2-methylphenyl)ethanone

[875-59-2] $C_9H_{10}O_2$ mol.wt. 150.18



Described [3734] **p. 765**

Syntheses

- Also obtained from pyrolysis behaviour of 4-oxo-β-damascone by on-line pyrolysis gas chromatography–mass spectrometry [4143].
- Also obtained by heating its methyl ether with pyridinium chloride for 7.5 min (56%) [3765].
- Also refer to: [3765,4018,4144,4145].

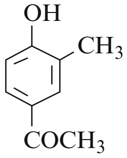
Methyl ether [24826-74-2] $C_{10}H_{12}O_2$ mol.wt. 164.20

- Obtained by reaction of acetic anhydride with 3-methylanisole in the presence of aluminium chloride in carbon disulfide (87%) [3966], at r.t., then at 30–35° until no more hydrochloric acid is evolved [3796,3797].
- Also obtained by reaction of acetic anhydride with 3-methylanisole in the presence of lithium perchlorate at 60° for 2 h (70%) [3798].
- Also refer to: [3765].

b.p.$_3$ 116.5° [3966], b.p.$_{735}$ 267° [3966];
^{1}H NMR [3796,3797], ^{13}C NMR [3858], IR [3796,3797].

1-(4-Hydroxy-3-methylphenyl)ethanone

[876-02-8] $C_9H_{10}O_2$ mol.wt. 150.18



Described [3734] **p. 766**

Syntheses

- Also obtained by heating its methyl ether with pyridinium chloride for 15 min (53%) [3765].
- Also refer to: [3743,4146–4148].

Methyl ether [10024-90-5] $C_{10}H_{12}O_2$ mol.wt. 164.20

- Preparation by reaction of acetic anhydride with 2-methylanisole in the presence of lithium perchlorate at 60° for 1.5 h (99%) [3798].
- Also obtained by Friedel–Crafts acetylation of o-methoxytoluene (73%) [3966], (21%) [4149].
- Refer to: [3765,3778,4150].

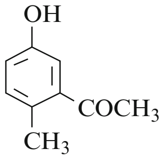
b.p.$_3$ 116° [3966]; m.p. 26–26.5° [3966], 24.5–25° [4149];
^{13}C NMR [3751,3754].

Benzyl ether [56443-69-7] $C_{16}H_{16}O_2$ mol.wt. 240.30

- Obtained by reaction of benzyl chloride with 4-hydroxy-3-methylacetophenone in the presence of KOH in acetonitrile. Then, the mixture was refluxed for 3 h [3978].

1-(5-Hydroxy-2-methylphenyl)ethanone

[40180-70-9] $C_9H_{10}O_2$ mol.wt. 150.18



Described [3734] **p. 767**

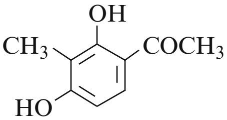
Methyl ether [110743-57-2] $C_{10}H_{12}O_2$ mol.wt. 164.20

- Refer to: [4151], (5%) [3917].

b.p.$_7$ 118–121° [4151];
^{1}H NMR [3917], ^{13}C NMR [3917], IR [3917].

1-(2,4-Dihydroxy-3-methylphenyl)ethanone

[10139-84-1] $C_9H_{10}O_3$ mol.wt. 166.18



Described [3734] **p. 768**

Syntheses

- Also refer to: [4152].[3999,4011,4153–4156]

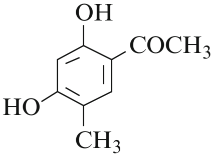
Isolation from natural sources

- From the volatile oil of *Dendrobium nobile* Lind [4157].
- From the volatile oil of *Dendrobium Ioddigesii* Rolfe [4157].

MS [4157]; GC-MS [4157].

1-(2,4-Dihydroxy-5-methylphenyl)ethanone

[93578-16-6] $C_9H_{10}O_3$ mol.wt. 166.18



Described to: [3734] **p. 769**

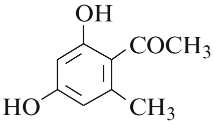
Synthesis

- Also obtained by heating its methylether with pyridinium chloride for 15 min (67%) [3765].

Dimethyl ether $C_{11}H_{14}O_3$ mol.wt. 194.23 [3765].

1-(2,4-Dihydroxy-6-methylphenyl)ethanone

[703-29-7] $C_9H_{10}O_3$ mol.wt. 166.18



Described [3734] **p. 769**

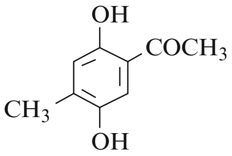
Dimethyl ether [6110-38-9] $C_{11}H_{14}O_3$ mol.wt. 194.23

- Obtained by reaction of acetyl chloride with orcinol dimethyl ether [4158],
  - in the presence of aluminium chloride [3953];
  - in the presence of stannic chloride [4159].
- Also obtained by reaction of dimethyl sulfate with 2,4-dihydroxy-6-methylacetophenone in the presence of sodium hydroxide in dilute methanol [4160].
- Also refer to: [4161].

b.p.$_{0.2}$ 98.5–99.5° [4159]; b.p.$_{15}$ 150–165° [3953];
m.p. 85° [3953], 48° [4160], 41–42° [4158,4162].
One of the reported melting points is obviously wrong.
^{1}H NMR [4158,4162,4163], ^{13}C NMR [4164],
IR [4162,4163], UV [4158,4159,4162], MS [4158].

1-(2,5-Dihydroxy-4-methylphenyl)ethanone

[54698-17-8] $C_9H_{10}O_3$ mol.wt. 166.18



Described [3734] **p. 771**

Syntheses

- Obtained by hydrolysis of 5-acetoxy-2-hydroxy-4-methyl-acetophenone with concentrated hydrochloric acid in methanol at r.t. for 1 h (27%) [4165].
- Also obtained by Fries rearrangement of 2,5-diacetoxytoluene,
  - in the presence of boron trifluoride dihydrate for 4 h at 130° (quantitative yield) [4166];
  - in the presence of aluminium chloride at 125–130° (41%) [4167].

Isolation from natural sources

- From the root bark of *Paeonia suffruticosa* Andrews (Paeoniaceae) [4168].

m.p. 146–147° [4167], 146° [4165];
^{1}H NMR [4166], ^{13}C NMR [4166], IR [4166], UV [4166], MS [4166].

Dimethyl ether [13720-58-6] $C_{11}H_{14}O_3$ mol.wt. 194.23

- Obtained by action of dimethyl sulfate with 2,5-dihydroxy-4-methylacetophenone in the presence of potassium carbonate in refluxing acetone for 7 h (50%) [4165].
- Also refer to: [4137].

m.p. 76° [4165].

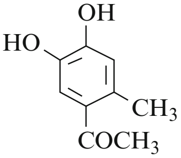
Dibenzyl ether $C_{23}H_{22}O_3$ mol.wt. 346.43

- To a solution of 2,5-dihydroxy-4-methylacetophenone in dimethoxyethane was added potassium carbonate, under nitrogen and the solution was stirred to 80° in nitrogen atmosphere. Then, benzyl bromide and N,N-dimethylformamide were added (95%) [4166].

^{1}H NMR [4166], ^{13}C NMR [4166], IR [4166], UV [4166], MS [4166].

1-(3,4-Dihydroxy-2-methylphenyl)ethanone

[18087-17-7] $C_9H_{10}O_3$ mol.wt. 166.18



Described [3734] **p. 774**

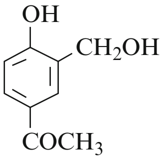
Dimethyl ether [24186-66-1] $C_{11}H_{14}O_3$ mol.wt. 194.23

- Obtained from 4-methylveratrole by reaction,
  - with acetyl chloride in the presence of aluminium chloride in carbon disulfide [4169,4170];
  - with acetic anhydride in the presence of aluminium chloride [4171] in tetrachloroethane [4172].
- Also obtained by treatment of ω-chloro-4,5-dimethoxyacetophenne with zinc in acetic acid [4173].
- Also refer to: [4174–4176].

b.p.$_{15}$ 175° [4170], b.p.$_{70}$ 204° [4169];
m.p. 76–77° [4170], 76° [4176], 75–76° [4175], 73–75° [4171,4172], 68° [4169,4173];
^{1}H NMR [4171], IR [4171], UV [4171].

1-[4-Hydroxy-3-(hydroxymethyl)phenyl]ethanone

[39235-58-0] $C_9H_{10}O_3$ mol.wt. 166.18



New compound

Syntheses

- Obtained by refluxing 3-chloromethyl-4-hydroxy-acetophenone with water for 3 h (70%) [4120].
- Also obtained by heating 6-acetyl-1,3-benzo(*4H*)dioxine with hydrochloric acid for 2 h (80%) [4177].
- Also obtained by treatment of 4-hydroxyacetophenone with poly(oxymethylene) (37%) [4177].
- Also refer to: [3978,4178–4180].

m.p. 124–125° [4181], 120.5–121.5° [4180], 116–118° [4178,4179], 116° [4120]; ^{1}H NMR [4120], IR [4120], MS [4120].

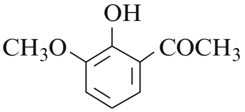
4-Benzyl ether [39235-59-1] $C_{16}H_{16}O_3$ mol.wt. 256.30 [3978]

- Obtained by reaction of benzyl chloride with 4-hydroxy-3-hydroxymethylacetophenone in the presence of potassium hydroxide and tetraethylammonium iodide in refluxing acetonitrile for 3 h (88%) [3978,4177].
- Preparation by reaction of benzyl bromide with the potassium salt of 5-acetyl-2-hydroxybenzyl alcohol [4180].

m.p. 125° [3978], 124–125° [4180], 122.5–124° [4177];
^{1}H NMR [3978,4177], ^{13}C NMR [3978,4177], IR [3978,4177], MS [3978].

1-(2-Hydroxy-3-methoxyphenyl)ethanone

[703-98-0] $C_9H_{10}O_3$ mol.wt. 166.18



Described [3734] **p. 775**

Syntheses

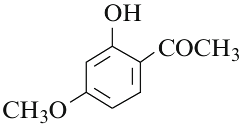
- Also obtained by action of hydrogen bromide with 2,3-dimethoxyacetophenone in acetic acid at 30° for 25 min (92%) [3989] or at r.t. (70%) [3984].
- Also refer to: [3744,3810].

m.p. 53–54° [3989,4182], 49–51.5° [3984];
UV [3955,3956].

Acetate $C_{11}H_{12}O_4$ mol.wt. 208.21 (m.p. 44.5–46.5°) [3984]

1-(2-Hydroxy-4-methoxyphenyl)ethanone *(Paeonol)*

[552-41-0] $C_9H_{10}O_3$ mol.wt. 166.18



Described [3734] **p. 775**

Synthesis

- Also obtained by action of hydrogen bromide with 2,4-dimethoxyacetophenone in acetic acid at r.t. (9%) [3984].

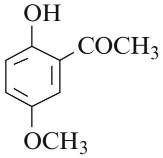
m.p. 41–50° [3984]; IR [3954], UV [3956,3963].

Acetate $C_{11}H_{12}O_4$ mol.wt. 208.21 m.p. 43–46° [3984].

Oxime [51864-08-5] $C_9H_{11}NO_3$ mol.wt. 181.19 [3737,3738]

1-(2-Hydroxy-5-methoxyphenyl)ethanone

[705-15-7] $C_9H_{10}O_3$ mol.wt. 166.18



Described [3734] **p. 777**

Syntheses

- Obtained by heating 2,5-dimethoxyacetophenone oxime with pyridinium chloride containing 10% of water (11%) [3980].
- Also obtained by action of hydrogen bromide with 2,5-diimethoxyacetophenone in acetic acid at r.t. (6%) [3984].
- Also refer to: [3744,3892,3997,3998,4011,4183].

b.p.$_{17}$ 145° [3980]; m.p. 46–47° [3984]; UV [3955,3963].
USE: Ultraviolet absorbers [4183].

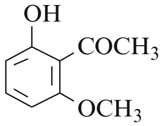
Acetate $C_{11}H_{12}O_4$ mol.wt. 208.21 m.p. 60–62° [3984].

Benzyl ether $C_{16}H_{16}O_3$ mol.wt. 256.30 [4184]

Oxime $C_9H_{11}NO_3$ mol.wt. 181.19 m.p. 113° [3980].

1-(2-Hydroxy-6-methoxyphenyl)ethanone

[703-23-1] $C_9H_{10}O_3$ mol.wt. 166.18



Described [3734] **p. 778**

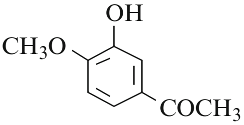
Syntheses

- Also refer to: [3787,3892,4042].

UV [3956,3963].

1-(3-Hydroxy-4-methoxyphenyl)ethanone *(Isoacetovanillone)*

[6100-74-9] $C_9H_{10}O_3$ mol.wt. 166.18



Described [3734] **p. 779**

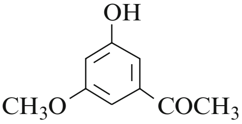
Synthesis

- Also refer to: [3738]; UV [3956].

Benzyl ether $C_{16}H_{16}O_3$ mol.wt. 256.30 [3947]

1-(3-Hydroxy-5-methoxyphenyl)ethanone

[35999-23-6] $C_9H_{10}O_3$ mol.wt. 166.18

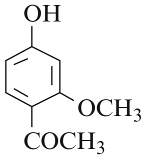


Described [3734] **p. 780**

UV [3956].

1-(4-Hydroxy-2-methoxyphenyl)ethanone *(Isopaenol)*

[493-33-4] $C_9H_{10}O_3$ mol.wt. 166.18



Described [3734] **p. 780**

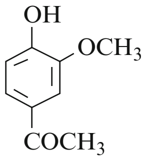
Synthesis

- Also refer to: [3788].

UV [3956].

1-(4-Hydroxy-3-methoxyphenyl)ethanone *(Acetoguaiacone, Acetovanillone)*

[498-02-2] $C_9H_{10}O_3$ mol.wt. 166.18



Described [3734] **p. 781**

Syntheses

- Also obtained by Fries rearrangement of guaiacol acetate in the presence of zinc chloride [3953].
- Also refer to: [4015].

m.p. 116° [3953]; UV [3956,3963,4015].

Isolation from natural sources

- From the fresh Iris rhizomes (2.69%) [4026].

Benzoate $C_{16}H_{14}O_4$ mol.wt. 270.28

- Obtained by reaction of benzoyl chloride with acetoguaiacone in the presence of pyridine (90%) [4015].

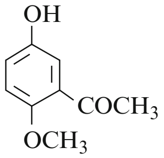
m.p. 108–109° [4015].

Acetate [54771-60-7] $C_{11}H_{12}O_4$ mol.wt. 208.21

- Refer to: [4185].

1-(5-Hydroxy-2-methoxyphenyl)ethanone

[31405-60-4] $C_9H_{10}O_3$ mol.wt. 166.18



Described [3734] **p. 782**

Synthesis

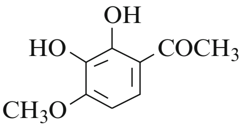
- Also prepared by treatment of 5-(pivaloyloxy)-2-methoxyacetophenone in DMF with a 2 M sodium hydroxide aqueous solution at 100° for 12 h (83%) [3818].

Yellow oil [3818];

^{1}H NMR [3818], ^{13}C NMR [3818], UV [3956], MS [3818].

1-(2,3-Dihydroxy-4-methoxyphenyl)ethanone

[708-53-2] $C_9H_{10}O_4$ mol.wt. 182.18



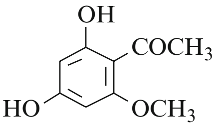
Described [3734] **p. 783**

Syntheses

- Also obtained by Fries rearrangement of 2,3-dihydroxy-3-methoxyphenyl diacetate with boron trifluoride etherate at 70° for 1 h, then hydrolyse with concentrated HCl at 60° for 2 h (75%) [4186].
- Also refer to: [4187].

1-(2,4-Dihydroxy-6-methoxyphenyl)ethanone

[3602-54-8] $C_9H_{10}O_4$ mol.wt. 182.18



Described [3734] **p. 785**

Synthesis

- Also obtained by reaction of dimethyl sulfate with phloroacetophenone in the presence of potassium carbonate in refluxing acetone for 5 h (14%) [4188].

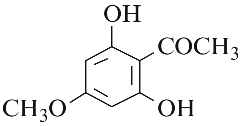
Isolation from natural sources

- From the aerial parts of *Artemisia scoparia* [4189].

m.p. 204–206° [4188].

1-(2,6-Dihydroxy-4-methoxyphenyl)ethanone

[7507-89-3] $C_9H_{10}O_4$ mol.wt. 182.18



Described [3734] **p. 787**

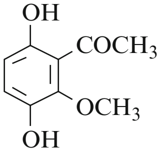
Syntheses

- Also obtained by reaction of dimethyl sulfate with phloroacetophenone in the presence of potassium carbonate in refluxing acetone for 5 h (21%) [4188].
- Also refer to: [4190,4191].

m.p. 140–141° [4188].

1-(3,6-Dihydroxy-2-methoxyphenyl)ethanone

[33539-20-7] $C_9H_{10}O_4$ mol.wt. 182.18



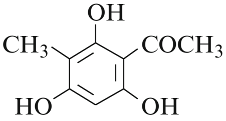
Described [3734] **p. 789**

Synthesis

- Also refer to: [4042].

1-(2,4,6-Trihydroxy-3-methylphenyl)ethanone

[2657-28-5] $C_9H_{10}O_4$ mol.wt. 182.18



Described [3734] **p. 790**

Syntheses

- Also obtained by treatment of its trimethyl ether with boron tribromide in methylene chloride at −78°, then at r.t. for 24–48 h (80%) [4192].
- Also refer to: [4193,4194].

^{1}H NMR [4192], ^{13}C NMR [4192], UV [4194], MS [4192].

Trimethyl ether [39701-13-8] $C_{12}H_{16}O_4$ mol.wt. 224.26

- Obtained by reaction of dimethyl sulfate with 4,6-dihydroxy-2-methoxy-3-methylacetophenone in the presence of potassium carbonate in boiling acetone for 2 h (quantitative yield) [4195].
- Also obtained by reaction of dimethyl sulfate with 2-hydroxy-4,6-dimethoxy-3-methyl-acetophenone,
  - in the presence of potassium carbonate in boiling acetone for 48 h (90%) [4195];
  - in the presence of 2 N sodium hydroxide at 100° for 30 min [4196].

- Also obtained by reaction of methyl iodide with 2,4,6-trihydroxy-3-methylacetophenone in the presence of potassium carbonate in DMF for 5 h [4197].
- Also obtained by reaction of dimethyl sulfate with 2,4,6-trihydroxy-3-methylacetophenone in the presence of potassium carbonate in boiling acetone for 3 h [4198].
- Also obtained by reaction of acetyl chloride with 2,4,6-trimethoxytoluene in methylene chloride in the presence of stannic chloride at −10° for 2–3 h (77%) [4192].
- Also refer to: [4193].

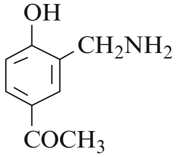
Isolation from natural sources

- From *Euphorbia portulacoides* [4199].

Colourless oil [4195,4196]; b.p.$_{12}$ 203–204° [4196];
m.p. 44–45° [4196], 44° [4195];
^{1}H NMR [4192,4196–4200], ^{13}C NMR [4192], IR [4196–4198], UV [4198], MS [4192,4197].

1-(3-Aminomethyl-4-hydroxyphenyl)ethanone

$C_9H_{11}NO_2$ mol.wt. 165.19



New compound

Synthesis

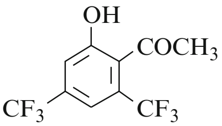
- Obtained by treatment of N-(5-acetyl-2-hydroxy-benzyl) phthalimide (m.p. 207°) with boiling 7% aqueous sodium hydroxide for 7 min [4201].

m.p. 202° (d) [4201].

Hydrochloride [109314-50-3] $C_9H_{11}NO_2$, HCl mol.wt. 201.65 (m.p. 253°) [4201]

1-[2-Hydroxy-4,6-bis(trifluoromethyl)phenyl]ethanone

[884851-57-4] $C_{10}H_6F_6O_2$ mol.wt. 272.15



New compound

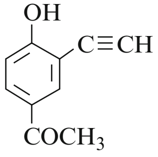
Synthesis

- To a CH_2Cl_2 solution of 1,3-bis-silyl enol ether (R^3 = CH_3) and 1,1,1,5,5,5-hexafluoro-4-silyloxy-3-penten-2-one was added $TiCl_4$ at −78° under argon atmosphere. The temperature of the reaction mixture was allowed to rise to 20° during 14 h and, subsequently, a saturated aqueous solution of $NaHCO_3$ was added (35%) [4202].

^{1}H NMR [4202], ^{13}C NMR [4202], ^{19}F NMR [4202]; TLC [4202].

1-(3-Ethynyl-4-hydroxyphenyl)ethanone

[370565-08-5] $C_{10}H_8O_2$ mol.wt. 160.17



New compound

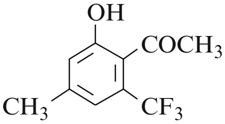
Synthesis

- Preparation from p-acetyl-o-[(trimethylsilyl)-ethynyl] phenol (m.p. 127–129°) via a Sonogashira coupling followed by desilylation [3920].

m.p. 100–102° [3920];
^{1}H NMR [3920], ^{13}C NMR [3920], MS [3920].

1-[2-Hydroxy-4-methyl-6-(trifluoromethyl)phenyl]ethanone

[884851-54-1] $C_{10}H_9F_3O_2$ mol.wt. 218.18



New compound

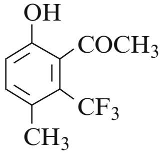
Synthesis

- To a CH_2Cl_2 solution of 1,3-bis-silyl enol ether ($R^3 = CH_3$) and 1,1,1-trifluoro-4-silyloxy-3-penten-2-one was added $TiCl_4$ at −78° under argon atmosphere. The temperature of the reaction mixture was allowed to rise to 20° during 14 h and, subsequently, a saturated aqueous solution of $NaHCO_3$ was added (61%) [4202].

^{1}H NMR [4202], ^{13}C NMR [4202], ^{19}F NMR [4202]; TLC [4202].

1-[6-Hydroxy-3-methyl-2-(trifluoromethyl)phenyl]ethanone

[884851-62-1] $C_{10}H_9F_3O_2$ mol.wt. 218.18



New compound

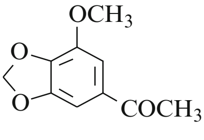
Synthesis

- To a CH_2Cl_2 solution of 1,3-bis-silyl enol ether ($R^3 = CH_3$) and 4-ethoxy-1,1,1-trifluoro-3-methyl-3-buten-2-one was added $TiCl_4$ at −78° under argon atmosphere. The temperature of the reaction mixture was allowed to rise to 20° during 14 h and, subsequently, an aqueous solution of 10% HCl was added (60%) [4202].

^{1}H NMR [4202], ^{13}C NMR [4202], ^{19}F NMR [4202], IR [4202]; TLC [4202].

1-(7-Methoxy-1,3-benzodioxol-5-yl)ethanone

[66922-69-8] $C_{10}H_{10}O_4$ mol.wt. 194.19



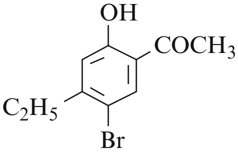
New compound

Synthesis

- Refer to: [4203].

1-(5-Bromo-4-ethyl-2-hydroxyphenyl)ethanone

[649551-87-1] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



New compound

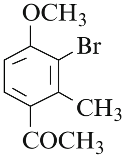
Synthesis

- Preparation by reaction of bromine with 4-ethyl-2-hydroxyacetophenone in chloroform at −12° for 50 min (76%) [3911].

Brown solid [3911]; MS [3911].

1-(3-Bromo-4-methoxy-2-methylphenyl)ethanone

[898538-41-5] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



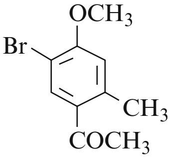
New compound

Synthesis

- Refer to: [4204].

1-(5-Bromo-4-methoxy-2-methylphenyl)ethanone

[898538-40-4] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



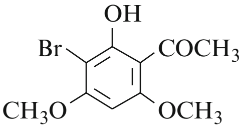
New compound

Synthesis

- Refer to: [4204].

1-(3-Bromo-2-hydroxy-4,6-dimethoxyphenyl)ethanone

[18064-89-6] $C_{10}H_{11}BrO_4$ mol.wt. 275.10



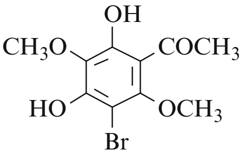
Described [3734] **p. 804**

Synthesis

- Also refer to: [4205].

1-(5-Bromo-2,4-dihydroxy-3,6-dimethoxyphenyl)ethanone

$C_{10}H_{11}BrO_5$ mol.wt. 291.10



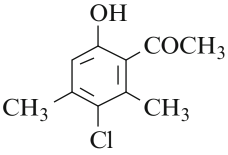
New compound

Synthesis

- Obtained by halogenation of 2,4-dihydroxy-3,6-dimethoxy-acetophenone with N-bromosuccinimide [4206].

1-(3-Chloro-6-hydroxy-2,4-dimethylphenyl)ethanone

[50343-13-0] $C_{10}H_{11}ClO_2$ mol.wt. 198.65

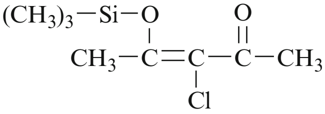


Described [3734] **p. 806**

Syntheses

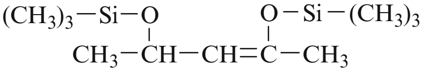
- Also obtained by [3 + 3] cyclisation of 2-chloro-3-(silyl-oxy)alk-2-en-1-one (**3a**) with 1,3-bis-silyl enol ether (**4b**) in the presence of titanium tetrachloride in methylene chloride, first at −78°, then to 20° for 20 h (50%) [4207], (49%) [4208].

3a = 3-chloro-4-(trimethylsilyloxy)-3-penten-2-one.



$$CH_3-\overset{\overset{(CH_3)_3-Si-O}{|}}{C}=\underset{\underset{Cl}{|}}{C}-\overset{\overset{O}{\|}}{C}-CH_3$$

4b = 2,4-bis(trimethylsilyloxy)-2-pentene.



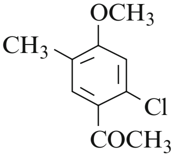
$$CH_3-\overset{\overset{(CH_3)_3-Si-O}{|}}{CH}-CH=\overset{\overset{O-Si-(CH_3)_3}{|}}{C}-CH_3$$

m.p. 99° [4208];

^{1}H NMR [4208], ^{13}C NMR [4208], IR [4208], MS [4208].

1-(2-Chloro-4-methoxy-5-methylphenyl)ethanone

[412021-93-3] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



New compound

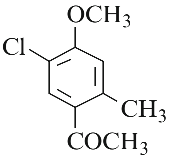
Synthesis

- Refer to: [4209].

USE: For preparation of phenylmethylbicyclocarboxamide derivatives as VR1 receptor inhibitors [4209].

1-(5-Chloro-4-methoxy-2-methylphenyl)ethanone

[103039-12-9] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



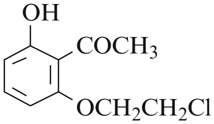
New compound

Synthesis

- Refer to: [4210].

1-[2-(2-Chloroethoxy)-6-hydroxyphenyl]ethanone

[870652-73-6] $C_{10}H_{11}ClO_3$ mol.wt. 214.65



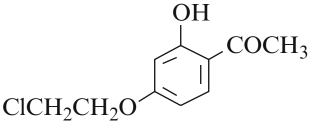
New compound

Synthesis

- Refer to: [4211].

1-[4-(2-Chloroethoxy)-2-hydroxyphenyl]ethanone

[109661-96-3] $C_{10}H_{11}ClO_3$ mol.wt. 214.65



Described [3734] **p. 807**

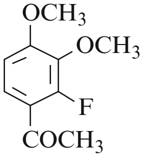
Syntheses

- Also obtained by reaction of 1-bromo-2-chloroethane with resacetophenone in the presence of potassium carbonate in boiling acetone for 24 h (50%) [4212].
- Also refer to: [4211].

m.p. 96–98° [4212]; ^1H NMR [4212].

1-(2-Fluoro-3,4-dimethoxyphenyl)ethanone

[158641-45-3] $C_{10}H_{11}FO_3$ mol.wt. 198.19



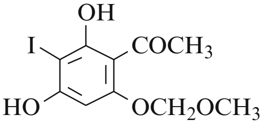
New compound

Synthesis

- Refer to: [3879].

1-[2,4-Dihydroxy-3-iodo-6-(methoxymethoxy)phenyl]ethanone

[321569-79-3] $C_{10}H_{11}IO_5$ mol.wt. 338.10



New compound

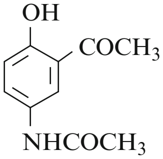
Synthesis

- Obtained by reaction of iodine and periodic acid mixture with 2,4-dihydroxy-6-methoxymethoxyacetophenone in dilute ethanol for 1 h at 40° (94%) [4213].

m.p. 162–164° [4213]; ^1H NMR [4213].

N-(3-Acetyl-4-hydroxyphenyl)acetamide

[7298-67-1] $C_{10}H_{11}NO_3$ mol.wt. 193.20



New compound

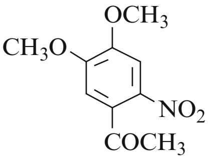
Synthesis

- Obtained by adding aluminium chloride to a suspension of N-(4-methoxyphenyl)acetamide and acetyl chloride then refluxing for 4.5 h (87%) [3911].

Pale green solid [3911];
^{1}H NMR [3911], ^{13}C NMR [3911], MS [3911].

1-(4,5-Dimethoxy-2-nitrophenyl)ethanone

[4101-32-0] $C_{10}H_{11}NO_5$ mol.wt. 225.20



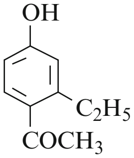
New compound

Synthesis

- Refer to: [4019].

1-(2-Ethyl-4-hydroxyphenyl)ethanone

[103323-98-4] $C_{10}H_{12}O_2$ mol.wt. 164.20



Described [3734] **p. 812**

Syntheses

- Also obtained by heating its methyl ether with pyridinium chloride for 15 min (61%) [3765].
- Also obtained by Fries rearrangement of 3-ethylphenyl acetate with aluminium chloride at 140° for 30 min (14%) [4214].

b.p.$_{11}$ 182–184° [4214]; m.p. 109–110° [4214].

2,4-Dinitrophenylhydrazone $C_{16}H_{16}N_4O_5$ mol.wt. 344.33

m.p. 206–207° [4214].

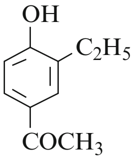
Methyl ether [41068-29-5] $C_{11}H_{14}O_2$ mol.wt. 178.23

- Obtained by reaction of acetic anhydride with 3-ethylanisole in the presence of aluminium chloride in carbon disulfide at r.t., then at 30–35° until no more hydrochloric acid is evolved [3796,3797].
- Also refer to: [3765].

^{1}H NMR [3796,3797], IR [3796,3797].

1-(3-Ethyl-4-hydroxyphenyl)ethanone

[22934-47-0] $C_{10}H_{12}O_2$ mol.wt. 164.20



Described [3734] **p. 813**

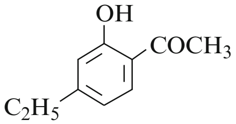
Methyl ether [29643-34-3] $C_{11}H_{14}O_2$ mol.wt. 178.23

- Obtained by reaction of acetyl chloride with 2-ethylanisole in the presence of aluminium chloride in carbon disulfide at r.t. overnight (45%) [4215].
- Also obtained by reaction of dimethyl sulfate with 3-ethyl-4-hydroxyacetophenone in the presence of aqueous sodium hydroxide [4216].
- Also obtained by Fries rearrangement of 2-ethylphenyl acetate in the presence of aluminium chloride in nitrobenzene [4216].
- Also refer to: [3778,4217,4218].

b.p.$_{0.5}$ 99–101° [4216], b.p.$_1$ 120° [4215], b.p.$_{25}$ 156–158° [4217];
IR [4217], UV [4217].

1-(4-Ethyl-2-hydroxyphenyl)ethanone

[5896-50-4] $C_{10}H_{12}O_2$ mol.wt. 164.20



Described [3734] **p. 813**

Syntheses

- Also obtained by Fries rearrangement of 3-ethylphenyl acetate (b.p.$_{21}$ 116–118°) with aluminium chloride at 140° for 30 min (77%) [4214] or at 130° for 2 h 30 min (97%) [3911].
- Also obtained by Friedel–Crafts acylation of m-ethylphenol with acetic acid in a mixture of graphite and methanesulfonic acid at 120° for 2.5 h (73%) [3889].
- Also refer to: [3912,4219,4220].

Brown oil [3911]; b.p.$_{13}$ 128–130° [4214];
^{1}H NMR [3889,3911], ^{13}C NMR [3889,3911], IR [3889], MS [3911].

Methyl ether $C_{11}H_{14}O_2$ mol.wt. 178.23

- Preparation by reaction of dimethyl sulfate with 4-ethyl-2-hydroxyacetophenone in the presence of aqueous sodium hydroxide solution (92%) [4221].

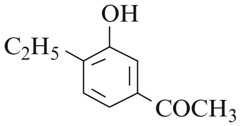
b.p.$_{15}$ 160° [4221].

2,4-Dinitrophenylhydrazone $C_{16}H_{16}N_4O_5$ mol.wt. 344.33

m.p. 211–212° [4214].

1-(4-Ethyl-3-hydroxyphenyl)ethanone

[73898-20-1] $C_{10}H_{12}O_2$ mol.wt. 164.20



Described [3734] **p. 814**

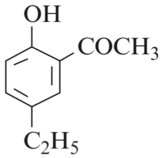
Synthesis

- Also refer to: [3905].

Methyl ether [947691-65-8] $C_{11}H_{14}O_2$ mol.wt. 178.23 [3905]

1-(5-Ethyl-2-hydroxyphenyl)ethanone

[24539-92-2] $C_{10}H_{12}O_2$ mol.wt. 164.20



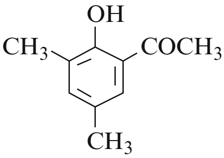
Described [3734] **p. 814**

Syntheses

- Also refer to: [3773,3887,4222].

1-(2-Hydroxy-3,5-dimethylphenyl)ethanone

[1198-66-9] $C_{10}H_{12}O_2$ mol.wt. 164.20



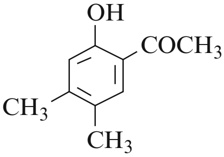
Described [3734] **p. 814**

Synthesis

- Also refer to: [3773].

1-(2-Hydroxy-4,5-dimethylphenyl)ethanone

[36436-65-4] $C_{10}H_{12}O_2$ mol.wt. 164.20



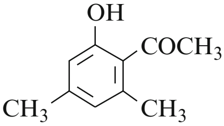
Described [3734] **p. 815**

Synthesis

- Also refer to: [4223].

1-(2-Hydroxy-4,6-dimethylphenyl)ethanone

[16108-50-2] $C_{10}H_{12}O_2$ mol.wt. 164.20



Described [3734] **p. 816**

Synthesis

- Also obtained by heating its methyl ether with pyridinium chloride for 15 min (51%) [3765].

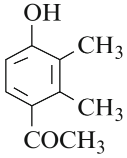
Methyl ether [21009-92-7] $C_{11}H_{14}O_2$ mol.wt. 178.23

- Obtained by reaction of acetyl chloride with 3,5-dimethylanisole in the presence of aluminium chloride in carbon disulfide at 25° [4224].
- Also obtained by reaction of dimethyl sulfate with 2-hydroxy-4,6-dimethylacetophenone in the presence of sodium hydroxide at 50° (60%) [4225].
- Also obtained by reaction of acetic anhydride with 3,5-dimethylanisole in the presence of aluminium chloride in carbon disulfide [4226].
- Also refer to: [3765,4227–4229].

b.p.$_{14}$ 135° [4224], b.p.$_{18}$ 140–144° [4226];
m.p. 48–49° [4224,4227];
^{1}H NMR [4225,4228,4229], IR [4225].

1-(4-Hydroxy-2,3-dimethylphenyl)ethanone

[5384-57-6] $C_{10}H_{12}O_2$ mol.wt. 164.20



Described [3734] **p. 818**

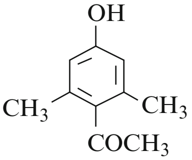
Syntheses

- Also obtained by heating its methyl ether with pyridinium chloride for 15 min (70%) [3765].
- Also refer to: [4230].

Methyl ether $C_{11}H_{14}O_2$ mol.wt. 178.23 [3765]

1-(4-Hydroxy-2,6-dimethylphenyl)ethanone

[91060-92-3] $C_{10}H_{12}O_2$ mol.wt. 164.20



Described [3734] **p. 818**

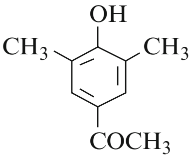
Methyl ether [60999-76-0] $C_{11}H_{14}O_2$ mol.wt. 178.23

- Obtained by treatment of 2,6-dimethyl-4-methoxybenzoyl chloride,
  - with dimethylcadmium in benzene [4231];
  - with methylmagnesium iodide [4231].
- Also obtained by treatment of 2-(1-ethoxyvinyl)-5-methoxy-1,3-dimethylbenzene with 50% sulfuric acid in dilute THF at r.t. [4232].
- Also obtained by reaction of dimethyl sulfate with 4-hydroxy-2,6-dimethylacetophenone in the presence of aqueous sodium hydroxide (84%) [4225].
- Also refer to: [4233,4234].

b.p.$_{0.08}$ 85–92° [4231]; m.p. 47° [4231], 46–48° [4234];
^{1}H NMR [4225,4234], ^{13}C NMR [3858], IR [4225,4234].

1-(4-Hydroxy-3,5-dimethylphenyl)ethanone

[5325-04-2] $C_{10}H_{12}O_2$ mol.wt. 164.20



Described [3734] **p. 818**

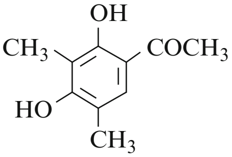
Methyl ether [60609-65-6] $C_{11}H_{14}O_2$ mol.wt. 178.23

- Obtained by reaction of dimethyl sulfate with 3,5-dimethyl-4-hydroxyacetophenone in aqueous sodium hydroxide solution [4235].
- Also obtained by reaction of acetyl chloride,
  - with 2-methoxy-1,3-dimethylbenzene in the presence of aluminium chloride in benzene [4236];
  - with 1-methoxy-2,6-dimethyl-4-tert-butylbenzene in the presence of aluminium chloride in nitromethane/methylene chloride mixture at r.t. for 12 h (80%) [4237].
- Preparation by the method of [4235], (77%) [4149].
- Also refer to: [3751–3756,4233,4238–4241].

b.p.$_1$ 105–110° [4241];
m.p. 47–48° [4235], 39.5–40.5° [4149], 39–40° [4236];
^{1}H NMR [3753,3756,4233,4241], ^{13}C NMR [3751,3754,4239], IR [4149,4240], UV [4240].

1-(2,4-Dihydroxy-3,5-dimethylphenyl)ethanone

[577-45-7] $C_{10}H_{12}O_3$ mol.wt. 180.20



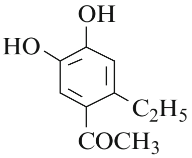
Described [3734] **p. 820**

Synthesis

- Also refer to: [4242].

1-(2-Ethyl-4,5-dihydroxyphenyl)ethanone

[267008-03-7] $C_{10}H_{12}O_3$ mol.wt. 180.20



New compound

Synthesis

- Obtained by treatment of 4,5-dimethoxy-2-ethyl-acetophenone with boron tribromide in methylene chloride at r.t. for 12 h (97%) [4243].

^{1}H NMR [4243], IR [4243].

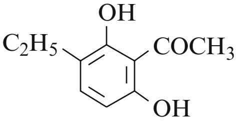
Dimethyl ether [105401-93-2] $C_{12}H_{16}O_3$ mol.wt. 208.26

- Obtained from 4-ethyl-1,2-dimethoxybenzene by treatment with,
  - acetyl chloride in the presence of ferric chloride in carbon disulfide [4244];
  - acetic anhydride in the presence of titanium tetrachloride (66%) [4243].
- Also refer to: [4174,4245,4246].

b.p.$_{10}$ 104–108° [4243];
m.p. 63° [4244], 62–63° [4245], 62° [4246]; ^{1}H NMR [4243], IR [4243].

1-(3-Ethyl-2,6-dihydroxyphenyl)ethanone

[54337-59-6] $C_{10}H_{12}O_3$ mol.wt. 180.20



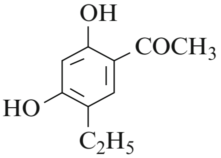
Described [3734] **p. 824**

Syntheses

- Also refer to: [3822,4033].

1-(5-Ethyl-2,4-dihydroxyphenyl)ethanone

[4460-42-8] $C_{10}H_{12}O_3$ mol.wt. 180.20



Described [3734] **p. 825**

Syntheses

- Also obtained by reaction of acetic acid with 4-ethyl-resorcinol in the presence of boron trifluoride etherate at 90° for 12 h under nitrogen (69%) [3829].
- Also refer to: [4247–4249].

Light-pink crystalline solid [3829]; m.p. 112° [4249];
^{1}H NMR [3829], MS [3829]; TLC [3829].
USE: Stabilization of PVC [4249].

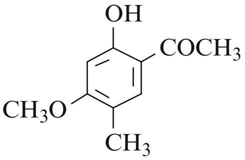
Dibenzyl ether [1001385-69-8] $C_{24}H_{24}O_3$ mol.wt. 360.45

- Obtained by reaction of benzyl bromide with 5-ethyl-2,4-dihydroxyacetophenone in the presence of potassium carbonate in DMF at r.t. for 3 h (70%) [3829].

Colourless solid [3829];
^{1}H NMR [3829], ^{13}C NMR [3829], MS [3829].

1-(2-Hydroxy-4-methoxy-5-methylphenyl)ethanone

[81511-52-6] $C_{10}H_{12}O_3$ mol.wt. 180.20



Described [3734] **p. 827**

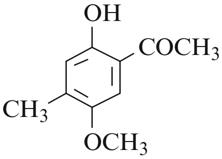
Synthesis

- Also obtained by reaction of acetic anhydride with N-(2,4-dimethoxybenzyl)-N-ethylethanamine in the presence of boron trifluoride etherate in methylene chloride (52%) [3830].

m.p. 64–65° [3830];
^{1}H NMR [3830], ^{13}C NMR [3830], MS [3830].

1-(2-Hydroxy-5-methoxy-4-methylphenyl)ethanone

[4223-84-1] $C_{10}H_{12}O_3$ mol.wt. 180.20



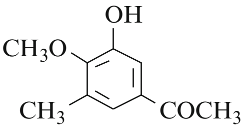
Described [3734] **p. 828**

Synthesis

- Also refer to: [4250].

1-(3-Hydroxy-4-methoxy-5-methylphenyl)ethanone

[741264-99-3] $C_{10}H_{12}O_3$ mol.wt. 180.20



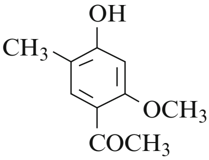
New compound

Synthesis

- Refer to: [3737].

1-(4-Hydroxy-2-methoxy-5-methylphenyl)ethanone

[868702-20-9] $C_{10}H_{12}O_3$ mol.wt. 180.20



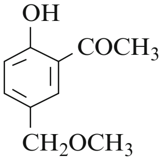
New compound

Synthesis

- Refer to: [3862].

1-[2-Hydroxy-5-(methoxymethyl)phenyl]ethanone

[60402-33-7] $C_{10}H_{12}O_3$ mol.wt. 180.20



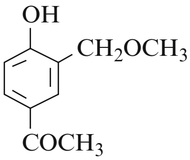
New compound

Synthesis

- Refer to: [3744].

1-[4-Hydroxy-3-(methoxymethyl)phenyl]ethanone

[65033-20-7] $C_{10}H_{12}O_3$ mol.wt. 180.20



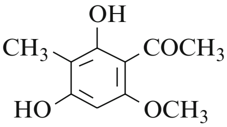
New compound

Syntheses

- Refer to: [4011,4251].

1-(2,4-Dihydroxy-6-methoxy-3-methylphenyl)ethanone

[83459-37-4] $C_{10}H_{12}O_4$ mol.wt. 196.20



Described [3734] **p. 831**

Syntheses

- Also refer to: [4193,4252].

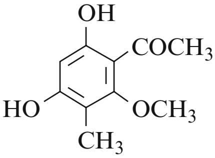
Isolation from natural sources

- From *Euphorbia ebracteolata* (Euphorbiaceae) [4253,4254].
- From the roots of *Euphorbia kansui* (Euphorbiaceae) [4255].
- From *Stellera chamaejasme* [4253].

^{1}H NMR [4193,4255], ^{13}C NMR [4193,4255],
UV [4253], MS [4253]; HPLC [4253], LC [4253].

1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)ethanone

[52200-61-0] $C_{10}H_{12}O_4$ mol.wt. 196.20



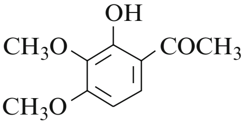
Described [3734] **p. 832**

Isolation from natural sources

- From the Lichen *Psoroma leprolomum* (compound **8**) [4193].

1-(2-Hydroxy-3,4-dimethoxyphenyl)ethanone

[5396-18-9] $C_{10}H_{12}O_4$ mol.wt. 196.20



Described [3734] **p. 834**

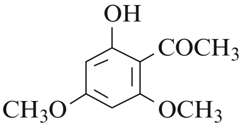
Syntheses

- Also obtained by action of hydrogen bromide with 2,3,4-trimethoxyacetophenone in acetic acid at r.t. (71%) [3984].
- Also refer to: [3810,4011,4042].

m.p. 65.5–71.5° [3984].

1-(2-Hydroxy-4,6-dimethoxyphenyl)ethanone

[90-24-4] $C_{10}H_{12}O_4$ mol.wt. 196.20



Described [3734] **p. 837**

Syntheses

- Also obtained by reaction of dimethyl sulfate with phloroacetophenone in the presence of potassiumcarbonate in refluxing acetone (97%) [4071] for 5 h (36%) [4188].
- Also obtained by action of hydrogen bromide with 2,4,6-trimethoxyacetophenone in acetic acid at r.t. (27%) [3984].
- Also refer to: [4205,4256,4257].

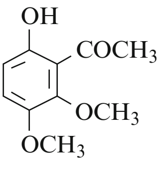
m.p. 80–82° [4188], 79–82° [3984], 78° [4071].

Acetate $C_{12}H_{14}O_5$ mol.wt. 238.24

m.p. 105–108° [3984].

1-(6-Hydroxy-2,3-dimethoxyphenyl)ethanone

[22248-13-1] $C_{10}H_{12}O_4$ mol.wt. 196.20



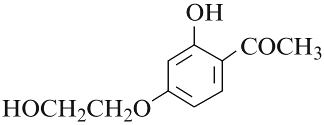
Described [3734] **p. 841**

Synthesis

- Also refer to: [4042].

1-[2-Hydroxy-4-(2-hydroxyethoxy)phenyl]ethanone

[17086-21-4] $C_{10}H_{12}O_4$ mol.wt. 196.20



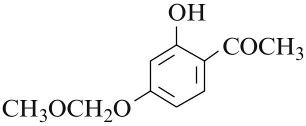
New compound

Synthesis

- Refer to: [4258].

1-[2-Hydroxy-4-(methoxymethoxy)phenyl]ethanone

[65490-08-6] $C_{10}H_{12}O_4$ mol.wt. 196.20



Described [3734] **p. 842**

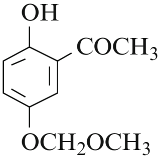
Syntheses

- Also obtained by reaction of methoxymethyl chloride with resacetophenone in the presence of potassium carbonate in acetone at r.t. for 24 h (87%) [4259].
- Also refer to: [4260].

m.p. 38–39° [4259];
^{1}H NMR [4259], ^{13}C NMR [4259], IR [4259], MS [4259].

1-[2-Hydroxy-5-(methoxymethoxy)phenyl]ethanone

[31405-69-3] $C_{10}H_{12}O_4$ mol.wt. 196.20



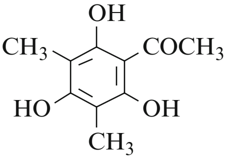
New compound

Synthesis

- Refer to: [4261].

1-(2,4,6-Trihydroxy-3,5-dimethylphenyl)ethanone

[13383-63-6] $C_{10}H_{12}O_4$ mol.wt. 196.20



Described [3734] **p. 843**

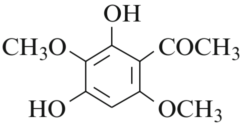
Synthesis

- Also refer to: [4194].

UV [4194].

1-(2,4-Dihydroxy-3,6-dimethoxyphenyl)ethanone

[7499-99-2] $C_{10}H_{12}O_5$ mol.wt. 212.20



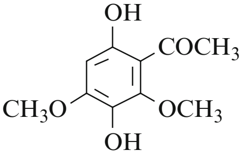
Described [3734] **p. 844**

Syntheses

- Also refer to: [4262,4263].

1-(3,6-Dihydroxy-2,4-dimethoxyphenyl)ethanone

[6962-57-8] $C_{10}H_{12}O_5$ mol.wt. 212.20



Described [3734] **p. 845**

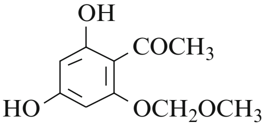
Syntheses

- Obtained by treatment of 2-hydroxy-4,6-dimethoxyacetophenone with an aqueous solution of potassium persulfate in 10% sodium hydroxide (58%) [4071].
- Also refer to: [4264].

m.p. 164° [4071].

1-[2,4-Dihydroxy-6-(methoxymethoxy)phenyl]ethanone

[71386-98-6] $C_{10}H_{12}O_5$ mol.wt. 212.20



New compound

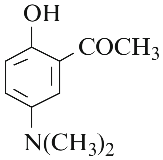
Synthesis

- Obtained from palladium-carbon-catalyzed hydrogenolysis of 2,4-bis(benzyloxy)-6-methoxymethoxyacetophenone in a mixture of methanol and ethyl acetate at 20° (95%) [4213].

m.p. 117–119° [4213]; ^{1}H NMR [4213].

1-[5-(Dimethylamino)-2-hydroxyphenyl]ethanone

[49619-68-3] $C_{10}H_{13}NO_2$ mol.wt. 179.22



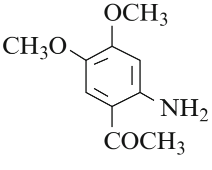
Described [3734] **p. 847**

Methyl ether $C_{11}H_{15}NO_2$ mol.wt. 193.25

- Refer to: [3858]; ^{13}C NMR [3858].

1-(2-Amino-4,5-dimethoxyphenyl)ethanone

[4101-30-8] $C_{10}H_{13}NO_3$ mol.wt. 195.25



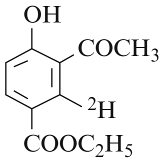
New compound

Syntheses

- Refer to: [4019,4265].

Ethyl 3-acetyl-2-deuterio-4-hydroxybenzoate

[78515-06-7] $C_{11}H_{11}DO_4$ mol.wt. 209.21



New compound

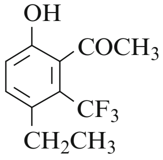
Synthesis

- Obtained by reaction of 5-formyl-1,3-dimethyluracil-d1 with 2,4-pentanedione in the presence of sodium ethoxide in ethanol on heating for 3 h [4098].

m.p. 65–67° [4098]; ^{1}H NMR [4098], UV [4098].

1-[3-Ethyl-6-hydroxy-2-(trifluoromethyl)phenyl]ethanone

[884851-64-3] $C_{11}H_{11}F_3O_2$ mol.wt. 232.20



New compound

Synthesis

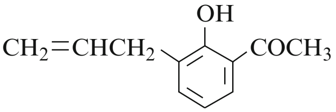
- To a CH_2Cl_2 solution of 1,3-bis-silyl enol ether ($R^3 = CH_3$) and 4-ethoxy-3-ethyl-1,1,1-trifluoro-3-buten-2-one was added $TiCl_4$ at −78° under argon atmosphere.

The temperature of the reaction mixture was allowed to rise to 20° during 14 h and, subsequently, an aqueous solution of 10% HCl was added (40%) [4202].

^{1}H NMR [4202], ^{13}C NMR [4202], ^{19}F NMR [4202], IR [4202]; TLC [4202].

1-[2-Hydroxy-3-(2-propen-1-yl)phenyl]ethanone

[58621-39-9] $C_{11}H_{12}O_2$ mol.wt. 176.22



Described [3734] **p. 850**

Syntheses

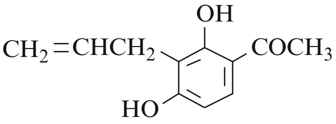
- Also obtained by thermal Claisen rearrangement of 2-(allyloxy)acetophenone,

- by heating at 200° for 44 h (quantitative yield) [3911];
- by irradiation in a microwave set at 230 W and 210° for 1 h with simultaneous air cooling (quantitative yield) [3911].

^{1}H NMR [3911], ^{13}C NMR [3911], MS [3911].

1-[2,4-Dihydroxy-3-(2-propen-1-yl)phenyl]ethanone

[38987-00-7] $C_{11}H_{12}O_3$ mol.wt. 192.21



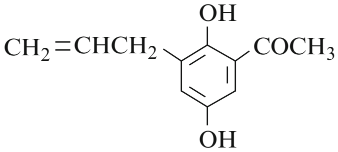
Described [3734] **p. 852**

Syntheses

- Also refer to: [4155,4266].

1-[2,5-Dihydroxy-3-(2-propen-1-yl)phenyl]ethanone

[956525-45-4] $C_{11}H_{12}O_3$ mol.wt. 192.21



New compound

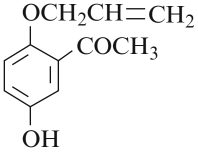
Synthesis

– Refer to: [3895].

Oxime [956525-49-8] $C_{11}H_{14}NO_4$ mol.wt. 224.24 [3895]

1-[5-Hydroxy-2-(2-propen-1-yloxy)phenyl]ethanone

[956525-48-7] $C_{11}H_{12}O_3$ mol.wt. 192.21



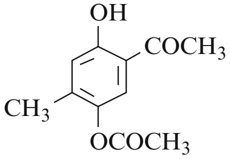
New compound

Synthesis

– Refer to: [3895].

1-[5-(Acetyloxy)-2-hydroxy-4-methylphenyl]ethanone

[126570-32-9] $C_{11}H_{12}O_4$ mol.wt. 208.21



Described [3734] **p. 854**

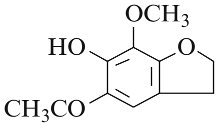
Synthesis

– Also obtained by Fries rearrangement of 2-methylhydroquinone diacetate with aluminium chloride at 120° for 15 min [4165].

m.p. 108° [4165].

1-(2,3-Dihydro-6-hydroxy-7-methoxy-5-benzofuranyl)ethanone

[88897-94-3] $C_{11}H_{12}O_4$ mol.wt. 208.21



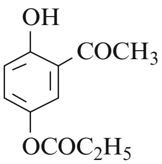
New compound

Synthesis

– Refer to: [4267].

1-[2-Hydroxy-5-(1-oxopropoxy)phenyl]ethanone

[1004985-99-2] $C_{11}H_{12}O_4$ mol.wt. 208.21



New compound

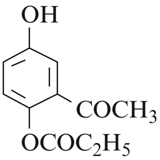
Synthesis

– Obtained by direct acylation of quinacetophenone with vinyl propionate in the presence of *Candida antarctica* lipase B (CAL-B) in diisopropyl ether at 45° for 1 day (73%) [4268].

^{1}H NMR [4268], ^{13}C NMR [4268].

1-[5-Hydroxy-2-(1-oxopropoxy)phenyl]ethanone

[1004986-06-4] $C_{11}H_{12}O_4$ mol.wt. 208.21



New compound

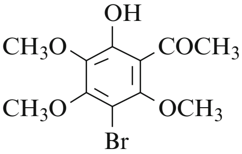
Synthesis

- Obtained by direct acylation of quinacetophenone with vinyl propionate in the presence of *Candida antarctica* lipase B (CAL-B) in diisopropyl ether at 45° for 1 day (8%) [4268].

^{1}H NMR [4268], ^{13}C NMR [4268].

1-(5-Bromo-2-hydroxy-3,4,6-trimethoxyphenyl)ethanone

$C_{11}H_{13}BrO_5$ mol.wt. 305.13



New compound

Synthesis

- Obtained by bromination of 2-hydroxy-3,4,6-trimethoxy-acetophenone with N-bromosuccinimide [4206].

m.p. 83° [4206].

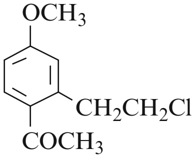
Methyl ether [92905-07-2] $C_{12}H_{15}BrO_5$ mol.wt. 397.13

- Obtained by treatment of 2-hydroxy-3,4,6-trimethoxyacetophenone with dimethyl sulfate in the presence of potassium carbonate in boiling acetone [4206].
- Also obtained by treatment of 2,4-dihydroxy-3,6-dimethoxyacetophenone with dimethyl sulfate in the presence of potassium carbonate in boiling acetone [4206].

m.p. 51° [4206].

1-[2-(2-Chloroethyl)-4-methoxyphenyl]ethanone

[960592-54-5] $C_{11}H_{13}ClO_2$ mol.wt. 198.65



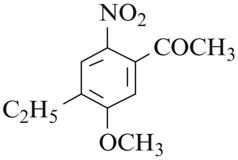
New compound

Synthesis

- Refer to: [4269].

1-(4-Ethyl-5-methoxy-2-nitrophenyl)ethanone

[947691-66-9] $C_{11}H_{13}NO_4$ mol.wt. 223.23



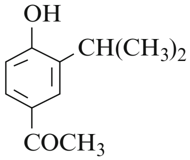
New compound

Synthesis

- Refer to: [3905].

1-[4-Hydroxy-3-(1-methylethyl)phenyl]ethanone

[1632-59-3] $C_{11}H_{14}O_2$ mol.wt. 178.23



Described [3734] **p. 867**

Synthesis

- Also obtained by heating its methyl ether with pyridinium chloride for 15 min (67%) [3765].

Methyl ether [1634-64-6] $C_{12}H_{16}O_2$ mol.wt. 192.26

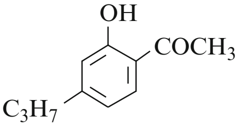
- Obtained by reaction of acetyl chloride with 2-isopropylanisole in the presence of aluminium chloride in carbon disulfide [4270], (64%) [4184].
- Also obtained by using the method [4271], (66%) [4272].
- Also refer to: [3765,4273].

b.p.$_{0.2}$ 108° [4184], b.p.$_{21}$ 165–166° [4270], b.p.$_{37}$ 170–178° [4272];

n_D^{23} = 1.5385 [4270]; m.p. 50–51° [4184].

1-(2-Hydroxy-4-propylphenyl)ethanone

[104175-20-4] $C_{11}H_{14}O_2$ mol.wt. 178.23



Described [3734] **p. 868**

Synthesis

- Also obtained by Fries rearrangement of 3-propylphenyl acetate (b.p.$_{16}$ 128–129°) with aluminium chloride at 130–135° for 90 min (78%) [4214].

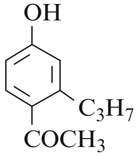
b.p.$_{18}$ 145–146° [4214].

2,4-Dinitrophenylhydrazone $C_{17}H_{18}N_4O_5$ mol.wt. 358.35

m.p. 198–199° [4214].

1-(4-Hydroxy-2-propylphenyl)ethanone

[104174-27-8] $C_{11}H_{14}O_2$ mol.wt. 178.23



Described [3734] **p. 868**

Synthesis

- Also obtained by Fries rearrangement of 3-propylphenyl acetate (b.p.$_{16}$ 128–129°) with aluminium chloride at 130–135° for 90 min (2%) [4214].

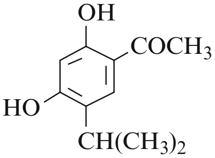
b.p.$_{760}$ 332–334° [4214]; m.p. 75–76° [4214].

2,4-Dinitrophenylhydrazone $C_{17}H_{18}N_4O_5$ mol.wt. 358.35

m.p. 163–164° [4214].

1-[2,4-Dihydroxy-5-(1-methylethyl)phenyl]ethanone

[747414-17-1] $C_{11}H_{14}O_3$ mol.wt. 194.23



New compound

Syntheses

- Obtained by reaction of acetic acid with 4-isopropylresorcinol in the presence of boron trifluoride etherate at 90° for 16 h (88%) [3829].
- Also refer to: [3870–3874].

Off-white solid [3829]; ^{1}H NMR [3829], MS [3829].

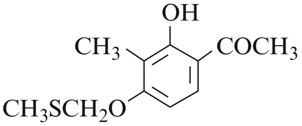
Dibenzyl ether [747414-18-2] $C_{25}H_{26}O_3$ mol.wt. 374.48

- Obtained by reaction of benzyl bromide with 5-isopropylresacetophenone in the presence of potassium carbonate in DMF at 150° for 16 h under nitrogen (88%) [3829].
- Also refer to: [3870–3874].

Colourless solid [3829]; ^{1}H NMR [3829], MS [3829].

1-[2-Hydroxy-3-methyl-4-[(methylthio)methoxy]phenyl]ethanone

[942133-87-1] $C_{11}H_{14}O_3S$ mol.wt. 226.30



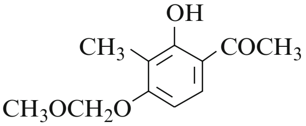
New compound

Synthesis

- Obtained by treatment of 2,4-dihydroxy-3-methyl-acetophenone with chloromethyl methyl sulfide in the presence of KOH/K_2CO_3 mixture in boiling 2-butanone [4274].

1-[2-Hydroxy-4-(methoxymethoxy)-3-methylphenyl]ethanone

[942133-85-9] $C_{11}H_{14}O_4$ mol.wt. 210.23



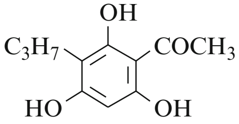
New compound

Synthesis

- Obtained by treatment of 2,4-dihydroxy-3-methyl-acetophenone with chloromethyl methyl ether in the presence of KOH/K_2CO_3 mixture in boiling 2-butanone [4274].

1-(2,4,6-Trihydroxy-3-propylphenyl)ethanone

[96756-28-4] $C_{11}H_{14}O_4$ mol.wt. 210.23



New compound

Synthesis

- Obtained by treatment of its trimethyl ether with boron tribromide in methylene chloride at −78°, then at r.t. for 24–48 h (77%) [4192].

^{1}H NMR [4192], ^{13}C NMR [4192], MS [4192].

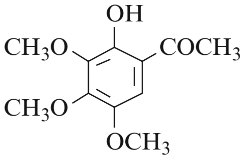
Trimethyl ether [916916-57-9] $C_{14}H_{20}O_4$ mol.wt. 252.31

- Obtained by reaction of acetyl chloride with 2,4,6-trimethoxy-n-propylbenzene in methylene chloride in the presence of stannic chloride at −10° for 2–3 h (80%) [4192].

^{1}H NMR [4192], ^{13}C NMR [4192], MS [4192].

1-(2-Hydroxy-3,4,5-trimethoxyphenyl)ethanone

[30225-96-8] $C_{11}H_{14}O_5$ mol.wt. 226.23



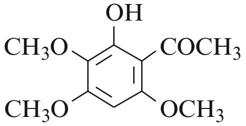
Described [3734] **p. 888**

Synthesis

- Also obtained by Friedel–Crafts reaction of 3,4,5-trimethoxy-phenol and acetic anhydride using zinc chloride in nitromethane (91%) [4275].

1-(2-Hydroxy-3,4,6-trimethoxyphenyl)ethanone

[7507-98-4] $C_{11}H_{14}O_5$ mol.wt. 226.23



Described [3734] **p. 888**

Syntheses

- Also obtained by reaction of acetyl chloride with 1,2,3,5-tetra-methoxybenzene in the presence of aluminium chloride in ethyl ether (55%) [4071] under argon, at 0° for 3 h, then at r.t. overnight (56%) [4276].

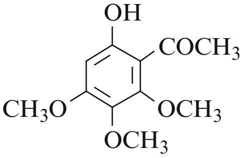
Isolation from natural sources

- From liverwort *Adelanthus decipiens* [4074].
- From liverwort *Plagiochila fasciculata* Lindenb. [4277].

m.p. 114–115° [4276], 112–113° [4071];
^{13}C NMR [4276,4278], NOE [4074].

1-(6-Hydroxy-2,3,4-trimethoxyphenyl)ethanone

[22248-14-2] $C_{11}H_{14}O_5$ mol.wt. 226.23



Described [3734] **p. 891**

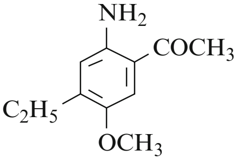
Syntheses

- Also obtained by reaction of dimethyl sulfate with 2,3,4,6-tetrahydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 3 h (41%) [4063].
- Also refer to: [4071].

m.p. 76° [4063];
^{1}H NMR [4063], ^{13}C NMR [4063], IR [4063], UV [4063], MS [4063]; TLC [4063].

1-(2-Amino-4-ethyl-5-methoxyphenyl)ethanone

[947691-67-0] $C_{11}H_{15}NO_2$ mol.wt. 193.25



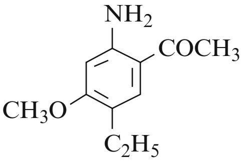
New compound

Syntheses

- Refer to: [3905,4279].

1-(2-Amino-5-ethyl-4-methoxyphenyl)ethanone

[947691-62-5] $C_{11}H_{15}NO_2$ mol.wt. 193.25



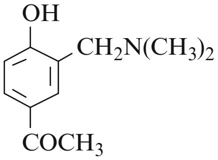
New compound

Syntheses

- Refer to: [3905,4279].

1-[3-[(Dimethylamino)methyl]-4-hydroxyphenyl]ethanone

[73096-98-7] $C_{11}H_{15}NO_2$ mol.wt. 193.25



New compound

Syntheses

- Obtained by adding dimethylamine to a solution of 3-chloromethyl-4-hydroxyacetophenone in benzene in the cold. Stirring was continued for 4–5 h, then treatment of the hydrochloride so obtained with sodium bicarbonate (60%) [4120].
- Also obtained by adding an aqueous solution of 37% formaldehyde to 4-hydroxyacetophenone and dimethylamine in dilute ethanol, then refluxing for 22 h (72%) [4280].
- Also obtained from 4-hydroxyacetophenone using Mannich reaction (55%) [4281].

- Also obtained by reaction of dimethylmethyliminium iodide with 4-hydroxyacetophenone in the presence of,
  - potassium carbonate in methylene chloride at r.t. for 7 h (85%) [4282];
  - carbonate exchange resin in methylene chloride at r.t. for 16 h (97%) [4283].
- Also refer to: (55%) [4281].

m.p. 74° [4282], 73° [4281], 72° [4120], 68° [4280];
^{1}H NMR [4120,4281,4282], IR [4120], MS [4120,4282]; pK$_a$ [4284].
BIOLOGICAL ACTIVITY: Insecticide [4120,4154].

Hydrochloride [91246-57-0] $C_{11}H_{15}NO_2$, HCl mol.wt. 229.71

m.p. 174–175° [4281], 170° [4280].

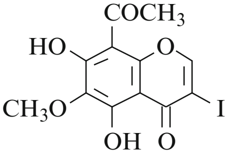
Methyl ether $C_{12}H_{17}NO_2$ mol.wt. 207.27

- A solution of (5-bromo-2-methoxybenzyl)dimethylamine, butoxyethene, palladium diacetate, 1,3-bis(diphenylphosphino)propane and potassium carbonate in dimethylformamide under argon was heated at 80° overnight. The mixture was poured into a solution of 2 N aqueous hydrochloric acid and stirred for 1 h. The solution was adjusted to basic pH using a solution of 2 N aqueous sodium hydroxide (42%) [4285].

Orange oil [4285]; ^{1}H NMR [4285]; GC-MS [4285].

8-Acetyl-5,7-dihydroxy-3-iodo-6-methoxy-4*H*-1-benzopyran-4-one

[870480-53-8] $C_{12}H_9IO_6$ mol.wt. 376.10



New compound

Synthesis

- Obtained by treatment of its dimethyl ether with titanium tetrachloride in toluene, first at r.t, then at 100° for 1 h (73%) [4275].

Colourless crystals; m.p. 224–226° [4275];
^{1}H NMR [4275], MS [4275].

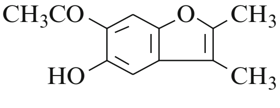
Dimethyl ether [870480-17-4] $C_{14}H_{13}IO_6$ mol.wt. 404.16

- Obtained by treatment of (*E*)-3′-acetyl-3-(dimethylamino)-2′-hydroxy-4′,5′,6′-trimethoxy-acrylophenone (m.p. 113–114°) with iodine in methylene chloride at r.t. for 2 h (83%) [4275].

m.p. 139–140° [4275]; ^{1}H NMR [4275], MS [4275].

1-(5-Hydroxy-2,3-dimethyl-6-benzofuranyl)ethanone

$C_{12}H_{12}O_3$ mol.wt. 204.23



New compound

Synthesis

- Also obtained by heating its methyl ether with pyridinium chloride for 30 min (71%) [3765].

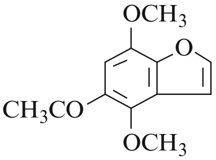
Methyl ether [4223-70-5] $C_{13}H_{14}O_3$ mol.wt. 218.25.

- Refer to: [3765,4286].

b.p.$_{12}$ 191–192° [4286]; m.p. 114° [4286].

1-(4,7-Dimethoxy-5-benzofuranyl)ethanone

[1025008-58-5] $C_{12}H_{12}O_4$ mol.wt. 220.22



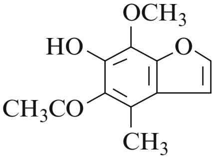
New compound

Synthesis

- Obtained from 5-acetyl-4,7-dimethoxy-6-benzofuranyl 1,1,1-trifluoromethanesulfonate [1025008-57-4], (89%) [4267].

1-(6-Hydroxy-7-methoxy-4-methyl-5-benzofuranyl)ethanone

[1017961-46-4] $C_{12}H_{12}O_4$ mol.wt. 220.22



New compound

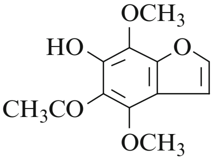
Synthesis

- Refer to: [4287].

USE: As potassium channel blockers [4287].

1-(6-Hydroxy-4,7-dimethoxy-5-benzofuranyl)ethanone *(Khellinone)*

[484-51-5] $C_{12}H_{12}O_5$ mol.wt. 236.22



New compound

Syntheses

- Obtained by slowly adding 3 M sodium hydroxide to a solution of 4,9-dimethoxy-7-methylfuro[3,2-g] chromen-5-one in refluxing dilute ethanol, then the resulting solution was stirred at 70° for 3 h [4288].
- Also obtained by treatment of 5-(β-dimethylaminocrotonyl)-4,7-dimethoxy-6-hydroxycoumarone with 3% sodium hydroxide for 30 min at r.t. [4289].
- Also refer to: [4287], **7** [4267].

m.p. 98–99° [4289].

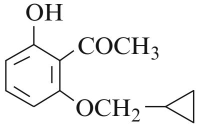
USE: As potassium channel blockers [4287].

Phenylhydrazone $C_{18}H_{18}N_2O_4$ mol.wt. 326.35

m.p. 167–168° [4289].

1-[(2-Cyclopropylmethoxy)-6-hydroxyphenyl]ethanone

[405239-70-5] $C_{12}H_{14}O_3$ mol.wt. 206.24



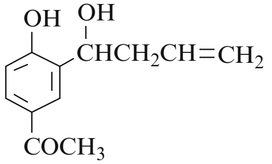
New compound

Synthesis

- Preparation by reaction of (bromomethyl)cyclopropane with 2,6-dihydroxyacetophenone in the presence of potassium carbonate in acetone at 50° for 2 days (83%) [4006].

1-[4-Hydroxy-3-(1-hydroxy-3-buten-1-yl)phenyl]ethanone

[1000781-23-6] $C_{12}H_{14}O_3$ mol.wt. 206.24



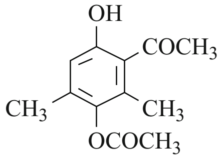
New compound

Synthesis

- Refer to: [4290].

1-[3-(Acetyloxy)-6-hydroxy-2,4-dimethylphenyl]ethanone

[334868-41-6] $C_{12}H_{14}O_4$ mol.wt. 222.24

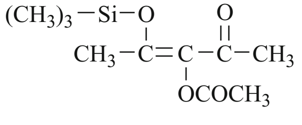


New compound

Syntheses

- Obtained by [3+3] cyclisation of 2-acetoxy-3-(silyloxy)alk-2-en-1-one (**4a**) with 1,3-bis-silyl enol ether (**5b**) in the presence of titanium tetrachloride in methylene chloride, first at −78°, then to 20° for 20 h (55%) [4207], (42%) [4291].
  Yellow crystals [4291];
  ^{1}H NMR [4291], ^{13}C NMR [4291], IR [4291], MS [4291].

4a = 3-(acetyloxy)-4-(trimethylsilyloxy)-3-penten-2-one.



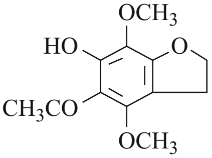
$$\mathrm{CH_3-\overset{\overset{(CH_3)_3-Si-O}{|}}{C}=\underset{\underset{OCOCH_3}{|}}{C}-\overset{\overset{O}{||}}{C}-CH_3}$$

5b = 2,4-bis(trimethylsilyloxy)-2-pentene.

$$\mathrm{CH_3-\overset{\overset{(CH_3)_3-Si-O}{|}}{C}H-CH=\overset{\overset{O-Si-(CH_3)_3}{|}}{C}-CH_3}$$

1-(2,3-Dihydro-6-hydroxy-4,7-dimethoxy-5-benzofuranyl)ethanone

[6938-22-3] $C_{12}H_{14}O_5$ mol.wt. 238.24



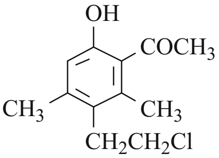
New compound

Synthesis

- Obtained by treatment of Khellinone in methanol with hydrogen in the presence of 10% Pd/C (86%) [4267].

1-[3-(2-Chloroethyl)-2,4-dimethyl-6-hydroxyphenyl]ethanone

$C_{12}H_{15}ClO_2$ mol.wt. 226.70



New compound

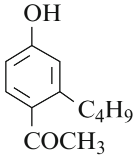
Syntheses

- Preparation by adding titanium tetrachloride in methylene chloride to a methylene chloride solution of 1,1-diacetyl-cyclopropane and 1-methyl-1, 3-bis (trimethylsilyloxy)-1,3-butadiene at −78° under argon atmosphere in the presence of molecular sieves (4 Å). The temperature of the reaction mixture was allowed to rise to 20° over 6 h, and stirred for an additional 6 h at 20° (68%) [4292], (82%) [4293].

^{1}H NMR [4293], ^{13}C NMR [4293], IR [4293], MS [4293]; TLC [4293].

1-(2-Butyl-4-hydroxyphenyl)ethanone

[105337-80-2] $C_{12}H_{16}O_2$ mol.wt. 192.26



New compound

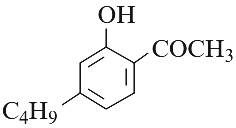
Synthesis

- Obtained by Fries rearrangement of 3-butylphenyl acetate (b.p.$_{12}$ 130–131°) with aluminium chloride at 130–135° for 60 min (9%) [4214].

b.p.$_{34}$ 226–230° [4214]; m.p. 61–62° [4214].

1-(4-Butyl-2-hydroxyphenyl)ethanone

[105337-19-7] $C_{12}H_{16}O_2$ mol.wt. 192.26



New compound

Synthesis

- Obtained by Fries rearrangement of 3-butylphenyl acetate (b.p.$_{12}$ 130–131°) with aluminium chloride at 130–135° for 60 min (73%) [4214].

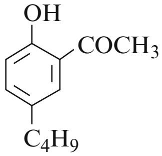
b.p.$_{34}$ 180–182° [4214].

2,4-Dinitrophenylhydrazone $C_{18}H_{20}N_4O_5$ mol.wt. 372.38

m.p. 187–188° [4214].

1-(5-Butyl-2-hydroxyphenyl)ethanone

[50743-14-1] $C_{12}H_{16}O_2$ mol.wt. 192.26

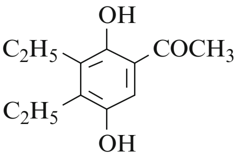


Described [3734] **p. 910**

Methyl ether [784177-14-6] $C_{13}H_{18}O_2$ mol.wt. 206.28 Refer to: [4294].

1-(3,4-Diethyl-2,5-dihydroxyphenyl)ethanone

[873222-91-4] $C_{12}H_{16}O_3$ mol.wt. 208.26



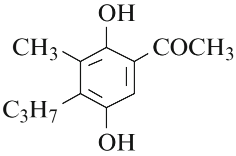
New compound

Synthesis

- Obtained by $[Cp^*RuCl_2]_2$-catalyzed cocyclisation of 3-hexyne (1,2-diethylacetylene), 3-buten-2-one and carbon monoxide in DMF at 140° for 20 h (60%) [4295].

1-(2,5-Dihydroxy-3-methyl-4-propylphenyl)ethanone

[873222-93-6] $C_{12}H_{16}O_3$ mol.wt. 208.26



New compound

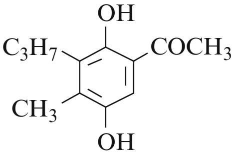
Synthesis

- Obtained by $[Cp^*RuCl_2]_2$-catalyzed cocyclisation of 2-hexyne, 3-buten-2-one and carbon monoxide in DMF at 140° for 20 h (27%) [4295].

^1H NMR [4295].

1-(2,5-Dihydroxy-4-methyl-3-propylphenyl)ethanone

[873222-92-5] $C_{12}H_{16}O_3$ mol.wt. 208.26



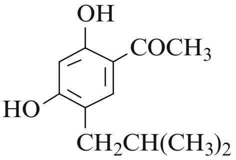
New compound

Synthesis

- Obtained by $[Cp^*RuCl_2]_2$-catalyzed cocyclisation of 2-hexyne, 3-buten-2-one and carbon monoxide in DMF at 140° for 20 h (35%) [4295].

^1H NMR [4295].

1-[2,4-Dihydroxy-5-(2-methylpropyl)phenyl]ethanone *(6-Acetyl-4-isobutylresorcinol)*



$C_{12}H_{16}O_3$ mol.wt. 208.26

New compound

Synthesis

- Obtained by reaction of acetic acid with 4-isobutylresorcinol in the presence of boron trifluoride etherate at 90° for 16 h [3829].

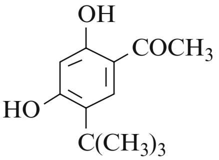
Dibenzyl ether $C_{26}H_{28}O_3$ mol.wt. 388.51

- Obtained by reaction of benzyl bromide with 5-isobutylresacetophenone in the presence of potassium carbonate in DMF under nitrogen at 150° for 16 h (25%) [3829].

Colourless crystals [3829]; ^{1}H NMR [3829], MS [3829].

1-[5-(1,1-Dimethylethyl)-2,4-dihydroxyphenyl]ethanone

[140660-31-7] $C_{12}H_{16}O_3$ mol.wt. 208.26



Described [3734] **p. 924**

Synthesis

- Also obtained by adding sulfuric acid to a suspension of resacetophenone in a mixture of 2-methyl-2-propanol and trifluoroacetic acid under nitrogen.

The resulting suspension was heated at 75° for 3 h (92%) [3829].

Pale orange powder [3829]; ^{1}H NMR [3829], MS [3829].

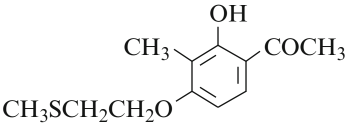
Dibenzyl ether [747414-06-8] $C_{26}H_{28}O_3$ mol.wt. 388.51

- Obtained by treatment of 5-tert-butylresacetophenone with benzyl bromide in the presence of potassium carbonate in DMF at r.t. for 4 h (36%) [3829].
- Also refer to: [3870–3874].

Pale-pink powder [3829]; ^{1}H NMR [3829], MS [3829].

1-[2-Hydroxy-3-methyl-4-[2-(methylthio)ethoxy]phenyl]ethanone

[942133-89-3] $C_{12}H_{16}O_3S$ mol.wt. 240.32



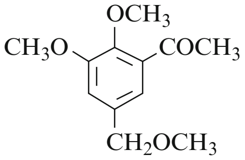
New compound

Synthesis

- Obtained by treatment of 2,4-dihydroxy-3-methyl-acetophenone with 2-chloroethyl methyl sulfide in the presence of KOH/K_2CO_3 mixture in boiling 2-butanone [4274].

1-[2,3-Dimethoxy-5-(methoxymethyl)phenyl]ethanone

[1004984-76-2] $C_{12}H_{16}O_4$ mol.wt. 224.26



New compound

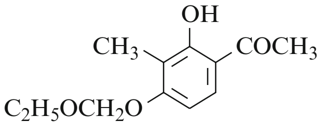
Synthesis

- Obtained by adding a solution of 1-(2,3-dimethoxy-5-methoxymethylphenyl)ethanol in methylene chloride to a suspension of PCC and silica gel in methylene chloride and stirring for 2 h at r.t. (98%) [4296].

Oil [4296];
^{1}H NMR [4296], ^{13}C NMR [4296], IR [4296], MS [4296].

1-[4-(Ethoxymethoxy)-2-hydroxy-3-methylphenyl]ethanone

[942133-88-2] $C_{12}H_{16}O_4$ mol.wt. 224.26



New compound

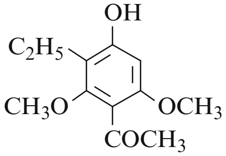
Synthesis

- Obtained by treatment of 2,4-dihydroxy-3-methyl-acetophenone with 2-chloroethyl methyl ether in the presence of KOH/K_2CO_3 mixture in boiling 2-butanone [4274].

1-(3-Ethyl-4-hydroxy-2,6-dimethoxyphenyl)ethanone

$C_{12}H_{16}O_4$ mol.wt. 224.26

Described [3734] **p. 929**



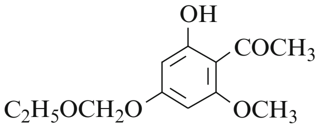
Acetate $C_{14}H_{18}O_5$ mol.wt. 266.29

- Obtained by reaction of acetic anhydride with 3-ethyl-4-hydroxy-2,6-dimethoxyacetophenone (80%) [4297].

b.p.$_{0.005}$ 180–200° [4297]; m.p. 59–60° [4297].

1-[4-(Ethoxymethoxy)-2-hydroxy-6-methoxyphenyl]ethanone

[158017-91-5] $C_{12}H_{16}O_5$ mol.wt. 240.26



New compound

Synthesis

- Obtained by reaction of ethoxymethyl chloride with 2,4-dihydroxy-6-methoxyacetophenone in the presence of potassium carbonate in acetone for 5–10 min [4298].

m.p. 95° [4298];
^{1}H NMR [4298], IR [4298], UV [4298]; LC [4298].

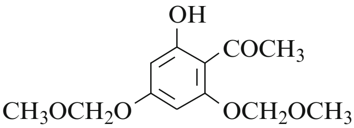
Methyl ether [158017-92-6] $C_{13}H_{18}O_5$ mol.wt. 254.28

- Obtained by reaction of dimethyl sulfate with 4-ethoxymethoxy-2-hydroxy-6-methoxy-acetophenone in the presence of potassium carbonate in refluxing acetone for 8 h (79%) [4298].
  Colourless oil [4298];

  ^{1}H NMR [4298], IR [4298]; TLC [4298].

1-[2-Hydroxy-4,6-bis(methoxymethoxy)phenyl]ethanone

[65490-09-7] $C_{12}H_{16}O_6$ mol.wt. 256.26



Described [3734] **p. 935**

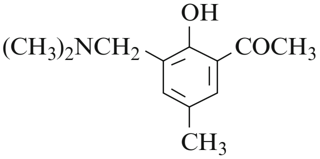
Synthesis

- Also obtained by reaction of chloromethyl methyl ether with phloroacetophenone monohydrate in the presence of potassium carbonate in refluxing acetone for 90 min (50%) [4299] according to [4300].

White solid [4299]; ^{1}H NMR [4299], ^{13}C NMR [4299].

1-[3-[(Dimethylamino)methyl]-2-hydroxy-5-methylphenyl]ethanone

[93201-29-7] $C_{12}H_{17}NO_2$ mol.wt. 207.27



New compound

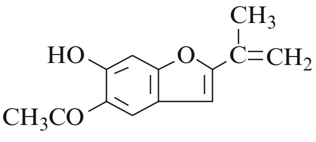
Synthesis

- Obtained by adding N,N-dimethylamine gas into a solution of 3-chloromethyl-2-hydroxy-5-m ethylacetophenone in cold benzene and stirring for 4–5 h. The solution of the hydrochloride obtained was neutralized with sodium bicarbonate solution to pH 7.0 (74%) [4154].

Oil [4154]; ^{1}H NMR [4154], UV [4154].
BIOLOGICAL ACTIVITY: Insecticide [4154].

1-[6-Hydroxy-2-(1-methylethenyl)-5-benzofuranyl]ethanone *(Euparin)*

[532-48-9] $C_{13}H_{12}O_3$ mol.wt. 216.24



New compound

Isolation from natural sources

- From the roots of *Ligularia stenocephala* MATSUM. et KOIDZ. (Compositae) [4301].
- From brittle bush (*Encelia farinosa* Gray) [4302].

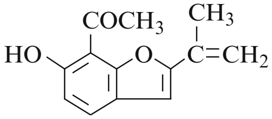
Yellow needles [4302]; m.p. 117–118° [4303], 116° [4302];
^{1}H NMR [4302], IR [4302], UV [4302], MS [4302].

Acetate [69309-25-7] $C_{15}H_{14}O_4$ mol.wt. 258.27

m.p. 80° [4304], 78–79° [4302].

1-[6-Hydroxy-2-(1-methylethenyl)-7-benzofuranyl]ethanone

[55682-75-2] $C_{13}H_{12}O_3$ mol.wt. 216.24



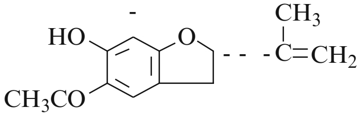
New compound

Synthesis

- Refer to: [4305] (Chinese paper).

1-[(2*R*)-2,3-Dihydro-6-hydroxy-2-(1-methylethenyl)-5-benzofuranyl]ethanone *(R)-(-)-Hydroxytremetone*

[21491-62-3] $C_{13}H_{14}O_3$ mol.wt. 218.25



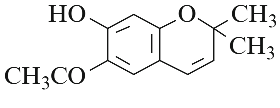
New compound

Isolation from natural sources

- From the roots of *Ligularia stenocephala* MATSUM. et KOIDZ. (Compositae) [4301].
- From the white snakeroot plant *(Eupatorium urticaefolium)* [4306].
- From the rayless goldenrod plant *(Aploppapus heterophyllus)* [4306].

1-(7-Hydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)ethanone *(Eupatoriochromene A)*

[19013-03-7] $C_{13}H_{14}O_3$ mol.wt. 218.25



New compound

Synthesis

– Obtained by dehydrogenation of 6-acetyl-7-hydroxy-2,2-dimethylchroman with DDQ in refluxing benzene for 1.5 h [4302].

m.p. 76–77° [4302], 76° [4307];
^{1}H NMR [4302,4307], IR [4302], MS [4302].

Isolation from natural sources

- From the genus of *Eupatorium riparium* Regel and *Eupatorium glandulosum* H.B. & K. (syn. *Eupatorium adenophorum* Spr.) (Eupatorieae) [4307].

Acetate $C_{15}H_{16}O_4$ mol.wt. 260.29

m.p. 75.5–76.5° [4302].

Methyl ether [20628-09-5] $C_{14}H_{16}O_3$ mol.wt. 232.28

(Encecalin)

- Obtained by treatment of eupatoriochromene A with dimethyl sulfate in the presence of potassium carbonate [4307].
- Also obtained by oxidation of encecalol [6-(1-hydroxyethyl)-7-methoxy-2,2-dimethylchromene] (encecalol) with manganese oxide in petroleum ether suspension for 2 h at r.t. (84%) [4302].

Isolation from the natural sources

- From the genus of *Eupatorium riparium* Regel and *Eupatorium glandulosum* H.B. & K. (syn. *Eupatorium adenophorum* Spr.) (Eupatorieae) [4307].
- From *Encelia californica* Nutt (tribe Heliantheae) [4084].
- From brittle bush (*Encelia farinosa* Gray) [4302].

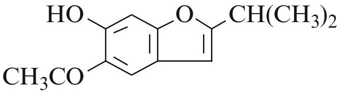
Yellow viscous oil [4302], oil [4307];
b.p.$_{0.05}$ 123° [4308], b.p.$_{0.11}$ 135–137° [4302];
^{1}H NMR [4302,4307], IR [4084,4302], MS [4084,4302].

Oxime (of the methyl ether) [23840-18-8] $C_{14}H_{17}NO_3$ mol.wt. 247.29

m.p. 140° [4084,4308], 136–137° [4302]; ^{1}H NMR [4084].

1-[6-Hydroxy-2-(1-methylethyl)-5-benzofuranyl]ethanone *(Isodihydroeuparin)*

[5207-55-6] $C_{13}H_{14}O_3$ mol.wt. 218.25



New compound

Syntheses

- Obtained by hydrogenation (1 atm) of euparin in ethanol in the presence of Raney nickel [4309].
- Also refer to: [4310].

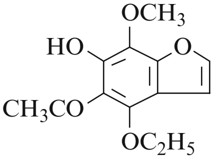
Isolation from natural sources

- From brittle bush (*Encelia farinosa* Gray) [4302].
- From the aerial parts of *Senecio graveolens* Wedd [4311].

m.p. 188–120° [4302], 62° [4309], 57–58.5° [4312].
One of the reported melting points is obviously wrong.
^{1}H NMR [4302,4309], UV [4302].

1-(4-Ethoxy-6-hydroxy-7-methoxy-5-benzofuranyl)ethanone

[75884-10-5] $C_{13}H_{14}O_5$ mol.wt. 250.25



New compound

Syntheses

- Obtained by slowly adding 3 M sodium hydroxide to a solution of 4-ethoxy-9-methoxy-7-methylfuro[3,2-g]-chromen-5-one (m.p. 104–106°) in refluxing dilute ethanol, then the resulting solution was stirred at 70° for 3 h (75%) [4288].

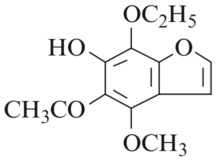
- Also obtained by treatment of 5-(β-ethylaminocrotonyl)-4-ethoxy-6-hydroxy-7-methoxy-coumarone with 3% potassium hydroxide for 30 min at r.t. [4289].

m.p. 94–95° [4288], 93–95° [4289];
^{1}H NMR [4288], ^{13}C NMR [4288], MS [4288].

Benzoate $C_{20}H_{18}O_6$ mol.wt. 354.36 m.p. 83–85° [4289].

1-(7-Ethoxy-6-hydroxy-4-methoxy-5-benzofuranyl)ethanone

[88897-98-7] $C_{13}H_{14}O_5$ mol.wt. 250.25



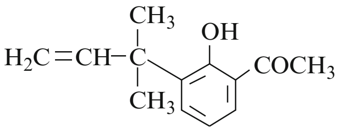
New compound

Synthesis

- Obtained by slowly adding 3 M sodium hydroxide to a solution of 9-ethoxy-4-methoxy-7-methylfuro[3,2-g]-chromen-5-one in refluxing dilute ethanol, then the resulting solution was stirred at 70° for 3 h [4288].

1-[3-(1,1-Dimethyl-2-propenyl)-2-hydroxyphenyl]ethanone

[873211-43-9] $C_{13}H_{16}O_2$ mol.wt. 204.27



New compound

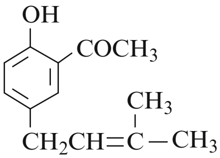
Synthesis

- Refer to: [4313].

BIOLOGICAL ACTIVITY: Antiproliferative and antimicrobial agent [4313].

1-[2-Hydroxy-5-(3-methyl-2-butenyl)phenyl]ethanone

[873211-41-7] $C_{13}H_{16}O_2$ mol.wt. 204.27



New compound

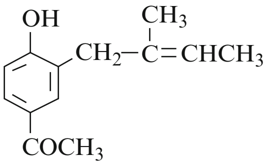
Synthesis

- Refer to: [4313].

BIOLOGICAL ACTIVITY: Antiproliferative and antimicrobial agent [4313].

1-[4-Hydroxy-3-[(2*E*)-2-methyl-2-butenyl]phenyl]ethanone

[603110-50-5] $C_{13}H_{16}O_2$ mol.wt. 204.27



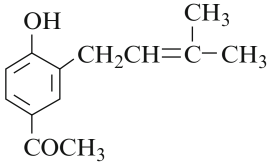
New compound

Synthesis

- Refer to: [4314].

1-[4-Hydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[26932-05-8] $C_{13}H_{16}O_2$ mol.wt. 204.27



Described [3734] **p. 939**

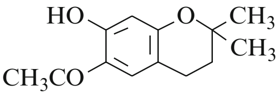
Synthesis

- Also refer to: [4313].

BIOLOGICAL ACTIVITY: Antiproliferative and antimicrobial agent [4313].

1-(3,4-Dihydro-7-hydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)ethanone

[31273-58-2] $C_{13}H_{16}O_3$ mol.wt. 220.27



New compound

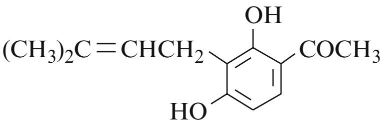
Syntheses

- Obtained by cyclization of 2,4-dihydroxy-5-prenyl-acetophenone with concentrated hydrochloric acid in refluxing ethanol for 3 h (87%) [4302].
- Also refer to: [4315].

m.p. 119° [4316], 117–118° [4302];
^{1}H NMR [4302], IR [4302].

1-[2,4-Dihydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[19825-40-2] $C_{13}H_{16}O_3$ mol.wt. 220.27



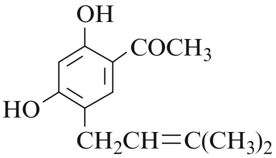
Described [3734] **p. 941**

Synthesis

- Also refer to: [4155].

1-[2,4-Dihydroxy-5-(3-methyl-2-butenyl)phenyl]ethanone

[28437-37-8] $C_{13}H_{16}O_3$ mol.wt. 220.27



Described [3734] **p. 942**

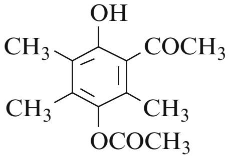
Syntheses

- Also obtained by adding 2-methyl-3-buten-2-ol over 30 min to a solution of resacetophenone in 80% formic acid at 50° (7%) [4302].
- Also obtained by reaction of prenyl bromide with resacetophenone in potassium hydroxide solution between 0° and 25° for 13 h (23%) [4317].
- Also refer to: [4155,4318–4320].

m.p. 144–145° [4302], 144° [4316], 139–141° [4320], 124–125° [4318].
One of the reported melting points is obviously wrong.
^{1}H NMR [4302,4317–4319], ^{13}C NMR [4318], IR [4302,4318,4319], UV [4155,4319].

1-[3-(Acetyloxy)-6-hydroxy-2,4,5-trimethylphenyl]ethanone

[66901-79-9] $C_{13}H_{16}O_4$ mol.wt. 236.27



New compound

Synthesis

- Readily obtained from TMHQ (trimethylhydroquinone) by treatment with BF_3–acetic acid complex [4321].

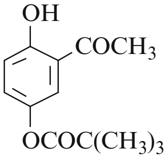
Isopropyl ether $C_{16}H_{22}O_4$ mol.wt. 278.35

- Preparation by reaction of isopropyl bromide with 1-(2,5-dihydroxy-3,4,6-trimethylphenyl)ethanone in the presence of potassium carbonate and potassium iodide in acetone at r.t. for 24 h [4321].

1-[2-Hydroxy-5-(2,2-dimethylpropanoyloxy)phenyl]ethanone

2,2-Dimethylpropionic acid 3-acetyl-4-hydroxyphenyl ester

$C_{13}H_{16}O_4$ mol.wt. 236.27



New compound

Synthesis

- Obtained by reaction of pivaloyl chloride with quinacetophenone in the presence of pyridine at r.t. for 1 h (79%) [3818].

Colourless oil [3818]; ^{1}H NMR [3818], ^{13}C NMR [3818], MS [3818].

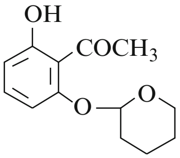
Methyl ether $C_{14}H_{18}O_4$ mol.wt. 250.29

- Obtained by reaction of methyl iodide with the ketone above mentioned in the presence of lithium carbonate in DMF at 120° for 4 h (82%) [3818].

Colourless oil [3818]; ^{1}H NMR [3818], ^{13}C NMR [3818], MS [3818].

1-[2-Hydroxy-6-[(tetrahydro-*2H*-pyran)-2-yl]phenyl]ethanone

[63854-17-1] $C_{13}H_{16}O_4$ mol.wt. 236.27



Described [3734] **p. 947**

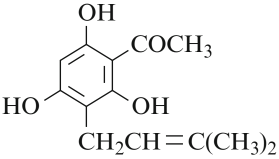
Synthesis

- Also obtained by reaction of 3,4-dihydro-*2H*-pyran with 2,6-dihydroxyacetophenone in the presence of p-toluenesulfonic acid in THF. The mixture was stirred under argon atmosphere overnight (55%) [4276].

Pale yellow crystals (unstable) [4276];
^{1}H NMR [4276], ^{13}C NMR [4276].

1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone *(6-Demethylacronylin)*

[27364-71-2] $C_{13}H_{16}O_4$ mol.wt. 236.27



Described [3734] **p. 947**

Synthesis

- Also obtained by reaction of 3-methyl-2-butenyl bromide with phloroacetophenone in the presence of aqueous potassium hydroxide at r.t. for 24 h (29%) [4322].

Isolation from natural sources.

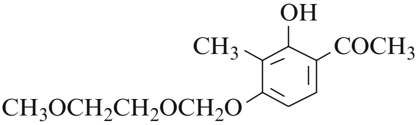
- From the root bark of *Acronychia laurifolia* B1 (Rutaceae) [4323].

m.p. 173–174° [4322], 154° (d) [4323]. One of the reported melting points is obviously wrong.

^{1}H NMR [4323], IR [4323], UV [4323], MS [4323].

1-[2-Hydroxy-4-[(2-methoxyethoxy)methoxy]-3-methylphenyl]ethanone

[886999-22-0] $C_{13}H_{18}O_5$ mol.wt. 254.28



New compound

Synthesis

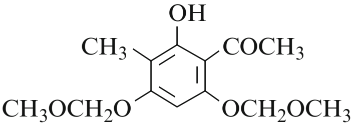
- Obtained by reaction of 2-methoxyethoxymethyl chloride with 2,4-dihydroxy-3-methyl acetophenone in the presence of DIEA in methylene chloride [4152].

m.p. 57–59° [4152];

^{1}H NMR [4152], IR [4152], MS [4152]; TLC [4152].

1-[2-Hydroxy-4,6-bis(methoxymethoxy)-3-methylphenyl]ethanone

[106929-57-1] $C_{13}H_{18}O_6$ mol.wt. 270.28



Described [3734] **p. 965**

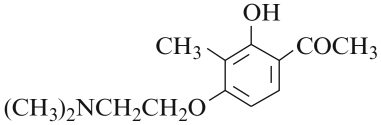
Synthesis

- Also obtained by reaction of chloromethyl methyl ether with 2,4,6-trihydroxy-3-methylacetophenone in the presence of potassium carbonate in refluxing acetone for 1 h (64%) [4192].

^{1}H NMR [4192], ^{13}C NMR [4192], MS [4192].

1-[4-[2-(Dimethylamino)ethoxy]-2-hydroxy-3-methylphenyl]ethanone

[942133-91-7] $C_{13}H_{19}NO_3$ mol.wt. 237.30



New compound

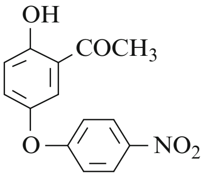
Synthesis

- Obtained by treatment of 2,4-dihydroxy-3-methyl-acetophenone with dimethylaminoethyl chloride in the presence of KOH/K_2CO_3 mixture in boiling 2-butanone [4274].

Hydrochloride (1:1) [942134-13-6] $C_{13}H_{19}NO_3$, HCl mol.wt. 273.76 [4274]

1-[2-Hydroxy-5-(4-nitrophenoxy)phenyl]ethanone

[1006063-13-3] $C_{14}H_{11}NO_5$ mol.wt. 273.35



New compound

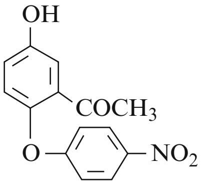
Synthesis

- Obtained by adding quinacetophenone to a mixture of 5 N sodium hydroxide and DMSO; then after 40 min, adding 4-fluoronitrobenzene and stirring the solution obtained at 50° for 5 h (20%) [4324].

Oil [4324]; ^{13}C NMR [4324], MS [4324].

1-[5-Hydroxy-2-(4-nitrophenoxy)phenyl]ethanone

[1006063-14-4] $C_{14}H_{11}NO_5$ mol.wt. 273.35



New compound

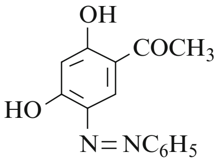
Synthesis

- Obtained by adding quinacetophenone to a mixture of 5 N sodium hydroxide and DMSO; then after 40 min, 4-fluoronitrobenzene and stirring the solution obtained at 50° for 5 h (<1%) [4324].

Oil [4324]; ^{13}C NMR [4324], MS [4324].

1-[2,4-Dihydroxy-5-(phenylazo)phenyl]ethanone *(E)*

[120196-22-7] $C_{14}H_{12}N_2O_3$ mol.wt. 256.26



New compound

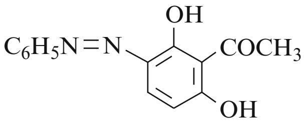
Syntheses

- Obtained by coupling resacetophenone with benzenediazonium chloride [4325,4326].

m.p. 198° [4326], 192° [4325].

Diacetate $C_{18}H_{16}N_2O_5$ mol.wt. 340.34 (m.p. 106–107°) [4325].

1-[2,6-Dihydroxy-3-(phenylazo)phenyl]ethanone



$C_{14}H_{12}N_2O_3$ mol.wt. 256.26

New compound

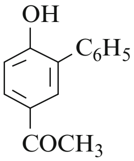
Synthesis

- Obtained by coupling 2-acetylresorcinol with benzenediazonium chloride [4325].

m.p. 150–151° [4325].

1-(6-Hydroxy[1,1′-biphenyl]-3-yl)ethanone

[20281-51-0] $C_{14}H_{12}O_2$ mol.wt. 212.25



Described [3734] **p. 972**

Benzyl ether $C_{21}H_{18}O_2$ mol.wt. 302.37

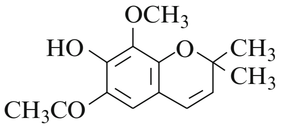
- Obtained by reaction of benzyl chloride with 4-hydroxy-3-phenylacetophenone in the presence of potassium hydroxide in refluxing methanol (88%) [4184].

m.p. 87–89° [4184].

1-(7-Hydroxy-8-methoxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)ethanone

(Ripariochromen A)

[20770-16-5] $C_{14}H_{16}O_4$ mol.wt. 248.28



New compound

Syntheses

- Obtained by treatment of 6-acetyl-7-hydroxy-8-methoxy-2,2-dimethyl-3,4-dihydro-2*H*-1-benzopyran with DDQ in boiling benzene for 8 h [4327,4328].
- Also prepared from 2-methyl-1,3-butadiene (multi-step reaction) [4327].
- Also prepared from gallacetophenone (multi-step reaction) [4327].

Isolation from natural sources

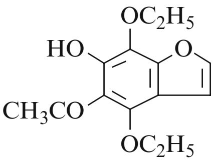
- From the roots of *Eupatorium riparium* Regel [4329].
- From the roots of *Ageratina riparia* [4083].

m.p. 90–91° [4330], 89.5–90.5° [4328,4331];
^{1}H NMR [4327,4328,4331], IR [4328,4330,4331], UV [4328,4330,4331].

BIOLOGICAL DATA: Antifungal activity [4329].

1-(4,7-Diethoxy-6-hydroxy-5-benzofuranyl)ethanone

[88349-53-5] $C_{14}H_{16}O_5$ mol.wt. 264.28



New compound

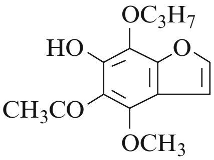
Synthesis

- Obtained by slowly adding 3 M sodium hydroxide to a solution of 4,9-diethoxy-7-methylfuro[3,2-g]chromen-5-one (m.p. 90°) in refluxing dilute ethanol, then the resulting solution was stirred at 70° for 3 h (55%) [4288].

m.p. 72–74° [4288]; ^{1}H NMR [4288], ^{13}C NMR [4288], MS [4288].

1-(6-Hydroxy-4-methoxy-7-propoxy-5-benzofuranyl)ethanone

[880479-07-2] $C_{14}H_{16}O_5$ mol.wt. 264.28



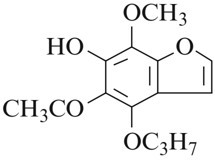
New compound

Synthesis

- Obtained by slowly adding 3 M sodium hydroxide to a solution of 4-methoxy-7-methyl-9-propoxyfuro[3,2-g]-chromen-5-one in refluxing dilute ethanol, then the resulting solution was stirred at 70° for 3 h [4288].

1-(6-Hydroxy-7-methoxy-4-propoxy-5-benzofuranyl)ethanone

$C_{14}H_{16}O_5$ mol.wt. 264.28



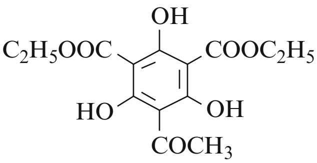
New compound

Synthesis

- Obtained by slowly adding 3 M sodium hydroxide to a solution of 9-methoxy-7-methyl-4-propoxyfuro[3,2-g]-chromen-5-one in refluxing dilute ethanol, then the resulting solution was stirred at 70° for 3 h [4288].

5-Acetyl-2,4,6-trihydroxyphenyl-1,3-dicarboxylic acid diethyl ester

$C_{14}H_{16}O_8$ mol.wt. 312.28



New compound

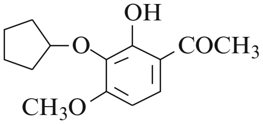
Syntheses

- Obtained by reaction of acetonitrile with 2,4,6-trihydroxy-phenyl-1,3-dicarboxylic acid diethyl ester in the presence of aluminium chloride and hydrogen chloride in ethyl ether (9%) [4297].
- Also refer to: [4332,4333].

m.p. 129° [4332,4333], 128–130° [4297].

1-[3-(Cyclopentyloxy)-2-hydroxy-4-methoxyphenyl]ethanone

[1001056-78-5] $C_{14}H_{18}O_4$ mol.wt. 250.29



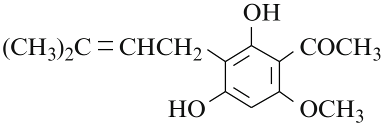
New compound

Synthesis

- Refer to: [4334].

1-[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[30403-01-1] $C_{14}H_{18}O_4$ mol.wt. 250.29



Described [3734] **p. 983**

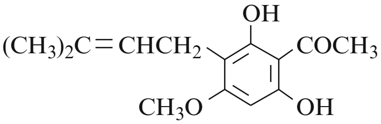
Syntheses

- Also obtained by reaction of diazomethane with 2,4,6-trihydroxy-3-(3-methyl-2-butenyl)-acetophenone in ethyl ether (19%) [4322].
- Also refer to: [4155,4335,4336].

m.p. 169–170° [4322], 164° [4335].

1-[2,6-Dihydroxy-4-methoxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[77179-30-7] $C_{14}H_{18}O_4$ mol.wt. 250.29



New compound

Syntheses

- Obtained by reaction of 4-bromo-2-methyl-2-butene with 2,6-dihydroxy-4-methoxyacetophenone in the presence of potassium hydroxide,
  - in methanol at 20° for 40 h (13%) [4337];
  - in ethanol at r.t. for 24 h [4338].
- Also obtained by reaction of α,α-dimethylallyl alcohol with 2,6-dihydroxy-4-methoxy-acetophenone in the presence of boron trifluoride etherate in dioxane for 2 h [4339].
- Also refer to: [4335,4336].

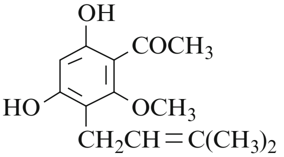
m.p. 164° [4335], 125–126° [4338], 43° [4337].
One of the reported melting points is obviously wrong.
^{1}H NMR [4337,4338], IR [4337,4338], UV [4337,4338].

N.B.:

- In the paper [4337], the “isomers” 3-C-prenyl (compound **C**, m.p. 43°) and 5-C-prenyl (compound **D**, m.p. 36°) are a single and same compound.
- The constitution of the title compound previously suggested by [4335] is incorrect [4336].

1-[4,6-Dihydroxy-2-methoxy-3-(3-methyl-2-butenyl)phenyl]ethanone *(Acronylin)*

[27364-64-3] $C_{14}H_{18}O_4$ mol.wt. 250.29



Described [3734] **p. 983**

Syntheses

- Also refer to: [4155,4340].

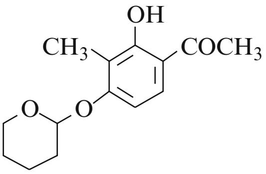
Isolation from natural sources

- From *Acronychia pedunculata* (Rutaceae) [4341].
- From the stem bark of *Acronychia laurifolia* (Rutaceae) [4342] also named *Acronychia pedunculata* [4323,4343].
- From the stem bark and Leaves of *Melicope stipitata* (Rutaceae) [4344].

Colourless needles [4343];
m.p. 128–130° [4343,4344], 127–128° [4345];
^{1}H NMR [4344], ^{13}C NMR [4345], IR [4344], UV [4155,4344], MS [4344].

1-[2-Hydroxy-3-methyl-4-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone

[200726-78-9] $C_{14}H_{18}O_4$ mol.wt. 250.29



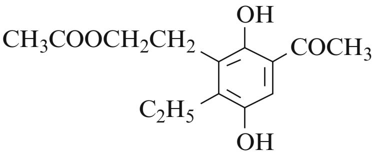
New compound

Synthesis

- Obtained by reaction of 2,3-dihydropyran with 2,4-dihydroxy-3-methylacetophenone in the presence of p-toluenesulfonic acid in ethyl ether at r.t. (83%) [4346].

1-[3-[2-(Acetyloxy)ethyl]-4-ethyl-2,5-dihydroxyphenyl]ethanone

[873222-94-7] $C_{14}H_{18}O_5$ mol.wt. 266.29



New compound

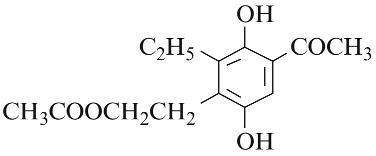
Synthesis

- Obtained by $[Cp^*RuCl_2]_2$-catalyzed cocyclisation of 1-acetoxy-3-hexyne, 3-buten-2-one and carbon monoxide in DMF at 140° for 20 h (45%) [4295].

^{1}H NMR [4295].

1-[4-[2-(Acetyloxy)ethyl]-3-ethyl-2,5-dihydroxyphenyl]ethanone

[873222-95-8]



$C_{14}H_{18}O_5$ mol.wt. 266.29

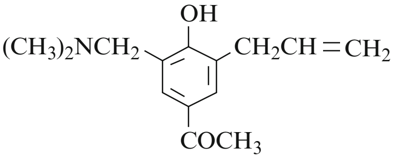
New compound

Synthesis

- Obtained by $[Cp^*RuCl_2]_2$-catalyzed cocyclisation of 1-acetoxy-3-hexyne, 3-buten-2-one and carbon monoxide in DMF at 140° for 20 h (30%) [4295].

^{1}H NMR [4295].

1-[3-Allyl-5-(dimethylaminomethyl)-4-hydroxyphenyl]ethanone



$C_{14}H_{19}NO_2$ mol.wt. 233.31

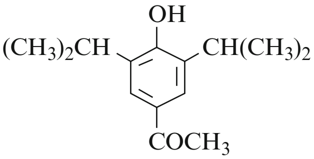
New compound

Synthesis

- Obtained by reaction of dimethylmethylimminium iodide with 3-allyl-4-hydroxyacetophenone in the presence of carbonate exchange resin in methylene chloride at r.t. for 16 h (87%) [4283].

1-[4-Hydroxy-3,5-bis(1-methylethyl)phenyl]ethanone

[720-19-4]



$C_{14}H_{20}O_2$ mol.wt. 220.31

New compound

Synthesis

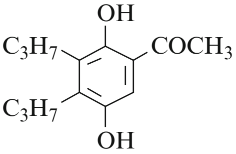
- Refer to: [4347].

Ethyl ether [1023740-57-9] $C_{16}H_{24}O_2$ mol.wt. 248.37

- Refer to: [4348].

1-(2,5-Dihydroxy-3,4-dipropylphenyl)ethanone

[873222-85-6]



$C_{14}H_{20}O_3$ mol.wt. 236.21

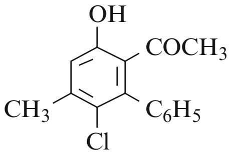
New compound

Synthesis

- Obtained by $[Cp^*RuCl_2]_2$-catalyzed cocyclisation of 4-octyne (1,2-dipropylacetylene), 3-buten-2-one and carbon monoxide in DMF at 140° for 20 h (79%) [4295].

1-(6-Chloro-3-hydroxy-5-methyl[1,1′-biphenyl]-2-yl)ethanone

[1001025-04-2] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



New compound

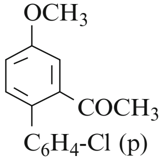
Synthesis

- Obtained by reaction of 3-chloro-4-phenyl-4-(trimethyl-silyloxy)-3-buten-2-one with 2,4-bis-(trimethylsilanyloxy)-1,3-pentadiene in the presence of titanium tetrachloride (43%) [4208].

Yellow crystals; m.p. 68° [4208];
^{1}H NMR [4208], ^{13}C NMR [4208], IR [4208], MS [4208].

1-(4′-Chloro-4-methoxy[1,1′-biphenyl]-2-yl)ethanone

[841298-81-5] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



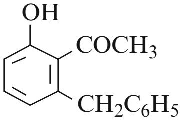
New compound

Syntheses

- Refer to: [4349,4350].

1-[2-Hydroxy-6-(phenylmethyl)phenyl]ethanone

$C_{15}H_{14}O_2$ mol.wt. 226.27



New compound

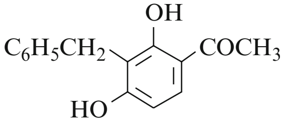
Synthesis

- Obtained by reaction of benzyl bromide with 2,6-dihydroxy-acetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone overnight under nitrogen (78%) [4010].

m.p. 109–111° [4010];
^{1}H NMR [4010], ^{13}C NMR [4010]; TLC [4010].

1-[2,4-Dihydroxy-3-(phenylmethyl)phenyl]ethanone

[67088-16-8] $C_{15}H_{14}O_3$ mol.wt. 242.27



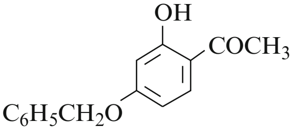
Described [3734] **p. 1001**

Synthesis

- Also refer to: [4155].

1-[2-Hydroxy-4-(phenylmethoxy)phenyl]ethanone

[29682-12-0] $C_{15}H_{14}O_3$ mol.wt. 242.27



Described [3734] **p. 1003**

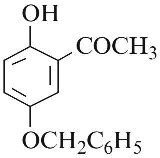
Syntheses

- Also obtained by reaction of benzyl bromide with resacetophenone in the presence of potassium carbonate in refluxing acetone for 18 h (87%) [3911].
- Also refer to: [4211].

Pink solid [3911]; ^{1}H NMR [3911], ^{13}C NMR [3911], MS [3911].

1-[2-Hydroxy-5-(phenylmethoxy)phenyl]ethanone

[30992-63-3] $C_{15}H_{14}O_3$ mol.wt. 242.27



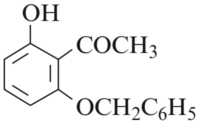
Described [3734] **p. 1004**

Synthesis

- Also refer to: [3908].

1-[2-Hydroxy-6-(phenylmethoxy)phenyl]ethanone

[4047-24-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



Described [3734] **p. 1004**

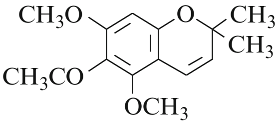
Synthesis

- Preparation by reaction of benzyl bromide with 2,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 12 h (82%) [4351].

m.p. 189° [4351].

1-(5,7-Dimethoxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)ethanone

[18780-97-7] $C_{15}H_{18}O_4$ mol.wt. 262.3



New compound

Syntheses

- Obtained by treatment of 6-acetyl-7-hydroxy-5-methoxy-2,2-dimethylchromene with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 12 h [4352,4353].
- Also obtained from 2,4-dihydroxy-5-prenyl-6-methoxyacetophenone (multi-step reaction) [4352].
- Also obtained from 2,4-dihydroxy-3-iodo-6-methoxyacetophenone (multi-step reaction) [4352].

- Also obtained from 2-hydroxy-3-iodo-4-O-(1,1-dimethylpropargyl)-6-methoxyacetophenone (multi-step reaction) [4352].
- From α,α-dimethylallyl alcohol (multi-step reaction) [4352].
- Also refer to: [4354,4355].

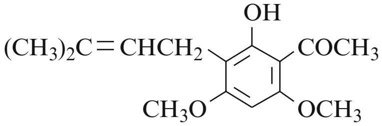
Isolation from natural sources

- From *Medicosma cunninghamii* [4355].
- From *Acradenia franklinii* [4356].
- From *Euodia lunu-ankenda* Merr [4357].
- From *Melicope stipitata* [4344].

m.p. 79–80° [4354], 79° [4353], 78–79° [4356], 78–78.5° [4355], 76–78° [4344], 76–77° [4357];
^{1}H NMR [4354], IR [4356], UV [4358].

1-[2-Hydroxy-4,6-dimethoxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[33523-62-5] $C_{15}H_{20}O_4$ mol.wt. 264.32



Described [3734] **p. 1011**

Syntheses

- Obtained by reaction of diazomethane with 2,4,6-trihydroxy-3-(3-methyl-2-butenyl)- acetophenone in ethyl ether [4335], (90%) [4359], (20%) [4322].
- Also obtained by reaction of prenyl bromide with 2-hydroxy-4,6-dimethoxyacetophenone in the presence of potassium carbonate in refluxing acetone for 8 h [4360,4361].
- Also obtained by reaction of 4-bromo-2-methyl-2-butene with xanthoxyline in the presence of potassium carbonate in boiling acetone for 8 h [4360].
- Also obtained by reaction of 50% potassium hydroxide with 7,7-methylglabranin in boiling methanol for 6 h [4362].
- Also obtained by refluxing 2,4-dimethoxy-6-(3,3-dimethylallyloxy)acetophenone with N,N-dimethylaniline for 5 h (3%) [4363].
- Also refer to: [4155,4298,4340].

m.p. 114° [4335], 113–114° [4345,4359,4361,4364], 113° [4322], 112–113° [4362], 109–110° [4360], 103–104° [4363];
^{1}H NMR [4359–4364], IR [4361,4363], UV [4361–4363].

Methyl ether [101253-53-6] $C_{16}H_{22}O_4$ mol.wt. 278.35

- Obtained by reaction of dimethyl sulfate with 4,6-dimethoxy-3-(3,3-dimethylallyl)-phloroacetophenone in the presence of potassium carbonate in boiling acetone [4335].

m.p. 50–51° [4335].

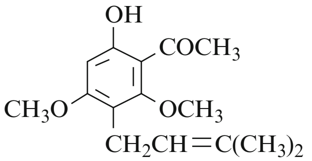
Benzoate [42344-99-0] $C_{22}H_{24}O_5$ mol.wt. 368.53

- Obtained by reaction of benzoyl chloride with 4,6-dimethoxy-2-hydroxy-3-(3-methyl-2-butenyl)-acetophenone in the presence of potassium carbonate in refluxing acetone for 6 h (90%) [4359].

m.p. 81–83° [4359], 81–82° [4335].

1-[6-Hydroxy-2,4-dimethoxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[4683-33-4] $C_{15}H_{20}O_4$ mol.wt. 264.32



Described [3734] **p. 1011**

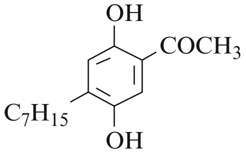
Syntheses

- Also obtained by refluxing 2,4-dimethoxy-6-(3,3-dimethyl-allyloxy)acetophenone with N,N-dimethylaniline for 5 h (3%) [4363].
- Also obtained by reaction of diazomethane with 2,4-dihydroxy-6-methoxy-5-prenylacetophenone in ethyl ether [4340].
- Also refer to: [4155].

m.p. 102–104° [4363];
^{1}H NMR [4340,4363], IR [4340,4363], UV [4155,4340,4363].

1-(4-Heptyl-2,5-dihydroxyphenyl)ethanone

$C_{15}H_{22}O_3$ mol.wt. 250.34



New compound

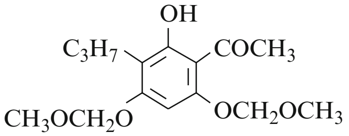
Syntheses

- Obtained by treatment of 4′-heptyl-2′,5′-dihydroxy-3,3-dimethylacrylophenone with aluminium chloride in refluxing benzene for 3 h [3996].
- Also obtained by reaction of acetyl chloride with 2-heptylhydroquin one dimethyl ether in the presence of aluminium chloride in carbon disulfide [3996].

b.p.$_{0.1}$ 143–147° [3996], b.p.$_7$ 170–180° [3996].

1-[2-Hydroxy-4,6-bis(methoxymethoxy)-3-propylphenyl]ethanone

[916916-81-9] $C_{15}H_{22}O_6$ mol.wt. 298.34



New compound

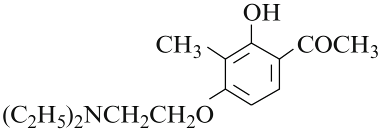
Synthesis

- Obtained by reaction of chloromethyl methyl ether with 2,4,6-trihydroxy-3-propylacetophenone in the presence of potassium carbonate in refluxing acetone for 1 h (68%) [4192].

^{1}H NMR [4192], ^{13}C NMR [4192], MS [4192].

1-[4-[2-(Diethylamino)ethoxy]-2-hydroxy-3-methylphenyl]ethanone

[942133-92-8] $C_{15}H_{23}NO_3$ mol.wt. 265.35



New compound

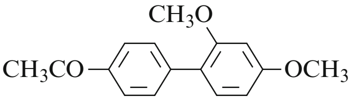
Synthesis

- Obtained by treatment of 2,4-dihydroxy-3-methyl-acetophenone with diethylaminoethyl chloride in the presence of KOH/K_2CO_3 mixture in boiling 2-butanone [4274].

Hydrochloride (1:1) [942134-14-7] $C_{15}H_{23}NO_3$, HCl mol.wt. 301.81 [4274]

1-(2′,4′-Dimethoxy[1,1′-biphenyl]-4-yl)ethanone

[178055-99-7] $C_{16}H_{16}O_3$ mol.wt. 256.30



New compound

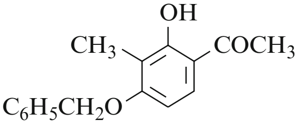
Synthesis

- Obtained by Suzuki coupling: p-chloroacetophenone (1 equiv), 2,4-dimethoxyphenylboronic acid (1.5 equiv) and sodium hydroxide (4 equiv) was added in water (20 ml). Then, Pd/PANI (1 mol%) was added to the solution at 100° and stirred at this temperature for 6–8 h (70%) [4365].

^{1}H NMR [4365].

1-[2-Hydroxy-3-methyl-4-(phenylmethoxy)phenyl]ethanone

[73640-74-1] $C_{16}H_{16}O_3$ mol.wt. 256.30



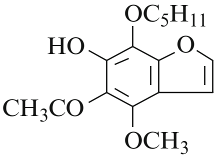
Described [3734] **p. 1018**

Syntheses

- Preparation by reaction of benzyl chloride with 2,4-dihydroxy-3-methylacetophenone in the presence of potassium carbonate and potassium iodide in acetone at 56° (88%) [4154].
- Also refer to: [4153].

1-[6-Hydroxy-4-methoxy-7-(pentyloxy)-5-benzofuranyl]ethanone

[880479-08-3] $C_{16}H_{20}O_5$ mol.wt. 292.92



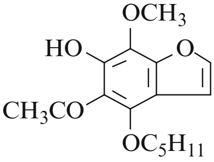
New compound

Synthesis

- Obtained by slowly adding 3 M sodium hydroxide to a solution of 4-methoxy-7-methyl-9-pentyloxyfuro [3,2-g]-chromen-5-one in refluxing dilute ethanol, then the resulting solution was stirred at 70° for 3 h [4288].

1-[6-Hydroxy-7-methoxy-4-(pentyloxy)-5-benzofuranyl]ethanone

[880479-11-8] $C_{16}H_{20}O_5$ mol.wt. 292.92



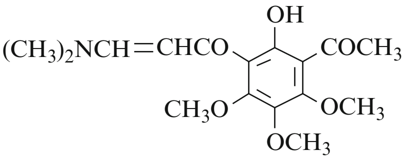
New compound

Synthesis

- Obtained by slowly adding 3 M sodium hydroxide to a solution of 9-methoxy-7-methyl-4-pentyloxyfuro[3,2-g]-chromen-5-one in refluxing dilute ethanol, then the resulting solution was stirred at 70° for 3 h [4288].

1-(3-Acetyl-2-hydroxy-4,5,6-trimethoxyphenyl)-3-(dimethylamino) (2*E*)-2-propen-1-one

[870480-50-5] $C_{16}H_{21}NO_6$ mol.wt. 323.34



New compound

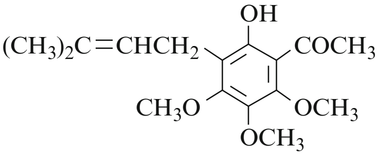
Synthesis

- Obtained by reaction of 2,6-diacetyl-3,4,5-trimethoxyphenol with N,N-dimethyl-formamide dimethyl acetal in DMF at 100° for 6 h (67%) [4275].

Yellow crystals; m.p. 113–114° [4275];
^{1}H NMR [4275], MS [4275].

1-[2-Hydroxy-4,5,6-trimethoxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[340816-26-4] $C_{16}H_{22}O_5$ mol.wt. 294.35



New compound

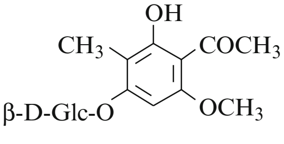
Synthesis

- Obtained by reaction of prenyl bromide with 6-hydroxy-2,3,4-trimethoxyacetophenone in the presence of potassium hydroxide in methanol for 20 h at r.t. (37%) [4063].

m.p. 112° [4063];
^{1}H NMR [4063], ^{13}C NMR [4063], IR [4063], UV [4063], MS [4063].

1-[4-(β-D-Glucopyranosyloxy)-2-hydroxy-6-methoxy-3-methylphenyl]ethanone

[145194-40-7] $C_{16}H_{22}O_9$ mol.wt. 358.35



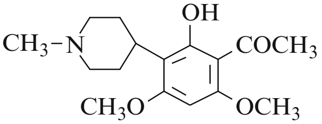
Described [3734] **p. 1025**

Isolation from natural sources

- Also obtained from the roots of *Euphorbia ebracteolata* Hayata (Euphorbiaceae) [4254,4366].

1-[2-Hydroxy-4,6-dimethoxy-3-(1-methyl-4-piperidinyl)phenyl]ethanone

[872057-13-1] $C_{16}H_{23}NO_4$ mol.wt. 293.36



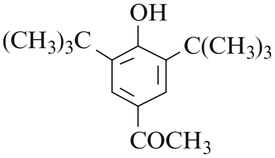
New compound

Synthesis

- Refer to: [3830].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]ethanone

[14035-33-7] $C_{16}H_{24}O_2$ mol.wt. 248.37



Described [3734] **p. 1026**

Syntheses

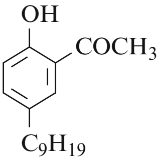
- Also obtained by reaction of acetyl chloride with 2,6-di-tert-butylphenol in the presence of titanium tetrachloride [3816].
- Also refer to: [4347,4348,4367].

Methyl ether [30492-50-3] $C_{17}H_{26}O_2$ mol.wt. 262.39

- Refer to: [4348,4367].

1-(2-Hydroxy-5-nonylphenyl)ethanone

[115851-77-9] $C_{17}H_{26}O_2$ mol.wt. 262.39



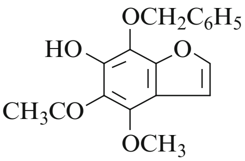
Described [3734] **p. 1041**

Synthesis

- Also obtained by reaction of acetyl chloride with 4-nonylphenol in the presence of aluminium chloride (98%) [4368].

1-[6-Hydroxy-4-methoxy-7-(phenylmethoxy)-5-benzofuranyl]ethanone

[880479-09-4] $C_{18}H_{16}O_5$ mol.wt. 312.32



New compound

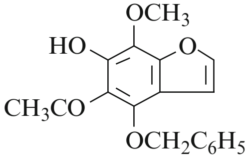
Synthesis

- Obtained by slowly adding 3 M sodium hydroxide to a solution of 4-methoxy-7-methyl-9-benzyloxy-furo[3,2-g]-chromen-5-one in refluxing dilute ethanol, then the resulting solution was stirred at 70° for 3 h (48%) [4288].

yellow solid; m.p. 78–80° [4288];
^1H NMR [4288], ^{13}C NMR [4288], MS [4288].

1-[6-Hydroxy-7-methoxy-4-(phenylmethoxy)-5-benzofuranyl]ethanone

[119104-31-3] $C_{18}H_{16}O_5$ mol.wt. 312.32



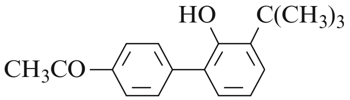
New compound

Synthesis

- Obtained by slowly adding 3 M sodium hydroxide to a solution of 9-methoxy-7-methyl-4-benzyloxy-furo[3,2-g]-chromen-5-one in refluxing dilute ethanol, then the resulting solution was stirred at 70° for 3 h [4288].

1-[3′-(1,1-Dimethylethyl)-2′-hydroxy[1,1′-biphenyl]-4-yl]ethanone

[521273-05-2] $C_{18}H_{20}O_2$ mol.wt. 268.36



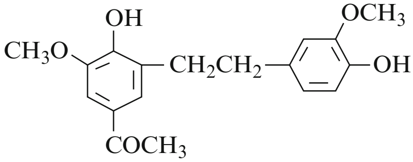
New compound

Synthesis

- Obtained by coupling of 2-tert-butylphenol (0.5 mmol) and 4-bromoacetophenone (0.6 mmol) in the presence of [[RhCl(COD)]$_2$] (5 mol% Rh), ClPi Pr$_2$ (10 mol%) and Cs_2CO_3 (0.85 mmol) in refluxing toluene for 18 h (89%) [4369].

1-[4-Hydroxy-3-[2-(4-hydroxy-3-methoxyphenyl)ethyl]-5-methoxyphenyl] ethanone

[75340-36-2] $C_{18}H_{20}O_5$ mol.wt. 316.35



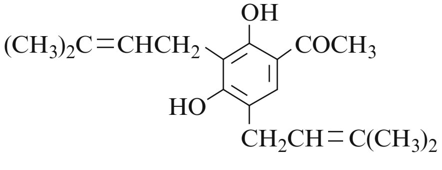
New compound

Isolation from natural sources

- Characterization in a pulp mill effluent [4370].

1-[2,4-Dihydroxy-3,5-bis(3-methyl-2-butenyl)phenyl]ethanone

[24672-82-0] $C_{18}H_{24}O_3$ mol.wt. 288.39



Described [3734] **p. 1048**

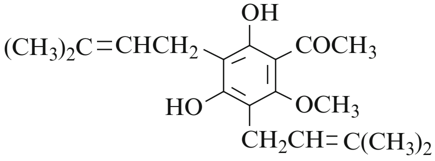
Syntheses

- Preparation by reaction of 2-methyl-but-3-en-2-ol with resacetophenone in the presence of boron trifluoride etherate in dioxane at r.t. for 1 h under nitrogen [4371].
- Also refer to: [4155].

1-[2,4-Dihydroxy-6-methoxy-3,5-bis(3-methyl-2-butenyl)phenyl]ethanone

[123999-38-2] $C_{19}H_{26}O_4$ mol.wt. 318.41

Described [3734] **p. 1059**



Isolation from natural sources

- From the leaves of *Acronychia pedunculata* (L.) Miq. (Rutaceae) [4372].

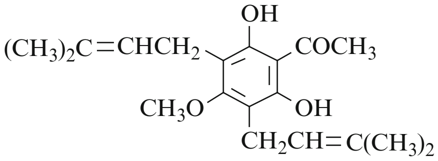
  Oil [4372]; ^{1}H NMR [4372], MS [4372].

Diacetate $C_{23}H_{30}O_6$ mol.wt. 402.49

- Preparation by acetylation of 1-[2,4-dihydroxy-6-methoxy-3,5-bis(3-methyl-2-butenyl) phenyl]ethanone with acetic anhydride in the presence of pyridine at 27° for 18 h (quantitative yield) [4343].

  Yellow oil [4343]; ^{1}H NMR [4343], IR [4343], MS [4343]

1-[2,6-Dihydroxy-4-methoxy-3,5-bis(3-methyl-2-butenyl)phenyl]ethanone



$C_{19}H_{26}O_4$ mol.wt. 318.41

New compound

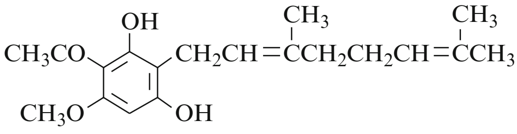
Synthesis

- Obtained by reaction of 4-bromo-2-methyl-2-butene with 2,6-dihydroxy-4-methoxy-acetophenone in the presence of potassium hydroxide in methanol at 20° for 40 h (19%) [4337].^{1}H NMR [4337], IR [4337], UV [4337].

1-[3-(3,7-Dimethyl-2,6-octadienyl)-2,4-dihydroxy-6-methoxyphenyl]ethanone *(E)*

[122585-64-2] $C_{19}H_{26}O_4$ mol.wt. 318.41

New compound

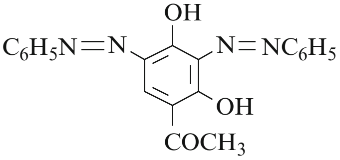


Isolation from natural sources

- From *Helichrysum cerastroides* DC subsp. *aurosicum* Merxm. et A. Schreiber (Compositae) [4373].

  ^{1}H NMR [4373], IR [4373], MS [4373].

1-[2,4-Dihydroxy-3,5-bis(phenylazo)phenyl]ethanone *(E)*



$C_{20}H_{16}N_4O_3$ mol.wt. 360.37

New compound

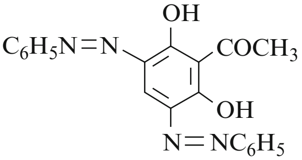
Syntheses

- Obtained by coupling resacetophenone with benzenediazonium chloride [4325,4374,4375].

m.p. 221–222° [4325], 220–221° [4374], 220° [4375].

Diacetate $C_{24}H_{20}N_4O_5$ mol.wt. 444.45 (m.p. 148°) [4325]

1-[2,6-Dihydroxy-3,5-bis(phenylazo)phenyl]ethanone *(E)*



$C_{20}H_{16}N_4O_3$ mol.wt. 360.37

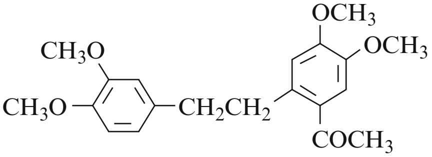
New compound

Syntheses

- Obtained by coupling 2-acetylresorcinol with benzenediazonium chloride [4325,4374].

m.p. 170° [4325], 169–170° [4374].

1-[4,5-Dimethoxy-2-[(3,4-dimethoxyphenyl)ethyl]phenyl]ethanone
2-Acetyl-4,5,3′,4′-tetramethoxydibenzyl



$C_{20}H_{24}O_5$ mol.wt. 344.41

New compound

Syntheses

- Obtained by reaction of acetyl chloride with *3,4,3′,4′-tetramethoxydibenzyl in the presence of aluminium chloride in benzene at r.t for 1 h, then at reflux for 2.5 h (13%) [4376,4377].

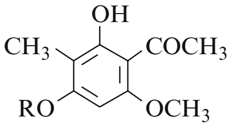
b.p.$_{20}$ 120–130° [4376]; m.p. 96–98° [4376]; UV [4376].
*1,2–(3,4-dimethoxyphenyl)ethane.

1-[4-[(6-O-L-Arabinofuranosyl-β-D-glucopyranosyl)oxy]-2-hydroxy-6-methoxy-3-methylphenyl]ethanone

1-[4-(-α-L-Arabinofuranosyl[1—>6]-β-D-glucopyranosyloxy)-2-hydroxy-6-methoxy-3-methylphenyl]ethanone

[883886-04-2] $C_{21}H_{30}O_{13}$ mol.wt. 490.46

New compound



Isolation from natural sources

- From the roots of *Euphorbia ebracteolata* Hayata (Euphorbiaceae) [4254].

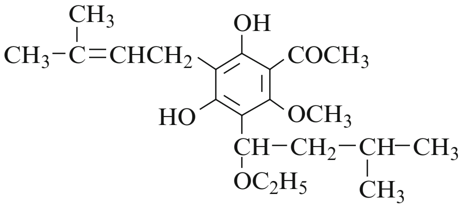
White needles [4254]; m.p. 256–258° [4254];
^{1}H NMR [4254], ^{13}C NMR [4254], IR [4254], MS [4254].

N.B.: R = α-D-Glucopyanosyl(6—>1)-β-L-arabinofuranosyl.

1-[3-(1-Ethoxy-3-methylbutyl)-4,6-dihydroxy-2-methoxy-5-(3-methyl-2-butenyl)-phenyl]ethanone

[253791-31-0] $C_{21}H_{32}O_5$ mol.wt. 364.48

New compound



Isolation from natural sources

- From the leaves of *Acronychia pedunculata* (L.) Miq. (Rutaceae) [4372].

Oil [4372];
^{1}H NMR [4372], ^{13}C NMR [4372], MS [4372].

Part VII
Monoketones Substituted on the Acetyl Groups

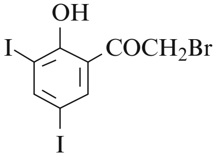
Chapter 11
Compounds Derived from Halogenoacetic Acids

11.1 Compounds Derived from Bromoacetic Acids

11.1.1 From Monobromoacetic Acid

2-Bromo-1-(2-hydroxy-3,5-diiodophenyl)ethanone

[32559-04-9] $C_8H_5BrI_2O_2$ mol.wt. 466.84



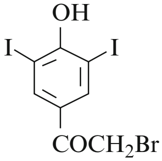
Synthesis

– Preparation by bromination of 2-hydroxy-3,5-diiodoacetophenone in acetic acid at 70–80°, under light irradiation (84%) [4378].

m.p. 150–151° [4378].

2-Bromo-1-(4-hydroxy-3,5-diiodophenyl)ethanone

[31827-97-1] $C_8H_5BrI_2O_2$ mol.wt. 466.84



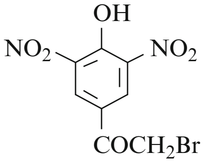
Synthesis

– Preparation by reaction of bromine on 4-hydroxy-3,5-di-iodoacetophenone in boiling chloroform under light irradiation (81–83%) [4379,4380].

m.p. 171–172° [4379,4380]; UV [4379].

2-Bromo-1-(4-hydroxy-3,5-dinitrophenyl)ethanone

[120388-18-3] $C_8H_5BrN_2O_6$ mol.wt. 305.04



Synthesis

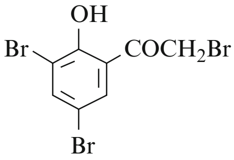
– Preparation by bromination of 4-hydroxy-3,5-dinitroacetophenone with cupric bromide in refluxing ethyl acetate (60%) [4381].

m.p. 92–94° [4381]; Crystal Data [4381].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_11,

2-Bromo-1-(3,5-dibromo-2-hydroxyphenyl)ethanone

[194226-52-3] $C_8H_5Br_3O_2$ mol.wt. 372.84



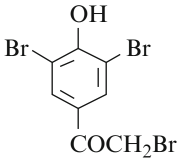
Syntheses

- Preparation by action of bromine with 3,5-dibromo-2-hydroxy-acetophenone in refluxing acetic acid for 2.5 h (55%) [4382].
- Also obtained by reaction of bromine with 2-hydroxyacetophenone in chloroform in an ice bath for 2 h [4383].

m.p. 107–108° [4382]; ^{1}H NMR [4382], IR [4382].

2-Bromo-1-(3,5-dibromo-4-hydroxyphenyl)ethanone

[34969-79-4] $C_8H_5Br_3O_2$ mol.wt. 372.84



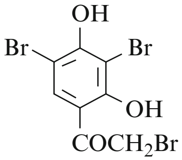
Synthesis

- Preparation by bromination of 3,5-dibromo-4-hydroxy-acetophenone in chloroform [4384–4386], (79%) [4386].

m.p. 137° [4386], 128° [4384,4385].

2-Bromo-1-(3,5-dibromo-2,4-dihydroxyphenyl)ethanone

$C_8H_5Br_3O_3$ mol.wt. 388.84



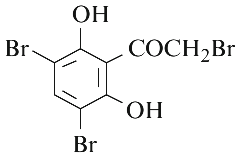
Synthesis

- Preparation by bromination of resacetophenone in acetic acid [4387].

m.p. 112–113° [4387].

2-Bromo-1-(3,5-dibromo-2,6-dihydroxyphenyl)ethanone

[74815-26-2] $C_8H_5Br_3O_3$ mol.wt. 388.84



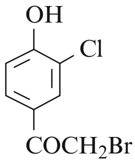
Synthesis

- Preparation by bromination of 2,6-dihydroxyacetophenone with cupric bromide in refluxing chloroform-ethyl acetate mixture [4388].

m.p. 150° [4388].

2-Bromo-1-(3-chloro-4-hydroxyphenyl)ethanone

[41877-19-4] $C_8H_6BrClO_2$ mol.wt. 249.49



Syntheses

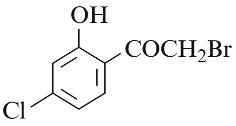
- Preparation by selective bromination of 3-chloro-4-hydroxy-acetophenone with dioxane dibromide in dioxane-ethyl ether mixture at r.t. (85%) [4389].

- Preparation by selective bromination of 3-chloro-4-hydroxy-acetophenone with cupric bromide in refluxing chloroform-ethyl acetate mixture [4390], (100%) [4391].

m.p. 128–130° [4389]; ^{1}H NMR [4389].

2-Bromo-1-(4-chloro-2-hydroxyphenyl)ethanone

[157068-00-3] $C_8H_6BrClO_2$ mol.wt. 249.49

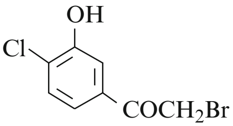


Synthesis

- Obtained by reaction of bromoacetyl bromide on 3-chloroanisole with aluminium chloride in carbon tetrachloride, first at 0°, then at r.t. (16%) [4392].

2-Bromo-1-(4-chloro-3-hydroxyphenyl)ethanone

[73898-34-7] $C_8H_6BrClO_2$ mol.wt. 249.49

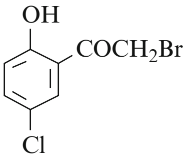


Synthesis

- Preparation by reaction of dioxane dibromide on 4-chloro-3-hydroxyacetophenone in dioxane-ethyl ether mixture at r.t. (84%) [4393].

2-Bromo-1-(5-chloro-2-hydroxyphenyl)ethanone

[52727-99-8] $C_8H_6BrClO_2$ mol.wt. 249.49



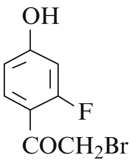
Syntheses

- Preparation by bromination of 5-chloro-2-hydroxy-acetophenone with cupric bromide in refluxing chloroform-ethyl acetate mixture (73%) [4394].
- Also refer to: [4395] (compound 1b).

m.p. 64–65° [4394]; ^{1}H NMR [4394].

2-Bromo-1-(2-fluoro-4-hydroxyphenyl)ethanone

[220131-30-6] $C_8H_6BrFO_2$ mol.wt. 233.04

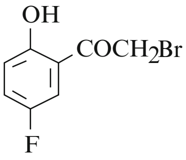


Synthesis

- Refer to: [4396].

2-Bromo-1-(5-fluoro-2-hydroxyphenyl)ethanone

[126581-65-5] $C_8H_6BrFO_2$ mol.wt. 233.04



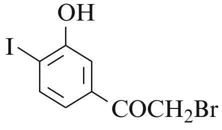
Synthesis

- Preparation by reaction of bromine on 5-fluoro-2-hydroxy-acetophenone in dioxane-ethyl ether mixture (80%) [4397,4398].

m.p. 86–87° [4397,4398]; ^{1}H NMR [4398].

2-Bromo-1-(3-hydroxy-4-iodophenyl)ethanone

[73898-36-9] $C_8H_6BrIO_2$ mol.wt. 340.94

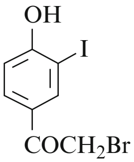


Synthesis

- Preparation by reaction of dioxane dibromide on 3-hydroxy-4-iodoacetophenone in dioxane-ethyl ether mixture at r.t. (75%) [4393].

2-Bromo-1-(4-hydroxy-3-iodophenyl)ethanone

[73898-29-0] $C_8H_6BrIO_2$ mol.wt. 340.94

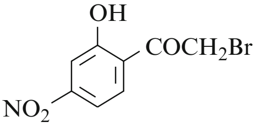


Synthesis

- Preparation by reaction of dioxane dibromide on 4-hydroxy-3-iodoacetophenone in dioxane-ethyl ether mixture at r.t. (87%) [4393].

2-Bromo-1-(2-hydroxy-4-nitrophenyl)ethanone

$C_8H_6BrNO_4$ mol.wt. 260.04



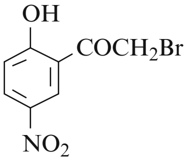
Synthesis

- Preparation by reaction of bromine on 2-hydroxy-4-nitroacetophenone in refluxing acetic acid (63%) [4399].

m.p. 112° [4399].

2-Bromo-1-(2-hydroxy-5-nitrophenyl)ethanone

[5037-70-7] $C_8H_6BrNO_4$ mol.wt. 260.04



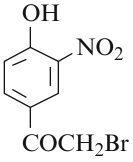
Synthesis

- Preparation by bromination of 2-hydroxy-5-nitroacetophenone in acetic acid (60%) [4399].

m.p. 127° [4399].

2-Bromo-1-(4-hydroxy-3-nitrophenyl)ethanone

[5029-61-8] $C_8H_6BrNO_4$ mol.wt. 260.04



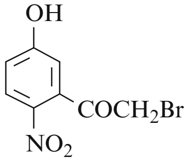
Syntheses

- Preparation by bromination of 4-hydroxy-3-nitroacetophenone in chloroform (74%) [4400], (71%) [4399] or in acetic acid (58%) [4401].
- Preparation by selective bromination of 4-hydroxy-3-nitroacetophenone with dioxane dibromide in dioxane-ethyl ether mixture at r.t. (94%) [4389].

m.p. 93° [4399,4401], 91°5–92° [4400], 80–82° [4389]; b.p.$_{0.2}$ 150–155° [4401].

2-Bromo-1-(5-hydroxy-2-nitrophenyl)ethanone

[50695-17-5] $C_8H_6BrNO_4$ mol.wt. 260.04



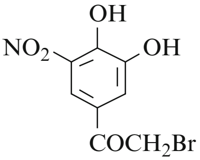
Synthesis

- Obtained by bromination of 5-hydroxy-2-nitroacetophenone in chloroform-carbon tetrachloride-ethyl acetate mixture at 61° (15%) [4400].

m.p. 112°5–113° [4400].

2-Bromo-1-(3,4-dihydroxy-5-nitrophenyl)ethanone

[134610-95-0] $C_8H_6BrNO_5$ mol.wt. 276.04



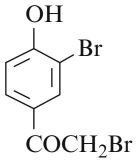
Synthesis

- Preparation by reaction of boron tribromide on 4-hydroxy-3-methoxy-5-nitro-α-bromoacetophenone in methylene chloride [4402].

m.p. 138–140° [4402].

2-Bromo-1-(3-bromo-4-hydroxyphenyl)ethanone

[41877-18-3] $C_8H_6Br_2O_2$ mol.wt. 293.94



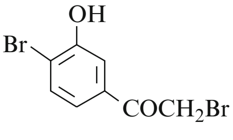
Syntheses

- Preparation by reaction of dioxane dibromide on 3-bromo-4-hydroxyacetophenone in dioxane-ethyl ether mixture at r.t. (82–85%) [4389,4393].
- Preparation by reaction of bromine on 4-hydroxyacetophenone in acetic acid at r.t. (37%) [4386].
- Preparation by reaction of phenyltrimethylammonium tribromide on 4-hydroxy-α-bromo-acetophenone [4403].
- Also refer to: [4404].

m.p. 143° [4386], 142–144° [4389], 140–142° [4405]; ^{1}H NMR [4389].

2-Bromo-1-(4-bromo-3-hydroxyphenyl)ethanone

[73898-35-8] $C_8H_6Br_2O_2$ mol.wt. 293.94

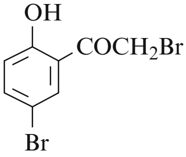


Synthesis

- Preparation by selective bromination of 4-bromo-3-hydroxy-acetophenone with dioxane dibromide in dioxane-ethyl ether mixture at r.t. (79%) [4393].

2-Bromo-1-(5-bromo-2-hydroxyphenyl)ethanone

[67029-74-7] $C_8H_6Br_2O_2$ mol.wt. 293.94



Syntheses

- Claimed to be prepared from 2-hydroxyacetophenone or 5-bromo-2-hydroxyacetophenone by reaction of bromine in glacial acetic acid and from 2-hydroxy-α-bromo-acetophenone by reaction ofbromine in 50% aqueous acetic acid (quantitative yields) (m.p. 107°) [4406]. No proof of structure was provided [4407]. Actually, it probably concerns 3,5-dibromo-2-hydroxyacetophenone (m.p. 108° [4408], 108–109° [4386]), as the use of acetic acid as solvent favours the aromatic ring bromination.
- Preparation by bromination of 5-bromo-2-hydroxyacetophenone with cupric bromide in refluxing chloroform-ethyl acetate mixture [4390,4407], (50%) [4407].

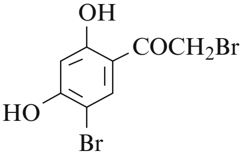
m.p. 107° [4406], 69° [4407].

One of the reported melting points is obviously wrong.

^{1}H NMR [4407], IR [4407].

2-Bromo-1-(5-bromo-2,4-dihydroxyphenyl)ethanone

[99657-26-8] $C_8H_6Br_2O_3$ mol.wt. 309.94

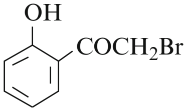


Synthesis

- Preparation by bromination of 5-bromo-2,4-dihydroxy-acetophenone with cupric bromide in refluxing chloroform-ethyl acetate mixture [4390,4409].

2-Bromo-1-(2-hydroxyphenyl)ethanone

[2491-36-3] $C_8H_7BrO_2$ mol.wt. 215.05



Syntheses

- Preparation by bromination of 2-hydroxyacetophenone with cupric bromide in refluxing chloroform-ethyl acetate mixture [4390,4391,4409–4413], (100%) [4390,4410], (56%) [4391], (36%) [4411].
- Preparation by reaction of bromine on 2-hydroxyacetophenone in acetic acid at r.t. (47%) [4406] or in a mixture of ethyl ether and chloroform [4413] according to [4414].
- Preparation by Fries rearrangement of phenyl bromoacetate with aluminium chloride without solvent at 120–140° (45–50%) [4415,4416].

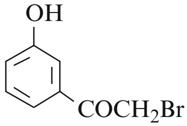
m.p. 70–71° [4416], 45° [4406], 44–45° [4391], 41–43° [4412], 40° [4390], 39–41° [4411].

One of the reported melting points is obviously wrong.

b.p.$_{7-10}$ 120–125° [4415], b.p.$_{18}$ 152–158° [4406];
^{1}H NMR [4411], UV [4417].

2-Bromo-1-(3-hydroxyphenyl)ethanone

[2491-37-4] $C_8H_7BrO_2$ mol.wt. 215.05



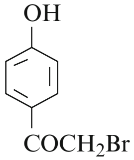
Syntheses

– Preparation by bromination of 3-hydroxyacetophenone,
  - with cupric bromide in refluxing chloroform-ethyl acetate mixture [4390,4391,4410,4412,4418], (quantitative yield) [4390,4391,4410];
  - with dioxane dibromide in dioxane-ethyl ether mixture at r.t. (91%) [4389];
  - in using silica gel coated with cupric bromide in refluxing ethyl acetate (94%) [4419];
  - with bromine in chloroform at 2° (96%) [4420].

m.p. 74–75° [4389], 70–72° [4420]; amorphous [4419];
^{1}H NMR [4389,4419], IR [4419,4420], UV [4420], MS [4419].

2-Bromo-1-(4-hydroxyphenyl)ethanone

[2491-38-5] $C_8H_7BrO_2$ mol.wt. 215.05



Syntheses

– Preparation by bromination of 4-hydroxyacetophenone with cupric bromide in refluxing chloroform-ethyl acetate mixture [4390,4391,4410–4413,4418,4421], (quantitative yield) [4390,4391,4410], (34–38%) [4411,4418], (15%) [4421].

– Preparation by reaction of dioxane dibromide on 4-hydroxyacetophenone in dioxane-ethyl ether mixture at r.t. (86%) [4389].

– Preparation by bromination of 4-hydroxyacetophenone in acetic acid (63%) [4422] or in a mixture of ethyl ether and chloroform [4413] according to [4414].

– Preparation by Fries rearrangement of phenyl bromoacetate with aluminium chloride without solvent between 120° and 140° (40%) [4415], (30%) [4416].

– Preparation by reaction of tetrabutylammonium tribromide, benzyltrimethylammonium tribromide or phenyltrimethylammonium tribromide on 4-hydroxyacetophenone in tetrahydrofuran [4403].

– Preparation by reaction of ammonium tribromide on 4-hydroxyacetophenone in methylene chloride-methanol mixture [4403].

– Preparation by reaction of silica gel coated with cupric bromide on 4-hydroxyacetophenone in refluxing ethyl acetate (95%) [4419].

– Preparation by sonochemical bromination of 4-hydroxyacetophenone using p-toluenesulfonic acid/N-bromosuccinimide in methanol for 6 h at 35–37° (97%) [4423].

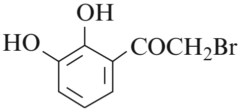
N.B.: In the absence of ultrasound the reaction takes place at the boiling point of methanol (65°) for 24 h (58%) [4423].

– Also refer to: [4424–4428].

m.p. 146° [4416], 132–133° [4429], 130–131° [4411], 130° [4422], 129–131° [4418], 128–130° [4403], 128–129° [4415], 126–127° [4421], 125–129° [4423], 124–126° [4389,4390], 121–122° [4419], 105–108° [4430];
^{1}H NMR [4389,4411,4419,4421,4423,4429], IR [4419,4423], UV [4421], MS [4419,4429].

2-Bromo-1-(2,3-dihydroxyphenyl)ethanone

[19278-79-6] $C_8H_7BrO_3$ mol.wt. 231.05



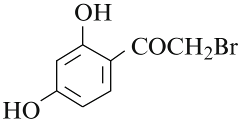
Synthesis

– Obtained by reaction of hydrobromic acid on 2,3-diacetoxy-α-bromoacetophenone in refluxing ethanol [4431].

m.p. 75–76° [4431]; UV [4417].

2-Bromo-1-(2,4-dihydroxyphenyl)ethanone

[2491-39-6] $C_8H_7BrO_3$ mol.wt. 231.05



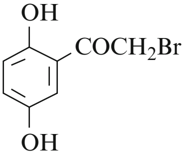
Syntheses

– Preparation by bromination of resacetophenone with cupric bromide in refluxing chloroform-ethyl acetate mixture (quantitative yield) [4390,4410], (3%) [4411].
– Also obtained by reaction of bromoacetonitrile on resorcinol with zinc chloride and hydrobromic acid in ethyl ether (Hoesch reaction) [4432,4433].
– Also obtained by reaction of bromoacetyl chloride on resorcinol with aluminium bromide or aluminium chloride in carbon disulfide [4421,4434], (80%) [4434], (12%) [4421].
– Also obtained (poor yield) by reaction of bromoacetic acid on resorcinol with zinc chloride or phosphorous oxychloride [4435,4436].

m.p. 144–145° [4390], 127° [4432,4433], 126–128° [4421], 118–119° [4411].
There is discrepancy between the different melting points.
^{1}H NMR [4411,4421], UV [4421].

2-Bromo-1-(2,5-dihydroxyphenyl)ethanone

[25015-91-2] $C_8H_7BrO_3$ mol.wt. 231.05



Syntheses

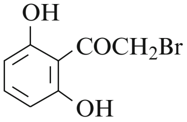
– Preparation by bromination of 2,5-dihydroxyacetophenone,
  - with cupric bromide in refluxing chloroform-ethyl acetate mixture (74–87%) [4390,4410,4437], (70%) [4438], (37%) [4411];
  - with bromine in acetic acid at 55–58° (16%) [4439].
– Preparation by reaction of hydrobromic acid on 5-α-bromoacetoxy-2-hydroxy-α-bromoacetophenone in methanol at r.t. (87%) [4440].

- Preparation by reaction of aluminium bromide on 2-hydroxy-5-methoxy-α-bromo-acetophenone in carbon disulfide at r.t. (87%) [4440].
- Also obtained by action of acetic acid saturated with hydrobromic acid (10 min, r.t.) on 2,5-di-acetoxy-α-diazoacetophenone, reduced pressure elimination of acetic acid, then action (overnight, r.t.) of a methanolic solution of hydrobromic acid (59%) [4440].
- Also obtained by reaction of aluminium bromide on 2,5-dimethoxy-α-chloroa cetophenone in refluxing carbon disulfide (28%) [4440].
- Also obtained by reaction of bromoacetyl bromide on 1,4-dimethoxybenzene with aluminium bromide at r.t. (11%) [4421], (2%) [4440].
- Also obtained by reaction of phenyltrimethylammonium bromide tribromide with 2,5-dihydroxy-acetophenone in THF at r.t. overnight (63%) [4441], according to [4442].

m.p. 120–121° [4439], 117°5–119° [4440], 117–119° [4421], 114–116° [4438], 113–115° [4411,4437], 112–113° [4390];
TLC [4441]; flash chromatography [4441];
^{1}H NMR [4411,4421,4438–4441], IR [4438,4441],
UV [4417,4421,4439,4441], MS [4438,4441].

2-Bromo-1-(2,6-dihydroxyphenyl)ethanone

[2491-40-9] $C_8H_7BrO_3$ mol.wt. 231.05



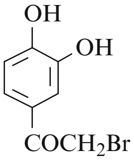
Syntheses

- Preparation by bromination of 2,6-dihydroxyacetophenone with cupric bromide in refluxing ethyl acetate (quantitative yield) [4391].
- Preparation by reaction of 40% hydrobromic acid on 2,6-diacetoxy-α-bromoacetophenone in refluxing 60% ethanol (73%) [4443].
- Refer to: [4444] (Japanese patent).

m.p. 143° [4443]; UV [4417].

2-Bromo-1-(3,4-dihydroxyphenyl)ethanone

[40131-99-5] $C_8H_7BrO_3$ mol.wt. 231.05



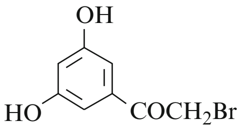
Syntheses

- Preparation by reaction of bromoacetic acid on pyrocatechol with phosphorous oxychloride [4445].
- Preparation by reaction of bromoacetyl bromide on pyrocatechol with aluminium bromide in carbon disulfide at r.t. (63%) [4446].
- Preparation by reaction of bromine on 3,4-dihydroxyacetophenone in chloroform at r.t. [4447,4448].
- Also refer to: [4404,4449–4453], and [4454] (Japanese patent).

m.p. 167° [4445], 61° [4446]. One of the reported melting points is obviously wrong.
^{1}H NMR [4446], IR [4446], UV [4446], MS [4446].

2-Bromo-1-(3,5-dihydroxyphenyl)ethanone

[62932-92-7] $C_8H_7BrO_3$ mol.wt. 231.05

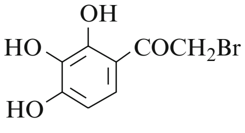


Syntheses

- Preparation by reaction of bromine on 3,5-dihydroxy-acetophenone in chloroform at r.t. [4447].
- Also refer to: [4404,4450].

2-Bromo-1-(2,3,4-trihydroxyphenyl)ethanone

[105190-52-1] $C_8H_7BrO_4$ mol.wt. 247.05



Syntheses

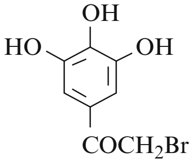
- Preparation by reaction of bromoacetyl bromide on pyrogallol with aluminium bromide in carbon disulfide at r.t. (42%) [4446].
- Also obtained by reaction of bromoacetic acid on pyrogallol with phosphorous oxychloride [4455], (poor yield) [4435,4436] or with zinc chloride (poor yield) [4435,4436].

m.p. 159° [4455], 138° [4446]. One of the reported melting points is obviously wrong.

^{1}H NMR [4446], IR [4446], UV [4446], MS [4446].

2-Bromo-1-(3,4,5-trihydroxyphenyl)ethanone

[111011-09-7] $C_8H_7BrO_4$ mol.wt. 247.05

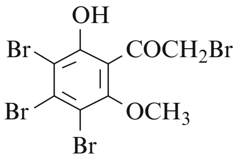


Synthesis

- Refer to: [4456].

2-Bromo-1-(3,4,5-tribromo-2-hydroxy-6-methoxyphenyl)ethanone

[98592-28-0] $C_9H_6Br_4O_3$ mol.wt. 481.76

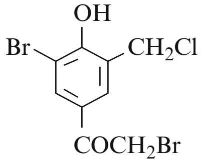


Synthesis not yet described

- Only one reference dealing with 2,3′,4′,5′-tetrabromo-2′-hydroxy-6′-methoxyacetophenone does exist. This reference [Chem. Abstr., Formula Index, page 112F, **52**, 2796h (1958)] is obviously erroneous. The described product is actually 2,2,3′,5′-tetrabromo-2′-hydroxy-6′-methoxyacet-ophenone, since in the original paper [4457] it is specified that two labile bromine atoms are present in the molecule.

2-Bromo-1-[3-bromo-5-(chloromethyl)-4-hydroxyphenyl]ethanone

[107700-04-9] $C_9H_7Br_2ClO_2$ mol.wt. 342.41



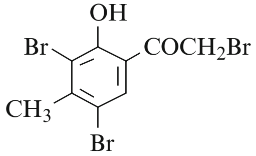
Synthesis

- Preparation by adding a methylene chloride solution of bromine to a methanol/methylene chloride solution of 3-(chloromethyl)-4-hydroxyacetophenone at r.t. (80%) [4458].

m.p. 95° [4458]; ^{1}H NMR [4458], IR [4458], MS [4458].

2-Bromo-1-(3,5-dibromo-2-hydroxy-4-methylphenyl)ethanone

[260435-53-8] $C_9H_7Br_3O_2$ mol.wt. 386.87



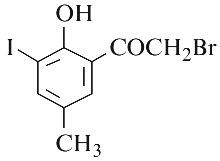
Syntheses

- Preparation by reaction of bromine with 2-hydroxy-4-methylacetophenone in chloroform/ethylene dichloride mixture, first in an ice-water bath, then for 2 h at r.t. (50%) [4459].
- Preparation by reaction of bromine with 3,5-dibromo-2-hydroxy-4-methylacetophenone in acetic acid first at r.t., then at reflux to discolouration (72%) [4460].

m.p. 139–140° [4459], 136–137° [4460];
^{1}H NMR [4459,4460], IR [4459,4460].

2-Bromo-1-(2-hydroxy-3-iodo-5-methylphenyl)ethanone

[194226-48-7] $C_9H_8BrIO_2$ mol.wt. 354.97

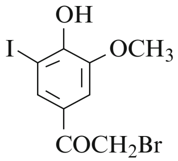


Synthesis

- Preparation [4461] (compound 1a) according to described procedure [4462] (Romanian patent).

2-Bromo-1-(4-hydroxy-3-iodo-5-methoxyphenyl)ethanone

[144978-69-8] $C_9H_8BrIO_3$ mol.wt. 370.97



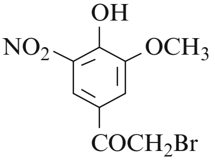
Syntheses

- Preparation by reaction of bromine with 5-iodoaceto-vanillone in chloroform (quantitative yield) [4463].
- Preparation from 4-hydroxy-3-methoxy-α-bromo-acetophenone by the oxidative procedure using chloramine T and sodium iodide in DMF, DMSO or acetonitrile [4463,4464].

^{1}H NMR [4463], ^{13}C NMR [4463], MS [4463].

2-Bromo-1-(4-hydroxy-3-methoxy-5-nitrophenyl)ethanone

[125629-36-9] $C_9H_8BrNO_5$ mol.wt. 290.07



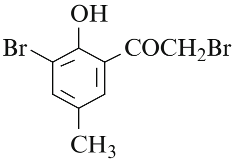
Synthesis

– Preparation by reaction of 96% nitric acid on 4-hydroxy-3-methoxy-α-bromoacetophenone in acetic acid at 20–25° [4402,4465], (72%) [4465].

m.p. 147–149° [4402,4465]; ^{1}H NMR [4465].

2-Bromo-1-(3-bromo-2-hydroxy-5-methylphenyl)ethanone

[194226-50-1] $C_9H_8Br_2O_2$ mol.wt. 307.97



Syntheses

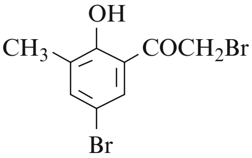
– Preparation by reaction of bromine on 2-hydroxy-5-methyl-α-bromoacetophenone in 50% aqueous acetic acid at 60° (75%) [4406].

– Also obtained by reaction of bromine with 2-hydroxy-5-methylacetophenone in chloroform in an ice bath for 2 h [4383].

m.p. 106° [4406].

2-Bromo-1-(5-bromo-2-hydroxy-3-methylphenyl)ethanone

[194226-51-2] $C_9H_8Br_2O_2$ mol.wt. 307.97



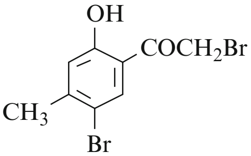
Synthesis

– Preparation by reaction of bromine (1 mol) with 5-bromo-2-hydroxy-3-methylacetophenone (1 mol) in acetic acid on a water bath for 20 min (47%) [4466].

m.p. 72–73° [4466]; ^{1}H NMR [4466], IR [4466].

2-Bromo-1-(5-bromo-2-hydroxy-4-methylphenyl)ethanone

[194226-49-8] $C_9H_8Br_2O_2$ mol.wt. 307.97

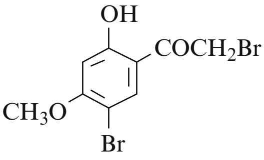


Synthesis

– Obtained by reaction of bromine with 2-hydroxy-4-methyl-acetophenone in chloroform in an ice bath for 2 h [4383].

2-Bromo-1-(5-bromo-2-hydroxy-4-methoxyphenyl)ethanone

$C_9H_8Br_2O_3$ mol.wt. 323.97



Syntheses

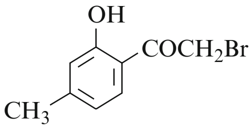
– Obtained by reaction of bromine with 3,3′-diacetyl-4,4′-di-hydroxy-6,6′-dimethoxydiphenyl ether in acetic acid in the presence of a crystal of iodine, first at 90°, then at r.t. overnight [4467].

- Also obtained (by-product) by reaction of bromine on 5-bromo-2-hydroxy-4-methoxyacetophenone in acetic acid [4468].

m.p. 178–180° [4468], 72–73° [4467]. One of the reported melting points is obviously wrong.

2-Bromo-1-(2-hydroxy-4-methylphenyl)ethanone

[144219-74-9] $C_9H_9BrO_2$ mol.wt. 229.07



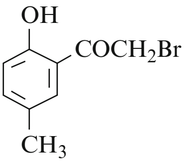
Synthesis

- Preparation by reaction of bromine with 2-acetoxy-4-methylacetophenone in refluxing chloroform (44%) [4469].

colourless oil [4469];
^{1}H NMR [4469], IR [4469], MS [4469].

2-Bromo-1-(2-hydroxy-5-methylphenyl)ethanone

[51317-87-4] $C_9H_9BrO_2$ mol.wt. 229.07



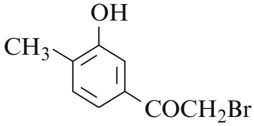
Syntheses

- Preparation by reaction of bromoacetic acid on p-cresol with boron trifluoride into an autoclave at 70° (90%) [4470].
- Preparation by Fries rearrangement of p-cresyl bromoacetate with aluminium chloride without solvent at 125° (47%) [4471].
- Preparation by reaction of bromine on 2-hydroxy-5-methylacetophenone in acetic acid at r.t. (39%) [4406].

m.p. 45°5–46°5 [4471], 44–45° [4470].

2-Bromo-1-(3-hydroxy-4-methylphenyl)ethanone

[73898-30-3] $C_9H_9BrO_2$ mol.wt. 229.07

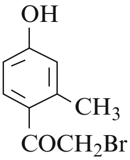


Synthesis

- Preparation by selective bromination of 3-hydroxy-4-methylacetophenone with dioxane dibromide in dioxane-ethyl ether mixture at r.t. (42%) [4393].

2-Bromo-1-(4-hydroxy-2-methylphenyl)ethanone

[41877-16-1] $C_9H_9BrO_2$ mol.wt. 229.07



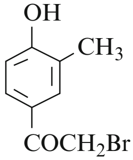
Synthesis

- Preparation by selective bromination of 4-hydroxy-2-methylacetophenone with dioxane dibromide in dioxane-ethyl ether mixture at r.t. (86%) [4389].

m.p. 122–124° [4389];
^{1}H NMR [4389].

2-Bromo-1-(4-hydroxy-3-methylphenyl)ethanone

[41877-17-2] $C_9H_9BrO_2$ mol.wt. 229.07



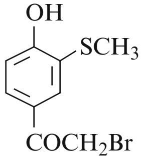
Syntheses

- Preparation by selective bromination of 4-hydroxy-3-methylacetophenone with dioxane dibromide in dioxane-ethyl ether mixture at r t. (98%) [4389], (78%) [4393].
- Preparation by selective bromination of 4-hydroxy-3-methyl-acetophenone with cupric bromide in refluxing chloroform-ethyl acetate mixture (quantitative yield) [4391].

m.p. 124–125° [4389]; ^{1}H NMR [4389].

2-Bromo-1-[4-hydroxy-3-(methylthio)phenyl]ethanone

[66265-63-2] $C_9H_9BrO_2S$ mol.wt. 261.14



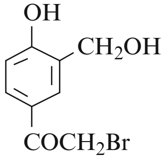
Synthesis

- Preparation by reaction of dioxane dibromide on 4-hydroxy-3-(methylthio)acetophenone in dioxane-ethyl ether mixture (75%) [4472,4473].

solid [4472].

2-Bromo-1-[4-hydroxy-3-(hydroxymethyl)phenyl]ethanone

[62932-94-9] $C_9H_9BrO_3$ mol.wt. 245.07

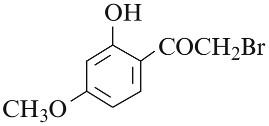


Syntheses

- Refer to: [4474–4482].

2-Bromo-1-(2-hydroxy-4-methoxyphenyl)ethanone

[60965-24-4] $C_9H_9BrO_3$ mol.wt. 245.07



Syntheses

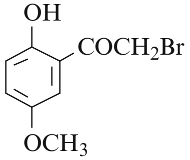
- Obtained by reaction of cupric bromide with 2-hydroxy-4-methoxyacetophenone, in refluxing chloroform-ethyl acetate mixture [4391,4413], (quantitative yield) [4391] according to [4390], (62%) [4483] or in refluxing dioxane (44%) [4484], (10%) [4411].
- Preparation by reaction of bromine with 2-hydroxy-4-methoxyacetophenone in a mixture of ethyl ether and chloroform [4413] according to [4414].
- Preparation from resorcinol dimethyl ether,
  - by reaction of bromoacetyl bromide with aluminium bromide at r.t. [4485];
  - by reaction of bromoacetyl chloride with aluminium chloride in carbon disulfide [4484,4486], (23%) [4484].

- Preparation by reaction of bromoacetonitrile on resorcinol dimethyl ether with hydrobromic acid gas in ethyl ether [4433].

m.p. 161° [4484], 92° [4485], 90–92° [4411], 70–72° [4483].
There is discrepancy between the different melting points.
^{1}H NMR [4411,4483], IR [4483], MS [4483].

2-Bromo-1-(2-hydroxy-5-methoxyphenyl)ethanone

[203524-87-2] $C_9H_9BrO_3$ mol.wt. 245.07



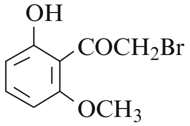
Syntheses

- Preparation by reaction of bromoacetyl bromide on 1,4-dimethoxybenzene with aluminium bromide at r.t. (18%) [4440].
- Also refer to: [4487].

m.p. 65–66° [4440].

2-Bromo-1-(2-hydroxy-6-methoxyphenyl)ethanone

[50879-47-5] $C_9H_9BrO_3$ mol.wt. 245.07



Synthesis

- Claimed to be prepared by bromination of 2-hydroxy-6-methoxyacetophenone in glacial acetic acid [4488].

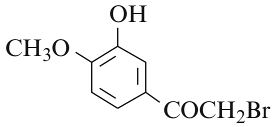
N.B.: It has been observed that glacial acetic acid promotes side chain bromination of 2-hydroxyacetophenones [4422].

However, actually, 3-bromo-2-hydroxy-6-methoxyacetophenone was obtained by bromination of 2-hydroxy-6-methoxyacetophenone in glacial acetic acid, dilute acetic acid (80%) or acetic anhydride [4489,4490].

m.p. 106° [4488,4490].

2-Bromo-1-(3-hydroxy-4-methoxyphenyl)ethanone

[90971-90-7] $C_9H_9BrO_3$ mol.wt. 245.07

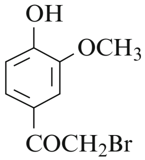


Synthesis

- Preparation by reaction of cupric bromide on 3-hydroxy-4-methoxyacetophenone in refluxing dioxane (80%) [4391].

2-Bromo-1-(4-hydroxy-3-methoxyphenyl)ethanone

[69638-06-8] $C_9H_9BrO_3$ mol.wt. 245.07



Syntheses

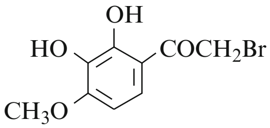
- Preparation by reaction of cupric bromide on 4-hydroxy-3-methoxyacetophenone in refluxing chloroform-ethyl acetate mixture (quantitative yield) [4391].
- Preparation by reaction of bromoacetyl bromide on guaiacol with aluminium chloride in carbon disulfide (75%) [4491].

- Preparation by reaction of bromine with 4-hydroxy-3-methoxyacetophenone [4429,4492] in ice cooled solution of ethyl ether and dioxane (quantitative yield) [4492].

m.p. 78–79° [4491]; ^{1}H NMR [4492], IR [4492], MS [4492].

2-Bromo-1-(2,3-dihydroxy-4-methoxyphenyl)ethanone

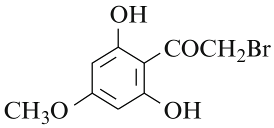
[204648-67-9] $C_9H_9BrO_4$ mol.wt. 261.07



Synthesis

- Refer to: [4493] (Japanese patent).

2-Bromo-1-(2,6-dihydroxy-4-methoxyphenyl)ethanone



$C_9H_9BrO_4$ mol.wt. 261.07

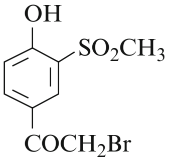
Synthesis

- Preparation by hydrolysis of 2,6-dihydroxy-4-methoxy-α-bromoacetophenone with 16% hydrobromic acid in refluxing ethanol (94%) [4494].

m.p. 139°5–140°5 (d) [4494].

2-Bromo-1-[4-hydroxy-3-(methylsulfonyl)phenyl]ethanone

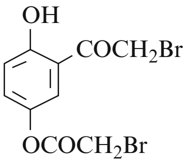
[66264-67-3] $C_9H_9BrO_4S$ mol.wt. 293.14



Synthesis

- Preparation by reaction of aluminium chloride on 4-methoxy-3-(methylsulfonyl)-α-bromoacetophenone in refluxing chlorobenzene (70%) [4473].
  crystalline solid [4473].

2-Bromo-1-[5-(2-bromoacetyloxy)-2-hydroxyphenyl]ethanone



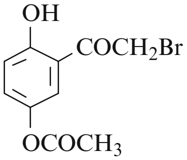
$C_{10}H_8Br_2O_4$ mol.wt. 352.00

Synthesis

- Preparation by reaction of bromoacetyl bromide on hydroquinone dimethyl ether with aluminium bromide (13–16%) [4421,4440].

m.p. 106–107° [4440], 105–107° [4421].

1-[5-(Acetyloxy)-2-hydroxyphenyl]-2-bromoethanone



$C_{10}H_9BrO_4$ mol.wt. 273.08

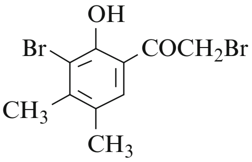
Synthesis

- Preparation from 2,5-dihydroxy-α-bromoacetophenone on heating with acetyl bromide (62%) [4440].

m.p. 143°5–144° [4440].

2-Bromo-1-(3-bromo-2-hydroxy-4,5-dimethylphenyl)ethanone

[319923-52-9] $C_{10}H_{10}Br_2O_2$ mol.wt. 322.00



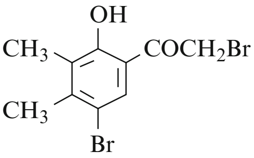
Synthesis

- Preparation by reaction of bromine with 3-bromo-2-hydroxy-4,5-dimethylacetophenone in acetic acid first at r.t., then at reflux to discolouration (2 min) (80%) [4495].

m.p. 109–110° [4495]; ^{1}H NMR [4495], IR [4495].

2-Bromo-1-(5-bromo-2-hydroxy-3,4-dimethylphenyl)ethanone

[260430-25-9] $C_{10}H_{10}Br_2O_2$ mol.wt. 322.00



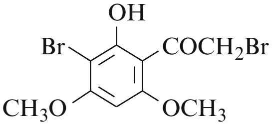
Syntheses

- Preparation by reaction of bromine with 2-hydroxy-3,4-dimethylacetophenone,
  - in chloroform/ethylene dichloride mixture, first in an ice-water bath, then for 1 h at r.t. (68%) [4459];
  - in chloroform in an ice-water bath for 2 h [4383].
- Preparation by reaction of bromine with 5-bromo-2-hydroxy-3,4-dimethylacetophenone in acetic acid first at r.t., then at reflux to discolouration (2 min) (68%) [4495].

m.p. 97–98° [4459,4495]; ^{1}H NMR [4459,4495], IR [4459,4495].

2-Bromo-1-(3-bromo-2-hydroxy-4,6-dimethoxyphenyl)ethanone

[29784-35-8] $C_{10}H_{10}Br_2O_4$ mol.wt. 354.00



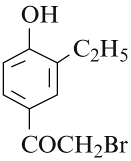
Synthesis

- Obtained by reaction of bromine on 2-hydroxy-4,6-di-methoxyacetophenone in chloroform (22%) [4489].

m.p. 194–195° [4489]; ^{1}H NMR [4489].

2-Bromo-1-(3-ethyl-4-hydroxyphenyl)ethanone

[73898-24-5] $C_{10}H_{11}BrO_2$ mol.wt. 243.10

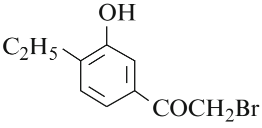


Syntheses

- Preparation by reaction of dioxane dibromide on 3-ethyl-4-hydroxyacetophenone in dioxane-ethyl ether mixture at r.t. (63%) [4393].
- Also refer to: [4496].

2-Bromo-1-(4-ethyl-3-hydroxyphenyl)ethanone

[73898-31-4] $C_{10}H_{11}BrO_2$ mol.wt. 243.10

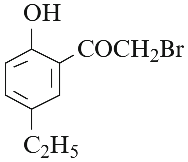


Synthesis

– Preparation by reaction of dioxane dibromide on 4-ethyl-3-hydroxyacetophenone in dioxane-ethyl ether mixture at r.t. (67%) [4393].

2-Bromo-1-(5-ethyl-2-hydroxyphenyl)ethanone

[180154-50-1] $C_{10}H_{11}BrO_2$ mol.wt. 243.10

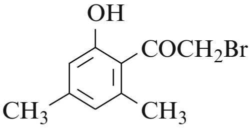


Synthesis

– Refer to: [4497].

2-Bromo-1-(2-hydroxy-4,6-dimethylphenyl)ethanone

[67029-80-5] $C_{10}H_{11}BrO_2$ mol.wt. 243.10

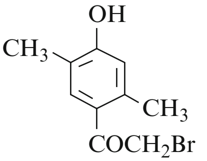


Synthesis

– Preparation by reaction of cupric bromide on 2-hydroxy-4,6-dimethylacetophenone in refluxing chloroform-ethyl acetate mixture [4407].

2-Bromo-1-(4-hydroxy-2,5-dimethylphenyl)ethanone

[107584-78-1] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



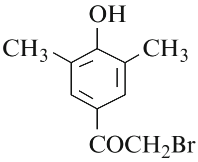
Synthesis

– Preparation from 4-hydroxy-2,5-dimethylacetophenone by standard bromination procedure according to [4492], (62%) [4429].

m.p. 129–131° [4429]; ^{1}H NMR [4429], MS [4429].

2-Bromo-1-(4-hydroxy-3,5-dimethylphenyl)ethanone

[157014-27-2] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



Syntheses

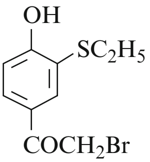
– Preparation by reaction of 4-hydroxy-3,5-dimethyl-α-chloroacetophenone in boiling methylene chloride with 46–48% aqueous hydrobromic acid in the presence of tetrabutylammonium bromide (0.3 M relative to the ketone) for 24 h, (87% yield) [4498].

– Also obtained from 4-(benzyloxy)-3,5-dimethyl-α-bromoacetophenone by the former treatment (83%) [4499].

- Also refer to: [4500].

m.p. 131° [4498], 130°6 [4499]; ^{1}H NMR [4498,4499], IR [4499], MS [4498,4499].

2-Bromo-1-[4-hydroxy-3-(ethylthio)phenyl]ethanone



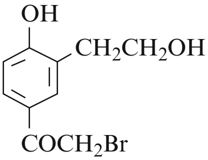
$C_{10}H_{11}BrO_2S$ mol.wt. 275.17

Synthesis

- Preparation by reaction of bromine on 3-(ethylthio)-4-hydroxyacetophenone in chloroform, in the presence of calcium carbonate at 25° [4435,4473].

2-Bromo-1-[4-hydroxy-3-(2-hydroxyethyl)phenyl]ethanone

[101386-50-9] $C_{10}H_{11}BrO_3$ mol.wt. 259.10

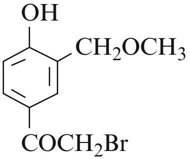


Syntheses

- Refer to: [4501,4502].

2-Bromo-1-[4-hydroxy-3-(methoxymethyl)phenyl]ethanone

[91363-39-2] $C_{10}H_{11}BrO_3$ mol.wt. 259.10

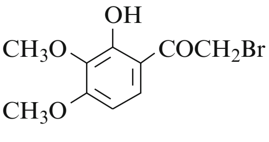


Synthesis

- Refer to: [4503].

2-Bromo-1-(2-hydroxy-3,4-dimethoxyphenyl)ethanone

[18064-92-1] $C_{10}H_{11}BrO_4$ mol.wt. 275.10



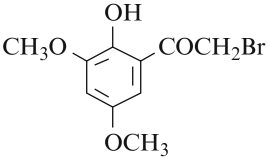
Syntheses

- Preparation by reaction of cupric bromide on 2-hydroxy-3,4-dimethoxyacetophenone [4504] or 2,3,4-trimethoxy-acetophenone [4505] in refluxing chloroform-ethyl acetate mixture (47%) [4504], (26%) [4505].
- Preparation by reaction of bromine on 2-hydroxy-3,4-dimethoxyacetophenone in chloroform-ethyl ether solution (44%) [4414].
- Preparation by reaction of bromoacetyl chloride with 1,2,3-trimethoxybenzene in the presence of aluminium chloride in methylene chloride at 20° (42%) [4506].

m.p. 144–145° [4414], 142° [4506], 140–142°5 [4505], 140–141° [4504]; ^{1}H NMR [4414,4504,4505], IR [4414,4506,4507].

2-Bromo-1-(2-hydroxy-3,5-dimethoxyphenyl)ethanone

[204648-51-1] $C_{10}H_{11}BrO_4$ mol.wt. 275.10

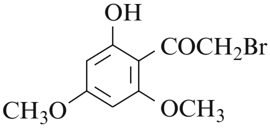


Synthesis

– Refer to: [4493] (Japanese patent).

2-Bromo-1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone

[18064-88-5] $C_{10}H_{11}BrO_4$ mol.wt. 275.10



Syntheses

– Preparation by reaction of aluminium bromide with 2,4,6-trimethoxy-α-bromoacetophenone at 120° [4508].

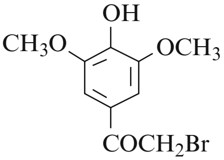
– Preparation by reaction of bromoacetyl bromide with phloroglucinol trimethyl ether according to [4509], but using aluminium bromide instead of aluminium chloride in carbon disulfide at r.t. [4508].

– Also obtained by reaction of cupric bromide on 2-hydroxy-4,6-dimethoxyacetophenone in refluxing chloroform-ethyl acetate mixture [4410,4505], (2%) [4505].

m.p. 130–131° [4508], 125–126° [4505]; ^{1}H NMR [4410,4505], MS [4410].

2-Bromo-1-(4-hydroxy-3,5-dimethoxyphenyl)ethanone

[51149-28-1] $C_{10}H_{11}BrO_4$ mol.wt. 275.10



Syntheses

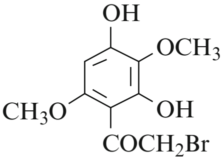
– Preparation by bromination of 4-hydroxy-3,5-dimethoxy-acetophenone with,
  - bromine in chloroform [4463,4510–4512], (90%) [4511], (27%) [4512];
  - cupric bromide in a refluxing mixture of ethyl acetate and chloroform [4513].

– Also refer to: [4514].

m.p. 130° [4511], 118–120° [4512]; ^{1}H NMR [4511,4512], IR [4512], MS [4513].

2-Bromo-1-(2,4-dihydroxy-3,6-dimethoxyphenyl)ethanone

$C_{10}H_{11}BrO_5$ mol.wt. 291.10



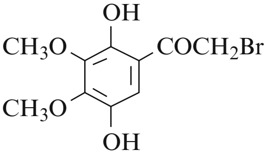
Synthesis

– Preparation by reaction of NBS on 2,4-dihydroxy-3,6-dimethoxyacetophenone in refluxing carbon tetrachloride (64%) [4515].

m.p. 159–160° [4515].

2-Bromo-1-(2,5-dihydroxy-3,4-dimethoxyphenyl)ethanone

[204648-54-4]



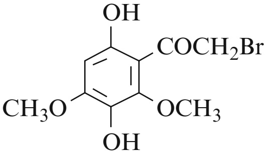
$C_{10}H_{11}BrO_5$ mol.wt. 291.10

Synthesis

- Refer to: [4493] (Japanese paper).

2-Bromo-1-(3,6-dihydroxy-2,4-dimethoxyphenyl)ethanone

[204648-57-7]



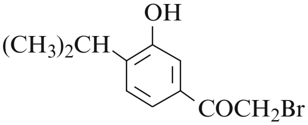
$C_{10}H_{11}BrO_5$ mol.wt. 291.10

Synthesis

- Refer to: [4493] (Japanese paper).

2-Bromo-1-[3-hydroxy-4-(1-methylethyl)phenyl]ethanone

[73898-32-5]



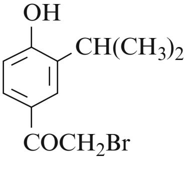
$C_{11}H_{13}BrO_2$ mol.wt. 257.13

Synthesis

- Preparation by bromination of 3-hydroxy-4-isopropyl-acetophenone with dioxane dibromide in dioxane-ethyl ether mixture at r.t. (78%) [4393].

2-Bromo-1-[4-hydroxy-3-(1-methylethyl)phenyl]ethanone

[73898-25-6]

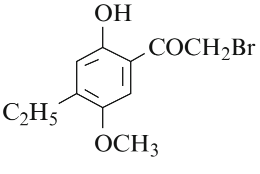


$C_{11}H_{13}BrO_2$ mol.wt. 257.13

Synthesis

- Preparation by bromination of 4-hydroxy-3-isopropyl-acetophenone with dioxane dibromide in dioxane-ethyl ether mixture at r.t. (55%) [4393].

2-Bromo-1-(4-ethyl-2-hydroxy-5-methoxyphenyl)ethanone



$C_{11}H_{13}BrO_3$ mol.wt. 273.13

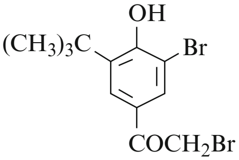
Synthesis

- Preparation by reaction of bromine on 4-ethyl-2-hydroxy-5-methoxyacetophenone in acetic acid at r.t. (59%) [4516].

m.p. 75–76° [4516].

2-Bromo-1-[3-bromo-5-(1,1-dimethylethyl)-4-hydroxyphenyl]ethanone

[153355-99-8] $C_{12}H_{14}Br_2O_2$ mol.wt. 350.05

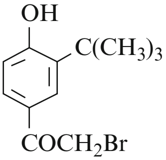


Synthesis

- Refer to: [4517] (Japanese patent).

2-Bromo-1-[3-(1,1-dimethylethyl)-4-hydroxyphenyl]ethanone

[118788-50-4] $C_{12}H_{15}BrO_2$ mol.wt. 271.15

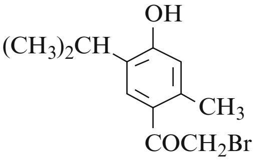


Synthesis

- Refer to: [4518].

2-Bromo-1-[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]ethanone

[205655-36-3] $C_{12}H_{15}BrO_2$ mol.wt. 271.15



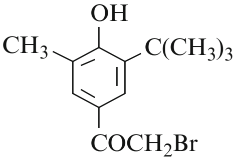
Synthesis

- Refer to: [4519].

N.B.: All reagents and starting materials were from commercial sources. This compound was called 4-hydroxy-3-isopropyl-6-methyl phenacyl bromide (p. 185).

2-Bromo-1-[3-(1,1-dimethylethyl)-4-hydroxy-5-methylphenyl]ethanone

[18611-32-0] $C_{13}H_{17}BrO_2$ mol.wt. 285.18



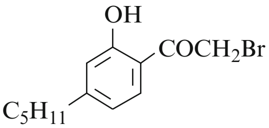
Syntheses

- Preparation by bromination of 3-tert-butyl-4-hydroxy-5-methylacetophenone in usual manner in benzene or chloroform [4520].
- Preparation by reaction of cupric bromide with 3-tert-butyl-4-hydroxy-5-methylacetophenone in refluxing ethyl acetate (72%) [4518].

m.p. 95–97° [4520], 90–92° [4518].

2-Bromo-1-(2-hydroxy-4-pentylphenyl)ethanone

[133301-45-8] $C_{13}H_{17}BrO_2$ mol.wt. 285.18



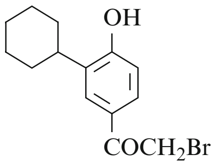
Synthesis

- Obtained by reaction of cupric bromide with 2-hydroxy-4-pentylacetophenone in refluxing ethyl acetate/chloroform mixture (1:1) for 4 h (18%) [4521].

^{1}H NMR [4521], IR [4521]; TLC [4521].

2-Bromo-1-(3-cyclohexyl-4-hydroxyphenyl)ethanone

[73898-26-7] $C_{14}H_{17}BrO_2$ mol.wt. 297.19

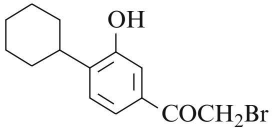


Synthesis

- Preparation by reaction of dioxane dibromide on 3-cyclohexyl-4-hydroxyacetophenone in dioxane and ethyl ether mixture at r.t. (95%) [4393].

2-Bromo-1-(4-cyclohexyl-3-hydroxyphenyl)ethanone

[73898-33-6] $C_{14}H_{17}BrO_2$ mol.wt. 297.19

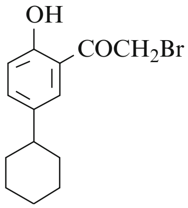


Synthesis

- Preparation by reaction of dioxane dibromide on 4-cyclohexyl-3-hydroxyacetophenone in dioxane and ethyl ether mixture at r.t. (51%) [4393].

2-Bromo-1-(5-cyclohexyl-2-hydroxyphenyl)ethanone

[74815-30-8] $C_{14}H_{17}BrO_2$ mol.wt. 297.19

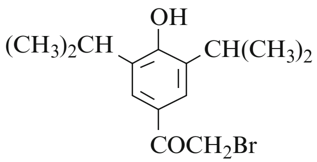


Synthesis

- Preparation by reaction of cupric bromide on 5-cyclohexyl-2-hydroxyacetophenone in refluxing ethyl acetate [4388].
- yellow oil [4388].

2-Bromo-1-[4-hydroxy-3,5-bis(1-methylethyl)phenyl]ethanone

[157014-26-1] $C_{14}H_{19}BrO_2$ mol.wt. 299.21

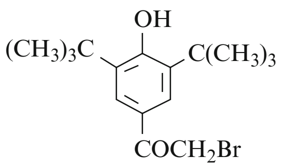


Synthesis

- Preparation by a Friedel–Crafts acylation of propofol (2,6-diisopropylphenol) using aluminium chloride and bromoacetyl bromide in methylene chloride (compound 6) [4522].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-2-bromoethanone

[14386-64-2] $C_{16}H_{23}BrO_2$ mol.wt. 327.26



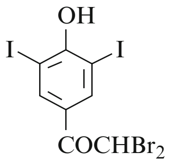
Synthesis

- Preparation by reaction of bromine with 3,5-di-tert-butyl-4-hydroxyacetophenone in ethanol under nitrogen bubbling for 2.5 h (73%) [4523] or in refluxing methylene chloride (67%) [4518].

m.p. 107°5–108°5 [4523], 105–108° [4518]; ^{1}H NMR [4523].

11.1.2 From Dibromoacetic Acid

2,2-Dibromo-1-(4-hydroxy-3,5-diiodophenyl)ethanone



$C_8H_4Br_2I_2O_2$ mol.wt. 545.74

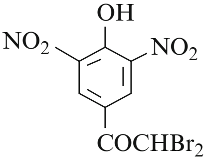
Synthesis

- Obtained by reaction of bromine on 4-hydroxy-3,5-diiodo-acetophenone with sunlight in chloroform at 50–60° (83%) [4524].

m.p. 132–133° [4524].

2,2-Dibromo-1-(4-hydroxy-3,5-dinitrophenyl)ethanone

[120388-19-4]



$C_8H_4Br_2N_2O_6$ mol.wt. 383.94

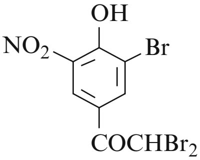
Synthesis

- Preparation by bromination of 4-hydroxy-3,5-dinitro-acetophenone with excess cupric bromide in refluxing ethyl acetate (66%) [4381].

m.p. 93–95° [4381].

2,2-Dibromo-1-(3-bromo-4-hydroxy-5-nitrophenyl)ethanone

[35928-54-2]



$C_8H_4Br_3NO_4$ mol.wt. 417.84

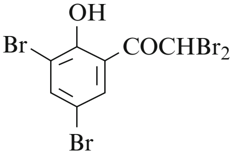
Synthesis

- Preparation by bromination of 3-bromo-4-hydroxy-5-nitro-acetophenone in acetic acid-sulfuric acid solution at 25° (78%) [4525].

m.p. 121° [4525]; ^{1}H NMR [4525].

2,2-Dibromo-1-(3,5-dibromo-2-hydroxyphenyl)ethanone

[49619-83-2]



$C_8H_4Br_4O_2$ mol.wt. 451.73

Syntheses

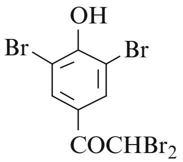
- Preparation by reaction of bromine on 2-hydroxy-acetophenone in acetic acid [4386,4526], (62%) [4386].
- Also obtained by reaction of aqueous sodium hypobromite on chromone-3-carboxaldehyde in acetic acid (24–30%) [4527,4528].
- Also obtained by reaction of sodium sulfite on 3,5-dibromo-2-hydroxy-α,α,α-tribromo-acetophenone, in boiling acetic acid [4529].

- Also obtained by reaction of bromine on 3,3′-diacetyl-4,4′-dihydroxydiphenyl thioether [4530].

m.p. 124–125° [4527], 122–123° [4529], 121–122° [4528], 120–121° [4386,4526,4530];

^{1}H NMR [4527,4528], IR [4527,4528], UV [4527], MS [4527,4528].

2,2-Dibromo-1-(3,5-dibromo-4-hydroxyphenyl)ethanone



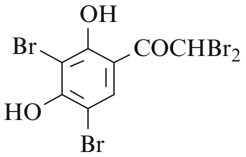
$C_8H_4Br_4O_2$ mol.wt. 451.73

Syntheses

- Preparation by reaction of bromine on 3,5-dibromo-4-hydroxyacetophenone [4385,4386].
- Preparation by reaction of bromine on 4-hydroxyacetophenone in acetic acid (60%) [4386].

m.p. 105–106° [4386], 105° [4385].

2,2-Dibromo-1-(3,5-dibromo-2,4-dihydroxyphenyl)ethanone



$C_8H_4Br_4O_3$ mol.wt. 467.73

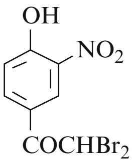
Synthesis

- Preparation by reaction of bromine on resacetophenone in acetic acid [4531].

m.p. 110–110°5 [4531].

2,2-Dibromo-1-(4-hydroxy-3-nitrophenyl)ethanone

[35928-53-1] $C_8H_5Br_2NO_4$ mol.wt. 338.94

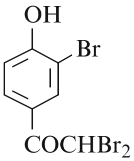


Syntheses

- Preparation by bromination of 4-hydroxy-3-nitro-acetophenone with excess cupric bromide in refluxing ethyl acetate (56–82%) [4381].
- Preparation by bromination of 4-hydroxy-3-nitro-acetophenone in acetic acid-sulfuric acid mixture at 25° (49%) [4525].

m.p. 63° [4525]; ^{1}H NMR [4525].

2,2-Dibromo-1-(3-bromo-4-hydroxyphenyl)ethanone



$C_8H_5Br_3O_2$ mol.wt. 372.84

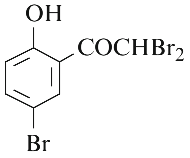
Synthesis

- Preparation by reaction of bromine on 4-hydroxy-acetophenone in acetic acid or chloroform (65%) [4386].

m.p. 139° [4386].

2,2-Dibromo-1-(5-bromo-2-hydroxyphenyl)ethanone

$C_8H_5Br_3O_2$ mol.wt. 372.84



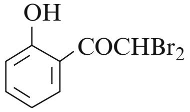
Synthesis

- Preparation by reaction of bromine on 2-hydroxy-acetophenone in chloroform or acetic acid (quantitative yield) [4386].

m.p. 103–104° [4386].

2,2-Dibromo-1-(2-hydroxyphenyl)ethanone

[54735-43-2] $C_8H_6Br_2O_2$ mol.wt. 293.94



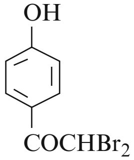
Syntheses

- Obtained by transformation of 4-hydroxycoumarin with tetrabutylammonium bromide promoted by hydrogen peroxide and vanadium pentoxide at 5° for 1 h (55%) [4532].
- Also refer to: [4533].

^{1}H NMR [4532], ^{13}C NMR [4532], UV [4533]; TLC [4532], GC [4532].

2,2-Dibromo-1-(4-hydroxyphenyl)ethanone

[92596-96-8] $C_8H_6Br_2O_2$ mol.wt. 293.94



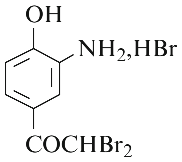
Syntheses

- Preparation by bromination of 4-hydroxyacetophenone with excess cupric bromide in refluxing ethyl acetate (56–82%) [4381].
  - Also obtained (by-product) by reaction of bromine-with p-hydroxyacetophenone in dioxane at r.t. for 40 min (<6%) [4534].

m.p. 121–122° [4534]; ^{1}H NMR [4534], IR [4534].

1-(3-Amino-4-hydroxyphenyl)-2,2-dibromoethanone *(Hydrobromide)*

$C_8H_7Br_2NO_2$, HBr mol.wt. 389.87



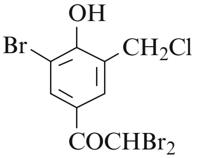
Synthesis

- Preparation by reaction of bromine on 3-amino-4-hydroxyacetophenone hydrobromide in acetic acid at 65° (60%) [4401].

m.p. 215° [4401].

2,2-Dibromo-1-[3-bromo-5-(chloromethyl)-4-hydroxyphenyl]ethanone

[107700-05-0] $C_9H_6Br_3ClO_2$ mol.wt. 421.31



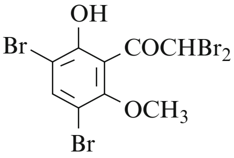
Synthesis

– Preparation by adding a methylene chloride solution of bromine to a methanol/methylene chloride solution of 3-(chloromethyl)-4-hydroxyacetophenone at r.t. (82%) [4458].

m.p. 140° [4458]; ^{1}H NMR [4458], IR [4458], MS [4458].

2,2-Dibromo-1-(3,5-dibromo-2-hydroxy-6-methoxyphenyl)ethanone

$C_9H_6Br_4O_3$ mol.wt. 481.76



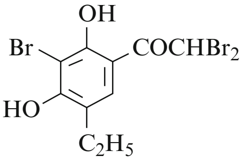
Synthesis

– Obtained by reaction of bromine on 3,3′-diacetyl-2,2′-dihydroxy-4,4′-dimethoxydiphenylsulfide in acetic acid, heated in a boiling water bath [4457].

m.p. 101–102° [4457].

2,2-Dibromo-1-(3-bromo-5-ethyl-2,4-dihydroxyphenyl)ethanone

$C_{10}H_9Br_3O_3$ mol.wt. 416.89



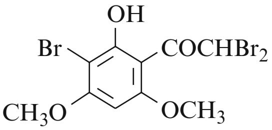
Synthesis

– Preparation by reaction of 5-ethyl-2,4-dihydroxy-acetophenone with an excess of bromine in chloroform [4535].

m.p. 144–145° [4535].

2,2-Dibromo-1-(3-bromo-2-hydroxy-4,6-dimethoxyphenyl)ethanone

[88503-19-9] $C_{10}H_9Br_3O_4$ mol.wt. 432.89



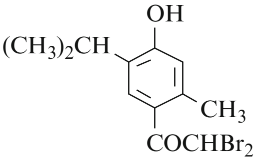
Synthesis

– Preparation by heating for 7 h on a steam bath a mixture of 2-hydroxy-3-iodo-4,6-dimethoxyacetophenone and bromine in acetic acid (67%) [4536].

m.p. 167–168° [4536].

2,2-Dibromo-1-[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]ethanone

[72235-94-0] $C_{12}H_{14}Br_2O_2$ mol.wt. 350.05

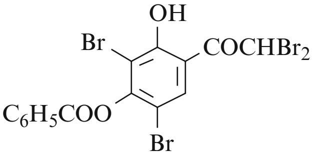


Synthesis

– Preparation by reaction of bromine on 4-hydroxy-2-methyl-5-isopropylacetophenone in acetic acid at 18° (45%) [4408].

m.p. 111° [4408].

1-[4-(Benzoyloxy)-3,5-dibromo-2-hydroxyphenyl]-2,2-dibromoethanone



$C_{15}H_8Br_4O_4$ mol.wt. 571.84

Synthesis

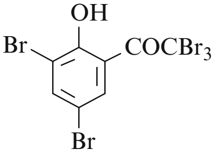
- Preparation by bromination of 4-(benzoyloxy)-2-hydroxy-acetophenone at r.t. [4537].

m.p. 69–70° [4537].

11.1.3 *From Tribromoacetic Acid*

2,2,2-Tribromo-1-(3,5-dibromo-2-hydroxyphenyl)ethanone

[98436-51-2]



$C_8H_3Br_5O_2$ mol.wt. 530.63

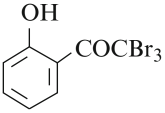
Synthesis

- Obtained by reaction of bromine on 4-hydroxycoumarin (benzotetronic acid) in methanol or acetic acid at r.t. [4529].

m.p. 125–126° [4529].

2,2,2-Tribromo-1-(2-hydroxyphenyl)ethanone

[101495-49-2]



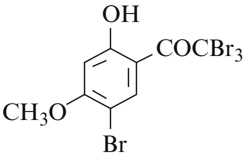
$C_8H_5Br_3O_2$ mol.wt. 372.84

Synthesis

- Obtained by reaction of bromine on 4-hydroxycoumarin (benzotetronic acid) in acetic acid–dioxane mixture at r.t. [4529].

m.p. 87° [4529].

2,2,2-Tribromo-1-(5-bromo-2-hydroxy-4-methoxyphenyl)ethanone



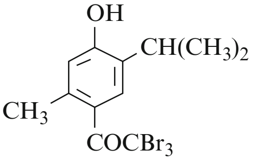
$C_9H_6Br_4O_3$ mol.wt. 481.76

Synthesis

- Preparation by reaction of bromine on bromopaeonol (5-bromo-2-hydroxy-4-methoxyacetophenone) in the presence of a crystal of iodine at r.t. [4538].

m.p. 123–124° [4538].

2,2,2-Tribromo-1-[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]ethanone



$C_{12}H_{13}Br_3O_2$ mol.wt. 428.95

Synthesis

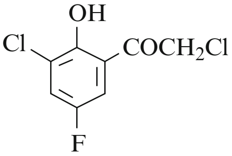
- Preparation by reaction of bromine on 4-hydroxy-2-methyl-5-isopropylacetophenone in acetic acid at 18° (90%) [4408].

m.p. 69° [4408].

11.2 Compounds Derived from Chloroacetic Acids

11.2.1 From Monochloroacetic Acid

2-Chloro-1-(3-chloro-5-fluoro-2-hydroxyphenyl)ethanone



$C_8H_5Cl_2FO_2$ mol.wt. 223.03

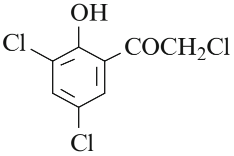
Synthesis

- Preparation by Fries rearrangement of 2-chloro-4-fluoro-phenyl chloroacetate with aluminium chloride without solvent at 130–140° (63%) [4539].

b.p.$_2$ 116° [4539].

2-Chloro-1-(3,5-dichloro-2-hydroxyphenyl)ethanone

[79214-30-5]



$C_8H_5Cl_3O_2$ mol.wt. 239.48

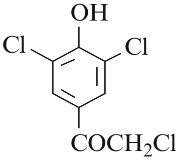
Syntheses

- Preparation by Fries rearrangement of 2,4-dichlorophenyl chloroacetate with aluminium chloride without solvent at 135–145° [4412,4540], (56%) [4540].
- Preparation by reaction of chloroacetyl chloride on 2,4-dichloroanisole with aluminium chloride in refluxing carbon disulfide (45%) [4405].

m.p. 136–136°8 [4540], 133° [4405], 132–134° [4412].

2-Chloro-1-(3,5-dichloro-4-hydroxyphenyl)ethanone

[220291-97-4]



$C_8H_5Cl_3O_2$ mol.wt. 239.48

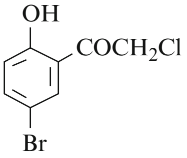
Syntheses

- Preparation by Fries rearrangement of 2,6-dichlorophenyl chloroacetate with aluminium chloride without solvent at 112–114° (77%) [4541].
- Also refer to: [4542] (Japanese patent).

m.p. 120–121° [4541].

1-(5-Bromo-2-hydroxyphenyl)-2-chloroethanone

[100959-21-5] $C_8H_6BrClO_2$ mol.wt. 249.49



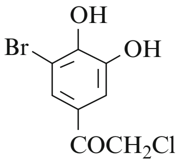
Synthesis

– Preparation by reaction of chloroacetyl chloride with 4-bromophenol in the presence of aluminium chloride at 40° (66%) [4543].

m.p. 73–74° [4543].

1-(3-Bromo-4,5-dihydroxyphenyl)-2-chloroethanone

$C_8H_6BrClO_3$ mol.wt. 265.50



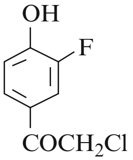
Synthesis

– Preparation by bromination of 3,4-dihydroxy-α-chloroacetophenone with bromine in the presence of quinoline sulfate while cooling (60%) [4544] according to the method [4545].

m.p. 137° [4544].

2-Chloro-1-(3-fluoro-4-hydroxyphenyl)ethanone

$C_8H_6ClFO_2$ mol.wt. 188.59



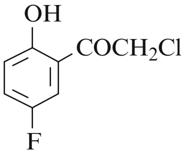
Synthesis

– Preparation by Fries rearrangement of 2-fluorophenyl chloroacetate with aluminium chloride without solvent at 135–140° (27–40%) [4546].

m.p. 101–102° [4546].

2-Chloro-1-(5-fluoro-2-hydroxyphenyl)ethanone

[2002-75-7] $C_8H_6ClFO_2$ mol.wt. 188.59



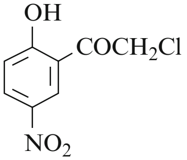
Synthesis

– Preparation by Fries rearrangement of 4-fluorophenyl chloroacetate with aluminium chloride without solvent at 130° (50%) [4547].

b.p.$_{10}$ 177° [4547].

2-Chloro-1-(2-hydroxy-5-nitrophenyl)ethanone

$C_8H_6ClNO_4$ mol.wt. 215.59

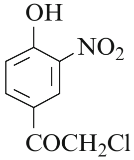


Synthesis

– Obtained by nitration of 2-hydroxy-α-chloroacetophenone in acetic acid [4548].

m.p. 163–164° [4548].

2-Chloro-1-(4-hydroxy-3-nitrophenyl)ethanone



$C_8H_6ClNO_4$ mol.wt. 215.59

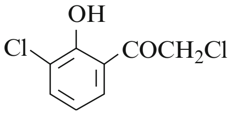
Syntheses

- Preparation by reaction of chlorine on 4-hydroxy-3-nitroacetophenone in acetic acid (45%) [4401].
- Also obtained by reaction of chloroacetyl chloride on 2-nitrophenol with aluminium chloride in nitrobenzene at 50–60° (19%) [4401].

m.p. 88° [4401]; b.p.$_{0.1}$ 135–140° [4401], b.p.$_{0.4}$ 140–145° [4401].

2-Chloro-1-(3-chloro-2-hydroxyphenyl)ethanone

[75717-49-6] $C_8H_6Cl_2O_2$ mol.wt. 205.04



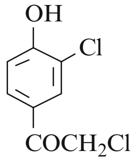
Synthesis

- Obtained by reaction of chloroacetonitrile on 2-chloro-phenol with aluminium chloride and boron trichloride mixture in ethylene dichloride at r.t. (21%) [4549,4550].

m.p. 72–73° [4549,4550]; ^{1}H NMR [4550].

2-Chloro-1-(3-chloro-4-hydroxyphenyl)ethanone

[39066-18-7] $C_8H_6Cl_2O_2$ mol.wt. 205.04



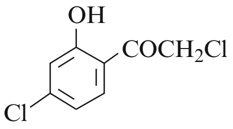
Synthesis

- Preparation by Fries rearrangement of 2-chlorophenyl chloroacetate with aluminium chloride without solvent at 135–140° (39–59%) [4546,4551,4552].

m.p. 141–142° [4546].

2-Chloro-1-(4-chloro-2-hydroxyphenyl)ethanone

[75717-50-9] $C_8H_6Cl_2O_2$ mol.wt. 205.04



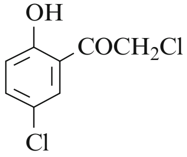
Synthesis

- Preparation by reaction of chloroacetonitrile on 3-chloro-phenol with aluminium chloride and boron trichloride mixture in refluxing ethylene dichloride (51%) [4549,4550].

m.p. 75–76° [4549,4550]; ^{1}H NMR [4550].

2-Chloro-1-(5-chloro-2-hydroxyphenyl)ethanone

[24483-75-8] $C_8H_6Cl_2O_2$ mol.wt. 205.04



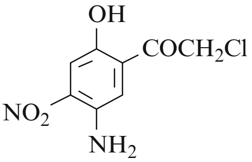
Syntheses

- Preparation by Fries rearrangement of 4-chlorophenyl chloroacetate with aluminium chloride without solvent at 140–150° [4412,4553,4554], (30%) [4554].
- Also obtained by reaction of chloroacetonitrile on 4-chlorophenol with aluminium chloride and boron trichloride mixture in ethylene dichloride [4549,4550], (18%) [4549].

m.p. 107–110° [4412], 65–66° [4549,4550], 65° [4553,4554]. One of the reported melting points is obviously wrong. ^1H NMR [4550].

1-(5-Amino-2-hydroxy-4-nitrophenyl)-2-chloroethanone

$C_8H_7ClN_2O_4$ mol.wt. 230.61



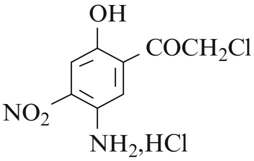
Synthesis

- Preparation by treatment of 5-acetamido-2-hydroxy-4-nitro-α-chloroacetophenone with boiling 25% aqueous hydrochloric acid solution [4555].

m.p. 145° (d) [4555].

1-(5-Amino-2-hydroxy-4-nitrophenyl)-2-chloroethanone *(Hydrochloride)*

$C_8H_7ClN_2O_4$, HCl mol.wt. 267.07



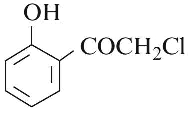
Synthesis

- Preparation from 5-amino-2-hydroxy-4-nitro-α-chloro-acetophenone [4555] (see above).

m.p. 210° (d) [4555].

2-Chloro-1-(2-hydroxyphenyl)ethanone

[53074-73-0] $C_8H_7ClO_2$ mol.wt. 170.60



Syntheses

- Preparation by reaction of acetonitrile on phenol with aluminium chloride and boron trichloride in refluxing ethylene dichloride (78%) [4549] or in methylene chloride at r.t. (85%) [4550].
- Preparation by halogenation of o-hydroxyacetophenone,
  - using EGDMA crosslinked polystyrene based benzyltriethylammonium dichloroiodate or tetrachloroiodate reagents in chloroform for 7–11 h at 30° (80%) [4556];

  - using 5% N,N′-MBA crosslinked polyacrylamide-based dichloroiodate or tetrachloroiodate reagents in chloroform at 30° for 8 h (77–79%) [4557];
  - with benzyltrimethylammonium dichloroiodate in refluxing methylene chloride/methanol mixture for 10 h (73%) [4558].

- Preparation by reaction of hexachloro-2,4-cyclohexadienone on 2-hydroxyacetophenone in refluxing ethanol (66%) [4559].
- Preparation by Fries rearrangement of phenyl monochloroacetate,
  - with aluminium chloride(50%) [4560], without solvent at 120° (50%) [4561] or at 140° (by-product) [4562];
  - with beryllium chloride without solvent at 130–140° (30%) [4563].
- Also obtained by reaction of chloroacetyl chloride on bromomagnesium phenolate in toluene at r.t. (17%) [4564].
- Also obtained by reaction of aluminium chloride on 2-chloroacetylanisole in refluxing carbon disulfide [4565,4566].
- Also obtained (by-product) by reaction of chloroacetyl chloride on phenol with aluminium chloride at 140° [4562].
- Also obtained (by-product) by treatment of anisole with chloroacetyl chloride in the presence of aluminium chloride in tetrachloroethane in a boiling water bath for 2–3 h [4567,4568].
- Also refer to: [4569–4577].

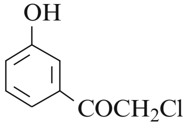
m.p. 101° [4566], 74–75° [4564], 74° [4557,4560–4562], 73–74° [4565], 73° [4558,4559,4563,4566–4568], 72–73° [4550], 72° [4556], 71–71°5 [4573];

One of the reported melting points is obviously wrong.

^{1}H NMR [4558,4559,4564], IR [4558,4559].

2-Chloro-1-(3-hydroxyphenyl)ethanone

[62932-90-5] $C_8H_7ClO_2$ mol.wt. 170.60



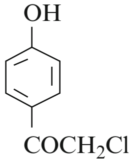
Synthesis

- Preparation by reaction of hexachloro-2,4-cyclohexadienone on 3-hydroxyacetophenone in refluxing ethanol (50%) [4559].

m.p. 93° [4559]; ^{1}H NMR [4559], IR [4559].

2-Chloro-1-(4-hydroxyphenyl)ethanone

[6305-04-0] $C_8H_7ClO_2$ mol.wt. 170.60



Syntheses

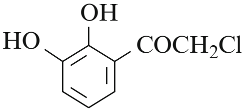
- Preparation by reaction of p-hydroxyacetophenone with benzyltrimethylammonium dichloroiodate in refluxing methylene chloride/methanol mixture for 10 h (95%) [4558] or for 12 h (92%) [4578].

- Preparation by reaction of chloroacetyl chloride,
  - with anisole in the presence of aluminium chloride in ligroin (44%) [4579], for 1 h in a water bath (50–55°) (32%) [4580], for 4 h (36%) [4581] or for 3 h (41–42%) [4582];
  - with anisole in the presence of aluminium chloride without solvent [4583], in carbon disulfide [4584] or in tetrachloroethane in a boiling water bath for 2–3 h [4567], (53%) [4568];
  - with phenol in the presence of aluminium chloride without solvent at 140° (71%) [4562] or in tetrachloroethane, first at 70° for 5 h, then at r.t. for 10 h [4585].
- Also obtained by Fries rearrangement of phenyl chloroacetate,
  - with aluminium chloride without solvent at 120–140° [4561,4562], (65%) [4562];
  - with beryllium chloride without solvent at 130–140° (23%) [4563].
- Preparation by reaction of hexachloro-2,4-cyclohexadienone on 4-hydroxyacetophenone in refluxing ethanol (77%) [4559].
- Also refer to: [4586–4588].

m.p. 151–152° [4573], 150–151° [4578], 150° [4558], 148° [4562,4579,4582–4584], 147–148° (d) [4580], 147°5 [4581], 147° [4567,4568], 145–146° [4563], 142° [4559];
^{1}H NMR [4558,4559,4578], IR [4558,4559].

2-Chloro-1-(2,3-dihydroxyphenyl)ethanone

[63704-55-2] $C_8H_7ClO_3$ mol.wt. 186.59

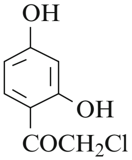


Synthesis

- Refer to: [4589] (Japanese patent).

2-Chloro-1-(2,4-dihydroxyphenyl)ethanone

[25015-92-3] $C_8H_7ClO_3$ mol.wt. 186.59



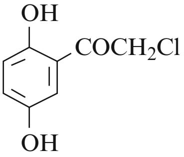
Syntheses

- Preparation by reaction of chloroacetonitrile on resorcinol with zinc chloride (Hoesch reaction) (90%) [4590], (82%) [4591] or with triflic acid (trifluoromethanesulfonic acid) (23%) [4592].
- Also obtained by reaction of chloroacetic acid on resorcinol with boron trifluoride (30%) [4593] or with zinc chloride or phosphorous oxychloride (poor yield) [4435,4436].

m.p. 132° [4593], 131° [4590], 130–132° [4592], 130° [4591];
^{1}H NMR [4592], IR [4592], UV [4417].

2-Chloro-1-(2,5-dihydroxyphenyl)ethanone

[60912-82-5] $C_8H_7ClO_3$ mol.wt. 186.59



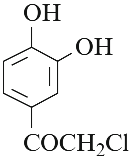
Syntheses

- Obtained by reaction of hydrochloric acid on 2,5-diacetoxy-α-chloroacetophenone in methanol at r.t. (85%) [4440].
- Preparation by reaction of aluminium bromide on 2,5-dimethoxy-α-chloroacetophenone in carbon disulfide at r.t. (72%) [4440].
- Also obtained by action of acetic acid saturated with hydrochloric acid (10 min, r.t.) on 2,5-di-acetoxy-α-diazoacetophenone, reduced pressure elimination of acetic acid, then action (overnight, r.t.) of a methanolic solution of hydrochloric acid (53%) [4440].
- Also obtained by reaction of sulfur dioxide on 2-chloroacetyl-1,4-benzoquinone in water [4440].

m.p. 132–133° [4440].

2-Chloro-1-(3,4-dihydroxyphenyl)ethanone

[99-40-1] $C_8H_7ClO_3$ mol.wt. 186.59



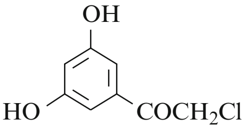
Syntheses

- Preparation by Fries rearrangement of pyrocatechol mono-chloroacetate with aluminium chloride in nitrobenzene at 100° (60%) [4594] or without solvent at 100° (25%) [4595].
- Preparation by reaction of chloroacetic acid on pyrocatechol with boron trifluoride in tetrachloroethane or in carbon tetrachloride at 65–85° (95–98%) [4593] or with phosphorous oxychloride [4445,4596–4599], (80%) [4445], (35–58%) [4596,4597,4599].
- Preparation by reaction of chloroacetyl chloride on pyrocatechol [4445].
- Preparation by reaction of chloroacetyl chloride on veratrole with aluminium chloride in nitrobenzene at 40° (82%) [4600].

m.p. 173° [4445,4593,4594,4596,4597,4600], 172° [4599,4601], 171° [4595], 169–170° [4437]; b.p.$_{12}$ 190° [4600].

2-Chloro-1-(3,5-dihydroxyphenyl)ethanone

[39878-43-8] $C_8H_7ClO_3$ mol.wt. 186.59



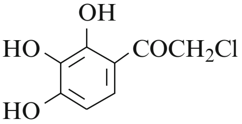
Synthesis

- Preparation by reaction of hydrochloric acid on 3,5-di-acetoxy-α-diazoacetophenone in aqueous methanol at reflux (97%) [4602].

m.p. 117° [4602].

2-Chloro-1-(2,3,4-trihydroxyphenyl)ethanone

[17345-68-5] $C_8H_7ClO_4$ mol.wt. 202.59



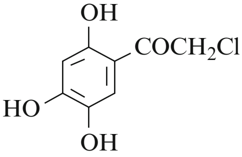
Syntheses

- Preparation by Fries rearrangement of 2,6-dimethoxyphenyl chloroacetate with aluminium chloride without solvent at 100° [4603].
- Preparation by reaction of chloroacetyl chloride on pyrogallol [4445].
- Preparation by reaction of chloroacetic acid on pyrogallol with phosphorous oxychloride [4435,4436,4455,4590,4598,4599,4604,4605], (55%) [4605], (40–41%) [4599,4604], with boron trifluoride [4593,4606] or with zinc chloride (poor yield) [4435,4436].
- Preparation by reaction of chloroacetic anhydride with pyrogallol in the presence of boron trifluoride in ethyl ether [4607].

m.p. 169° [4604], 167–168° [4455], 167° [4593], 166° [4599], 163–165° [4437].

2-Chloro-1-(2,4,5-trihydroxyphenyl)ethanone

[14771-02-9] $C_8H_7ClO_4$ mol.wt. 202.59



Synthesis

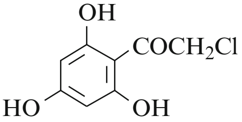
- Preparation by reaction of chloroacetonitrile on 1,2,4-benzenetriol (hydroxyhydroquinone) with zinc chloride in ethyl ether (Hoesch reaction) [4431,4608,4609].

m.p. 182° [4431], 139–140° [4609]. One of the reported melting points is obviously wrong.

^1H NMR [4431], IR [4431,4609], UV [4431,4609].

2-Chloro-1-(2,4,6-trihydroxyphenyl)ethanone

[110865-03-7] $C_8H_7ClO_4$ mol.wt. 202.59



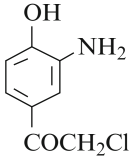
Syntheses

- Preparation by reaction of chloroacetyl chloride on phloroglucinol with aluminium chloride in nitromethane (86%) [4606].
- Preparation by reaction of chloroacetonitrile on phloro-glucinol (Hoesch reaction) (88%) [4610], (68%) [4611].
- Also refer to: [4612] (compound **1**).

m.p. 190° [4606], 188–191° [4610,4611]; ^1H NMR [4611], IR [4611], MS [4611].

1-(3-Amino-4-hydroxyphenyl)-2-chloroethanone

[108708-12-9] $C_8H_8ClNO_2$ mol.wt. 185.61



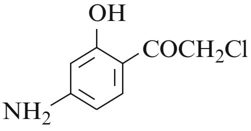
Synthesis

- Preparation by Friedel–Crafts chloroacetylation of N-acetyl-o-anisidine followed by hydrolysis with concentrated hydrochloric acid in ethanol [4613].

m.p. 113° [4613].

1-(4-Amino-2-hydroxyphenyl)-2-chloroethanone

[108708-13-0] $C_8H_8ClNO_2$ mol.wt. 185.61



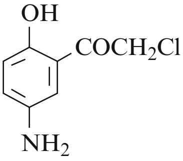
Synthesis

- Preparation by Friedel–Crafts chloroacetylation of N-acetyl-m-anisidine followed by hydrolysis with concentrated hydrochloric acid in ethanol [4613].

m.p. 214° (d) [4613].

1-(5-Amino-2-hydroxyphenyl)-2-chloroethanone

[108708-11-8] $C_8H_8ClNO_2$ mol.wt. 185.61



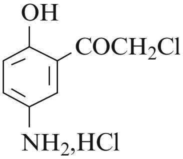
Synthesis

- Preparation by Friedel–Crafts chloroacetylation of N-acetyl-p-anisidine followed by hydrolysis with concentrated hydrochloric acid in ethanol [4555,4613,4614].

m.p. 135° [4555,4614], 128° [4613].

1-(5-Amino-2-hydroxyphenyl)-2-chloroethanone *(Hydrochloride)*

$C_8H_8ClNO_2$, HCl mol.wt. 222.07



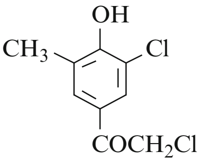
Synthesis

- Obtained by reaction of gaseous hydrochloric acid on 5-amino-2-hydroxy-α-chloroacetophenone in ethyl ether [4555].

m.p. 210° (d) [4555].

2-Chloro-1-(3-chloro-4-hydroxy-5-methylphenyl)ethanone

$C_9H_8Cl_2O_2$ mol.wt. 219.07



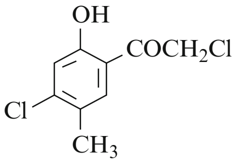
Synthesis

- Preparation by Fries rearrangement of 2-chloro-6-methylphenyl chloroacetate with aluminium chloride at 140° [4615].

m.p. 98°5–99°5 [4615]; b.p.$_{12}$ 172–180° [4615].

2-Chloro-1-(4-chloro-2-hydroxy-5-methylphenyl)ethanone

[22307-95-5] $C_9H_8Cl_2O_2$ mol.wt. 219.07

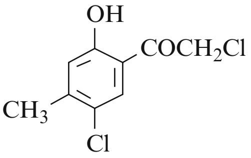


Synthesis not yet described

- See [4616]; this reference mentioned in the Chem. Abstr., **70**, 11442s (1969) is erroneous. Actually, the ketone described here as the 2,4′-dichloro-2′-hydroxy-5′-methylacetophenone should be the 2,5′-dichloro-2′-hydroxy-4′-methyl-acetophenone. In fact, on one hand, it is obtained by Fries rearrangement of 4-chloro-3-methylphenyl chloroacetate and on the other hand, it provides the 5-chloro-6-methyl-3-[2*H*]-benzofuranone by cyclisation (see below).

2-Chloro-1-(5-chloro-2-hydroxy-4-methylphenyl)ethanone

$C_9H_8Cl_2O_2$ mol.wt. 219.07



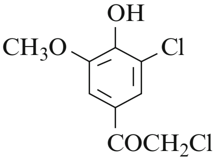
Synthesis

- Preparation by Fries rearrangement of 4-chloro-3-methyl-phenyl chloroacetate with aluminium chloride without solvent at 150° [4616].

m.p. 111° [4616]; ^{1}H NMR [4616], IR [4616].

2-Chloro-1-(3-chloro-4-hydroxy-5-methoxyphenyl)ethanone

[160925-81-5] $C_9H_8Cl_2O_3$ mol.wt. 235.07



Syntheses

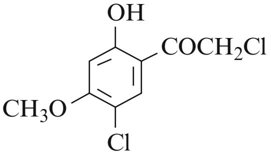
- Preparation by chlorination of acetoguaiacone (4-hydroxy-3-methoxyacetophenone) in dioxane with 2.5 mol equiv chlorine in acetic acid (67%) [4617].

- Also refer to: [4618].

m.p. 149–150° [4617]; ^{1}H NMR [4617], ^{13}C NMR [4617], MS [4617].

2-Chloro-1-(5-chloro-2-hydroxy-4-methoxyphenyl)ethanone

$C_9H_8Cl_2O_3$ mol.wt. 235.07



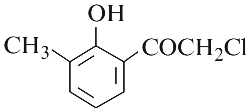
Syntheses

- Preparation by reaction of chloroacetyl chloride on 4-chlororesorcinol dimethyl ether with aluminium chloride in carbon disulfide (66%) [4486].
- Preparation by reaction of chlorine on 2-hydroxy-4-methoxy-α-chloroacetophenone in chloroform (40–45%) [4486].

m.p. 178°5–180° [4486].

2-Chloro-1-(2-hydroxy-3-methylphenyl)ethanone

[75717-51-0] $C_9H_9ClO_2$ mol.wt. 184.62



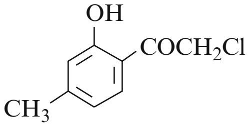
Syntheses

- Preparation by reaction of chloroacetonitrile on o-cresol with aluminium chloride and boron trichloride mixture in ethylene dichloride at r.t. (75%) [4549,4550] or with only boron trichloride in methylene chloride at r.t. under nitrogen (18%) [4619].
- Preparation by Fries rearrangement of 2-methylphenyl chloroacetate with aluminium chloride without solvent at 140° (50%) [4620], (20%) [4621].

m.p. 67° [4621], 66–67° [4620], 65–66° [4549,4550], 63–64° [4619];
^{1}H NMR [4550,4619], IR [4619], UV [4619], MS [4619].

2-Chloro-1-(2-hydroxy-4-methylphenyl)ethanone

[20834-75-7] $C_9H_9ClO_2$ mol.wt. 184.62



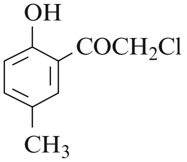
Syntheses

- Preparation by Fries rearrangement of 3-methylphenyl chloroacetate with aluminium chloride without solvent between 140° and 150° [4616,4620,4622–4624], (70%) [4620], (50%) [4623,4624], (12%) [4622].
- Preparation by reaction of chloroacetonitrile on m-cresol with boron trichloride and aluminium chloride in refluxing ethylene dichloride (quantitative yield) [4549].
- Also refer to: [4388,4575].

m.p. 102–102°5 [4620], 102° [4616], 101° [4623], 100° [4622], 95–96° [4549];
^{1}H NMR [4616], IR [4616].

2-Chloro-1-(2-hydroxy-5-methylphenyl)ethanone

[22307-94-4] $C_9H_9ClO_2$ mol.wt. 184.62



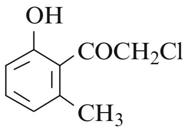
Syntheses

- Preparation by Fries rearrangement of p-tolyl chloroacetate with aluminium chloride without solvent at 140° [4412,4562,4620,4623], (90–93%) [4620,4623], (37%) [4562].
- Preparation by reaction of chloroacetic acid on p-cresol with boron trifluoride etherate (66%) or boron trifluoride (46%) in an autoclave at 70° [4470].
- Preparation by reaction of chloroacetyl chloride on 4-methylanisole with aluminium chloride in refluxing carbon disulfide (50–60%) [4625].
- Preparation by reaction of chloroacetyl chloride on p-cresol with aluminium chloride without solvent at 140° (31%) [4562].

m.p. 65° [4562,4623], 63° [4625], 62–63° [4412,4620], 62° [4470].

2-Chloro-1-(2-hydroxy-6-methylphenyl)ethanone

[73331-41-6] $C_9H_9ClO_2$ mol.wt. 184.62

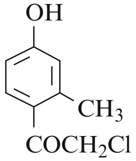


Synthesis

– Refer to: [4626].

2-Chloro-1-(4-hydroxy-2-methylphenyl)ethanone

[37904-71-5] $C_9H_9ClO_2$ mol.wt. 184.62



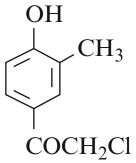
Synthesis

– Preparation by Fries rearrangement of 3-methylphenyl chloroacetate with aluminium chloride without solvent at 140° [4620,4622], (30%) [4620].

m.p. 148°5–149° [4620], 147°5 [4622].

2-Chloro-1-(4-hydroxy-3-methylphenyl)ethanone

[40943-24-6] $C_9H_9ClO_2$ mol.wt. 184.62



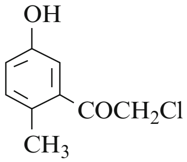
Synthesis

– Preparation by Fries rearrangement of 2-methylphenyl chloroacetate with aluminium chloride without solvent at 140° [4620,4621], (50%) [4620].

m.p. 144–145° [4621], 144–144°5 [4620].

2-Chloro-1-(5-hydroxy-2-methylphenyl)ethanone

$C_9H_9ClO_2$ mol.wt. 184.62



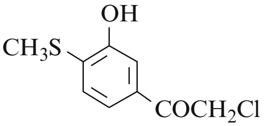
Synthesis

– Obtained (by-product) by Fries rearrangement of 4-methylphenyl chloroacetate with aluminium chloride without solvent at 140° (7%) [4620].

m.p. 155°5–156° [4620].

2-Chloro-1-[3-hydroxy-4-(methylthio)phenyl]ethanone

[151792-80-2] $C_9H_9ClO_2S$ mol.wt. 216.69



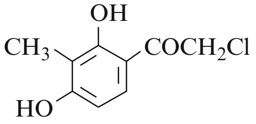
Synthesis

– Preparation by hydrolysis of 3-hydroxy-4-methylthio-α-chloroacetophenone chloroacetate with 1 N sodium hydroxide in methanol at r.t. (94%) [4627].

m.p. 140° [4627]; ^{1}H NMR [4627], IR [4627], MS [4627].

2-Chloro-1-(2,4-dihydroxy-3-methylphenyl)ethanone

[21861-21-2] $C_9H_9ClO_3$ mol.wt. 200.62



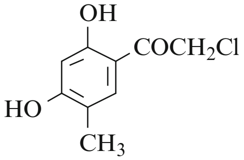
Synthesis

- Preparation by reaction of chloroacetonitrile with 2-methyl-resorcinol (72%) (Hoesch reaction) [4628].

m.p. 155° [4628].

2-Chloro-1-(2,4-dihydroxy-5-methylphenyl)ethanone

$C_9H_9ClO_3$ mol.wt. 200.62



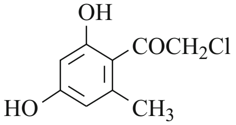
Syntheses

- Preparation by reaction of chloroacetonitrile with 4-methyl-resorcinol (Hoesch reaction) [4629].
- Also obtained by Friedel–Crafts acylation of 4-methylresorcinol with chloroacetyl chloride in the presence of aluminium chloride in nitrobenzene [4630].

m.p. 157° [4630], 156° [4629].

2-Chloro-1-(2,4-dihydroxy-6-methylphenyl)ethanone

[22670-61-7] $C_9H_9ClO_3$ mol.wt. 200.62



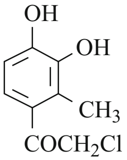
Synthesis

- Preparation by reaction of chloroacetonitrile with orcinol (68%) (Hoesch reaction) [4628].

m.p. 151° [4628].

2-Chloro-1-(3,4-dihydroxy-2-methylphenyl)ethanone

$C_9H_9ClO_3$ mol.wt. 200.62



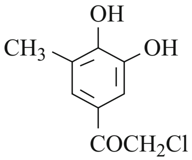
Synthesis

- Preparation by reaction of chloroacetyl chloride on 3-methylpyrocatechol with aluminium chloride in refluxing carbon disulfide (59%) [4631].

m.p. 178° [4631]; ^{1}H NMR [4631], IR [4631].

2-Chloro-1-(3,4-dihydroxy-5-methylphenyl)ethanone

$C_9H_9ClO_3$ mol.wt. 200.62

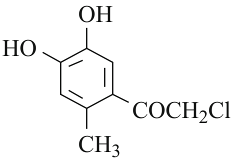


Synthesis

- Preparation by reaction of chloroacetic acid on 3-methylpyrocatechol with phosphorous oxychloride in refluxing benzene (28%) [4631].

m.p. 180° [4631]; ^{1}H NMR [4631], IR [4631].

2-Chloro-1-(4,5-dihydroxy-2-methylphenyl)ethanone



$C_9H_9ClO_3$ mol.wt. 200.62

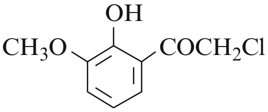
Syntheses

- Preparation by reaction of chloroacetyl chloride on homoveratrole (3,4-dimethoxytoluene) with aluminium chloride in carbon disulfide or nitrobenzene at 40° (82%) [4600].
- Also obtained (very low yield) by Fries rearrangement of creosol chloroacetate with aluminium chloride at 100° [4595].

m.p. 130° [4595], 128° [4600]; b.p.$_{14}$ 187° [4600].

2-Chloro-1-(2-hydroxy-3-methoxyphenyl)ethanone

[75717-52-1]



$C_9H_9ClO_3$ mol.wt. 200.62

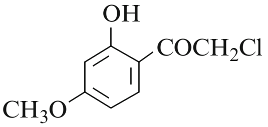
Synthesis

- Preparation by reaction of chloroacetonitrile on guaiacol with aluminium chloride and boron trichloride in refluxing ethylene dichloride (42%) [4549,4553].

m.p. 113–114° [4549,4550]; ^{1}H NMR [4550].

2-Chloro-1-(2-hydroxy-4-methoxyphenyl)ethanone

[60965-23-3]



$C_9H_9ClO_3$ mol.wt. 200.62

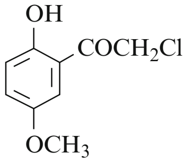
Syntheses

- Preparation by reaction of diazomethane on 2,4-dihydroxy-α-chloroacetophenone in ethyl ether (60%) [4590].
- Preparation by reaction of chloroacetyl chloride on 2,4-dimethoxybenzene with aluminium chloride in refluxing carbon disulfide (86%) [4486], (55%) [4632] or in ethyl ether at r.t. (57%) [4411].
- Preparation by reaction of chloroacetyl chloride on bromomagnesium 3-methoxyphenolate in toluene at r.t. (84%) [4564].
- Preparation by reaction of chloroacetonitrile on 3-methoxyphenol with aluminium chloride and boron trichloride in methylene chloride at r.t. (81%) [4549,4550] or with zinc chloride and hydrochloric acid in ethyl ether (by-product) [4433].
- Also refer to: [4575].

m.p. 117–118° [4549,4550], 116° [4411,4486], 115–117° [4564], 115–116° [4632], 114° [4590];
^{1}H NMR [4411,4564], MS [4564].

2-Chloro-1-(2-hydroxy-5-methoxyphenyl)ethanone

[75717-53-2] $C_9H_9ClO_3$ mol.wt. 200.62



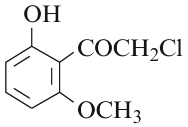
Syntheses

- Preparation by reaction of chloroacetyl chloride on hydroquinone dimethyl ether with aluminium chloride in refluxing carbon disulfide (65%) [4486].
- Preparation by reaction of chloroacetonitrile on hydroquinone monomethyl ether with boron trichloride and aluminium chloride in ethylene dichloride (67%) [4549,4553].
- Also refer to: [4575].

m.p. 83–84° [4549,4550], 81–81°5 [4486]; ^{1}H NMR [4550].

2-Chloro-1-(2-hydroxy-6-methoxyphenyl)ethanone

[75717-59-8] $C_9H_9ClO_3$ mol.wt. 200.62



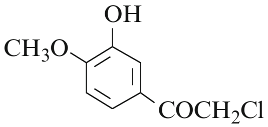
Syntheses

- Obtained (by-product) by reaction of chloroacetyl chloride on bromomagnesium 3-methoxyphenolate in toluene at r.t. (10%) [4564].
- Also obtained (by-product) by reaction of chloroacetonitrile on 3-methoxyphenol with aluminium chloride and boron trichloride mixture in ethylene dichloride at r.t. (5%) [4550].

m.p. 105–109° [4564]; ^{1}H NMR [4564], IR [4564].

2-Chloro-1-(3-hydroxy-4-methoxyphenyl)ethanone

[55761-46-1] $C_9H_9ClO_3$ mol.wt. 200.62



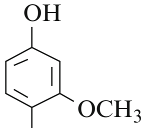
Syntheses

- Preparation by Fries rearrangement of guaiacol chloroacetate with aluminium chloride in refluxing carbon disulfide (63%) [4633], (49%) [4634,4635].
- Also obtained by reaction of 5 N sodium hydroxide with 5-chloroacetylguaiacol chloroacetate in dioxane, the mixture being gently warmed [4636].

m.p. 122–123° [4635], 121–122° [4636], 116–118° [4633].

2-Chloro-1-(4-hydroxy-2-methoxyphenyl)ethanone

[104691-67-0] $C_9H_9ClO_3$ mol.wt. 200.62



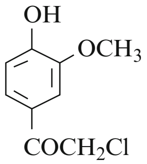
Synthesis

- Preparation by reaction of chloroacetonitrile on resorcinol monomethyl ether (Hoesch reaction) (major product, good yield) [4433].

m.p. 173–174° [4433].

2-Chloro-1-(4-hydroxy-3-methoxyphenyl)ethanone

[6344-28-1] $C_9H_9ClO_3$ mol.wt. 200.62



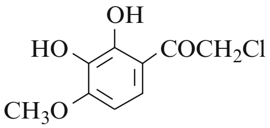
Syntheses

- Preparation by reaction of chloroacetyl chloride on veratrole with aluminium chloride in carbon disulfide (72%) [4637].
- Preparation by reaction of chloroacetyl chloride withguaiacol in the presence of aluminium chloride in boiling carbon disulfide (56%) [4638]; the same yield was obtained using tetrachloroethane as solvent [4638].
- Preparation by chlorination of the acetate of acetoguaiacone (4-acetoxy-3-methoxyacetophenone) with 1.1 mol equiv chlorine in chloroform and subsequent hydrolysis (67%) [4617].

m.p. 102–104° [4636], 102° [4637], 100–102° [4617,4638];
^{1}H NMR [4617], ^{13}C NMR [4617], MS [4381].

2-Chloro-1-(2,3-dihydroxy-4-methoxyphenyl)ethanone

$C_9H_9ClO_4$ mol.wt. 216.62

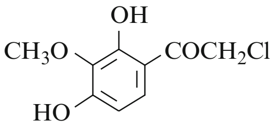


Synthesis

- Preparation from 2,3-dihydroxyanisole by Hoesch acylation by means of chloroacetonitrile (70%) [4607].

2-Chloro-1-(2,4-dihydroxy-3-methoxyphenyl)ethanone

[69151-93-5] $C_9H_9ClO_4$ mol.wt. 216.62



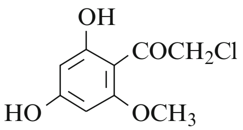
Synthesis

- Preparation by reaction of chloroacetonitrile with 2,6-di-hydroxyanisole (Hoesch reaction) [4639].

m.p. 71–72° [4639].

2-Chloro-1-(2,4-dihydroxy-6-methoxyphenyl)ethanone

[70651-70-6] $C_9H_9ClO_4$ mol.wt. 216.62



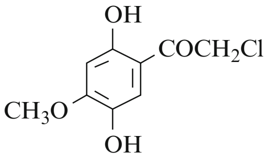
Syntheses

- Obtained by treatment of 2-hydroxy-4,6-dimethoxy-α-chloroacetophenone with aluminium chloride in refluxing chlorobenzene for 1 h (85%) [4640].
- Preparation by reaction of chloroacetonitrile with phloroglucinol monomethyl ether (Hoesch reaction) (41%) [4641].
- Also refer to: [4642,4643].

m.p. 184–186° (d) [4641], 174–175° [4640].
One of the reported melting points is obviously wrong.

2-Chloro-1-(2,5-dihydroxy-4-methoxyphenyl)ethanone

[163980-43-6] $C_9H_9ClO_4$ mol.wt. 216.62



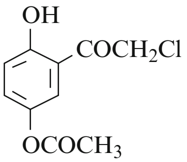
Synthesis

- Preparation by reaction of chloroacetonitrile with methoxy-hydroquinone (Hoesch reaction) (84%) [4644].

light brown crystals [4644]; ^{1}H NMR [4644], MS [4644].

1-[5-(Acetyloxy)-2-hydroxyphenyl]-2-chloroethanone

$C_{10}H_9ClO_4$ mol.wt. 228.63



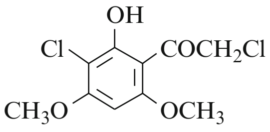
Syntheses

- 12 Preparation by reaction of acetyl chloride on 2,5-dihydroxy-α-chloroacetophenone at reflux (73%) [4440].
- Also obtained (by-product) by reaction of 2,5-diacetoxy-α-chloroacetophenone with sodium acetate (7%) [4440].

m.p. 151–152° [4440].

2-Chloro-1-(3-chloro-2-hydroxy-4,6-dimethoxyphenyl)ethanone

[72565-72-1] $C_{10}H_{10}Cl_2O_4$ mol.wt. 265.09



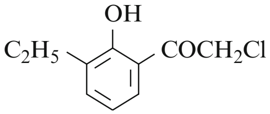
Synthesis

- Preparation by reaction of chloroacetyl chloride with 2-chloro-3,5-dimethoxyphenol in the presence of aluminium chloride in nitrobenzene at r.t. [4645,4646,4647], (88%) [4647], (70%) [4646].

m.p. 211° (d) [4646]; ^{1}H NMR [4647], IR [4646].

2-Chloro-1-(3-ethyl-2-hydroxyphenyl)ethanone

$C_{10}H_{11}ClO_2$ mol.wt. 198.65



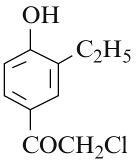
Synthesis

- Preparation by Fries rearrangement of 2-ethylphenyl chloroacetate with aluminium chloride without solvent at 140° (39%) [4648].

oil [4648].

2-Chloro-1-(3-ethyl-4-hydroxyphenyl)ethanone

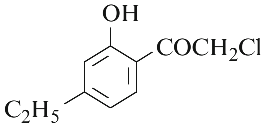
[145736-97-6] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



Synthesis

- Refer to: [4649].

2-Chloro-1-(4-ethyl-2-hydroxyphenyl)ethanone



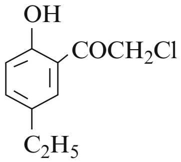
$C_{10}H_{11}ClO_2$ mol.wt. 198.65

Syntheses

- Preparation by Fries rearrangement of 3-ethylphenyl chloroacetate with aluminium chloride without solvent at 140° (49%) [4648].
- Also obtained also by Fries rearrangement of 4-ethylphenylchloroacetate with aluminium chloride without solvent at 140°, on account of a migration of the ethyl group (46%) [4648].

m.p. 69–70° [4648].

2-Chloro-1-(5-ethyl-2-hydroxyphenyl)ethanone



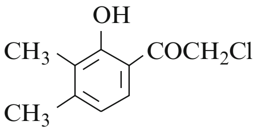
$C_{10}H_{11}ClO_2$ mol.wt. 198.65

Synthesis

- Preparation by reaction of chloroacetyl chloride on 4-ethylanisole with aluminium chloride in carbon disulfide (32%) [4648].

m.p. 50–52° [4648].

2-Chloro-1-(2-hydroxy-3,4-dimethylphenyl)ethanone



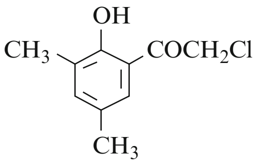
$C_{10}H_{11}ClO_2$ mol.wt. 198.65

Synthesis

- Preparation by reaction of chloroacetonitrile on 2,3-di-methylphenol with aluminium chloride and boron trifluoride in refluxing ethylene dichloride (quantitative yield) [4550].

m.p. 95–96° [4550].

2-Chloro-1-(2-hydroxy-3,5-dimethylphenyl)ethanone



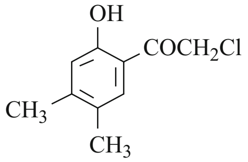
$C_{10}H_{11}ClO_2$ mol.wt. 198.65

Synthesis

- Preparation by reaction of chloroacetyl chloride on 2,4-dimethylanisole with aluminium chloride in carbon disulfide (20%) [4650].

2-Chloro-1-(2-hydroxy-4,5-dimethylphenyl)ethanone

[22307-96-6] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



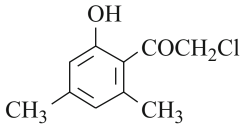
Synthesis

- Preparation by Fries rearrangement of 3,4-dimethylphenyl chloroacetate with aluminium chloride without solvent at 150° [4616].

m.p. 101° [4616]; ^{1}H NMR [4616], IR [4616].

2-Chloro-1-(2-hydroxy-4,6-dimethylphenyl)ethanone

$C_{10}H_{11}ClO_2$ mol.wt. 198.65



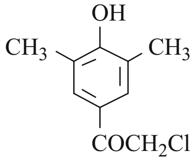
Synthesis

- Preparation by Fries rearrangement of 3,5-dimethylphenyl chloroacetate with aluminium chloride without solvent at 135–140° [4621,4623], (good yield) [4623].

m.p. 92° [4623].

2-Chloro-1-(4-hydroxy-3,5-dimethylphenyl)ethanone

[40943-25-7] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



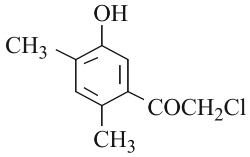
Syntheses

- Preparation by reaction of chloroacetyl chloride with 2,6-di-methylphenol in the presence of aluminium chloride in nitrobenzene at 60° (43%) [4498].
- Also obtained (poor yield) by reaction of aluminium chloride on 2,6-dimethyl-4-ethylphenyl chloroacetate without solvent at 150° [4615].

m.p. 107°7 [4498]; ^{1}H NMR [4498], IR [4498], MS [4498].

2-Chloro-1-(5-hydroxy-2,4-dimethylphenyl)ethanone

$C_{10}H_{11}ClO_2$ mol.wt. 198.65

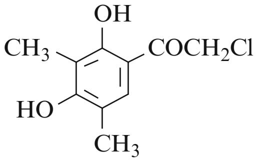


Synthesis

- Preparation (by-product) by reaction of chloroacetyl chloride on 2,4-dimethylanisole with aluminium chloride in carbon disulfide (10%) [4650].

m.p. 107–107°5 [4650].

2-Chloro-1-(2,4-dihydroxy-3,5-dimethylphenyl)ethanone



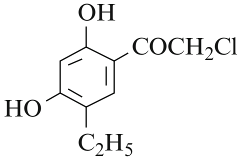
$C_{10}H_{11}ClO_3$ mol.wt. 214.65

Synthesis

- Preparation by reaction of acetonitrile on 2,4-dimethyl-resorcinol (Hoesch reaction) (93%) [4632].

m.p. 126–127° [4632].

2-Chloro-1-(5-ethyl-2,4-dihydroxyphenyl)ethanone



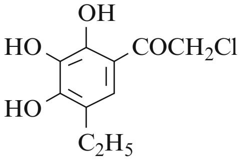
$C_{10}H_{11}ClO_3$ mol.wt. 214.65

Syntheses

- Preparation by reaction of 4-ethylresorcinol with chloro-acetonitrile (Hoesch reaction) [4629], (95%) [4651], (88%) [4652].
- Preparation by Friedel–Crafts acylation of 4-ethylresorcinol with chloroacetyl chloride in nitrobenzene in the presence of aluminium chloride for 24 h at r.t. (70%) [4630].

m.p. 163–165° [4630], 161–161°5 [4651], 161° [4629,4652].

2-Chloro-1-(5-ethyl-2,3,4-trihydroxyphenyl)ethanone



$C_{10}H_{11}ClO_4$ mol.wt. 230.65

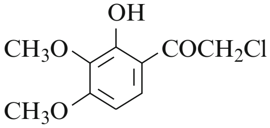
Synthesis

- Preparation by reaction of chloroacetonitrile with 4-ethyl-pyrogallol (m.p. 105–106°) (Hoesch reaction) (60%) [4652].

m.p. 131–132°5 [4652].

2-Chloro-1-(2-hydroxy-3,4-dimethoxyphenyl)ethanone

[7507-92-8]



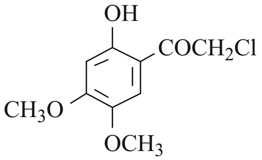
$C_{10}H_{11}ClO_4$ mol.wt. 230.65

Synthesis

- Preparation by reaction of chloroacetyl chloride with 1,2,3-trimethoxybenzene in the presence of aluminium chloride in ethylene dichloride first at 0°, then at r.t. (70%) [4653] or without solvent at 100° (16%) [4654].

m.p. 161°5 [4654], 160–162° [4653]; ^{1}H NMR [4653], MS [4653].

2-Chloro-1-(2-hydroxy-4,5-dimethoxyphenyl)ethanone



$C_{10}H_{11}ClO_4$ mol.wt. 230.65

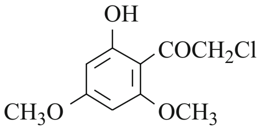
Synthesis

- Preparation by reaction of chloroacetonitrile on 3,4-di-methoxyphenol (Hoesch reaction) (47%) [4655].

m.p. 154–155° [4655].

2-Chloro-1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone

[103040-51-3]



$C_{10}H_{11}ClO_4$ mol.wt. 230.65

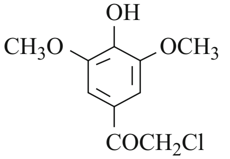
Syntheses

- Preparation by reaction of chloroacetonitrile on phloroglucinol dimethyl ether (Hoesch reaction) [4611].
- Preparation by reaction of chloroacetyl chloride on phloroglucinol trimethyl ether with aluminium chloride in boiling ligroin [4656].
- Preparation by partial demethylation of 2,4,6-trimethoxy-α-chloroacetophenone by heating with aluminium chloride without solvent at 120° (89%) [4508].

m.p. 144–146° [4508], 142–144° [4656], 136–140° [4611];
^{1}H NMR [4611], IR [4611], MS [4611].

2-Chloro-1-(4-hydroxy-3,5-dimethoxyphenyl)ethanone

[59719-58-3]

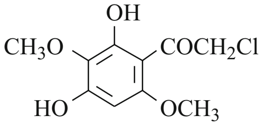


$C_{10}H_{11}ClO_4$ mol.wt. 230.65

Synthesis

- Preparation by chlorination of 4-(benzyloxy)-3,5-di-methoxybenzoylacetic acid methyl ester with sulfuryl chloride in acetic acid at r.t., and subsequent hydrolysis of the keto ester so obtained by refluxing in hydrochloric acid (90%) [4657].

2-Chloro-1-(2,4-dihydroxy-3,6-dimethoxyphenyl)ethanone



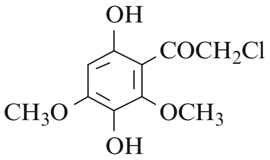
$C_{10}H_{11}ClO_5$ mol.wt. 246.65

Synthesis

- Preparation by reaction of chloroacetonitrile on 2,5-dimethoxyresorcinol (Hoesch reaction) (52%) [4515], (25%) [4658].

m.p. 150–151°5 [4658], 148–149° [4515].

2-Chloro-1-(3,6-dihydroxy-2,4-dimethoxyphenyl)ethanone



$C_{10}H_{11}ClO_5$ mol.wt. 246.65

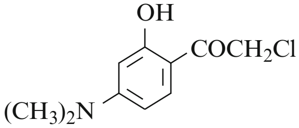
Synthesis

- Obtained by reaction of chloroacetyl chloride with 1,4-di-hydroxy-2,6-dimethoxybenzene in ethyl ether in the presence of aluminium chloride, cooling in ice and standing overnight (45%) [4659].

m.p. 154° [4659].

2-Chloro-1-[4-(dimethylamino)-2-hydroxyphenyl]ethanone

[127354-36-3] $C_{10}H_{12}ClNO_2$ mol.wt. 213.66



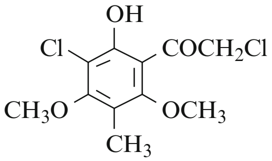
Synthesis

- Preparation by reaction of chloroacetyl chloride on bromomagnesium 3-(N,N-dimethylamino) phenolate in toluene at r.t. (92%) [4564].

m.p. 96–98° (d) [4564]; ^{1}H NMR [4564], IR [4564], MS [4564].

2-Chloro-1-(3-chloro-2-hydroxy-4,6-dimethoxy-5-methylphenyl)ethanone

[201288-73-5] $C_{11}H_{12}Cl_2O_4$ mol.wt. 279.12



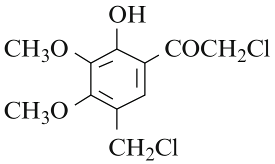
Synthesis

- Preparation by reaction of chloroacetyl chloride with 2-chloro-3,5-dimethoxy-4-methylphenol in ethyl ether in the presence of aluminium chloride, first at 0° under an argon atmosphere, then at r.t. for 0.5 h and at reflux for 3 h (90%) [4660].

m.p. 144–146° [4660]; ^{1}H NMR [4660], IR [4660], MS [4660].

2-Chloro-1-[5-(chloromethyl)-2-hydroxy-3,4-dimethoxyphenyl]ethanone

[76439-46-8] $C_{11}H_{12}Cl_2O_4$ mol.wt. 279.12

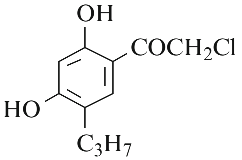


Synthesis

- Preparation by reaction of paraformaldehyde with 2-hydroxy-3,4-dimethoxy-α-chloroacetophenone in the presence of hydrochloric acid gas in acetic acid between 60° and 70° (77%) [4653].

m.p. 130–131° [4653]; ^{1}H NMR [4653], MS [4653].

2-Chloro-1-(2,4-dihydroxy-5-propylphenyl)ethanone



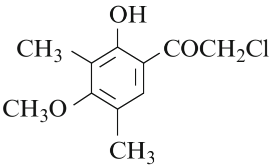
$C_{11}H_{13}ClO_3$ mol.wt. 22.8.68

Synthesis

- Preparation by reaction of chloroacetonitrile with 4-propyl-resorcinol (Hoesch reaction) [4629].

m.p. 156–157° [4629].

2-Chloro-1-(2-hydroxy-4-methoxy-3,5-dimethylphenyl)ethanone



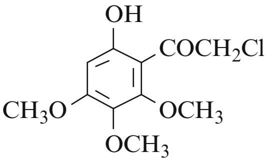
$C_{11}H_{13}ClO_3$ mol.wt. 228.68

Synthesis

- Preparation by reaction of chloroacetyl chloride on 2,4-dimethylresorcinol dimethyl ether with aluminium chloride in refluxing carbon disulfide (42%) [4632].

m.p. 158–160° [4632].

2-Chloro-1-(6-hydroxy-2,3,4-trimethoxyphenyl)ethanone



$C_{11}H_{13}ClO_5$ mol.wt. 260.67

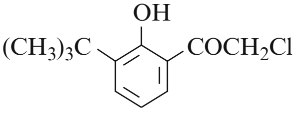
Synthesis

- Preparation by reaction of chloroacetonitrile on 3,4,5-trimethoxyphenol (antiarol) (Hoesch reaction) (42%) [4658].

m.p. 107–107°5 [4658].

2-Chloro-1-[3-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone

[127354-33-0]



$C_{12}H_{15}ClO_2$ mol.wt. 226.70

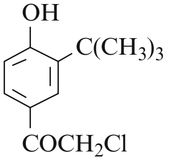
Synthesis

- Preparation by reaction of chloroacetyl chloride with aluminium or titanium 2-tert-butylphenolate in toluene at r.t. (98% and 70% yields, respectively) [4564].

m.p. 52–53° [4564]; ^{1}H NMR [4564], IR [4564], MS [4564].

2-Chloro-1-[3-(1,1-dimethylethyl)-4-hydroxyphenyl]ethanone

[127354-34-1] $C_{12}H_{15}ClO_2$ mol.wt. 226.70

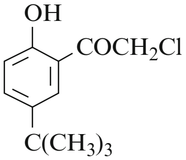


Synthesis

– Obtained by reaction of chloroacetyl chloride with dichloroaluminium 2-tert-butylphenolate or with trichloro (2-tert-butylphenoxy)titanium in toluene at r.t. (50% and 52% yields, respectively) [4564].

2-Chloro-1-[5-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone

[75060-43-4] $C_{12}H_{15}ClO_2$ mol.wt. 226.70



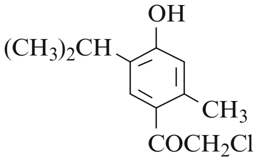
Synthesis

– Obtained by reaction of chloroacetyl chloride on 4-tert- butylanisole with aluminium chloride in methylene chloride at r.t. (10%) [4661].

m.p. 51–52° [4661]; ^{1}H NMR [4661], IR [4661].

2-Chloro-1-[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]ethanone

[72235-89-3] $C_{12}H_{15}ClO_2$ mol.wt. 226.70



Syntheses

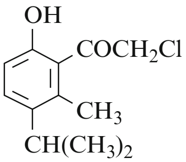
– Obtained by reaction of chloroacetyl chloride on thymol with aluminium chloride in nitrobenzene at 50° (21%) [4662].

– Also obtained by Fries rearrangement of thymyl chloroacetate with aluminium chloride in nitrobenzene at r.t. (15%) [4408].

m.p. 133° [4662], 132° [4408]; b.p.$_{0.0018}$ 175–178° [4662].

2-Chloro-1-[6-hydroxy-2-methyl-3-(1-methylethyl)phenyl]ethanone

[23053-74-9] $C_{12}H_{15}ClO_2$ mol.wt. 226.70

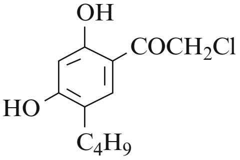


Synthesis

– Obtained by reaction of chloroacetyl chloride on 3-methyl-4-isopropylanisole (p-thymol methyl ether) with aluminium chloride in carbon disulfide [4663].

m.p. 68°5 [4663].

1-(5-Butyl-2,4-dihydroxyphenyl)-2-chloroethanone



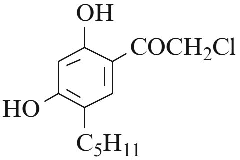
$C_{12}H_{15}ClO_3$ mol.wt. 242.70

Synthesis

- Preparation by reaction of chloroacetonitrile with 4-butyl-resorcinol (Hoesch reaction) [4629].

m.p. 155–156° [4629].

2-Chloro-1-(2,4-dihydroxy-5-pentylphenyl)ethanone



$C_{13}H_{17}ClO_3$ mol.wt. 256.73

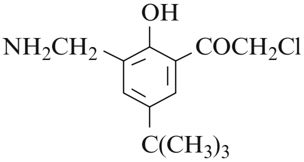
Synthesis

- Preparation by reaction of chloroacetonitrile with 4-pentyl-resorcinol (Hoesch reaction) [4629].

m.p. 148–149° [4629].

1-[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-2-chloroethanone

[75060-96-7]



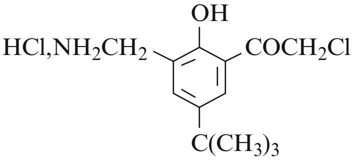
$C_{13}H_{18}ClNO_2$ mol.wt. 255.74

Synthesis

- From the corresponding hydrochloride (see below) [4661].

1-[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-2-chloroethanone *(Hydrochloride)*

[75060-68-3]



$C_{13}H_{18}ClNO_2$, HCl mol.wt. 292.20

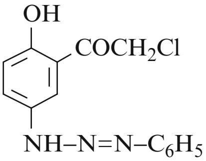
Synthesis

- Preparation by reaction of concentrated hydrochloric acid on 2-chloroacetyl-4-tert-butyl-6-(N-chloro-acetylaminomethyl)phenol in refluxing ethanol (64%) [4661].

m.p. 160° (d) [4661]; ^{1}H NMR [4661], IR [4661].

2-Chloro-1-[2-hydroxy-5-(1-triazene-3-phenyl)phenyl]ethanone

$C_{14}H_{12}ClN_3O_2$ mol.wt. 289.72



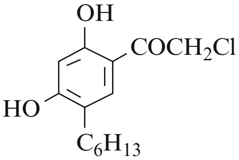
m.p. 127° [4555].

Synthesis

- Obtained by reaction of benzenediazonium chloride on 5-amino-2-hydroxy-α-chloroacetophenone in the presence of an aqueous sodium acetate solution [4555].

2-Chloro-1-(5-hexyl-2,4-dihydroxyphenyl)ethanone

$C_{14}H_{19}ClO_3$ mol.wt. 270.76



m.p. 145° [4629].

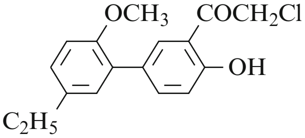
Synthesis

- Preparation by reaction of chloroacetonitrile with 4-hexyl-resorcinol (Hoesch reaction) [4629].

2-Chloro-1-(5′-ethyl-4-hydroxy-2′-methoxy[1,1′-biphenyl]-3-yl)ethanone

[131845-71-1]

$C_{17}H_{17}ClO_3$ mol.wt. 304.77



Synthesis

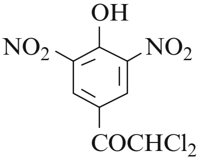
- Refer to: [4664].

11.2.2 From Dichloroacetic Acid

2,2-Dichloro-1-(4-hydroxy-3,5-dinitrophenyl)ethanone

[52129-63-2]

$C_8H_4Cl_2N_2O_6$ mol.wt. 295.04

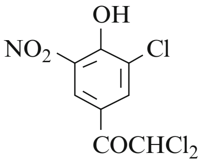


Synthesis

- Preparation by reaction of chlorine on 4-hydroxy-3,5-di-nitroacetophenone in methylene chloride-ethanol mixture (93%) [4665,4666].

2,2-Dichloro-1-(3-chloro-4-hydroxy-5-nitrophenyl)ethanone

[52501-35-6] $C_8H_4Cl_3NO_4$ mol.wt. 284.48

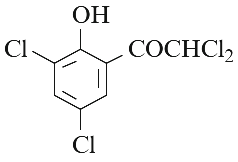


Synthesis

- Preparation by reaction of chlorine on 4-hydroxy-3-nitroacetophenone with ferric chloride in methylene chloride-ethanol mixture (94%) [4665,4666].

2,2-Dichloro-1-(3,5-dichloro-2-hydroxyphenyl)ethanone

$C_8H_4Cl_4O_2$ mol.wt. 273.93



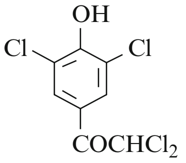
Syntheses

- Preparation by Fries rearrangement of 2,4-dichlorophenyl dichloroacetate with aluminium chloride without solvent at 120° (50%) [4667].
- Also obtained when 4-hydroxycoumarin in aceticacid was treated with an excess of gaseous chlorine at 10–15° and the polychlorinated product so formed hydrolyzed [4668,4669].

m.p. 90° [4667], 70° [4668,4669].

2,2-Dichloro-1-(3,5-dichloro-4-hydroxyphenyl)ethanone

$C_8H_4Cl_4O_2$ mol.wt. 273.93



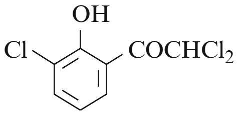
Synthesis

- Obtained (by-product) by Fries rearrangement of 2,6-di-chlorophenyl dichloroacetate with aluminium chloride without solvent at 135° (9%) [4541].

m.p. 92°5–94°5 [4541].

2,2-Dichloro-1-(3-chloro-2-hydroxyphenyl)ethanone

$C_8H_5Cl_3O_2$ mol.wt. 239.48



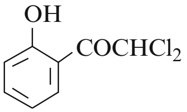
Synthesis

- Preparation by Fries rearrangement of 2-chlorophenyl dichloroacetate with aluminium chloride without solvent at 120° (76%) [4667].

b.p.$_8$ 173° [4667].

2,2-Dichloro-1-(2-hydroxyphenyl)ethanone

[29003-58-5] $C_8H_6Cl_2O_2$ mol.wt. 205.04



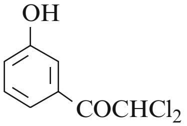
Synthesis

- Preparation by Fries rearrangement of phenyl dichloroacetate with aluminium chloride without solvent at 120° (50%) [4670].

b.p.$_{0.1}$ 65–72° [4670].

2,2-Dichloro-1-(3-hydroxyphenyl)ethanone

[85299-04-3] $C_8H_6Cl_2O_2$ mol.wt. 205.04



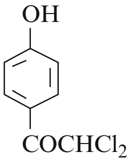
Synthesis

– Preparation by reaction of hexachloro-2,4-cyclohexadienone on 3-hydroxyacetophenone in refluxing ethanol (34%) [4559].

pale yellow oil [4559]; ^{1}H NMR [4559], IR [4559].

2,2-Dichloro-1-(4-hydroxyphenyl)ethanone

$C_8H_6Cl_2O_2$ mol.wt. 205.04



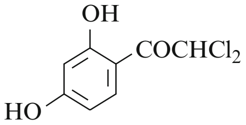
Synthesis

– Preparation by reaction of dichloroacetyl chloride on anisole with aluminium chloride in carbon disulfide at 25–30° (34%) [4671].

m.p. 98° [4671].

2,2-Dichloro-1-(2,4-dihydroxyphenyl)ethanone

[29003-59-6] $C_8H_6Cl_2O_3$ mol.wt. 221.04

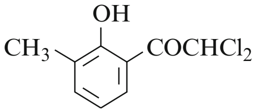


Synthesis

– Refer to: [4672].

2,2-Dichloro-1-(2-hydroxy-3-methylphenyl)ethanone

[145818-23-1] $C_9H_8Cl_2O_2$ mol.wt. 219.07



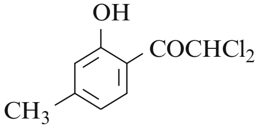
Synthesis

– Preparation by reaction of dichloroacetonitrile on o-cresol (Hoesch reaction) (25%) [4619].

m.p. 33°5 [4619]; ^{1}H NMR [4619], IR [4619], UV [4619], MS [4619].

2,2-Dichloro-1-(2-hydroxy-4-methylphenyl)ethanone

[116046-02-7] $C_9H_8Cl_2O_2$ mol.wt. 219.07



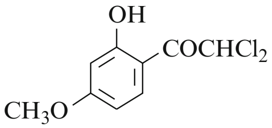
Synthesis

– Preparation by reaction of dichloroacetyl chloride with 3-methylanisole in the presence of aluminium chloride at 80° (30%) [4673].

m.p. 125–126° [4673]; ^{1}H NMR [4673], IR [4673], MS [4673].

2,2-Dichloro-1-(2-hydroxy-4-methoxyphenyl)ethanone

[95235-25-9] $C_9H_8Cl_2O_3$ mol.wt. 235.07

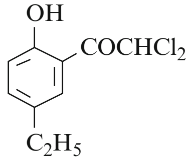


Syntheses

- Preparation by Fries rearrangement of 3-methoxyphenyl dichloroacetate with aluminium chloride without solvent at 120° (42%) [4667].
- Also obtained (by-product) by reaction of dichloroacetyl chloride with resorcinol dimethyl ether in the presence of aluminium chloride at 0–10° (9%) [4673].

m.p. 86° [4673], 84° [4667].

2,2-Dichloro-1-(5-ethyl-2-hydroxyphenyl)ethanone



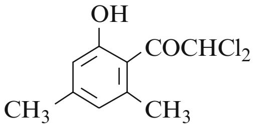
$C_{10}H_{10}Cl_2O_2$ mol.wt. 233.09

Synthesis

- Preparation by Fries rearrangement of 4-ethylphenyl dichloroacetate with aluminium chloride without solvent at 120° (53%) [4667].

b.p.$_8$ 110° [4667].

2,2-Dichloro-1-(2-hydroxy-4,6-dimethylphenyl)ethanone



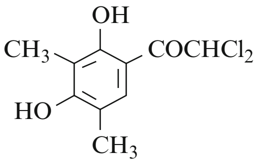
$C_{10}H_{10}Cl_2O_2$ mol.wt. 233.09

Synthesis

- Preparation by Fries rearrangement of 3,5-dimethylphenyl dichloroacetate with aluminium chloride without solvent at 120° (58%) [4667].

b.p.$_4$ 150° [4667].

2,2-Dichloro-1-(2,4-dihydroxy-3,5-dimethylphenyl)ethanone



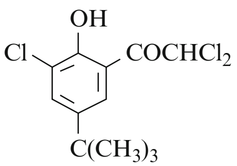
$C_{10}H_{10}Cl_2O_3$ mol.wt. 249.09

Synthesis

- Preparation by reaction of dichloroacetonitrile on 2,4-di-methylresorcinol (Hoesch reaction) [4632].

m.p. 123° [4632]; b.p.$_{0.001}$ 150° [4632].

2,2-Dichloro-1-[3-chloro-5-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone



$C_{12}H_{13}Cl_3O_2$ mol.wt. 295.59

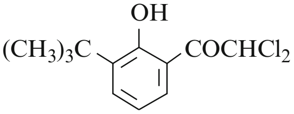
Synthesis

- Preparation by Fries rearrangement of 4-tert-butyl-2-chlorophenyl dichloroacetate with aluminium chloride at 120° (79%) [4667].

b.p.$_{20}$ 175° [4667].

2,2-Dichloro-1-[3-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone

[127354-38-5] $C_{12}H_{14}Cl_2O_2$ mol.wt. 261.15



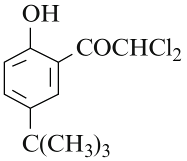
Synthesis

– Preparation by reaction of dichloroacetyl chloride on bromomagnesium 2-tert-butylphenolate in toluene at r.t. (58%) [4564]. yellow oil [4564];

^{1}H NMR [4564], IR [4564], MS [4564].

2,2-Dichloro-1-[5-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone

$C_{12}H_{14}Cl_2O_2$ mol.wt. 261.15



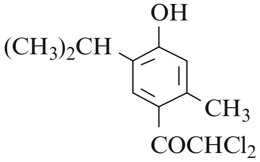
Synthesis

– Preparation by Fries rearrangement of 4-tert-butylphenyl dichloroacetate with aluminium chloride at 120° (86%) [4667].

b.p.$_{10}$ 120° [4667].

2,2-Dichloro-1-[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]ethanone

[72235-91-7] $C_{12}H_{14}Cl_2O_2$ mol.wt. 261.15



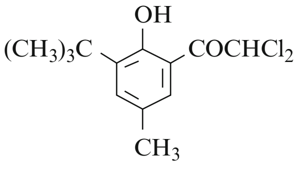
Synthesis

– Preparation by reaction of dichloroacetyl chloride on thymol with aluminium chloride in nitrobenzene at r.t., via a Fries rearrangement (20%) [4408].

m.p. 110° [4408].

2,2-Dichloro-1-[3-(1,1-dimethylethyl)-2-hydroxy-5-methylphenyl]ethanone

[127354-45-4] $C_{13}H_{16}Cl_2O_2$ mol.wt. 275.17



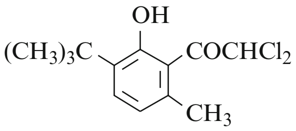
Syntheses

– Preparation by reaction of dichloroacetyl chloride,
  - on aluminium tri 2-tert-butyl-4-methylphenolate in toluene at r.t. (98%) [4564];
  - on bromomagnesium 2-tert-butyl-4-methylphenolate in toluene at r.t. (78%) [4564].

m.p. 55–59° [4564]; ^{1}H NMR [4564], IR [4564], MS [4564].

2,2-Dichloro-1-[3-(1,1-dimethylethyl)-2-hydroxy-6-methylphenyl]ethanone

$C_{13}H_{16}Cl_2O_2$ mol.wt. 275.17

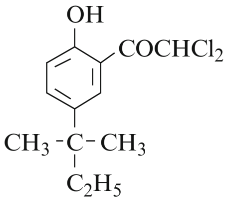


Synthesis

– Preparation by Fries rearrangement of 2-tert-butyl-5-methyl-phenyl dichloroacetate without solvent at 120° (60%) [4667].

b.p.$_6$ 88° [4667].

2,2-Dichloro-1-[5-(1,1-dimethylpropyl)-2-hydroxyphenyl]ethanone



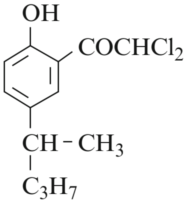
$C_{13}H_{16}Cl_2O_2$ mol.wt. 275.17

Synthesis

- Preparation by Fries rearrangement of 4-tert-pentylphenyl dichloroacetate with aluminium chloride without solvent at 120° (54%) [4667].

b.p.$_2$ 122° [4667].

2,2-Dichloro-1-[2-hydroxy-5-(1-methylbutyl)phenyl]ethanone



$C_{13}H_{16}Cl_2O_2$ mol.wt. 275.17

Synthesis

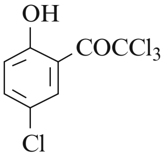
- Preparation by Fries rearrangement of 5-sec-pentylphenyl dichloroacetate with aluminium chloride without solvent at 120° (43%) [4667].

b.p.$_{10}$ 125° [4667].

11.2.3 From Trichloroacetic Acid

2,2,2-Trichloro-1-(5-chloro-2-hydroxyphenyl)ethanone

[145818-26-4]



$C_8H_4Cl_4O_2$ mol.wt. 273.93

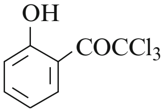
Syntheses

- Preparation by Fries rearrangement of 4-chlorophenyl trichloroacetate with aluminium chloride without solvent at 125–130° (25%) [4674,4675].
- Preparation by reaction of trichloroacetonitrile on 4-chlorophenol (Hoesch reaction) [4619].

b.p.$_{44}$ 129–132° [4619,4674,4675]; ^{1}H NMR [4619], IR [4619], UV [4619], MS [4619].

2,2,2-Trichloro-1-(2-hydroxyphenyl)ethanone

[75717-55-4]



$C_8H_5Cl_3O_2$ mol.wt. 239.48

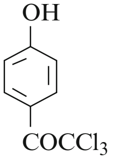
Syntheses

- Preparation by reaction of trichloroacetonitrile on phenol with aluminium chloride and boron trichloride mixture in refluxing methylene chloride (92%) [4549,4550].
- Preparation by reaction of trichloroacetonitrile on phenol (Hoesch reaction) (64%) [4619].

m.p. 53–54° [4619]; ^{1}H NMR [4550,4619], IR [4619], UV [4619], MS [4619].

2,2,2-Trichloro-1-(4-hydroxyphenyl)ethanone

[131170-16-6] $C_8H_5Cl_3O_2$ mol.wt. 239.48



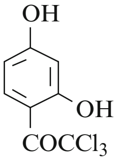
Synthesis

- Preparation by reaction of trichloroacetonitrile on phenol (Hoesch reaction) (95%) [4665,4666,4676,4677], (30%) [4619].

  m.p. 99–99°5 [4619,4676,4677]; b.p.$_{0.5}$ 170° [4676,4677];
  ^{1}H NMR [4619], IR [4619], UV [4619], MS [4619].

2,2,2-Trichloro-1-(2,4-dihydroxyphenyl)ethanone

[76569-42-1] $C_8H_5Cl_3O_3$ mol.wt. 255.48



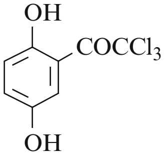
Synthesis

- Preparation by reaction of trichloroacetonitrile with resorcinol in the presence of triflic acid (52%) [4592] or zinc chloride (55%) [4678].

m.p. 142°5 [4678], 138–141° [4592];
^{1}H NMR [4592], IR [4592], UV [4592], MS [4592].

2,2,2-Trichloro-1-(2,5-dihydroxyphenyl)ethanone

[145818-27-5] $C_8H_5Cl_3O_3$ mol.wt. 255.48



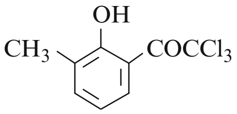
Synthesis

- Preparation by reaction of trichloroacetonitrile on hydroquinone (Hoesch reaction) (40%) [4619].

  m.p. 123–124° [4619];
  ^{1}H NMR [4619], IR [4619], UV [4619], MS [4619].

2,2,2-Trichloro-1-(2-hydroxy-3-methylphenyl)ethanone

[145818-22-0] $C_9H_7Cl_3O_2$ mol.wt. 253.51

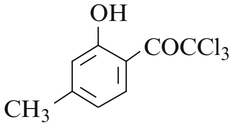


Synthesis

- Preparation by reaction of trichloroacetonitrile on o-cresol (Hoesch reaction) (70%) [4619].

oil [4619]; ^{1}H NMR [4619], IR [4619], UV [4619], MS [4619].

2,2,2-Trichloro-1-(2-hydroxy-4-methylphenyl)ethanone



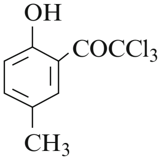
$C_9H_7Cl_3O_2$ mol.wt. 253.51

Synthesis

- Preparation by reaction of trichloroacetonitrile on m-cresol with aluminium chloride and gaseous hydrochloric acid in chlorobenzene at r.t. (37%) [4677].

b.p.$_{17}$ 162–163° [4677].

2,2,2-Trichloro-1-(2-hydroxy-5-methylphenyl)ethanone

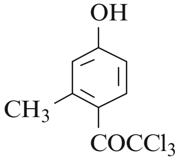


$C_9H_7Cl_3O_2$ mol.wt. 253.51

Synthesis

- Obtained by reaction of trichloroacetonitrile on p-cresol with aluminium chloride and gazeous hydrochloric acid in chlorobenzene at 50–60° (11%) [4677].

2,2,2-Trichloro-1-(4-hydroxy-2-methylphenyl)ethanone



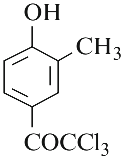
$C_9H_7Cl_3O_2$ mol.wt. 253.51

Synthesis

- Preparation by reaction of trichloroacetonitrile on m-cresol with aluminium chloride and gazeous hydrochloric acid in chlorobenzene at r.t. (36%) [4677].

m.p. 84–87° [4677].

2,2,2-Trichloro-1-(4-hydroxy-3-methylphenyl)ethanone



$C_9H_7Cl_3O_2$ mol.wt. 253.51

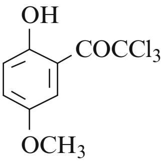
Synthesis

- Preparation by reaction of trichloroacetonitrile on o-cresol with aluminium chloride and gaseous hydrochloric acid in chlorobenzene at 60° (90%) [4677].

m.p. 90–91° [4677].

2,2,2-Trichloro-1-(2-hydroxy-5-methoxyphenyl)ethanone

[145818-25-3]



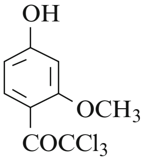
$C_9H_7Cl_3O_3$ mol.wt. 269.51

Synthesis

- Preparation by reaction of trichloroacetonitrile on 4-methoxyphenol (Hoesch reaction) (73%) [4619].

m.p. 65–66° [4619];
^{1}H NMR [4619], IR [4619], UV [4619], MS [4619].

2,2,2-Trichloro-1-(4-hydroxy-2-methoxyphenyl)ethanone



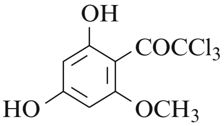
$C_9H_7Cl_3O_3$ mol.wt. 269.51

Synthesis

- Preparation by reaction of trichloroacetonitrile on 3-methoxyphenol with zinc chloride and gaseous hydrochloric acid in ethyl ether at 0° (Hoesch reaction) (34%) [4679].

m.p. 144° [4679].

2,2,2-Trichloro-1-(2,4-dihydroxy-6-methoxyphenyl)ethanone



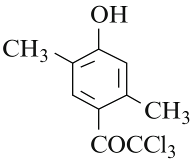
$C_9H_7Cl_3O_4$ mol.wt. 285.51

Synthesis

- Preparation by reaction of trichloroacetonitrile on phloroglucinol monomethyl ether with zinc chloride and gaseous hydrochloric acid in ethyl ether at 0° (Hoesch reaction) (39–49%) [4679].

m.p. 152° [4679].

2,2,2-Trichloro-1-(4-hydroxy-2,5-dimethylphenyl)ethanone



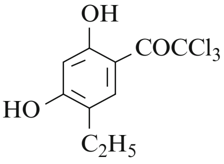
$C_{10}H_9Cl_3O_2$ mol.wt. 267.54

Synthesis

- Preparation by reaction of trichloroacetonitrile on 2,5-dimethylphenol with aluminium chloride and gaseous hydrochloric acid in chlorobenzene at r.t. (70%) [4676].

m.p. 85–86° [4676].

2,2,2-Trichloro-1-(5-ethyl-2,4-dihydroxyphenyl)ethanone



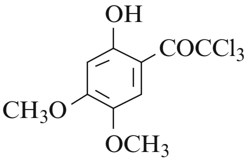
$C_{10}H_9Cl_3O_3$ mol.wt. 283.54

Synthesis

- Preparation by reaction of trichloroacetonitrile on 4-ethylresorcinol (Hoesch reaction) (52%) [4678].

m.p. 138° [4678].

2,2,2-Trichloro-1-(2-hydroxy-4,5-dimethoxyphenyl)ethanone



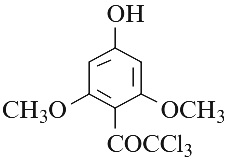
$C_{10}H_9Cl_3O_4$ mol.wt. 299.54

Synthesis

- Preparation by reaction of trichloroacetonitrile on 3,4-di-methoxylphenol (Hoesch reaction) (26%) [4679].

m.p. 107° [4679].

2,2,2-Trichloro-1-(4-hydroxy-2,6-dimethoxyphenyl)ethanone



$C_{10}H_9Cl_3O_4$ mol.wt. 299.54

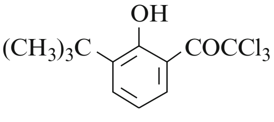
Synthesis

- Preparation by reaction of trichloroacetonitrile on phloroglucinol dimethyl ether (Hoesch reaction) (38%) [4680].

m.p. 117° [4680].

2,2,2-Trichloro-1-[3-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone

[111422-36-7] $C_{12}H_{13}Cl_3O_2$ mol.wt. 295.59



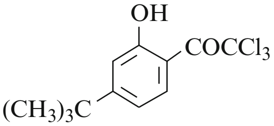
Synthesis

- Preparation by reaction of trichloroacetyl chloride on bromomagnesium 2-tert-butylphenolate in toluene at r.t. (65%) [4564].

m.p. 54–58° [4564]; ^1H NMR [4564], IR [4564], MS [4564].

2,2,2-Trichloro-1-[4-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone

[145818-24-2] $C_{12}H_{13}Cl_3O_2$ mol.wt. 295.59

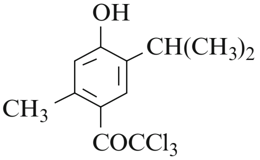


Synthesis

- Preparation by reaction of trichloroacetonitrile on 3-tert- butylphenol (Hoesch reaction) (74%) [4619].

m.p. 46–47°5 [4619]; ^1H NMR [4619], IR [4619], UV [4619], MS [4619].

2,2,2-Trichloro-1-[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]ethanone



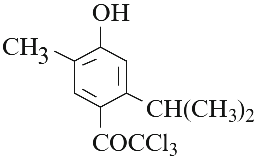
$C_{12}H_{13}Cl_3O_2$ mol.wt. 295.59

Synthesis

- Preparation by reaction of trichloroacetonitrile on thymol (Hoesch reaction) (73%) [4681].

m.p. 99–100° [4681].

2,2,2-Trichloro-1-[4-hydroxy-5-methyl-2-(1-methylethyl)phenyl]ethanone

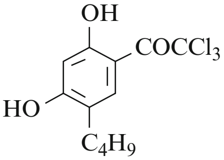


$C_{12}H_{13}Cl_3O_2$ mol.wt. 295.59

Synthesis

- Preparation by reaction of trichloroacetonitrile on carvacrol (Hoesch reaction) (75%) [4681]. oil [4681].

1-(5-Butyl-2,4-dihydroxyphenyl)-2,2,2-trichloroethanone



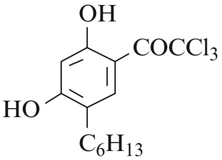
$C_{12}H_{13}Cl_3O_3$ mol.wt. 311.59

Synthesis

- Preparation by reaction of trichloroacetonitrile on 4-n-butylresorcinol (Hoesch reaction) (57%) [4678].

m.p. 95–98° [4678].

2,2,2-Trichloro-1-(5-hexyl-2,4-dihydroxyphenyl)ethanone



$C_{14}H_{17}Cl_3O_3$ mol.wt. 339.65

Synthesis

- Preparation by reaction of trichloroacetonitrile on 4-n-hexylresorcinol (Hoesch reaction) (56%) [4678].

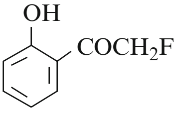
m.p. 71–73° [4678].

11.3 Compounds Derived from Fluoroacetic Acids

11.3.1 From Monofluoroacetic Acid

2-Fluoro-1-(2-hydroxyphenyl)ethanone

[83505-27-5]



$C_8H_7FO_2$ mol.wt. 154.14

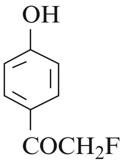
Synthesis

- When refluxed with water, 3-fluoro-4-hydroxycoumarin, undergoes hydrolytic ring opening and decarboxylation to give 2-fluoro-2′-hydroxyacetophenone (32%) [4682].

m.p. 67–69° [4682]; ^1H NMR [4682], ^{19}F NMR [4682], IR [4682], MS [4682].

2-Fluoro-1-(4-hydroxyphenyl)ethanone

[295779-85-0] $C_8H_7FO_2$ mol.wt. 154.14



Synthesis

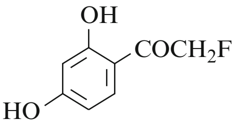
- Preparation by fluorination of p-hydroxyacetophenone (2 mmol) with 1-fluoro-4-hydroxy-1,4-diazoniabicyclo-[2.2.2] octane bis(tetrafluoroborate)(Accufluor ™ NFTh) (2.1 mmol of active compound) in refluxing methanol for 0.5–4 h until potassium iodide starch paper showed the consumption of the fluorinating reagent. Then, hydrolysis of the dimethylketal formed with 10% hydrochloric acid solution in acetonitrile (75–85%) [4683].

N.B.: Accufluor ™ NFTh is commercially available as 50% w/w on alumina.

m.p. 180–183°5 [4684];
^{1}H NMR [4684], ^{13}C NMR [4684], ^{19}F NMR [4684], IR [4684], MS [4684].

1-(2,4-Dihydroxyphenyl)-2-fluoroethanone

[147220-82-4] $C_8H_7FO_3$ mol.wt. 170.14

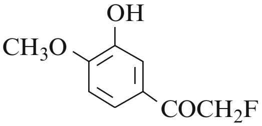


Synthesis

- Refer to: [4685] (compound Io).

2-Fluoro-1-(3-hydroxy-4-methoxyphenyl)ethanone

[85465-61-8] $C_9H_9FO_3$ mol.wt. 184.17



Synthesis

- Preparation by heating a mixture of 3-acetoxy-4-methoxy-α-bromoacetophenone and potassium hydrogen fluoride in diethylene glycol for 8 h at 100° (61%) [4686,4687].

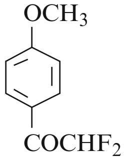
m.p. 70–71° [4686,4687].

11.3.2 From Difluoroacetic Acid

There is no hydroxyketone derived from difluoroacetic acid such as described up to December 2003. Only, a methyl ether, the α,α-difluoro-4-methoxyacetophenone [4688] (compound 1G), is mentioned in 1988.

2,2-Difluoro-1-(4-methoxyphenyl)ethanone

[114829-07-1] $C_9H_8F_2O_2$ mol.wt. 186.16



Synthesis

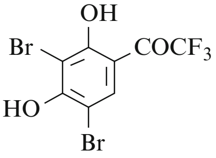
– Preparation by reaction of difluoroacetyl chloride with anisole in the presence of aluminium chloride in methylene chloride, first at 4°, then at 20° (56%) [4688].

m.p. 39–40° [4688]; b.p.$_{25}$ 134–135° [4688];
^{1}H NMR [4688].

11.3.3 *From Trifluoroacetic Acid*

1-(3,5-Dibromo-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65240-11-1] $C_8H_3Br_2F_3O_3$ mol.wt. 363.91



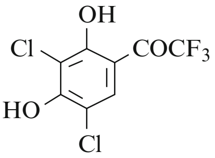
Synthesis

– Preparation by reaction of bromine on 2,4-dihydroxy-α,α,α-trifluoroacetophenone in acetic acid at r.t. (49%) [4689].

m.p. 81° [4689].

1-(3,5-Dichloro-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65240-09-7] $C_8H_3Cl_2F_3O_3$ mol.wt. 275.01



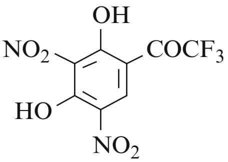
Synthesis

– Preparation by reaction of sulfuryl chloride on 2,4-dihydroxy-α,α,α-trifluoroacetophenone at r.t. [4689].

m.p. 101° [4689].

1-(2,4-Dihydroxy-3,5-dinitrophenyl)-2,2,2-trifluoroethanone

[65240-17-7] $C_8H_3F_3N_2O_7$ mol.wt. 296.12



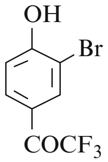
Synthesis

– Preparation by reaction of 65% nitric acid on 2,4-dihydroxy-α,α,α-trifluoroacetophenone in concentrated sulfuric acid [4689].

m.p. 68° [4689].

1-(3-Bromo-4-hydroxyphenyl)-2,2,2-trifluoroethanone

[303143-05-7] $C_8H_4BrF_3O_2$ mol.wt. 269.02

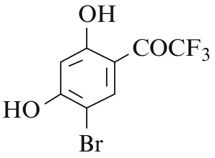


Synthesis

– Obtained by demethylation of 3-bromo-4-methoxy-α,α,α-trifluoroacetophenone with lithium chloride in refluxing DMF for 2 h (93%) [4690].

1-(5-Bromo-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65239-86-3] $C_8H_4BrF_3O_3$ mol.wt. 285.02



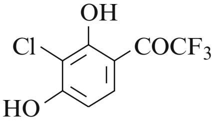
Synthesis

– Preparation by reaction of trifluoroacetic anhydride on 4-bromoresorcinol with aluminium chloride in ethylene dichloride at r.t. (88%) [4689].

m.p. 81° [4689].

1-(3-Chloro-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65239-93-2] $C_8H_4ClF_3O_3$ mol.wt. 240.57



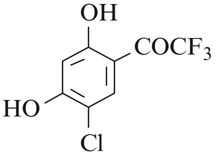
Synthesis

– Preparation by reaction of trifluoroacetic anhydride on 2-chlororesorcinol with aluminium chloride in ethylene dichloride at r.t. (83%) [4689].

m.p. 113° [4689].

1-(5-Chloro-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65233-63-8] $C_8H_4ClF_3O_3$ mol.wt. 240.57



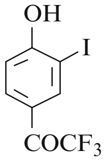
Synthesis

– Preparation by reaction of trifluoroacetic anhydride on 4-chlororesorcinol with aluminium chloride in ethylene dichloride at r.t. (90%) [4689].

m.p. 110° [4689].

2,2,2-Trifluoro-1-(4-hydroxy-3-iodophenyl)ethanone

[303143-06-8] $C_8H_4F_3IO_2$ mol.wt. 316.02

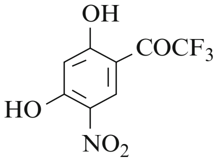


Synthesis

– Obtained by demethylation of 3-iodo-4-methoxy-α,α,α-tri-fluoroacetophenone with lithium chloride in refluxing DMF for 2 h (87%) [4690].

1-(2,4-Dihydroxy-5-nitrophenyl)-2,2,2-trifluoroethanone

[65240-16-6] $C_8H_4F_3NO_5$ mol.wt. 251.12



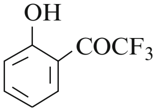
Synthesis

- Preparation by reaction of 26% nitric acid on 2,4-dihydroxy-α,α,α-trifluoroacetophenone in acetic acid at 0° [4689].

m.p. 81° [4689].

2,2,2-Trifluoro-1-(2-hydroxyphenyl)ethanone

[25666-51-7] $C_8H_5F_3O_2$ mol.wt. 190.12



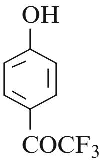
Synthesis

- Preparation by Fries rearrangement of phenyl trifluoroacetate with aluminium chloride without solvent at 90° (42%) [4691].

b.p.$_{55}$ 92° [4691]; ^{1}H NMR [4691], IR [4691].

2,2,2-Trifluoro-1-(4-hydroxyphenyl)ethanone

[1823-63-8] $C_8H_5F_3O_2$ mol.wt. 190.12



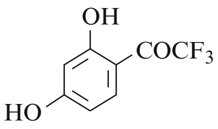
Syntheses

- Preparation by reaction of trifluoroacetic acid with phenol in hydrofluoric acid at 100° (75%) [4692].
- Obtained in small amount by Fries rearrangement of phenyl trifluoroacetate with aluminium chloride without solvent at 90° [4691].

m.p. 105°5–106° [4691]; ^{1}H NMR [4692], ^{19}F NMR [4692], IR [4691,4692].

1-(2,4-Dihydroxyphenyl)-2,2,2-trifluoroethanone

[315-44-6] $C_8H_5F_3O_3$ mol.wt. 206.12



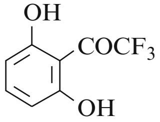
Syntheses

- Preparation by reaction of trifluoroacetonitrile on resorcinol (Hoesch reaction) (62%) [4679,4693,4694].
- Preparation by reaction of trifluoroacetic anhydride on resorcinol with aluminium chloride in ethylene dichloride at r.t. (73–83%) [4695].

m.p. 106–108° [4695], 103° [4679,4693].

1-(2,6-Dihydroxyphenyl)-2,2,2-trifluoroethanone

[70211-42-6] $C_8H_5F_3O_3$ mol.wt. 206.12



Syntheses

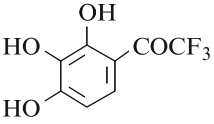
- Obtained by total demethylation of 2,6-dimethoxy-α,α,α-trifluoroacetophenone with boron tribromide in methylene chloride, first at 0° for 15 min, then at r.t. overnight (22%) [4696].
- Also refer to: [4697].

N.B.: There is also an erroneous reference. It concerns the 2′,6′-dihydroxytrifluoroacetanilide [4698].

^{1}H NMR [4696], MS [4696].

2,2,2-Trifluoro-1-(2,3,4-trihydroxyphenyl)ethanone

[65239-87-4] $C_8H_5F_3O_4$ mol.wt. 222.12



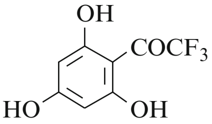
Synthesis

- Preparation by reaction of trifluoroacetic anhydride on pyrogallol with aluminium chloride in ethylene dichloride at r.t. (75%) [4689].

m.p. 134° [4689].

2,2,2-Trifluoro-1-(2,4,6-trihydroxyphenyl)ethanone

[13340-79-9] $C_8H_5F_3O_4$ mol.wt. 222.12



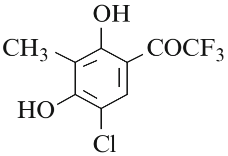
Synthesis

- Preparation by reaction of trifluoroacetic anhydride on phloroglucinol with aluminium chloride in ethylene dichloride at r.t. (40%) [4695].

m.p. 174–177° [4695]; ^{1}H NMR [4695], ^{19}F NMR [4695], IR [4695].

1-(5-Chloro-2,4-dihydroxy-3-methylphenyl)-2,2,2-trifluoroethanone

[65240-08-6] $C_9H_6ClF_3O_3$ mol.wt. 254.59



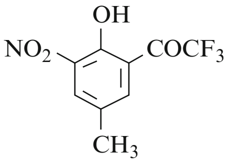
Synthesis

- Preparation by reaction of sulfuryl chloride on 2,4-di-hydroxy-3-methyl-α,α,α-trifluoroacetophenone in ethylene dichloride at r.t. (76%) [4689].

m.p. 96° [4689].

2,2,2-Trifluoro-1-(2-hydroxy-5-methyl-3-nitrophenyl)ethanone

[70978-48-2] $C_9H_6F_3NO_4$ mol.wt. 249.15



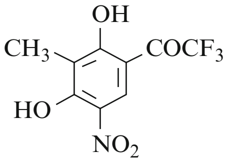
Synthesis

– Preparation by reaction of fuming nitric acid (d = 1.5) on 2-hydroxy-5-methyl-α,α,α-trifluoroacetophenone in acetic acid-methylene chloride mixture at r.t. [4699,4700], (44%) [4699].

m.p. 94–96° [4699], 49–50° [4700]. One of the reported melting points is obviously wrong.

1-(2,4-Dihydroxy-3-methyl-5-nitrophenyl)-2,2,2-trifluoroethanone

[65240-15-5] $C_9H_6F_3NO_5$ mol.wt. 265.15



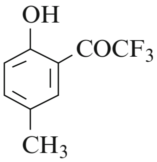
Synthesis

– Obtained by reaction of 26% nitric acid on 2,4-dihydroxy-3-methyl-α,α,α-trifluoroacetophenone in acetic acid at 0° (21%) [4689].

m.p. 104° [4689].

2,2,2-Trifluoro-1-(2-hydroxy-5-methylphenyl)ethanone

[70978-57-3] $C_9H_7F_3O_2$ mol.wt. 204.15



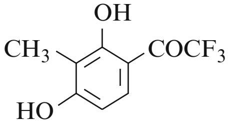
Synthesis

– Preparation by Fries rearrangement of 4-methylphenyl trifluoroacetate with aluminium chloride without solvent at 115° (25%) [4700].

m.p. 40°5–42° [4700].

1-(2,4-Dihydroxy-3-methylphenyl)-2,2,2-trifluoroethanone

[65233-60-5] $C_9H_7F_3O_3$ mol.wt. 220.15



Syntheses

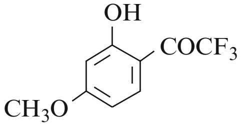
– Preparation by reaction of trifluoroacetic anhydride on 2-methylresorcinol with aluminium chloride in ethylene dichloride at r.t. (90%) [4689].

– Preparation by Fries rearrangement of 2-methylresorcinol monotrifluoroacetate with aluminium chloride in nitrobenzene or without solvent at 120° [4689].

m.p. 101° [4689].

2,2,2-Trifluoro-1-(2-hydroxy-4-methoxyphenyl)ethanone

[123716-19-8] $C_9H_7F_3O_3$ mol.wt. 220.15



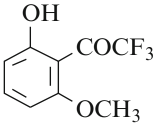
Synthesis

– Obtained by refluxing α-(difluoronitromethyl)-2-hydroxy-4-methoxy-α-(trifluoromethyl)benzenemethanol (SM) in hexane for 6 h in the presence of activated charcoal (91%). SM was prepared by reaction of NPFA with resorcinol monomethyl ether in carbon tetrachloride or nitromethane for 12 h at 20° (99%, m.p. 67–69°) [4701].

m.p. 62–64° [4701];
^{1}H NMR [4701], ^{13}C NMR [4701], ^{19}F NMR [4701]; TLC [4701].

2,2,2-Trifluoro-1-(2-hydroxy-6-methoxyphenyl)ethanone

[193738-66-8] $C_9H_7F_3O_3$ mol.wt. 220.15



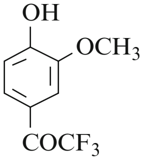
Synthesis

– Obtained by partial demethylation of 2,6-dimethoxy-α,α,α-trifluoroacetophenone with boron tribromide in methylene chloride, first at 0° for 15 min, then at r.t. overnight (17%) [4696].

yellow oil [4696]; ^{1}H NMR [4696], MS [4696].

2,2,2-Trifluoro-1-(4-hydroxy-3-methoxyphenyl)ethanone

[188194-66-3] $C_9H_7F_3O_3$ mol.wt. 220.15

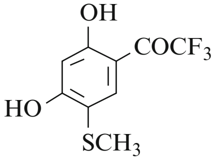


Synthesis

– This compound (**6**) [4702] was synthesized according to Dudley [4703].

1-[2,4-Dihydroxy-5-(methylthio)phenyl]-2,2,2-trifluoroethanone

[65239-90-9] $C_9H_7F_3O_3S$ mol.wt. 252.21



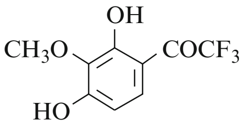
Synthesis

– Preparation by reaction of trifluoroacetic anhydride on 4-methylthioresorcinol with aluminium chloride in ethylene dichloride at r.t. (68%) [4689].

m.p. 57° [4689].

1-(2,4-Dihydroxy-3-methoxyphenyl)-2,2,2-trifluoroethanone

[65239-88-5] $C_9H_7F_3O_4$ mol.wt. 236.15



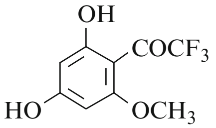
Synthesis

- Preparation by reaction of trifluoroacetic anhydride on 2-methoxyresorcinol with aluminium chloride in ethylene dichloride at r.t. (78%) [4689].

m.p. 79° [4689].

1-(2,4-Dihydroxy-6-methoxyphenyl)-2,2,2-trifluoroethanone

$C_9H_7F_3O_4$ mol.wt. 236.15



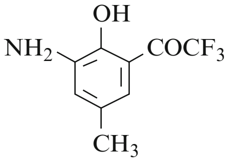
Synthesis

- Preparation by reaction of trifluoroacetonitrile on phloroglucinol monomethyl ether with zinc chloride and gaseous hydrochloric acid in ethyl ether at 0° (Hoesch reaction) (33%) [4679].

m.p. 154° [4679].

1-(3-Amino-2-hydroxy-5-methylphenyl)-2,2,2-trifluoroethanone

[70977-83-2] $C_9H_8F_3NO_2$ mol.wt. 219.16



Synthesis

- Preparation by catalytic hydrogenation of 2-hydroxy-5-methyl-3-nitro-α,α,α-trifluoroacetophenone in the presence of 5% Pt/C in ethanol at 25° [4699,4700], (57%) [4699].

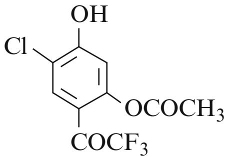
m.p. 87–88° [4699,4700].

1-[2-(Acetyloxy)-5-chloro-4-hydroxyphenyl]-2,2,2-trifluoroethanone

or

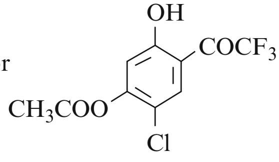
1-[4-(Acetyloxy)-5-chloro-2-hydroxyphenyl]-2,2,2-trifluoroethanone

[65233-64-9] $C_{10}H_6ClF_3O_4$
mol.wt. 282.60



(I)

or



(II)

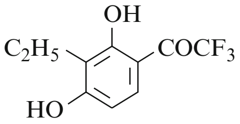
Synthesis

- Preparation by reaction of acetyl chloride on 5-chloro-2,4-dihydroxy-α,α,α-trifluoroacetophenone with pyridine in benzene at r.t. (72%) [4689].
  m.p. 80–83° [4689].

N.B.: The 4-(Acetyloxy)-5-chloro-2-hydroxy-α,α,α-trifluoroacetophenone (**II**) is the most likely formula, for two reasons: On the one hand, there is a strong chelation between hydroxyl and keto groups in the raw material and, on the other hand, the esterification was carried out at r.t.

1-(3-Ethyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[577-54-8] $C_{10}H_9F_3O_3$ mol.wt. 234.17



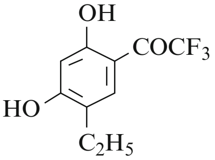
Synthesis

- Preparation by reaction of trifluoroacetonitrile on 2-ethyl-resorcinol (Hoesch reaction) (74%) [4679].

m.p. 139° [4679].

1-(5-Ethyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[584-41-8] $C_{10}H_9F_3O_3$ mol.wt. 234.17



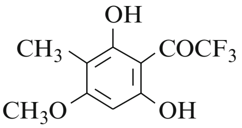
Synthesis

- Preparation by reaction of trifluoroacetonitrile on 4-ethyl-resorcinol (Hoesch reaction) (71%) [4679].

  m.p. 99° [4679].

1-(2,6-Dihydroxy-4-methoxy-3-methylphenyl)-2,2,2-trifluoroethanone

$C_{10}H_9F_3O_4$ mol.wt. 250.17



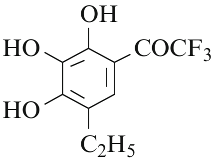
Synthesis

- Preparation by reaction of trifluoroacetonitrile on 1,3-dihydroxy-5-methoxy-4-methylbenzene(Hoesch reaction) (22%) [4680].

m.p. 145° [4680].

1-(5-Ethyl-2,3,4-trihydroxyphenyl)-2,2,2-trifluoroethanone

[65239-92-1] $C_{10}H_9F_3O_4$ mol.wt. 250.17

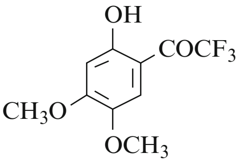


Synthesis

- Preparation by reaction of trifluoroacetic anhydride on 4-ethylpyrogallol with aluminium chloride in ethylene dichloride at r.t. (80%) [4689].

m.p. 82° [4689].

2,2,2-Trifluoro-1-(2-hydroxy-4,5-dimethoxyphenyl)ethanone



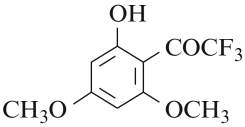
$C_{10}H_9F_3O_4$ mol.wt. 250.17

Synthesis

- Preparation by reaction of trifluoroacetonitrile on 3,4-di-methoxyphenol (Hoesch reaction) (62%) [4679].

m.p. 82° [4679].

2,2,2-Trifluoro-1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone



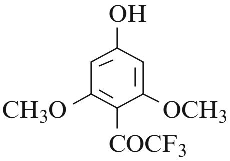
$C_{10}H_9F_3O_4$ mol.wt. 250.17

Synthesis

- Obtained (by-product) by reaction of trifluoroacetonitrile on phloroglucinol dimethyl ether (Hoesch reaction) (5%) [4680].

m.p. 87° [4680].

2,2,2-Trifluoro-1-(4-hydroxy-2,6-dimethoxyphenyl)ethanone



$C_{10}H_9F_3O_4$ mol.wt. 250.17

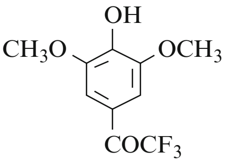
Synthesis

- Preparation by reaction of trifluoroacetonitrile on phloroglucinol dimethyl ether (Hoesch reaction) (25%) [4680].

m.p. 155° [4680].

2,2,2-Trifluoro-1-(4-hydroxy-3,5-dimethoxyphenyl)ethanone

[188194-67-4]



$C_{10}H_9F_3O_4$ mol.wt. 250.17

Synthesis

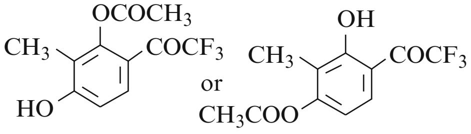
- This compound (**7**) [4702] was synthesized according to Dudley [4703].

1-[2-(Acetyloxy)-4-hydroxy-3-methylphenyl]-2,2,2-trifluoroethanone

or

1-[4-(Acetyloxy)-2-hydroxy-3-methylphenyl]-2,2,2-trifluoroethanone

[65233-62-7]



(I) **(II)**

$C_{11}H_9F_3O_4$ mol.wt. 262.19

Synthesis

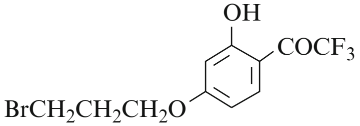
- Preparation by reaction of acetyl chloride on 2,4-dihydroxy-3-methyl-α,α,α-trifluoroacetophenone with pyridine in benzene at r.t. (87%) [4689].

m.p. 49–50° [4689].

N.B.: The 1-[4-(Acetyloxy)-2-hydroxy-3-methylphenyl]-2,2,2-trifluoroethanone (**II**) is the most likely formula. This hypothesis is supported by the fact that, considering the precursor (2,4-di-hydroxy-3-methyl-α,α,α-trifluoroacetophenone), the hydroxy group in the 4-position is less hindered than the hydroxyl substituent in the 2-position which is furthermore chelated with the vicinal carbonyl group. In addition, the reported melting point (49–50°) is in good agreement with those generally measured for o-hydroxyketones (below 80°) compared to those of p-hydroxy-ketones which are considerably higher (usually 120–200°).

1-[4-(3-Bromopropoxy)-2-hydroxyphenyl]-2,2,2-trifluoroethanone

[125617-37-0]



$C_{11}H_{10}BrF_3O_3$ mol.wt.327.10

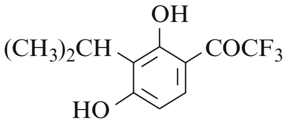
Synthesis

- Preparation by reaction of 1,3-dibromopropane with 2,4-dihydroxy-α,α,α-trifluoroacetophenone in the presence of potassium carbonate and potassium iodide in refluxing methyl ethyl ketone [4704].

pale yellow oil [4704]; IR [4704].

1-[2,4-Dihydroxy-3-(1-methylethyl)phenyl]-2,2,2-trifluoroethanone

[65239-70-5]



$C_{11}H_{11}F_3O_3$ mol.wt. 248.20

Syntheses

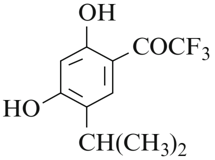
- Preparation by reaction of trifluoroacetic anhydride on 2-isopropylresorcinol with aluminium chloride in ethylene dichloride at r.t. (85%) [4689].

- Preparation by reaction of isopropanol on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with polyphosphoric acid at 80° (30%) [4689].
- Preparation by reaction of propylene on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with phosphorous oxychloride and phosphoric anhydride at 50° [4689].
- Preparation by Fries rearrangement of 2-isopropylresorcinol trifluoroacetate with aluminium chloride without solvent or in nitrobenzene at 120° [4689].

m.p. 145° [4689].

1-[2,4-Dihydroxy-5-(1-methylethyl)phenyl]-2,2,2-trifluoroethanone

[65239-68-1] $C_{11}H_{11}F_3O_3$ mol.wt. 248.20



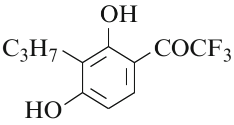
Synthesis

- Preparation by reaction of trifluoroacetic anhydride on 4-isopropylresorcinol with aluminium chloride in chloroform at r.t. (70%) [4689].

m.p. 97° [4689].

1-(2,4-Dihydroxy-3-propylphenyl)-2,2,2-trifluoroethanone

[65239-69-2] $C_{11}H_{11}F_3O_3$ mol.wt. 248.20



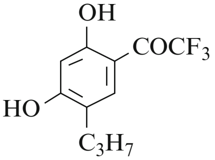
Syntheses

- Preparation by reaction of trifluoroacetic anhydride on 2-propylresorcinol with aluminium chloride in ethylene dichloride at r.t. (88%) [4689].
- Preparation by reaction of trifluoroacetonitrile on 2-propylresorcinol (Hoesch reaction) (66%) [4705].

m.p. 114° [4689], 110–112° [4705]; ^{1}H NMR [4705].

1-(2,4-Dihydroxy-5-propylphenyl)-2,2,2-trifluoroethanone

[65239-67-0] $C_{11}H_{11}F_3O_3$ mol.wt. 248.20



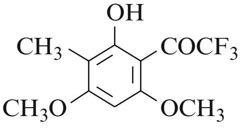
Synthesis

- Preparation by reaction of trifluoroacetic anhydride on 4-propylresorcinol with aluminium chloride in ethylene dichloride at r.t. (87%) [4689].

m.p. 95° [4689].

2,2,2-Trifluoro-1-(2-hydroxy-4,6-dimethoxy-3-methylphenyl)ethanone

$C_{11}H_{11}F_3O_4$ mol.wt. 264.20



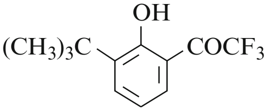
Synthesis

- Preparation by reaction of trifluoroacetonitrile on 3,5-dimethoxy-2-methylphenol (Hoesch reaction) (43%) [4680].

m.p. 100° [4680].

1-[3-(1,1-Dimethylethyl)-2-hydroxyphenyl]-2,2,2-trifluoroethanone

[111422-37-8] $C_{12}H_{13}F_3O_2$ mol.wt. 246.23



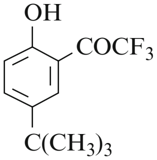
Synthesis

– Preparation by reaction of trifluoroacetyl chloride on bromomagnesium 2-tert-butylphenolate in toluene at r.t. (74%) [4564].

yellow oil [4564]; ^{1}H NMR [4564], IR [4564], MS [4564].

1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-2,2,2-trifluoroethanone

[75060-56-9] $C_{12}H_{13}F_3O_2$ mol.wt. 246.23



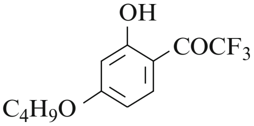
Synthesis

– Preparation by demethylation of 5-tert-butyl-2-methoxy-α,α,α-trifluoroacetophenone with 47% hydrobromic acid and 57% hydriodic acid in refluxing acetic acid (70%) [4661].

^{1}H NMR [4661], IR [4661], MS [4661].

1-(4-Butoxy-2-hydroxyphenyl)-2,2,2-trifluoroethanone

[65240-27-9] $C_{12}H_{13}F_3O_3$ mol.wt. 262.23



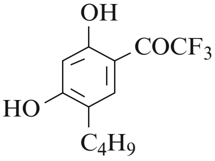
Synthesis

– Obtained by reaction of butyl iodide on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with potassium carbonate in refluxing acetone (29%) [4689].

m.p. 66° [4689].

1-(5-Butyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65239-71-6] $C_{12}H_{13}F_3O_3$ mol.wt. 262.23



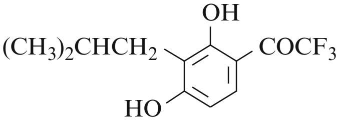
Synthesis

– Preparation by reaction of trifluoroacetic anhydride on 4-butylresorcinol with aluminium chloride in ethylene dichloride at r.t. (82%) [4689].

m.p. 96° [4689].

1-[2,4-Dihydroxy-3-(2-methylpropyl)phenyl]-2,2,2-trifluoroethanone

[65239-73-8] $C_{12}H_{13}F_3O_3$ mol.wt. 262.23



Syntheses

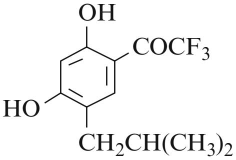
– Preparation by reaction of trifluoroacetic anhydride on 2-isobutylresorcinol with aluminium chloride in ethylene dichloride at r.t. (78%) [4689].

- Preparation by Fries rearrangement of 2-isobutylresorcinol monotrifluoroacetate with aluminium chloride in nitro-benzene at 120° [4689].
- Preparation by reaction of isobutyl alcohol on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with polyphosphoric acid at 80° [4689].
- Preparation by reaction of isobutylene on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with phosphorous pentoxide and phosphorous oxychloride at 50° [4689].

m.p. 114° [4689].

1-[2,4-Dihydroxy-5-(2-methylpropyl)phenyl]-2,2,2-trifluoroethanone

[65239-72-7] $C_{12}H_{13}F_3O_3$ mol.wt. 262.23



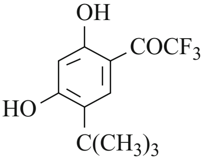
Syntheses

- Preparation by reaction of trifluoroacetic anhydride on 4-isobutylresorcinol with aluminium chloride in ethylene dichloride at r.t. (78–84%) [4689].
- Preparation by reaction of isobutyl alcohol on 2,4-di-hydroxy-α,α,α-trifluoroacetophenone with polyphosphoric acid at 80° [4689].

m.p. 90° [4689]; b.p.$_{0.1}$ 90° [4689].

1-[5-(1,1-Dimethylethyl)-2,4-dihydroxyphenyl]-2,2,2-trifluoroethanone

[65239-74-9] $C_{12}H_{13}F_3O_3$ mol.wt. 262.23



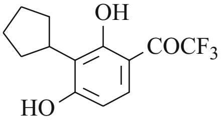
Syntheses

- Preparation by reaction of trifluoroacetic anhydride on 4-tert-butylresorcinol with aluminium chloride in ethylene dichloride at r.t. (80%) [4689].
- Preparation by reaction of tert-butyl alcohol on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with polyphosphoric acid at 80° [4689].

m.p. 159° [4689].

1-(3-Cyclopentyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65240-20-2] $C_{13}H_{13}F_3O_3$ mol.wt. 274.24



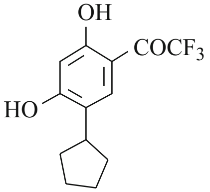
Synthesis

- Preparation by reaction of cyclopentene on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with phosphorous oxychloride-phosphorous pentoxide mixture at 50° [4689].

m.p. 166° [4689].

1-(5-Cyclopentyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65239-76-1] $C_{13}H_{13}F_3O_3$ mol.wt. 274.24



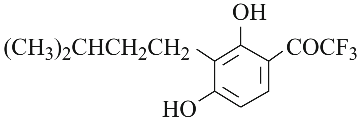
Synthesis

– Preparation by reaction of trifluoroacetic anhydride on 4-cyclopentylresorcinol with aluminium chloride in ethylene dichloride at r.t. (75%) [4689].

m.p. 94° [4689].

1-[2,4-Dihydroxy-3-(3-methylbutyl)phenyl]-2,2,2-trifluoroethanone

[65239-77-2] $C_{13}H_{15}F_3O_3$ mol.wt. 276.26



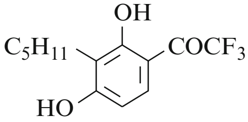
Synthesis

– Preparation by reaction of trifluoroacetic anhydride on 2-isopentylresorcinol with aluminium chloride in ethylene dichloride at r.t. (84%) [4689].

m.p. 101° [4689].

1-(2,4-Dihydroxy-3-pentylphenyl)-2,2,2-trifluoroethanone

[65239-78-3] $C_{13}H_{15}F_3O_3$ mol.wt. 276.26



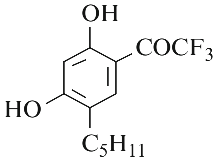
Synthesis

– Preparation by reaction of trifluoroacetic anhydride on 2-pentylresorcinol with aluminium chloride in ethylene dichloride at r.t. (87%) [4689].

m.p. 105° [4689].

1-(2,4-Dihydroxy-5-pentylphenyl)-2,2,2-trifluoroethanone

[65239-75-0] $C_{13}H_{15}F_3O_3$ mol.wt. 276.26



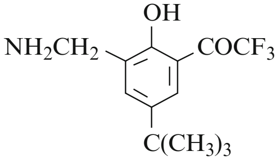
Synthesis

– Preparation by reaction of trifluoroacetic anhydride on 4-pentylresorcinol with aluminium chloride in ethylene dichloride at r.t. (86%) [4689].

m.p. 97° [4689].

1-[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-2,2,2-trifluoroethanone

[75060-97-8] $C_{13}H_{16}F_3NO_2$ mol.wt. 275.27

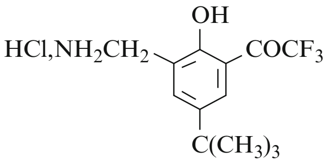


Synthesis

– Preparation from the corresponding hydrochloride (see below) [4661].

1-[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-2,2,2-trifluoroethanone *(Hydrochloride)*

[75060-74-1] $C_{13}H_{16}F_3NO_2$, HCl mol.wt. 311.73



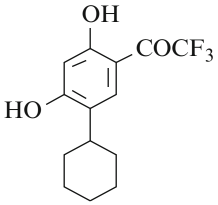
Synthesis

– Preparation by reaction of concentrated hydrochloric acid on 4-tert-butyl-6-(N-chloroacetylaminomethyl)-2-(trifluoroacetyl) phenol in refluxing ethanol (83%) [4661].

m.p. 180–186° [4661]; ^{1}H NMR [4661], IR [4661].

1-(5-Cyclohexyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65239-79-4] $C_{14}H_{15}F_3O_3$ mol.wt. 288.27



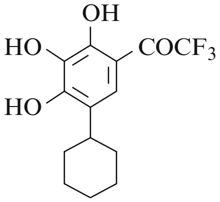
Synthesis

– Preparation by reaction of trifluoroacetic anhydride with 4-cyclohexylresorcinol in the presence of aluminium chloride in ethylene dichloride at r.t. (78%) [4689].

m.p. 80° [4689].

1-(5-Cyclohexyl-2,3,4-trihydroxyphenyl)-2,2,2-trifluoroethanone

[65239-91-0] $C_{14}H_{15}F_3O_4$ mol.wt. 304.27



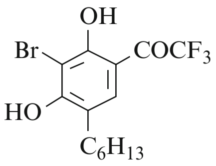
Synthesis

– Preparation by reaction of trifluoroacetic anhydride with 4-cyclohexylpyrogallol in the presence of aluminium chloride in ethylene dichloride at r.t. (78%) [4689].

m.p. 128° [4689].

1-(3-Bromo-5-hexyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65240-12-2] $C_{14}H_{16}BrF_3O_3$ mol.wt. 369.18



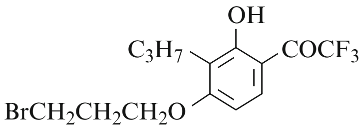
Synthesis

– Preparation by reaction of bromine on 5-n-hexyl-2,4-dihydroxy-α,α,α-trifluoroacetophenone in acetic acid at r.t. (43%) [4689].

m.p. 39° [4689].

1-[4-(3-Bromopropoxy)-2-hydroxy-3-propylphenyl]-2,2,2-trifluoroethanone

[125617-40-5] $C_{14}H_{16}BrF_3O_3$ mol.wt. 369.18



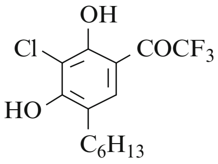
Synthesis

- Preparation by reaction of 1,3-dibromopropane with 2,4-dihydroxy-3-propyl-α,α,α-trifluoroacetophenone in the presence of potassium carbonate and potassium iodide in refluxing methyl ethyl ketone [4704].

yellow oil [4704]; IR [4704].

1-(3-Chloro-5-hexyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65240-10-0] $C_{14}H_{16}ClF_3O_3$ mol.wt. 324.73



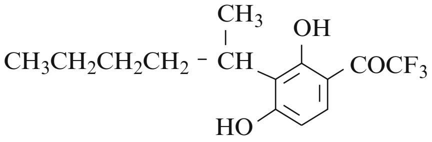
Synthesis

- Preparation by reaction of sulfuryl chloride on 5-n-hexyl-2,4-dihydroxy-α,α,α-trifluoroacetophenone at r.t. [4689].

m.p. 40° [4689].

1-[2,4-Dihydroxy-3-(1-methylpentyl)phenyl]-2,2,2-trifluoroethanone

[65240-18-8] $C_{14}H_{17}F_3O_3$ mol.wt. 290.28



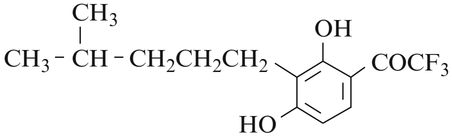
Synthesis

- Preparation by reaction of 1-hexene on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with phosphorous oxychloride and phosphorous pentoxide at 50° (30%) [4689].

m.p. 97° [4689].

1-[2,4-Dihydroxy-3-(4-methylpentyl)phenyl]-2,2,2-trifluoroethanone

[65240-07-5] $C_{14}H_{17}F_3O_3$ mol.wt. 290.28



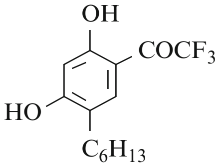
Synthesis

- Preparation by Fries rearrangement of 2-iso- hexyl-3-hydroxyphenyl trifluoroacetate with aluminium chloride in nitrobenzene or without solvent at 120° [4689].

m.p. 97° [4689].

2,2,2-Trifluoro-1-(5-hexyl-2,4-dihydroxyphenyl)ethanone

[65233-68-3] $C_{14}H_{17}F_3O_3$ mol.wt. 290.28



Syntheses

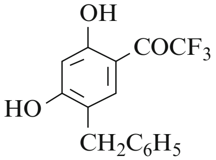
- Preparation by Fries rearrangement of 4-hexyl-3-hydroxyphenyl trifluoroacetate with aluminium chloride at 120°, in nitrobenzene (47%) or without solvent (38%) [4689].
- Preparation by reaction of trifluoroacetamide on 4-hexyl-resorcinol at reflux, with boron trifluoride etherate (55%) or with p-toluenesulfonic acid (20%) [4689].
- Preparation by reaction of trifluoroacetonitrile on 4-hexylresorcinol, in the presence of hydrochloric acid,
  - with zinc chloride in ethyl ether at 0° (Hoesch reaction) (69%) [4689];
  - with aluminium chloride (Houben reaction), in ethylene dichloride (60%), in phosphorous oxychloride (55%), in toluene (20%) or in nitrobenzene (15%) [4689].
- Preparation by reaction of trifluoroacetyl chloride on 4-hexylresorcinol at r.t. [4689],
  - with aluminium chloride in ethylene dichloride (92%) or in phosphorous oxychloride (20%);
  - with boron trifluoride etherate in ethylene dichloride (40%);
  - with zinc chloride in ethylene dichloride (30%).
- Preparation by reaction of trifluoroacetic acid on 4-hexylresorcinol in ethylene dichloride [4689],
  - with phosphorous pentachloride (54%) or p-toluenesulfonic acid (30%) at r.t.;
  - with boron trifluoride etherate at reflux (40%).
- Also obtained by reaction of ethyl trifluoroacetate on 4-hexylresorcinol with p-toluenesulfonic acid at reflux (25%) [4689].
- Preparation by reaction of trifluoroacetic anhydride on 4-hexylresorcinol at r.t. (see table below) [4689].

Catalyst	Solvent	Yield (%)
$AlCl_3$	$POCl_3$	80
	$C_6H_5CH_3$	10
	$C_6H_5NO_2$	75
	$CH_2Cl–CH_2Cl$	85
$ZnCl_2$	$POCl_3$	60
	$C_6H_5CH_3$	65
	$C_6H_5NO_2$	83
	$CH_2Cl–CH_2Cl$	87
	$(C_2H_5)_2O$	15
$FeCl_3$	$POCl_3$	30
	$C_6H_5CH_3$	30
	$C_6H_5NO_2$	45
	$CH_2Cl–CH_2Cl$	68
PCl_5	$POCl_3$	60 (at 100°)
	$C_6H_5CH_3$	20
	$C_6H_5NO_2$	20
4-$CH_3C_6H_4SO_3H$	$C_6H_5CH_3$	25
	$C_6H_5NO_2$	25
	CH_2Cl-CH_2Cl	22
BF_3-Et_2O	$C_6H_5CH_3$	35
	$CH_2Cl–CH_2Cl$	60
	$(C_2H_5)_2O$	70

m.p. 90° [4689].

1-[2,4-Dihydroxy-5-(phenylmethyl)phenyl]-2,2,2-trifluoroethanone

[65239-81-8] $C_{15}H_{11}F_3O_3$ mol.wt. 296.25



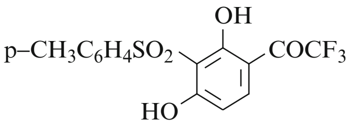
Synthesis

- Preparation by reaction of trifluoroacetic anhydride on 4-benzylresorcinol with aluminium chloride in ethylene dichloride at r.t. (80%) [4689].

m.p. 114° [4689].

1-[2,4-Dihydroxy-3-[(4-methylphenyl)sulfonyl]phenyl]-2,2,2-trifluoroethanone

[65240-14-4] $C_{15}H_{11}F_3O_5S$ mol.wt. 360.31



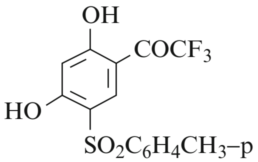
Synthesis

- Obtained by reaction of p-toluenesulfonyl chloride on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with aluminium chloride in phosphorous oxychloride (22%) [4689].

m.p. 127° [4689].

1-[2,4-Dihydroxy-5-[(4-methylphenyl)sulfonyl]phenyl]-2,2,2-trifluoroethanone

[65240-13-3] $C_{15}H_{11}F_3O_5S$ mol.wt. 360.31



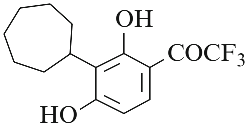
Synthesis

- Preparation by reaction of p-toluenesulfonyl chloride on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with ferric chloride in phosphorous oxychloride at 120° (40%) [4689].

m.p. 145° [4689].

1-(3-Cycloheptyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65240-21-3] $C_{15}H_{17}F_3O_3$ mol.wt. 302.29



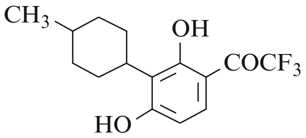
Synthesis

- Preparation by reaction of cycloheptene on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with phosphorous trichloride and phosphorous pentoxide at 50° [4689].

m.p. 174° [4689].

1-[2,4-Dihydroxy-3-(4-methylcyclohexyl)phenyl]-2,2,2-trifluoroethanone

[65239-82-9] $C_{15}H_{17}F_3O_3$ mol.wt. 302.29



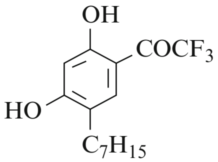
Syntheses

- Preparation by reaction of trifluoroacetic anhydride on 2-(4-methylcyclohexyl)resorcinol with aluminium chloride in ethylene dichloride at r.t. (76%) [4689].
- Preparation by Fries rearrangement of 3-hydroxy-2-(4-methylcyclohexyl)phenyl trifluoroacetate with aluminium chloride in nitrobenzene or without solvent at 120° [4689].

m.p. 143° [4689].

2,2,2-Trifluoro-1-(5-heptyl-2,4-dihydroxyphenyl)ethanone

[65239-80-7] $C_{15}H_{19}F_3O_3$ mol.wt. 304.31



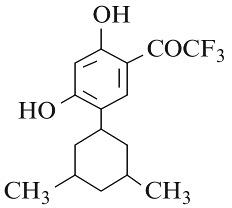
Syntheses

- Preparation by reaction of trifluoroacetic anhydride on 4-heptylresorcinol with aluminium chloride in ethylene dichloride at r.t. (79%) [4689].
- Preparation by Fries rearrangement of 4-heptyl-3-hydroxy-phenyl-α,α,α-trifluoroacetate with aluminium chloride in nitrobenzene or without solvent at 120° [4689].

m.p. 85° [4689].

1-[5-(3,5-Dimethylcyclohexyl)-2,4-dihydroxyphenyl]-2,2,2-trifluoroethanone

[65239-83-0] $C_{16}H_{19}F_3O_3$ mol.wt. 304.31



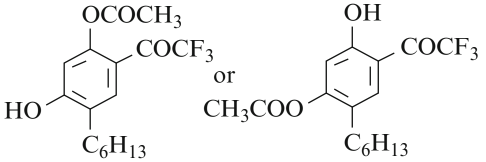
Synthesis

- Preparation by acylation of 4-(3,5-dime-thylcyclohexyl)-resorcinol (1 mol) with trifluoroacetic anhydride (1.2 mol) in the presence of aluminium chloride (2 mol) in ethylene dichloride at r.t. (79%) [4689].

m.p. 126° [4689].

1-[2 (or 4)-(Acetyloxy)-5-hexyl-4 (or 2)-hydroxyphenyl]-2,2,2-trifluoroethanone

[65233-69-4] $C_{16}H_{19}F_3O_4$ mol.wt. 332.32



(I) **(II)**

Synthesis

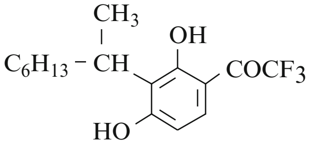
- Preparation by reaction of acetyl chloride on 5-hexyl-2,4-dihydroxy-α,α,α-tri-fluoroacetophenone with pyridine in benzene at r.t. (83%) [4689].

m.p. 30° [4689].

N.B.: The 1-[4-(acetyloxy)-5-hexyl-2-hydroxyphenyl]-2,2,2-trifluoroethanone (**II**) is the most likely formula. This hypothesis is supported by the fact that, considering the precursor (5-hexyl-2,4-di-hydroxy-α,α,α-trifluoroacetophenone), the hydroxy group in the 2-position is chelated with the carbonyl group. In addition, the reported melting point (30°) is in good agreement with those generally measured for o-hydroxyketones (below 80°) compared to those of p-hydroxyketones which are considerably higher (usually 120–200°).

1-[2,4-Dihydroxy-3-(1-methylheptyl)phenyl]-2,2,2-trifluoroethanone

[65240-22-4] $C_{16}H_{21}F_3O_3$ mol.wt. 318.34

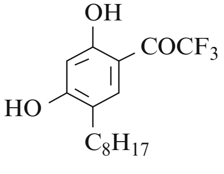


Synthesis

- Refer to: [4689].

1-(2,4-Dihydroxy-5-octylphenyl)-2,2,2-trifluoroethanone

[65239-94-3] $C_{16}H_{21}F_3O_3$ mol.wt. 318.34



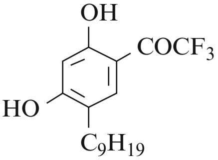
Synthesis

- Preparation by reaction of trifluoroacetic anhydride on 4-octylresorcinol with aluminium chloride in ethylene dichloride at r.t. (74%) [4689].

m.p. 87° [4689].

1-(2,4-Dihydroxy-5-nonylphenyl)-2,2,2-trifluoroethanone

[65239-84-1] $C_{17}H_{23}F_3O_3$ mol.wt. 332.36



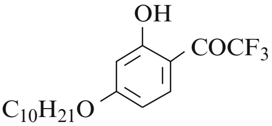
Synthesis

– Preparation by reaction of trifluoroacetic anhydride with 4-nonylresorcinol in the presence of aluminium chloride in ethylene dichloride at r.t. (85%) [4689].

m.p. 87° [4689].

1-[4-(Decyloxy)-2-hydroxyphenyl]-2,2,2-trifluoroethanone

[65240-25-7] $C_{18}H_{25}F_3O_3$ mol.wt. 346.39



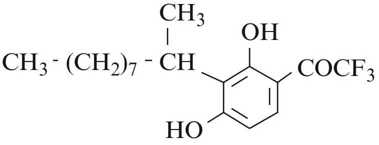
Synthesis

– Preparation by reaction of decyl iodide on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with potassium carbonate in refluxing acetone (53%) [4689].

m.p. 27–28° [4689]; b.p.$_{0.1}$ 150° [4689].

1-[2,4-Dihydroxy-3-(1-methylnonyl)phenyl]-2,2,2-trifluoroethanone

[65134-36-3] $C_{18}H_{25}F_3O_3$ mol.wt. 346.39



Synthesis

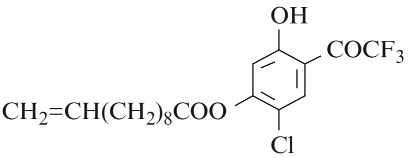
– Preparation by reaction of 1-decene on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with phosphorous oxychloride and phosphorous pentoxide at 50° [4689].

m.p. 98° [4689].

1-[5-Chloro-2-hydroxy-4-(10-undecenoyloxy)phenyl]-2,2,2-trifluoroethanone

10-Undecenoic acid, 2 (or 4)-Chloro-5-hydroxy-4 (or 2)-(trifluoroacetyl)phenyl ester

[65233-67-2] $C_{19}H_{22}ClF_3O_4$ mol.wt. 406.83



Synthesis

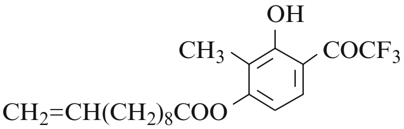
– Preparation by reaction of 10-undecenoyl chloride on 5-chloro-2,4-dihydroxy-α,α,α-trifluoro-acetophenone with pyridine in benzene at r.t. (65%) [4689].

b.p.$_{0.07}$ 168° [4689].

1-[2-Hydroxy-3-methyl-4-(10-undecenoyloxy)phenyl]-2,2,2-trifluoroethanone

10-Undecenoic acid, 3-Hydroxy-2-methyl-4 (or 6)-(trifluoroacetyl)phenyl ester

[65233-66-1]



b.p.$_{0.07}$ 165° [4689].

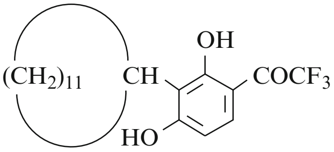
$C_{20}H_{25}F_3O_4$ mol.wt. 386.41

Synthesis

- Preparation by reaction of 10-undecenoyl chloride on 2,4-dihydroxy-3-methyl-α,α,α-trifluoro-acetophenone with pyridine in benzene at r.t. (64%) [4689].

1-(3-Cyclododecyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65240-19-9]



m.p. 166° [4689].

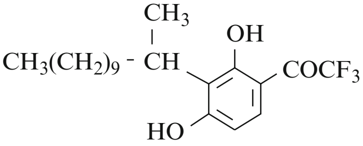
$C_{20}H_{27}F_3O_3$ mol.wt. 372.43

Synthesis

- Preparation by reaction of cyclododecene on 2,4-di-hydroxy-α,α,α-trifluoroacetophenone with phosphorous oxychloride and phosphorous pentoxide at 50° [4689].

1-[2,4-Dihydroxy-3-(1-methylundecyl)phenyl]-2,2,2-trifluoroethanone

[65134-37-4]



m.p. 96° [4689].

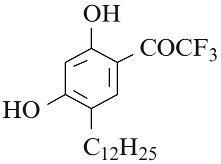
$C_{20}H_{29}F_3O_3$ mol.wt. 374.44

Synthesis

- Preparation by reaction of 1-dodecene on 2,4-di-hydroxy-α,α,α-trifluoroacetophenone with phosphorous oxychloride and phosphorous pentoxide at 50° [4689].

1-(5-Dodecyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone

[65239-85-2]



m.p. 92° [4689].

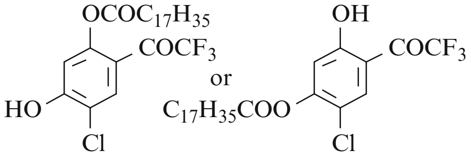
$C_{20}H_{29}F_3O_3$ mol.wt. 374.44

Synthesis

- Preparation by reaction of trifluoroacetic anhydride on 4-dodecylresorcinol with aluminium chloride in ethylene dichloride at r.t. (84%) [4689].

1-[5-Chloro-2 (or 4)-hydroxy-4 (or 2)-(octadecanoyloxy)phenyl]-2,2,2-trifluoroethanone

[65233-65-0]



(I) **(II)**

$C_{26}H_{38}ClF_3O_4$ mol.wt. 507.03

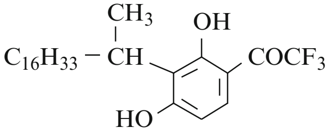
Synthesis

- Preparation by reaction of stearoyl chloride on 5-chloro-2,4-dihydroxy-α,α,α-trifluoroacetophenone with pyridine in benzene at r.t. (40%) [4689].

m.p. 51° [4689].

N.B.: The 1-[5-Chloro-2-hydroxy-4-(octadecanoyloxy)phenyl]-2,2,2-trifluoroethanone (**II**) is the most likely formula.

1-[2,4-Dihydroxy-3-(1-methylheptadecyl)phenyl]-2,2,2-trifluoroethanone



$C_{26}H_{41}F_3O_3$ mol.wt. 458.60

Synthesis

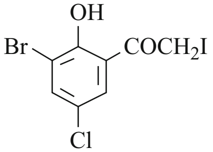
- Preparation by reaction of 1-octadecene on 2,4-dihydroxy-α,α,α-trifluoroacetophenone with phosphorous oxychloride and phosphorous pentoxide at 50° [4689].

m.p. 98° [4689].

11.4 Compounds Derived from Iodoacetic Acids

11.4.1 From Monoiodoacetic Acid

1-(3-Bromo-5-chloro-2-hydroxyphenyl)-2-iodoethanone

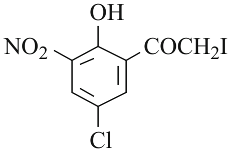


$C_8H_5BrClIO_2$ mol.wt. 375.39

Synthesis

- Obtained by reaction of iodine monochloride with 3-bromo-5-chloro-2-hydroxyacetophenone in boiling acetic acid [4706].

1-(5-Chloro-2-hydroxy-3-nitrophenyl)-2-iodoethanone



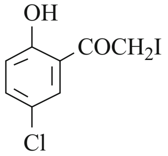
$C_8H_5ClINO_4$ mol.wt. 309.49

Synthesis

- Obtained by reaction of iodine monochloride with 5-chloro-2-hydroxy-3-nitroacetophenone in boiling acetic acid [4706].

1-(5-Chloro-2-hydroxyphenyl)-2-iodoethanone

[438625-16-2] $C_8H_6ClIO_2$ mol.wt. 296.49

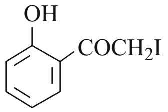


Synthesis

– Obtained by reaction of iodine monochloride with 5-chloro-2-hydroxyacetophenone in boiling acetic acid [4706].

1-(2-Hydroxyphenyl)-2-iodoethanone

[99233-30-4] $C_8H_7IO_2$ mol.wt. 262.05



Syntheses

– Refer to: [4567,4568,4707–4709].

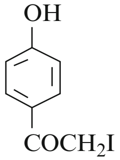
N.B.: The iodination kinetics of o-hydroxy-acetophenone have been investigated at 25° in aqueous buffer solutions.

The foregoing analysis assumes that only one mol of iodine reacts with each mol of o-hydroxyacetophenone, which may be incorrect. The title substance does not appear to have been isolated [4569].

m.p. 65° [4567,4568].

1-(4-Hydroxyphenyl)-2-iodoethanone

[99233-31-5] $C_8H_7IO_2$ mol.wt. 262.05



Syntheses

– Obtained by reaction of iodine with p-hydroxyacetophenone in the presence of 1-fluoro-4-chloromethyl-1,4-diazoniabicyclo[2.2.2]octane bis(tetrafluoroborate)* in methanol for 19 h at r.t. (64%) [4708].

- This reagent is known under the commercial name of Selectfluor™ F-TEDA-BF_4.

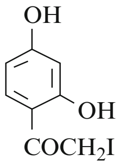
– Also refer to: [4567,4568,4707,4710].

m.p. 130° [4567,4568]; 126–128°8 [4708]; TLC [4708]; Crystal data [4567,4568];

^{1}H NMR [4708], IR [4708], MS [4708].

1-(2,4-Dihydroxyphenyl)-2-iodoethanone

$C_8H_7IO_3$ mol.wt. 278.05



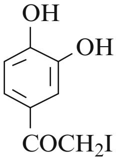
Synthesis

– Preparation by reaction of sodium iodide on 2,4-dihydroxy-α-chloroacetophenone in acetone at r.t. (70%) [4432].

m.p. 141° [4432].

1-(3,4-Dihydroxyphenyl)-2-iodoethanone

[105174-59-2] $C_8H_7IO_3$ mol.wt. 278.05



Syntheses

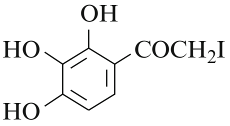
- Preparation by reaction of sodium iodide on 3,4-dihydroxy-α-chloroacetophenone in acetone at r.t. (34%) [4446].
- Also refer to: [4711].

no m.p.: This compound progressively decomposed from 140° and gave a black residue at 162–163° [4446];

^{1}H NMR [4446], IR [4446], UV [4446], MS [4446].

2-Iodo-1-(2,3,4-trihydroxyphenyl)ethanone

[105174-62-7] $C_8H_7IO_4$ mol.wt. 294.05



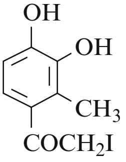
Synthesis

- Preparation by reaction of sodium iodide on 2,3,4-trihydroxy-α-chloroacetophenone in acetone at r.t. (63%) [4446].

m.p. 144–145° [4446]; ^{1}H NMR [4446], IR [4446], UV [4446], MS [4446].

1-(3,4-Dihydroxy-2-methylphenyl)-2-iodoethanone

$C_9H_9IO_3$ mol.wt. 292.07



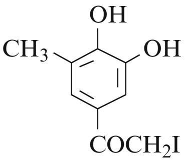
Synthesis

- Preparation by reaction of sodium iodide on 3,4-dihydroxy-2-methyl-α-chloroacetophenone in acetone at r.t. (62%) [4631].

m.p. 171–172° [4631].

1-(3,4-Dihydroxy-5-methylphenyl)-2-iodoethanone

$C_9H_9IO_3$ mol.wt. 292.07



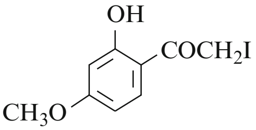
Synthesis

- Preparation by reaction of sodium iodide on 2,4-dihydroxy-5-methyl-α-chloroacetophenone in acetone [4631].

m.p. 177° [4631].

1-(2-Hydroxy-4-methoxyphenyl)-2-iodoethanone

$C_9H_9IO_3$ mol.wt. 292.07

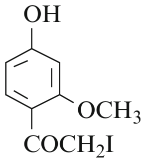


Synthesis

- Preparation by heating 2-hydroxy-4-methoxy-α-bromo-acetophenone with concentrated aqueous potassium iodide solution [4485].

m.p. 102° [4485].

1-(4-Hydroxy-2-methoxyphenyl)-2-iodoethanone



$C_9H_9IO_3$ mol.wt. 292.07

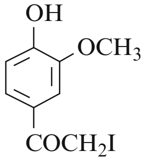
Synthesis

- Preparation by reaction of sodium iodide on 4-hydroxy-2-methoxy-α-chloroacetophenone in acetone at r.t. [4432].

m.p. 128° [4432].

1-(4-Hydroxy-3-methoxyphenyl)-2-iodoethanone

[105174-52-5] $C_9H_9IO_3$ mol.wt. 292.07



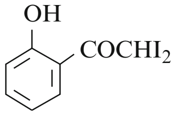
Synthesis

- Preparation by reaction of sodium iodide on 4-hydroxy-3-methoxy-α-bromoacetophenone in acetone at r.t. (52%) [4446].

m.p. 103° [4446]; ^{1}H NMR [4446], IR [4446], UV [4446], MS [4446].

11.4.2 From Diiodoacetic Acid

2,2-Diiodo-1-(2-hydroxyphenyl)ethanone



$C_8H_6I_2O_2$ mol.wt. 387.94

Synthesis not yet described

N.B.: Not obtained.

- The iodination kinetics of o-hydroxyacetophenone have been investigated at 25° in aqueous buffer solutions. The foregoing analysis assumes that only 1 mol of iodine reacts with each mol of o-hydroxyacetophenone, which may be incorrect. Cyclisation probably predominates over the introduction of a second iodine atom into o-hydroxyacetophenone, at least in the early stages of the reaction. There is formation of 3-coumaranone [4569].

11.4.3 From Triiodoacetic Acid

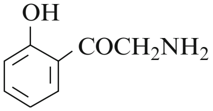
There is no hydroxyketone derived from triiodoacetic acid such as described up to December 2003.

Chapter 12
Compounds Derived from Aminoacetic Acids

12.1 Compounds Derived from Aminoacetic Acid

2-Amino-1-(2-hydroxyphenyl)ethanone

[72481-17-5] $C_8H_9NO_2$ mol.wt. 151.17

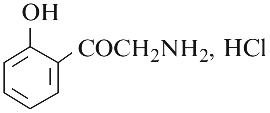


Syntheses

- Obtained by treatment of 3-nitro-4-hydroxycoumarin—m.p. 177° (d)—with refluxing in a mixture of 58% hydriodic acid solution and acetic acid for 15 min. The iodine produced during the reaction was reduced with hypophosphorous acid [4712].
- Also refer to: [4713,4714].

2-Amino-1-(2-hydroxyphenyl)ethanone *(Hydrochloride)*

$C_8H_9NO_2$, HCl mol.wt. 187.63



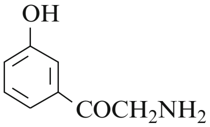
Synthesis

- Preparation by treatment of 3-nitro-4-hydroxycoumarin—m.p. 177° (d)—with refluxing in a mixture of 58% hydriodic acid solution and acetic acid for 15 min. The iodine produced during the reaction was reduced with hypophosphorous acid. Then, recrystallisation of the obtained base from concentrated hydrochloric acid (66%) [4712].

m.p. 229–230° [4712].

2-Amino-1-(3-hydroxyphenyl)ethanone

[90005-54-2] $C_8H_9NO_2$ mol.wt. 151.17



Syntheses

- Obtained by adding ammonia to an aqueous solution of its hydrochloride (63%) [4715].
- Also refer to: [4713,4716–4719].

m.p. 217–220° [4717], 215–235° [4715].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_12, © Springer Science+Business Media B.V. 2011

2-Amino-1-(3-hydroxyphenyl)ethanone *(Hydrochloride)*

[14665-75-9] $C_8H_9NO_2$, HCl mol.wt. 187.63

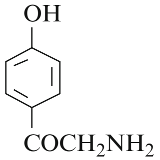
Syntheses

- Preparation by hydrolysis of m-(benzoyloxy)-α-amino-acetophenone hydrochloride (SM) (m.p. 202–205°) [4717], (m.p. 206°) [4715],
  - with refluxing 10% hydrochloric acid (quantitative yield) [4715], for 2 h (80%) [4717];
  - with 37% hydrochloric acid in chlorobenzene at 90° for 3 h (90%) [4718]. SM was obtained by reaction of hexamethylenetetramine with m-(benzoyloxy)-α-bromoacetophenone (m.p. 162°) in ethanol in the presence of 37% hydrochloric acid for 6 h at r.t. (75%) [4718].
- Also obtained by hydrolysis of 3,6-bis(3-hydroxyphenyl)-2,5-dihydropyrazine in aqueous suspension with hydrochloric acid at r.t. [4720].
- Also obtained by reaction of 3-acetoxy-α-bromoacetophenone (m.p. 71–72°) with hexamethylenetetramine in chloroform, followed by acetoxy group elimination in the obtained compound with hydrochloric acid [4721].
- Also obtained by reaction of 3-hydroxy-α-iodoacetophenone with hexamethylenetetramine, followed by transformation of the obtained iodo derivative (m.p. 138–139°) into hydrochloride salt [4722].
- Also refer to: [4716].

m.p. 221–222° [4720–4722], 219–220° [4715], 218–220° (d) [4718], 217–220° [4717].

2-Amino-1-(4-hydroxyphenyl)ethanone

[77369-38-1] $C_8H_9NO_2$ mol.wt. 151.17



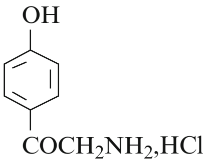
Syntheses

- Obtained by oxidation of the biogenic amine 1-(4-hydroxy-phenyl)-2-aminoethanol at high pH [4723].
- Also obtained by hydrogenation of p-hydroxyisonitroso-acetophenone—so called p-hydroxy-α-(hydroximino)-acetophenone—over Pd/C in acetic acid at a temperature <60° (91%) [4717].
- Also obtained from the corresponding hydrochloride aqueous solution with ammonia [4597].
- Also refer to: [4713,4724–4728].

N.B.: For the acetate [172417-70-8], refer to: [4728,4729]; pK_B [4730].

2-Amino-1-(4-hydroxyphenyl)ethanone *(Hydrochloride)*

[19745-72-3] $C_8H_9NO_2$, HCl mol.wt. 187.63



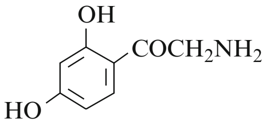
Syntheses

- Preparation by treatment of α-amino-p-hydroxy-acetophenone with hydrogen chloride in DMF (70%) [4714].
- Preparation by hydrogenation of p-hydroxy-α-(hydroximino)acetophenone (SM) over Pd/C in DMF (70%). SM was obtained by adding tert-butyl nitrite to a mixture of p-hydroxyacetophenone, hydrogen chloride and DMF at 40–45° [4731].
- Preparation from α-amino-p-benzoyloxyacetophenone hydrochloride with refluxing 20% hydrochloric acid solution for 7 h (80%) [4724].
- Preparation by condensation of phenol with aminoacetonitrile hydrochloride (Houben–Hoesch reaction) (51%) [4732].
- Also obtained by demethylation of α-amino-p-methoxyacetophenone (m.p. 197°) with 38% hydrochloric acid at 160–170° for 2 h [4597].
- Also refer to: [4733].

m.p. 249–251° [4732], 242° [4597], 241–245° (d) [4724].

2-Amino-1-(2,4-dihydroxyphenyl)ethanone

$C_8H_9NO_3$ mol.wt. 167.16



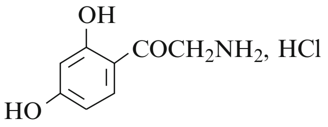
Syntheses

- Obtained from its hydriodide (m.p. 258°) or its hydrochloride (m.p. 280°) by addition of a hot concentrated solution of sodium carbonate [4566].
- Also refer to: [4734,4735].

m.p. 310° (d) [4566].

2-Amino-1-(2,4-dihydroxyphenyl)ethanone *(Hydrochloride)*

$C_8H_9NO_3$, HCl mol.wt. 203.61



Syntheses

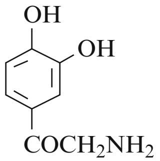
- Obtained by refluxing a mixture of 2,4-dihydroxy-α-[(ethoxycarbonyl)amino] acetophenone (m.p. 156–157°) and hydrochloric acid (1:1) for 2 h [4432].
- Also obtained by addition of concentrated hydrochloric acid to an alcoholic solution of the corresponding hydriodide (SM). SM—m.p. 128° (d)—was prepared from 2,4-dimethoxy-α-phthaliminoacetophenone (m.p. 188°) with boiling concentrated hydriodic acid containing some acetic acid [4566].
- Also obtained by treatment of 3-acetamido-4,7-dihydroxycoumarin (m.p. 268°) with 10% hydrochloric acid for 1 h [4734].
- Also refer to: [4735].

m.p. 280° (d) [4566], 271° [4734], 257° [4432].

One of the reported melting points is obviously wrong.

2-Amino-1-(3,4-dihydroxyphenyl)ethanone *(Arterenone; Noradrenalone; ART)*

[499-61-6] $C_8H_9NO_3$ mol.wt. 167.16



Syntheses

- Preparation by reaction of 35% aqueous ammonia with 3,4-dihydroxy-α-chloroacetophenone in methanol or in ethanol [4736], (71–73%) [4737], (67%) [4738].
- Also obtained by adding ammonia to an aqueous solution of the corresponding hydrochloride [4730,4739], (60–75%) [4597].
- Also obtained by adding sodium carbonate to an aqueous solution of its hydriodide (m.p. 247–248°) (SM). SM was prepared from m,m′,p,p′-tetramethoxy-2,5-diphenylpyrazine by boiling for 2 h with a mixture of acetic acid and concentrated hydriodic acid [4566].
- Preparation by hydrogenolysis of α-dibenzylamino-3,4-dihydroxyacetophenone hydrochloride in water in the presence of Pd/C under hydrogen atmosphere for 5 h. Then, treatment of the concentrated solution with 28% ammonia (85%) [4580].
- Also refer to: [4713,4720,4740,4741].

Isolation from natural sources

- From insect cuticle [4742,4743].
- Also obtained by mild acid hydrolysis of sclerotized cuticles from locusts *(Schistocerca gregaria)* and beetles *(Pachynoda sinuata)* [4744].
- From acid hydrolysates of insect sclerotized cuticle in refluxing 1 N formic acid for 1 h or in boiling methanolic hydrochloric acid. The cuticle used was obtained from the desert locust *Schistocerca gregaria* [4745].
- Also obtained by hydrolysis of 2-(3′,4′-dihydroxyphenyl)-3-acetylamino-6 (or 7)-(N-acetyl-2″-aminoethyl)-2,3-dihydro-1,4-benzodioxin (SM) with 6 N hydrochloric acid at 110° for 3 h. SM was formed by incubation of N-acetyldopamine with locust cuticle [4746].
- ART was the major identified catechol recovered from strong acid hydrolysates of tanning pharate pupae cuticle from *Manduca sexta* [4747].

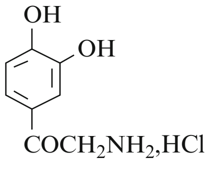
m.p. 300° (d) [4738], 235° (d) [4566,4580], >200° (d) (not melted) [4597,4739];

One note a very large dispersion of the various melting points.

^1H NMR [4746], UV [4744,4745], MS [4744,4748]; HPLC [4747]; pK_B [4730]; column chromatography [4744]; TLC [4744].

2-Amino-1-(3,4-dihydroxyphenyl)ethanone *(Hydrochloride)*

[5090-29-9] $C_8H_9NO_3$, HCl mol.wt. 203.61



Syntheses

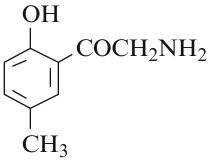
- Preparation by demethylation of α-amino-3,4-dimethoxy-acetophenone on heating with 37% hydrochloric acid for 2.5 h at 160–165° under carbon dioxide (85%) [4739].

- Preparation by dissolving the corresponding base in a mixture of concentrated hydrochloric acid/methanol and allowing to stand several hours at −10° (82%) [4736].
- Also obtained by hydrogenation of 3,4-dihydroxy-α-azidoacetophenone (m.p. 132°) in an ethanol and concentrated hydrochloric acid solution under hydrogen in the presence of 4% Pd/C for 7 h (65%) [4715].
- Also obtained from the addition compound (SM) of 3,4-diacetoxy-α-chloroacetophenone and hexamethylene tetramine in chloroform at r.t. for 24 h (40%). SM in ethanolic solution was treated with 38% hydrochloric acid at r.t. for 3 days [4597].
- Also obtained from hydrolysis of 3,6-bis(3,4-dihydroxyphenyl)-2,5-dihydropyrazine –m.p. 250° (d)—in aqueous suspension with hydrochloric acid at r.t. [4720].
- Also refer to: [4737].

m.p. 270° [4720], 260° [4738], 259° [4730], 256° (d) [4715,4739], 255° [4736], 252° [4597].

One note a very large dispersion of the various melting points.

2-Amino-1-(2-hydroxy-5-methylphenyl)ethanone

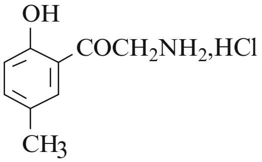


$C_9H_{11}NO_2$ mol.wt. 165.19

Synthesis

- Obtained by hydrogenation of the complex formed by addition of 2-(benzyloxy)-5-methyl-α-bromoacetophenone and hexamethylenetetramine in ethanol in the presence of Pd/C [4749].

2-Amino-1-(2-hydroxy-5-methylphenyl)ethanone *(Hydrochloride)*



$C_9H_{11}NO_2$, HCl mol.wt. 201.66

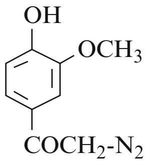
Synthesis

- Obtained by hydrogenolysis of 2-(benzyloxy)-5-methyl-α-aminoacetophenone hydrochloride (m.p. 191–192°) (SM) with hydrogen in the presence of Pd/C in 95% ethanol.
- SM was prepared by reaction of 2-(benzyloxy)-5-methyl-α-bromoacetophenonewith hexamethylene-tetramine, followed by treatment with ethanolic hydrogen chloride [4749].

m.p. 222–225° (d) [4749].

12.2 Compounds Derived from Substituted Aminoacetic Acids

2-Diazo-1-(4-hydroxy-3-methoxyphenyl)ethanone

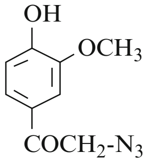


$C_9H_9N_2O_3$ mol.wt. 193.28

Synthesis

- Preparation by reaction of potassium hydroxide with 4-acetoxy-3-methoxy-α-diazoacetophenone (m.p. 92–93°) in methanol at 20° for 15 h (quantitative yield) [4750].
- amorphous solid [4750].

2-Azido-1-(4-hydroxy-3-methoxyphenyl)ethanone



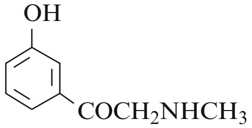
$C_9H_9N_3O_3$ mol.wt. 207.19

Synthesis

- Obtained by reaction of 4-hydroxy-3-methoxy-α-chloro-acetophenone with an alkali metal azide in dilute alcohols (C_1-C_5) [4751].

1-(3-Hydroxyphenyl)-2-(methylamino)ethanone *(Phenylephrone)*

[52093-42-2]



$C_9H_{11}NO_2$ mol.wt. 165.19

Syntheses

- Preparation by reductive condensation of m-hydroxyphenyl-glyoxal with methylamine in ethanol under saturated hydrogen atmosphere in the presence of Raney nickel at 45° (55%) [4752].
- Also obtained by action of potassium N-methyl-p-toluene sulfonamide with m-acetoxy-α-bromoacetophenone in acetone during some hours. Then, the resulting intermediate compound (m.p. 120–121°) was treated with boiling 55% aqueous hydriodic acid for 1 h [4753–4755].
- Also obtained by reaction of methylamine with α-bromo-m-benzoyloxyacetophenone in isopropanol, and subsequent treatment with aqueous hydrochloric acid [4756].
- Also obtained by reaction of methylamine with α-bromo-m-hydroxyacetophenone in dilute ethanol [4757,4758].
- Also refer to: [4713,4719,4759,4760].

m.p. 135° [4753–4755], 128° [4752].

1-(3-Hydroxyphenyl)-2-(methylamino)ethanone *(Hydrochloride)*

[94240-17-2]

$C_9H_{11}NO_2$, HCl mol.wt. 201.65

Syntheses

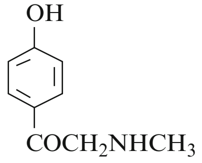
- Preparation by conversion of the base with 35% ethanolic hydrogen chloride [4752].

- Also obtained by hydrogenolysis of 2-(benzyl-methyl-amino)-1-(3-hydroxyphenyl) ethanone hydrochloride with hydrogen in the presence of Pd-black in ethanol [4759].
- Also refer to: [4753,4761].

m.p. 238° [4757,4758], 234° [4752–4755]; IR [4759].

1-(4-Hydroxyphenyl)-2-(methylamino)ethanone

[21213-89-8] $C_9H_{11}NO_2$ mol.wt. 165.19



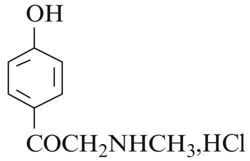
Syntheses

- Preparation by reductive condensation of p-hydroxy-phenyl-glyoxal with methylamine in ethanol under saturated hydrogen atmosphere in the presence of Raney nickel at 45° [4752].
- Also obtained by reductive condensation of p-hydroxy-phenylglyoxal potassium bisulfite ($C_8H_7O_6SK$, preparation given) with methylamine in dilute ethanol under saturated hydrogen atmosphere and cooling with ice (71%) [4752].
- Also obtained by reduction of potassium 2-(4-hydroxyphenyl)-2-oxo-1-methyl-aminoethane sulfonate ($C_9H_{10}NO_5SK$, preparation given) in dilute ethanol with hydrogen in the presence of Raney nickel (65%) [4752].
- Also obtained by reaction of potassium N-methyl-p-toluenesulfonamide with p-acetoxy-α-bromoacetophenone in acetone during some hours. Then, the resulting intermediate compound was treated with boiling 55% aqueous hydriodic acid for 1 h [4753].
- Also obtained by degradation of p-toluenesulfonamide (prepared from α-methylamino-p-methoxy-acetophenone) on heating with 37% hydrochloric acid for 2 h at 150° under carbon dioxide [4739].
- Also obtained by treatment of p-hydroxy-α-bromoacetophenone in ethanol with a 40% methylamine solution, first in an ice bath, then at r.t. overnight [4762].
- Also obtained by reaction of methylaminoacetonitrile with phenol (Houben–Hoesch reaction) (75%) [4732].
- Also refer to: [4713,4724,4761].

m.p. 148° [4753], 147–148° [4762], 147° [4739,4752], 142–144° (d) [4732]; pK_B [4730].

1-(4-Hydroxyphenyl)-2-(methylamino)ethanone *(Hydrochloride)*

[67828-68-6] $C_9H_{11}NO_2$, HCl mol.wt. 201.65



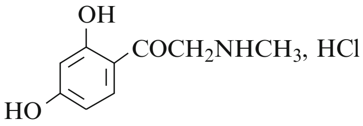
Syntheses

- Obtained by reaction of hydrochloric acid with the corresponding base [4739], (83%) [4732], (50%) [4724].
- Also obtained by treatment of its oxalate (m.p. 166°) with ethanolic hydrogen chloride (72%) [4752].

- Also obtained by reaction of methylaminoacetonitrile hydrochloride with phenol (Hoesch reaction) (88%) [4763].
- Also refer to: [4753,4764–4766].

m.p. 261–263° (d) (pure) [4724], 244–246° [4763], 242–244° (d) [4732], 242° (d) [4739], 241–243° (d) [4724], 239–240° [4762], 238–240° [4753], 238–239° [4752].

1-(2,4-Dihydroxyphenyl)-2-(methylamino)ethanone *(Hydrochloride)*



$C_9H_{11}NO_3$, HCl mol.wt. 217.65

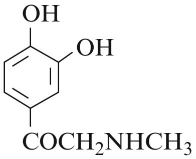
Synthesis

- Preparation by successively adding methylamino-acetonitrile hydrochloride and resorcinol to a solution of aluminium chloride in nitrobenzene, then bubbling hydrogen chloride for 6–8 h through the reaction mixture at 20–30° (73%) [4763].

m.p. 265–267° [4763].

1-(3,4-Dihydroxyphenyl)-2-(methylamino)ethanone *(Adrenalone)*

[99-45-6] $C_9H_{11}NO_3$ mol.wt. 181.19



Syntheses

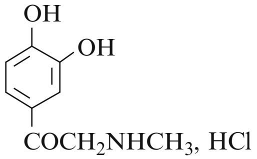
- Preparation by reaction of excess 33% aqueous methylamine with 3,4-dihydroxy-α-chloroacetophenone [4767].
- Preparation by adding an aqueous solution of 40% methyl-amine to an ethanolic solution of 3,4-dihydroxy-α-chloro-acetophenone. Then, adding of ammonia to a solution of recrystallized hydrochloride so formed, (71–73%) [4737], (62%) [4738].
- Preparation by reductive condensation of 3,4-dihydroxyphenylglyoxal with methylamine in ethanol under hydrogen atmosphere in the presence of 14% Pd/C [4752].
- Also obtained by degradation of p-toluenesulfonamide, prepared from 3,4-dimethoxy-α-methyl-aminoacetophenone, with refluxing 37% hydrochloric acid (150–160°) for 2 h under carbon dioxide [4739].
- Also obtained by treatment of 3,4-diacetoxy-α-chloroacetophenone (m.p. 110°) with 30% methylamine solution (good yield, not specified) [4768].
- Also refer to: [4713,4719,4740,4769–4774].

m.p. 232° [4767], 230° [4738], 229° (d) [4739], 215° (d) [4775]; UV [4776,4777]; pK_B [4730,4778];

micellar liquid chromatography [4779–4781]; electrophoresis [4782].

1-(3,4-Dihydroxyphenyl)-2-(methylamino)ethanone *(Hydrochloride) (Stryphnon)*

[62-13-5] $C_9H_{11}NO_3$, HCl mol.wt. 217.65



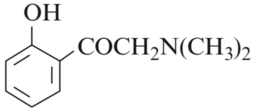
Syntheses

- Obtained by action of hydrochloric acid on 3,4-dihydroxy-α-methylaminoacetophenone (65%) [4737], in methanol [4738,4739].
- Also obtained (poor yield) from the base by saturation of its aqueous solution with hydrogen chloride (7%) [4752].
- Also refer to: [4761,4765–4767].

m.p. 248° [4730], 241° (d) [4739,4752], 240° (d) [4738], 237–243° (d) [4775]; UV [4773].

2-(Dimethylamino)-1-(2-hydroxyphenyl)ethanone

$C_{10}H_{13}NO_2$ mol.wt. 179.22

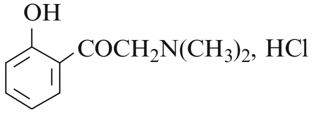


Synthesis

- Obtained by adding a solution of dimethylamine (2 mol) in ethyl ether to a cold solution of o-hydroxy-α-chloro-acetophenone (m.p. 71–71°5) (1 mol) and sodium iodide (1 mol) in acetone. The mixture was then allowed to stand for 14 h at 0° [4573].

2-(Dimethylamino)-1-(2-hydroxyphenyl)ethanone *(Hydrochloride)*

$C_{10}H_{13}NO_2$, HCl mol.wt. 215.69



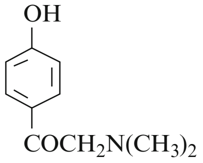
Synthesis

- Obtained by adding of ethanolic hydrochloric acid to a solution of the corresponding base in acetone (61%) [4573].

m.p. 105–107° [4573].

2-(Dimethylamino)-1-(4-hydroxyphenyl)ethanone

$C_{10}H_{13}NO_2$ mol.wt. 179.22



Syntheses

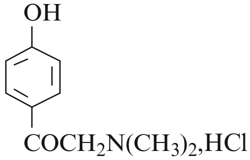
- Obtained by adding a solution of dimethylamine (2 mol) in ethyl ether to a cold solution of p-hydroxy-α-chloro-acetophenone (m.p. 151–152°) (1 mol) and sodium iodide (1 mol) in acetone. The mixture was then allowed to stand for 14 h at 0° [4573].
- Also obtained by hydrolysis of 1-[4-(benzoyloxy)phenyl]-2-dimethylaminoethanone with aqueous hydrochloric acid [4783].

- Also obtained by reaction of dimethylamine with p-(benzoyloxy)-α-bromoacetophenone in isopropanol [4724].
- Also refer to: [4566].

m.p. 142° [4566].

2-(Dimethylamino)-1-(4-hydroxyphenyl)ethanone *(Hydrochloride)*

[2970-79-8] $C_{10}H_{13}NO_2$, HCl mol.wt. 215.69



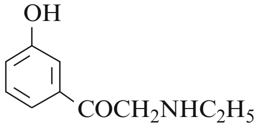
Syntheses

- Obtained by reaction of dimethylamine with p-acetoxy-α-bromoacetophenone in benzene, followed by treatment with dilute hydrochloric acid [4784].
- Also obtained by reaction of dimethylamine with p-benzoyl-oxy-α-bromoacetophenone in isopropanol [4724] or in benzene [4783] and subsequent treatment with hydrochloric acid (88%) [4783], (47%) [4724].
- Also obtained by adding ethanolic hydrochloric acid to a solution of the corresponding base in acetone (43%) [4573].
- Also refer to: [4785].

m.p. 242–243° [4573], 235° [4784], 234–237° [4783], 233–235° [4724].

2-(Ethylamino)-1-(3-hydroxyphenyl)ethanone

[22510-12-9] $C_{10}H_{13}NO_2$ mol.wt. 179.22



Syntheses

- Preparation by reductive condensation of 3-hydroxyphenyl-glyoxal with ethylamine in ethanol under saturated hydrogen atmosphere in the presence of Raney nickel at 45° (49%) [4752].
- Also obtained by reaction of ethylamine with 1-(3-acetoxy-phenyl)-2-bromoethanone in aqueous isopropanol [4786].

m.p. 203–205° [4752].

2-(Ethylamino)-1-(3-hydroxyphenyl)ethanone *(Hydrochloride)*

[22510-04-9] $C_{10}H_{13}NO_2$, HCl mol.wt. 215.69

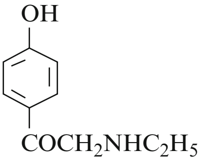
Syntheses

- Preparation by crystallization of the base in 2 N hydrochloric acid [4752].
- Also obtained by reaction of 40% ethylamine solution with m-acetoxy-α-bromoacetophenone in isopropanol, first at 0°, then at 40° for 10 min, followed by treatment with hydrochloric acid [4786].

m.p. 221–222° [4752], 212–215° (d) [4786].

2-(Ethylamino)-1-(4-hydroxyphenyl)ethanone

[99075-26-0] $C_{10}H_{13}NO_2$ mol.wt. 179.22



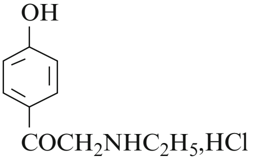
Syntheses

- Preparation by reductive condensation of 4-hydroxy-phenyl-glyoxal with ethylamine in ethanol under saturated hydrogen atmosphere in the presence of Raney nickel at 45° [4752].
- Also obtained by reductive condensation of 4-hydroxy-phenylglyoxal potassium bisulfite ($C_8H_7O_6SK$, preparation given) with ethylamine [4752].
- Also obtained by reaction of ethylamine with 1-[4-(benzoyloxy)phenyl]-2-bromoethanone in isopropanol [4724].

$pK_B = 6.23$ [4730].

2-(Ethylamino)-1-(4-hydroxyphenyl)ethanone *(Hydrochloride)*

[74730-79-3] $C_{10}H_{13}NO_2$, HCl mol.wt. 215.69



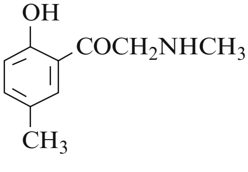
Syntheses

- Preparation by treatment of the oxalate with 28% ethanolic hydrogen chloride (65%) [4752].
- Also obtained by reaction of α-bromo-p-benzoyloxy-acetophenone with ethylamine in isopropanol and subsequent treatment with hydrochloric acid (51%) [4724].

m.p. 228–231° (d) [4724], 221° [4752].

1-(2-Hydroxy-5-methylphenyl)-2-(methylamino)ethanone

$C_{10}H_{13}NO_2$ mol.wt. 179.22

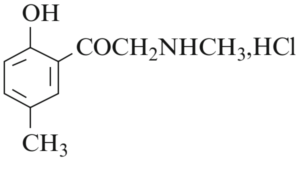


Synthesis

- Preparation by hydrogenolysis of 2-(benzyl-methylamino)-1-(2-hydroxy-5-methylphenyl) ethanone with hydrogen in the presence of Pd/C as catalyst [4749].

1-(2-Hydroxy-5-methylphenyl)-2-(methylamino)ethanone *(Hydrochloride)*

$C_{10}H_{13}NO_2$, HCl mol.wt. 215.69

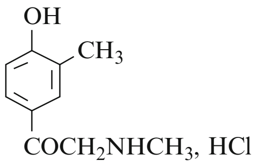


Synthesis

- Obtained by hydrogenolysis of 2-(benzyloxy)-5-methyl-N-benzylmethylaminoacetophenone hydrochloride or of 2-hydroxy-5-methyl-N-benzylmethylaminoacetophenone hydrochloride with hydrogen in the presence of Pd/C in 95% ethanol [4749].

m.p. 204–206° [4749].

1-(4-Hydroxy-3-methylphenyl)-2-(methylamino)ethanone *(Hydrochloride)*



$C_{10}H_{13}NO_2$, HCl mol.wt. 215.69

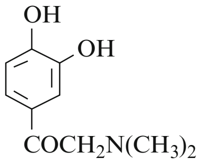
Synthesis

- Preparation by successively adding methylaminoacetonitrile hydrochloride and o-cresol to a solution of aluminium chloride in nitrobenzene, then bubbling hydrogen chloride for 6–8 h through the reaction mixture at 20–30° (90%) [4763].

m.p. 237–238° [4763].

1-(3,4-Dihydroxyphenyl)-2-(dimethylamino)ethanone

[150-10-7]



$C_{10}H_{13}NO_3$ mol.wt. 195.22

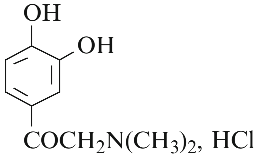
Syntheses

- Preparation by reaction of 3,4-dihydroxy-α-chloroacetophenone with dimethylamine [4787,4788], in ethanol at 40° for 75 min [4789,4790] or at 60° for 5 h [4791].
- Also obtained by action of sodium ethoxide with N-methyl-adrenalone hydrochloride in boiling ethanol (72–74%) [4737].
- Also obtained by demethylation of 2-dimethylamino-1-(3-hydroxy-4-methoxyphenyl)ethanone with concentrated aqueous hydrochloric acid at 130° [4792].
- Also obtained by reaction of dimethylamine with α-chloro-3-acetoxy-4-hydroxyacetophenone in aqueous ethanol [4793].

m.p. >130° (d) [4737].

1-(3,4-Dihydroxyphenyl)-2-(dimethylamino)ethanone *(Hydrochloride)*

[16899-83-5]



$C_{10}H_{13}NO_3$, HCl mol.wt. 231.69

Syntheses

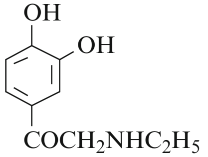
- Preparation by reaction of the corresponding base (SM) with hydrogen chloride in ethanol, [4787], (66%) [4791], (62%) [4771], (54%) [4792], (50%) [4789,4790]. SM was obtained by reaction of 3,4-dihydroxy-α-chloroacetophenone with concentrated aqueous dimethylamine.
- Direct preparation by reaction of 3,4-dihydroxy-α-chloro-acetophenone with dimethylamine in methanol for 20–30 min at <5° (73–76%) [4737] or in absolute ethanol for 2 h at 60° (45%) [4788].
- Also obtained by treatment of 3,4-dimethoxy-α-dimethylaminoacetophenone (SM) with concentrated hydrochloric acid for 2.5 h at 150–160° in a sealed tube (41%) [4794] or for 2 h at 130° (25%) [4792]. SM was prepared by reaction of 3,4-dimethoxy-α-chloroacetophenone with dimethylamine in benzene at r.t. overnight (91%, b.p.$_6$ 155–157°) [4794].

- Also obtained by treatment of 3-hydroxy-4-methoxy-α-dimethylaminoacetophenone with concentrated hydrochloric acid for 2 h at 130°, (65%) [4792].
- Also refer to: [4761].

m.p. 236–237° (d) [4789,4790], 234–236° (d) [4737,4771], 232° (d) [4794], 231–232° (d) [4792], 225–227° (d) [4787,4788], 213–214° [4791].

One note a very large dispersion of the various melting points. ^1H NMR [4788], IR [4788].

1-(3,4-Dihydroxyphenyl)-2-(ethylamino)ethanone



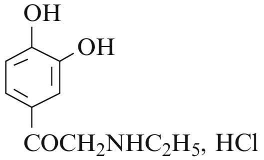
$C_{10}H_{13}NO_3$ mol.wt. 195.22

Syntheses

- Obtained by adding an aqueous solution of 40% ethylamine to an ethanolic solution of α-chloro-3,4-dihydroxyacetophenone, then adding ammonia in a solution of the recrytallized hydrochloride so formed (50%) [4738].
- Also refer to: [4740].

m.p. 185° [4738]; pK_B [4730].

1-(3,4-Dihydroxyphenyl)-2-(ethylamino)ethanone *(Hydrochloride)*



$C_{10}H_{13}NO_3$, HCl mol.wt. 231.69

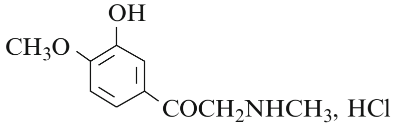
Syntheses

- Obtained by condensation of 3,4-dihydroxy-α-chloro-acetophenone with ethylamine in ethanol or isopropanol at 60–80°. The amino ketone base which separated was treated with concentrated hydrochloric acid [4795].
- Also obtained by total demethylation of 1-(3,4-dimethoxyphenyl)-2-(ethylamino)ethanone hydrochloride (m.p. 190–192°), (71%) [4773] according to [4763].

m.p. 260° (d) [4738], 255–257° (d) [4773], 240–242° (d) [4795].

One of the reported melting points is obviously wrong.

1-(3-Hydroxy-4-methoxyphenyl)-2-(methylamino)ethanone *(Hydrochloride)*



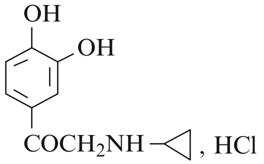
$C_{10}H_{13}NO_3$, HCl mol.wt. 231.69

Synthesis

- Preparation by successively adding methylamino-acetonitrile hydrochloride and guaiacol to a solution of aluminium chloride in nitrobenzene, then bubbling hydrogen chloride for 6–8 h through the reaction mixture at 20–30° (25%) [4763].

m.p. 230–230°5 [4763].

2-(Cyclopropylamino)-1-(3,4-dihydroxyphenyl)ethanone *(Hydrochloride)*



$C_{11}H_{13}NO_3$, HCl mol.wt. 243.69

Synthesis

- Obtained (poor yield) by treatment of cyclopropylamine salt of 4-chloroacetylcatechol (m.p. 95–97°) in refluxing isopropanol under nitrogen for 3 h. The formed free base in methanol was treated with ethanolic hydrochloric acid [4796].

m.p. 200–204° [4796].

1-(3-Hydroxyphenyl)-2-[(1-methylethyl)amino]ethanone

$C_{11}H_{15}NO_2$ mol.wt. 193.25

Syntheses

- Preparation by reductive condensation of 3-hydroxyphenyl-glyoxal with isopropylamine in ethanol under saturated hydrogen atmosphere in the presence of Raney nickel at 45° [4752].
- Also obtained by reaction of isopropylamine with α-bromo-m-hydroxyacetophenone in ethanol [4797].

1-(3-Hydroxyphenyl)-2-[(1-methylethyl)amino]ethanone *(Hydrochloride)*

[101241-90-1]

$C_{11}H_{15}NO_2$, HCl mol.wt. 229.71

Syntheses

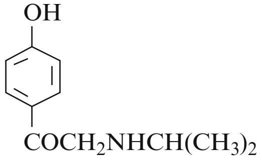
- Preparation by conversion of the oxalate in 28% ethanolic hydrogen chloride (35%) [4752].
- Also obtained by [4797] according to [4724,4795].

m.p. 226–227° [4752], 213–216° [4797].

One of the reported melting points is obviously wrong.

1-(4-Hydroxyphenyl)-2-[(1-methylethyl)amino]ethanone

[99985-57-6]



$C_{11}H_{15}NO_2$ mol.wt. 193.25

Syntheses

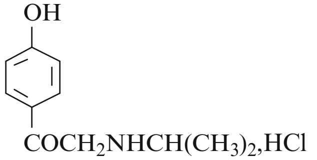
- Preparation by reductive condensation of 4-hydroxyphenyl-glyoxal with isopropylamine in ethanol under saturated hydrogen atmosphere in the presence of Raney nickel at 45° (86%) [4752].
- Also obtained by reductive condensation of 4-hydroxy-phenylglyoxal potassium bisulfite ($C_8H_7O_6SK$, preparation given) with isopropylamine [4752].

- Also obtained by reaction of isopropylaminoacetonitrile with benzyl phenyl ether (m.p. 39–41°) [4763] or with phenol [4732] in the presence of aluminium chloride and hydrogen chloride in nitrobenzene.
- Also obtained by reaction of isopropylamine with α-bromo-4-benzoyloxyacetophenone in isopropanol [4724].
- Also refer to: [4798].

m.p. 120–121° [4752]; pK_B [4730,4799].

1-(4-Hydroxyphenyl)-2-[(1-methylethyl)amino]ethanone ***(Hydrochloride)***

[69716-74-1] $C_{11}H_{15}NO_2$, HCl mol.wt. 229.71



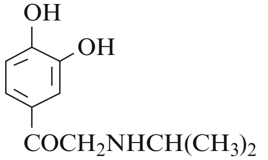
Syntheses

- Preparation from the base with aqueous hydrochloric acid (84%) [4752].
- Also obtained by reaction of α-bromo-p-benzoyloxy-acetophenone with isopropylamine in isopropanol between 20° and 30° for 2 h, then treatment of the formed base with refluxing 15% hydrochloric acid solution (64%) [4724].
- Also obtained by reaction of isopropylaminoacetonitrile hydrochloride (m.p. 166–167°),
  - with phenol (Houben–Hoesch reaction) (61%) [4732], (42%) [4798];
  - with phenyl benzyl ether in the presence of aluminium chloride in nitrobenzene at 20–30°, then bubbling hydrogen chloride for 6 h (39%) [4763].

m.p. 272–273° (d) [4763], 263° (d) [4798], 258–260° (d) [4732], 250–252° [4724], 248–249° [4752]. One note a very large dispersion of the various melting points.

1-(3,4-Dihydroxyphenyl)-2-[(1-methylethyl)amino]ethanone ***(Isoproterenone)***

[121-28-8] $C_{11}H_{15}NO_3$ mol.wt. 209.25



Syntheses

- Obtained by reductive condensation of 3,4-dihydroxy-phenylglyoxal with isopropylamine in ethanol under hydrogen atmosphere in the presence of 14% Pd/C [4752].

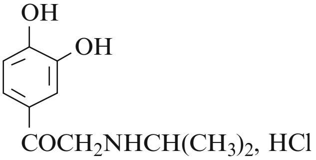
N.B.: In the same manner, the substance can also be obtained from 3,4-bis(benzyloxy)phenylglyoxal [4800].

- Also obtained by reaction of 3,4-dihydroxy-α-chloroacetophenone with excess isopropylamine in refluxing ethanol for 2.5 h (76%) [4801].
- Also obtained from the corresponding sulfate by action of a hot sodium bicarbonate aqueous solution (60°) (80%) [4761].
- Also obtained by treatment of the corresponding hydrochloride in concentrated aqueous solution at 0° with ammonia [4802].

m.p. 173° [4761], 168–169° [4801], 96° [4802]. One of the reported melting points is obviously wrong. pK_B [4730];
^{1}H NMR [4801], IR [4801], UV [4801].

1-(3,4-Dihydroxyphenyl)-2-[(1-methylethyl)amino]ethanone *(Hydrochloride)*

[16899-81-3] $C_{11}H_{15}NO_3$, HCl mol.wt. 245.71



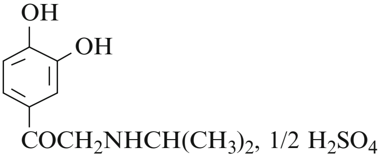
Syntheses

- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with isopropylamine [4803] in isopropanol at 65–70°. The amino ketone which separated was treated with concentrated hydrochloric acid (54%) [4795], (46%) [4804].
- Also obtained by demethylation of 3-methoxy-4-hydroxy-α-isopropylaminoacetophenone hydrochloride with concentrated hydrochloric acid at 140° for 6 h in a sealed tube (73%) [4802].
- Also refer to: [4763,4805–4807].

m.p. 257–259° [4763], 255–257° [4802], 239–242° (d) [4795,4804].
One of the reported melting points is obviously wrong.
^{1}H NMR [4804], ^{13}C NMR [4804], UV [4773].

1-(3,4-Dihydroxyphenyl)-2-[(1-methylethyl)amino]ethanone *(Sulfate)*

[27693-62-5] $C_{11}H_{15}NO_3$, 1/2 H_2SO_4 mol.wt. 258.28



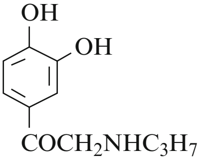
Synthesis

- Preparation by reaction of 5 N ethanolic sulfuric acid with the crude base (58%) [4752].

m.p. 243° [4752].

1-(3,4-Dihydroxyphenyl)-2-(propylamino)ethanone

$C_{11}H_{15}NO_3$ mol.wt. 209.25

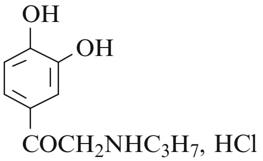


Syntheses

- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with propylamine in ethanol or isopropanol at 60–80° [4795].
- Also obtained by treatment of 1-(3,4-dimethoxyphenyl)-2-(propylamino)ethanone with aqueous hydrobromic acid [4773].

pK = 6.2 [4730].

1-(3,4-Dihydroxyphenyl)-2-(propylamino)ethanone *(Hydrochloride)*



$C_{11}H_{15}NO_3$, HCl mol.wt. 245.71

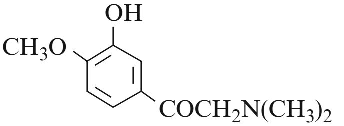
Syntheses

- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with propylamine in ethanol or isopropanol at 60–80°. The amino base which separated was treated with concentrated hydrochloric acid [4795].
- Also obtained by total demethylation of 1-(3,4-dimethoxyphenyl)-2-(propylamino)ethanone hydrochloride (m.p. 193–194°), (82%) [4773] according to [4763].

m.p. 240–241° (d) [4773], 234–236° (d) [4795].

2-(Dimethylamino)-1-(3-hydroxy-4-methoxyphenyl)ethanone

[55761-48-3]

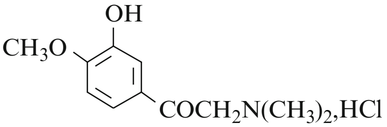


$C_{11}H_{15}NO_3$ mol.wt. 209.25

Synthesis

- Obtained by reaction of dimethylamine with α-chloro-3-hydroxy-4-methoxyacetophenone in benzene [4792].

2-(Dimethylamino)-1-(3-hydroxy-4-methoxyphenyl)ethanone *(Hydrochloride)*



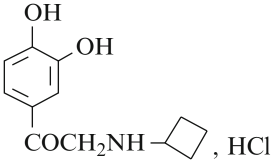
$C_{11}H_{15}NO_3$, HCl mol.wt. 245.71

Synthesis

- Obtained by reaction of α-chloro-3-hydroxy-4-methoxyacetophenone with dimethylamine in benzene, first at r.t. overnight, then at 50–60° for 1 h, followed by treatment of the isolated base with hydrochloric acid in ethyl ether (41%) [4792].

m.p. 220–221° (d) [4792].

2-(Cyclobutylamino)-1-(3,4-dihydroxyphenyl)ethanone *(Hydrochloride)*



$C_{12}H_{15}NO_3$, HCl mol.wt. 257.72

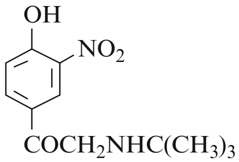
Synthesis

- Obtained by treatment of 4-chloroacetylcatechol cyclobutylamine salt (m.p. 100–104°) in refluxing isopropanol for 3 h (21%) or in a sealed tube at 100° for 1.5 h (28%) [4796].

m.p. 225–228° [4796]; IR [4796], UV [4796].

2-[(1,1-Dimethylethyl)amino]-1-(4-hydroxy-3-nitrophenyl)ethanone

$C_{12}H_{16}N_2O_4$ mol.wt. 252.27

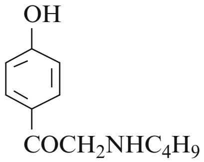


Synthesis

- Obtained by reaction of aqueous nitric acid with α-tert-butylamino-p-hydroxyacetophennone [4808].

2-(Butylamino)-1-(4-hydroxyphenyl)ethanone

[18986-11-3] $C_{12}H_{17}NO_2$ mol.wt. 207.27



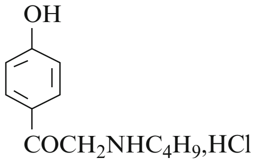
Syntheses

- Preparation by reductive condensation of 4-hydroxyphenyl-glyoxal with n-butylamine in ethanol under saturated hydrogen chloride atmosphere in the presence of Raney nickel at 45° (75%) [4752].
- Also obtained by reductive condensation of 4-hydroxy-phenylglyoxal potassium bisulfite ($C_8H_7O_6SK$, preparation given) with n-butylamine (85%) [4752].
- Also obtained by reaction of n-butylamine with α-bromo-p-benzoyloxyacetophenone in isopropanol [4724].
- Also obtained by reaction of n-butylaminoacetonitrile with phenol in the presence of aluminium chloride and hydrogen chloride in nitrobenzene [4763,4809].
- Also obtained from the corresponding hydrochloride with ammonia [4809].

m.p. 119–120° [4752,4809]; $pK_B = 5.45$ [4730].

2-(Butylamino)-1-(4-hydroxyphenyl)ethanone *(Hydrochloride)*

[28836-20-6] $C_{12}H_{17}NO_2$, HCl mol.wt. 243.73



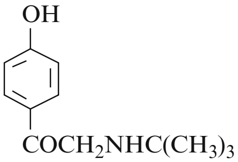
Syntheses

- Preparation from the base with hydrochloric acid (92%) [4752].
- Preparation by reaction of n-butylaminoacetonitrile hydrochloride (m.p. 101–102°5) [4763], (m.p. 95–96°) [4809] with phenol in nitrobenzene in the presence of hydrogen chloride and aluminium chloride as catalyst (Houben–Hoesch reaction), (78%) [4763], (66%) [4809].
- Also obtained by reaction of α-bromo-p-benzoyloxyacetophenone with n-butylamine in isopropanol and subsequent treatment with hydrochloric acid (44%) [4724].

m.p. 231° [4752,4809], 228–230° [4763], 228–229° [4724].

2-[(1,1-Dimethylethyl)amino]-1-(4-hydroxyphenyl)ethanone

[60853-18-1] $C_{12}H_{17}NO_2$ mol.wt. 207.27



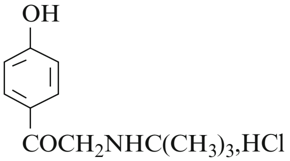
Syntheses

- Obtained by reaction of tert-butylamine with α-bromo-p-benzoyloxyacetophenone in isopropanol [4724].
- Also obtained by reaction of tert-butylaminoacetonitrile hydrochloride with phenol in the presence of aluminium chloride and hydrogen chloride in nitrobenzene (modified Hoesch reaction) [4763,4798].
- Also refer to: [4810].

$pK_B = 6.1$ [4730].

2-[(1,1-Dimethylethyl)amino]-1-(4-hydroxyphenyl)ethanone *(Hydrochloride)*

[41489-87-6] $C_{12}H_{17}NO_2$, HCl mol.wt. 243.73



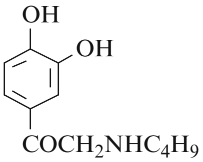
Syntheses

- Preparation by successively adding tert-butylamino-acetonitrile hydrochloride and phenol to a solution of aluminium chloride in nitrobenzene, then bubbling hydrogen chloride for 6–8 h through the reaction mixture at 20–30° (75%) [4763], (63%) [4798].
- Also obtained by reaction of α-bromo-p-benzoyloxyacetophenone with tert-butylamine in isopropanol and subsequent treatment with hydrochloric acid to complete hydrolysis (25%) [4724].
- Also refer to: [4810].

m.p. 268–270° (d) [4763,4810], 254–257° (d) [4724], 253–255° [4798].
One of the reported melting points is obviously wrong.

2-(Butylamino)-1-(3,4-dihydroxyphenyl)ethanone

[33406-44-9] $C_{12}H_{17}NO_3$ mol.wt. 223.27



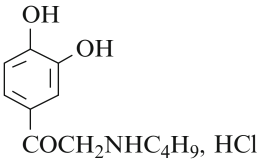
Synthesis

- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with butylamine in ethanol or isopropanol at 60–80° [4795].

pK_B [4730].

2-(Butylamino)-1-(3,4-dihydroxyphenyl)ethanone *(Hydrochloride)*

$C_{12}H_{17}NO_3$, HCl mol.wt. 259.73



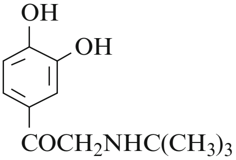
Synthesis

- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with butylamine in ethanol or isopropanol at 60–80°. The amino base which separated was treated with concentrated hydrochloric acid [4795].

m.p. 206–208° (d) [4795].

1-(3,4-Dihydroxyphenyl)-2-[(1,1-dimethylethyl)amino]ethanone

[105644-17-5] $C_{12}H_{17}NO_3$ mol.wt. 223.27



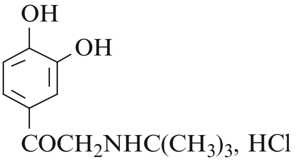
Syntheses

- Preparation by demethylation of 2-tert-butylamino-1-(3,4-dimethoxyphenyl)ethanone hydrochloride with aqueous hydrobromic acid [4763].
- Also obtained by reaction of tert-butylamine with 2-chloro-1-(3,4-dihydroxyphenyl)ethanone in dioxane [4795].
- Also refer to: [4811,4812].

m.p. 199–201° [4811]; pK_B [4730].

1-(3,4-Dihydroxyphenyl)-2-[(1,1-dimethylethyl)amino]ethanone *(Hydrochloride)*

[34715-64-5] $C_{12}H_{17}NO_3$, HCl mol.wt. 259.73



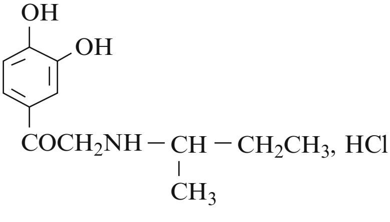
Syntheses

- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with tert-butylamine in dioxane at 60–80°. The amino ketone base which separated was treated with concentrated hydrochloric acid [4795].
- Also refer to: [4806,4807,4813].

m.p. 233–235° (d) [4795].

1-(3,4-Dihydroxyphenyl)-2-[(1-methylpropyl)amino]ethanone *(Hydrochloride)*

$C_{12}H_{17}NO_3$, HCl mol.wt. 259.73



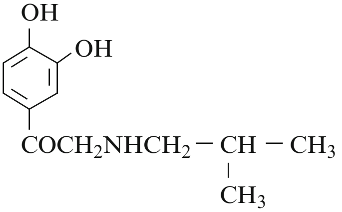
Synthesis

- Obtained by condensation of α-chloro-3,4-di-hydroxyacetophenone with sec-butylamine in ethanol or isopropanol at 60–80°. The amino base which separated was treated with concentrated hydrochloric acid [4795].

m.p. 226–227° [4795].

1-(3,4-Dihydroxyphenyl)-2-[(2-methylpropyl)amino]ethanone

$C_{12}H_{17}NO_3$ mol.wt. 223.27

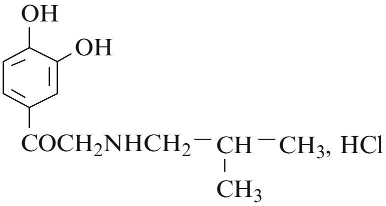


Synthesis

- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with isobutylamine in ethanol or isopropanol at 60–80° [4795].

$pK_B = 6.52$ [4730].

1-(3,4-Dihydroxyphenyl)-2-[(2-methylpropyl)amino]ethanone *(Hydrochloride)*



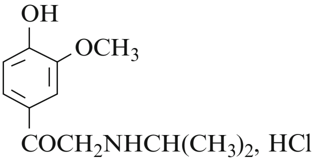
$C_{12}H_{17}NO_3$, HCl mol.wt. 259.73

Synthesis

- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with isobutylamine in ethanol or isopropanol at 60–80°. The amino ketone base which separated was treated with concentrated hydrochloric acid [4795].

m.p. 214–216° [4795].

1-(4-Hydroxy-3-methoxyphenyl)-2-[(1-methylethyl)amino]ethanone *(Hydrochloride)*



$C_{12}H_{17}NO_3$, HCl mol.wt. 259.73

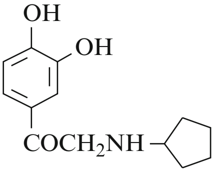
Synthesis

- Preparation by treatment of the corresponding oxalate (SM) with 26% ethanolic hydrogen chloride (61% yield). SM was obtained in two steps. First, gradual addition of a 3-methoxy-4-hydroxyphenylglyoxal potassium bisulfite and isopropylamine solution in dilute ethanol to a suspension of Raney nickel in 84% ethanol maintained at 45° under excess hydrogen. Then, after catalyst elimination, addition of oxalic acid to the obtained solution [4802].

m.p. 236° (d) [4802].

2-(Cyclopentylamino)-1-(3,4-dihydroxyphenyl)ethanone

[16149-16-9] $C_{13}H_{17}NO_3$ mol.wt. 235.28



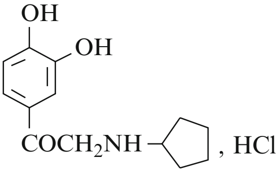
Synthesis

- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with cyclopentylamine in ethanol or isopropanol at 60–80° [4795] or in boiling isopropanol for 30 min (98%) [4796].

hemihydrate: m.p. 182° [4796]; UV [4796].

2-(Cyclopentylamino)-1-(3,4-dihydroxyphenyl)ethanone *(Hydrochloride)*

[16149-17-0] $C_{13}H_{17}NO_3$, HCl mol.wt. 271.74



Syntheses

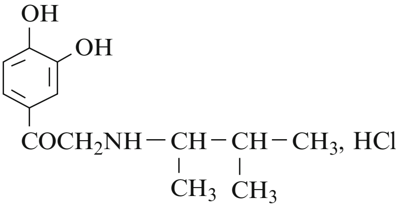
- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with cyclopentylamine in ethanol or isopropanol at 60–80°. The amino ketone base which separated was treated with concentrated hydrochloric acid [4795].

- Obtained by reaction of N-cyclopentylnoradrenalone with hydrochloric acid (44%) [4796].

m.p. 213–214° (d) [4795], 205–207° [4796].

1-(3,4-Dihydroxyphenyl)-2-(1,2-dimethylpropylamino)ethanone *(Hydrochloride)*

$C_{13}H_{19}NO_3$, HCl mol.wt. 273.76



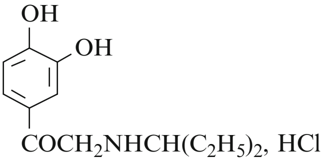
Synthesis

- Obtained by condensation of α-chloro-3,4-di-hydroxyacetophenone with 1,2-dimethylpropylamine in ethanol or isopropanol at 60–80°. The amino ketone base which separated was treated with concentrated hydrochloric acid [4795].

m.p. 231–233° [4795].

1-(3,4-Dihydroxyphenyl)-2-(1-ethylpropylamino)ethanone *(Hydrochloride)*

$C_{13}H_{19}NO_3$, HCl mol.wt. 273.76



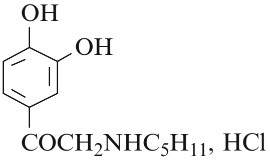
Synthesis

- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with 1-ethylpropylamine in methanol or isopropanol at 60–80°. The amino ketone base which separated was treated with concentrated hydrochloric acid [4795].

m.p. 198–201° [4795].

1-(3,4-Dihydroxyphenyl)-2-(pentylamino)ethanone *(Hydrochloride)*

$C_{13}H_{19}NO_3$, HCl mol.wt. 273.76



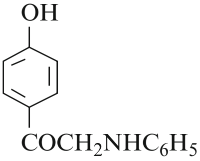
Synthesis

- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with pentylamine in ethanol or isopropanol at 60–80°. The amino ketone base which separated was treated with concentrated hydrochloric acid [4795].

m.p. 201–202° (d) [4795].

1-(4-Hydroxyphenyl)-2-(phenylamino)ethanone

[100866-41-9] $C_{14}H_{13}NO_2$ mol.wt. 227.26



Syntheses

- Preparation by adding aniline (0.1 ml) and rhodium (II) acetate dimer (2 mg) to a suspension of resin **6** (52 mg) in benzene and the mixture stirred at 85° for 2 h. The compound was isolated and purified by preparative TLC (51%) [4814].

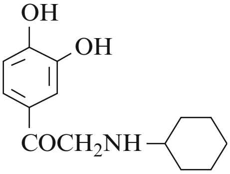
N.B.: Resin **6** (resin-bound α-TMS diazoketone **6**) (preparation given).

- Also refer to: [4815].

^{1}H NMR [4814], ^{13}C NMR [4814], IR [4814], MS [4814].

2-(Cyclohexylamino)-1-(3,4-dihydroxyphenyl)ethanone

[16149-18-1] $C_{14}H_{19}NO_3$ mol.wt. 249.31



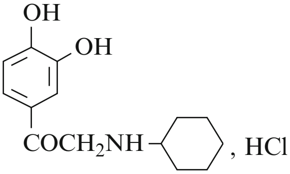
Syntheses

- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with cyclohexylamine in ethanol or isopropanol at 60–80° [4795] or in boiling isopropanol for 30 min [4796].
- Also obtained by reaction of 3,4-diacetoxy-α-iodo-acetophenone with cyclohexylamine in the presence of potassium carbonate in boiling acetone for 4 h (24%) [4796].

hemihydrate [4796]; m.p. 187–188° [4796]; IR [4796], UV [4796].

2-(Cyclohexylamino)-1-(3,4-dihydroxyphenyl)ethanone *(Hydrochloride)*

[16149-19-2] $C_{14}H_{19}NO_3$, HCl mol.wt. 285.77



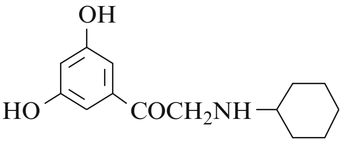
Synthesis

- Obtained by condensation of α-chloro-3,4-dihydroxy-acetophenone with cyclohexylamine in ethanol or isopropanol at 60–80°. The amino ketone base which separated was treated with concentrated hydrochloric acid [4795,4796].

m.p. 256–258° (d) [4795], 242–245° [4796].

2-(Cyclohexylamino)-1-(3,5-dihydroxyphenyl)ethanone

[161040-30-8] $C_{14}H_{19}NO_3$ mol.wt. 249.31

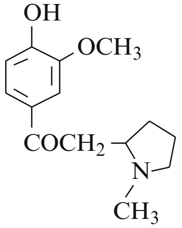


Synthesis

- Obtained by treatment of 3,5-diacetoxy-α-bromo-acetophenone with cyclohexylamine in ethyl acetate and then refluxing with hydrochloric acid [4816].

1-(4-Hydroxy-3-methoxyphenyl)-2-(1-methyl-2-pyrrolidinyl)ethanone (-) *(Phyllostone)*

[126262-24-6] $C_{14}H_{19}NO_3$ mol.wt. 249.31

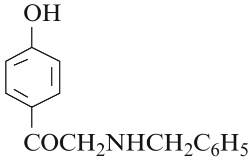


Isolation from natural sources

- From the New Caledonian lauraceous plant *Cryptocarya phyllostemon* [4817].

Gum [4817]; $(\alpha)_D = -5°$ (ethanol);
^1H NMR [4817], IR [4817], UV [4817], MS [4817].

1-(4-Hydroxyphenyl)-2-[(phenylmethyl)amino]ethanone



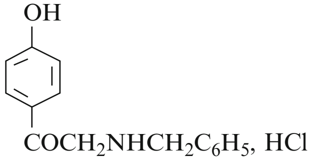
$C_{15}H_{15}NO_2$ mol.wt. 241.29

Synthesis

- Obtained by adding ammonia to an aqueous solution of its hydrochloride [4818].

m.p. 132–133° [4818].

1-(4-Hydroxyphenyl)-2-[(phenylmethyl)amino]ethanone *(Hydrochloride)*



$C_{15}H_{15}NO_2$, HCl mol.wt. 277.75

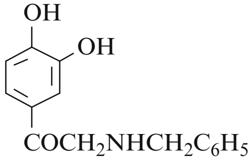
Synthesis

- Obtained by reductive condensation of p-hydroxyphenyl-glyoxal hydrate and benzylamine under hydrogen in the presence of Raney nickel in ethanol at 40°. Then, treatment of the mixture with 6 N ethanolic hydrogen chloride (82%) [4818].

N.B.: The same reaction from p-hydroxyphenylglyoxal-potassium bisulfite at 45° gave a 79% yield [4818].

m.p. 240° [4818].

1-(3,4-Dihydroxyphenyl)-2-[(phenylmethyl)amino]ethanone



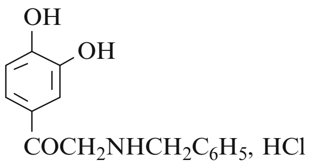
$C_{15}H_{15}NO_3$ mol.wt. 257.29

Syntheses

- Obtained by reaction of α-chloro-3,4-dihydroxy-acetophenone with benzylamine [4740].
- Also obtained by adding ammonia to an aqueous solution of its hydrochloride [4818].

m.p. 147–148° [4818].

1-(3,4-Dihydroxyphenyl)-2-[(phenylmethyl)amino]ethanone *(Hydrochloride)*



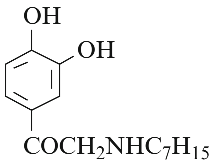
$C_{15}H_{15}NO_3$, HCl mol.wt. 293.75

Synthesis

- Obtained by reductive condensation of 3,4-dihydroxyphenyl-glyoxal and benzylamine under hydrogen in the presence of Raney nickel in ethanol at 45°. Then, treatment of the mixture with ethanolic hydrogen chloride (73%) [4818].

m.p. 220–221° [4818].

1-(3,4-Dihydroxyphenyl)-2-(heptylamino)ethanone



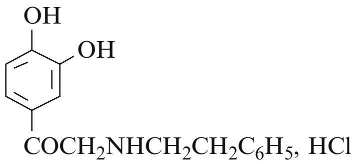
$C_{15}H_{23}NO_3$ mol.wt. 265.35

Synthesis

- Preparation by reaction of excess heptylamine with α-chloro-3,4-diacetoxyacetophenone [4740].

m.p. 125° [4740].

1-(3,4-Dihydroxyphenyl)-2-[2-(phenylethyl)amino]ethanone *(Hydrochloride)*



$C_{16}H_{17}NO_3$, HCl mol.wt. 307.78

Synthesis

- Obtained by total demethylation of 1-(3,4-dimethoxy-phenyl)-2-[(phenylethyl)amino]ethanone hydrochloride (m.p. 219–222°), (79%) [4773] according to [4763].

monohydrate [4773]; m.p. 220–222° (d) [4773].

1-(3-Hydroxy-4-methoxyphenyl)-2-[(phenylmethyl)amino]ethanone *(Hydrochloride)*

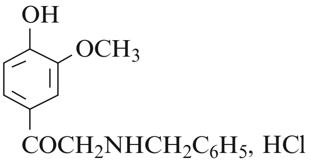
$C_{16}H_{17}NO_3$, HCl mol.wt. 307.78

Synthesis

- Obtained by reductive condensation of 3-hydroxy-4-methoxyphenylglyoxal and benzylamine under hydrogen in the presence of Raney nickel in ethanol at 45° for 45 min. Then, elimination of the catalyst and acidification of the mixture with hydrochloric acid (54%) [4818].

m.p. 226° [4818].

1-(4-Hydroxy-3-methoxyphenyl)-2-[(phenylmethyl)amino]ethanone *(Hydrochloride)*



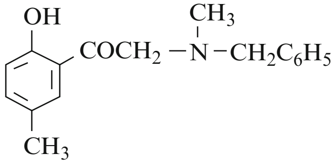
$C_{16}H_{17}NO_3$, HCl mol.wt. 307.78

Synthesis

- Preparation by reductive condensation of 4-hydroxy-3-methoxyphenylglyoxal-potassium-bisulfite and benzylamine under hydrogen in the presence of Raney nickel in dilute ethanol at 45° for 1.75 h. Then, elimination of the catalyst and acidification of the mixture with hydrochloric acid (76%) [4818].

m.p. 230° [4818].

2-(Benzyl-methyl-amino)-1-(2-hydroxy-5-methylphenyl)ethanone

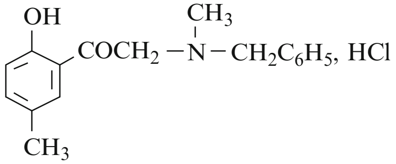


$C_{17}H_{19}NO_2$ mol.wt. 269.34

Synthesis

- Obtained by condensation of 2-hydroxy-5-methyl-α-bromoacetophenone with benzyl methyl amine in ethyl ether at r.t. for 24–72 h [4749].

2-(Benzyl-methyl-amino)-1-(2-hydroxy-5-methylphenyl)ethanone *(Hydrochloride)*



$C_{17}H_{19}NO_2$, HCl mol.wt. 305.80

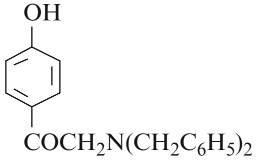
Synthesis

- Obtained by reaction of 2-hydroxy-5-methyl-α-bromoacetophenone with N-benzylmethylamine in ethyl ether at r.t. for 24–72 h, followed by treatment with hydrochloric acid [4749].

m.p. 86°5–187° [4749].

2-[Bis(phenylmethyl)amino]-1-(4-hydroxyphenyl)ethanone

[88693-95-2]

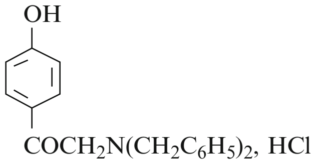


$C_{22}H_{21}NO_2$ mol.wt. 331.41

Synthesis

- Preparation by reaction of p-hydroxyphenacyl chloride (1 mol) with dibenzylamine (2 mol) in refluxing ethanol for 4 h [4580].
  uncrystallizable oil [4580].

2-[Bis(phenylmethyl)amino]-1-(4-hydroxyphenyl)ethanone *(Hydrochloride)*



m. p. 239–241° (d) [4580].

$C_{22}H_{21}NO_2$, HCl mol.wt. 367.87

Synthesis

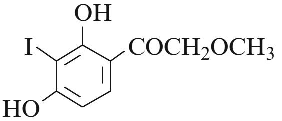
- Preparation by adding a solution of ethanolic hydrogen chloride to a solution of α-dibenzylamino-p-hydroxy-acetophenone in chloroform (77%) [4580].

Chapter 13
Compounds Derived from Alkoxyacetic Acids

13.1 Compounds Derived from Methoxyacetic Acids

1-(2,4-Dihydroxy-3-iodophenyl)-2-methoxyethanone

[72511-78-5] $C_9H_9IO_4$ mol.wt. 308.07



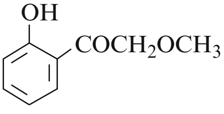
Synthesis

– Obtained by iodination of 2,4-dihydroxy-α-methoxy-acetophenone with iodine and periodic acid in ethanol for 2 h at r.t. (74%) [4819].

m.p. M157–158° [4819].

1-(2-Hydroxyphenyl)-2-methoxyethanone

[138206-45-8] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

– Preparation by hydrogenolysis of 2-(benzyloxy)-α-methoxyacetophenone (SM) in the presence of Pd/C in ethanol for 1 h in hydrogen atmosphere (96%). SM was obtained by treatment of 2-(benzyloxy)phenylmagnesium bromide with methoxyacetonitrile in THF, first in an ice bath, then stirred for 2 h at r.t. (56%, colourless oil) [4395].

– Also obtained by decomposition of 1-(2-acetoxyphenyl)-2-diazoethanone in methanol with copper bronze (54%). The diazoketone (deep red thick oil) was prepared by reaction of diazomethane with 2-acetoxybenzoyl chloride in ethyl ether [4820].

colourless oil [4395], pale yellow liquid [4820]; b.p.$_{0.6–0.8}$ 76–77° [4820];
^{1}H NMR [4395], IR [4395], MS [4395].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
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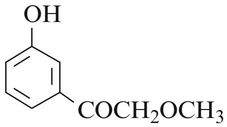
1-(3-Hydroxyphenyl)-2-methoxyethanone

[54794-31-9] $C_9H_{10}O_3$ mol.wt. 166.18



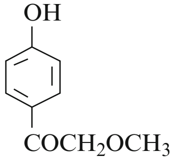
Syntheses

- Refer to: [4821] and [4822] (Polish patent).

N.B.: K salt [4823].

1-(4-Hydroxyphenyl)-2-methoxyethanone

[32136-81-5] $C_9H_{10}O_3$ mol.wt. 166.18



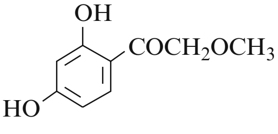
Syntheses

- Obtained by scission of 5-hydroxy-4-(4-hydroxyphenyl) 5*H*-furan-2-one with potassium hydroxide in methanol at 20° for 24 h (85%) [4824].
- Also obtained by catalytic debenzylation of 1-(4-benzyloxyphenyl)-2-methoxyethanone in methanol under hydrogen (five bars) in the presence of 5% Pd/C for 24 h (81%) [4824].
- Also obtained by methoxylation of the trimethylsilyl enol ether of 4-acetoxyacetophenone (SM) according to the procedure [4825], iodosobenzenediacetate replaced iodosobenzene, followed by hydrolysis of the ester complex formed (40%). SM was prepared in two steps from p-hydroxy-acetophenone, namely acetylation, then trimethylsilylation (80%) [4826].
- Also obtained by reaction of 2-chloro-1-(4-hydroxyphenyl)ethanone (m.p. 151°) with methanolic sodium methoxide at r.t. for 24 h (90%) [4827].
- Also refer to: [4828–4833].

m.p. 133–135° [4827], 128–130° [4824];
^{1}H NMR [4824,4827], ^{13}C NMR [4824], IR [4824,4827],
MS [4824,4827].

1-(2,4-Dihydroxyphenyl)-2-methoxyethanone

[57280-75-8] $C_9H_{10}O_4$ mol.wt. 182.18

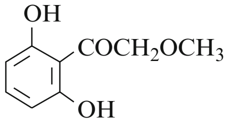


Syntheses

- Obtained by reaction of methoxyacetonitrile with resorcinol and subsequent hydrolysis of the ketimine hydrochloride (m.p. 205–207°) formed (Hoesch reaction) [4834–4837].
- Also obtained by decomposition of 1-(2,4-diacetoxyphenyl)-2-diazoethanone in methanol with copper bronze (43%). The diazoketone (brownish yellow glassy solid) was prepared by reaction of diazomethane with 2,4-diacetoxybenzoyl chloride in ethyl ether [4820].
- Also obtained by alkaline degradation of 7-acetoxy-3,4-dimethoxycoumarin (m.p. 123–124°) with sodium hydroxide or sodium carbonate [4838].
- Also refer to: [4640,4819,4839–4848].

m.p. 138–139° [4836], 136–138° [4820], 136° [4837];
^{1}H NMR [4836], ^{13}C NMR [4836], MS [4836].

1-(2,6-Dihydroxyphenyl)-2-methoxyethanone



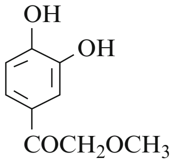
$C_9H_{10}O_4$ mol.wt. 182.18

Synthesis

- Obtained by Fries rearrangement of 4-methylumbelliferone methoxyacetate, followed by alkaline hydrolysis of the resulting 8-(2-methoxyacetyl)-4-methylumbelliferone [4849].

1-(3,4-Dihydroxyphenyl)-2-methoxyethanone

[64349-40-2] $C_9H_{10}O_4$ mol.wt. 182.18

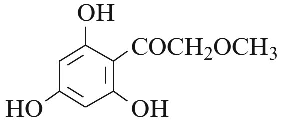


Syntheses

- Obtained by methoxylation of the trimethylsilyl enol ether of 3,4-diacetoxyacetophenone (SM) according to the procedure [4825], iodosobenzenediacetate replaced iodosobenzene, followed by hydrolysis of the ester
- complex formed (40%). SM was prepared in two steps from 3,4-dihydroxy-acetophenone, namely acetylation (70%), then trimethyl-silylation (88%) [4850].
- Also refer to: [4828,4851].

2-Methoxy-1-(2,4,6-trihydroxyphenyl)ethanone

[55317-02-7] $C_9H_{10}O_5$ mol.wt. 198.18



Syntheses

- Preparation by reaction of methoxyacetonitrile with phloro-glucinol (Hoesch reaction) [4834, 4837,4852–4856], (80%) [4857], (79%) [4858], (77%) [4836], (75–80%) [4859], (50%) [4860].
- Also refer to: [4839,4840,4842–4844,4861–4878].

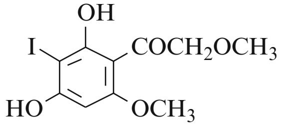
monohydrate [328074-83-5]: [4837,4856]; Crystal data [4856];

m.p. 195–196° [4854], 192–194° [4836,4857], 192° [4837,4879], 191–194° [4860];

^{1}H NMR [4836,4857], ^{13}C NMR [4836,4857,4880], MS [4836,4857].

1-(2,4-Dihydroxy-3-iodo-6-methoxyphenyl)-2-methoxyethanone

[74047-42-0] $C_{10}H_{11}IO_5$ mol.wt. 338.10



Synthesis

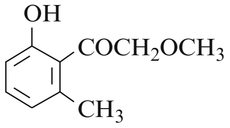
- Preparation by iodination of 2,4-dihydroxy-6, α-dimethoxy-acetophenone with iodine and periodic acid in dilute ethanol for 2 h at 60–70° (78%) [4881].

m.p. 191–193° [4881];

N.B.: This ketone was characterized by its corresponding diacetate: m.p. 112–114° and ^{1}H NMR [4881].

1-(2-Hydroxy-6-methylphenyl)-2-methoxyethanone

[75278-05-6] $C_{10}H_{12}O_3$ mol.wt. 180.20



Synthesis

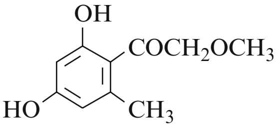
– Obtained by fission of 3-methoxy-5-methylflavone (m.p. 113–115°) with ethanolic potash (50%) [4882].

N.B.: This compound could not be prepared by Hoesch condensation of m-cresol with methoxyacetonitrile [4882].

oil [4882]; ^{1}H NMR [4882].

1-(2,4-Dihydroxy-6-methylphenyl)-2-methoxyethanone

[75278-00-1] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

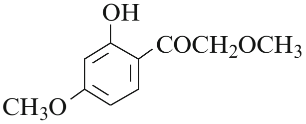
– Preparation by Hoesch condensation of orcinol with methoxyacetonitrile (53%) [4882].

– Also refer to: [4863].

m.p. 182–183° [4882]; ^{1}H NMR [4882].

1-(2-Hydroxy-4-methoxyphenyl)-2-methoxyethanone *(Fisetol dimethyl ether)*

[4940-44-7] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

– Preparation by reaction of methoxyacetonitrile with resorcinol monomethyl ether (Hoesch reaction) [4837], (80%) [4883].

– Also obtained by decomposition of 1-(2-acetoxy-4-methoxy-phenyl)-2-diazoethanone in methanol with copper bronze for 30 min at 50–55°, followed by hydrolysis of the acetyl derivative (57%). The diazoketone (m.p. 102–105°) was prepared by reaction of diazomethane with 2-acetoxy-4-methoxybenzoyl chloride in ethyl ether for 12 h at −5° [4820].

– Obtained by alkaline degradation of different polymethoxyflavones with potassium hydroxide,
  - From *fisetin tetramethyl ether* (3,7,3′,4′-tetramethoxyflavone) [4837,4884–4890];
  - From 3,7,3′,4′,5′-pentamethoxyflavone (SM) (m.p. 149°) [4891], (81%) [4890]. SM was prepared by methylation of 3,7,3′,4′,5′-pentahydroxyflavone, itself isolated from *Robinia pseudacacia* [4890];
  - From *kanugin* (3,7,3′-trimethoxy-4′,5′-methylenedioxyflavone) (m.p. 203–205°), isolated from the root bark of *Pongamia glabra* [4892];
  - From *demethoxykanugin* (3,7-dimethoxy-3′,4′-methylenedioxyflavone) (SM) (m.p. 142°), [4893], (86%) [4894]. SM was isolated from the seed oil of karanja (*Pongamia glabra*) [4894] or from fresh root bark and the stem bark of *Pongamia glabra* [4893].

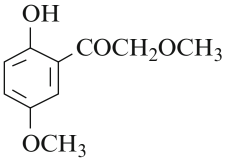
- Also obtained by alkaline degradation of 3,7-dimethoxychromone with sodium ethoxide [4895].
- Also obtained by alkaline degradation of 3,4,7-trimethoxycoumarin (m.p. 113–115°) with refluxing 5% aqueous sodium hydroxide for 1 h (60%) [4838].
- Also obtained by partial methylation of α-methoxyresacetophenone with dimethyl sulfate,
  - in the presence of potassium carbonate in refluxing benzene for 12 h (83%) [4848] or for 10 h (78%) [4846];
  - in 5% aqueous sodium hydroxide [4837,4896], (70%) [4835].
- Also refer to: [4640,4842,4897].

m.p. 132° [4883], 69–70° [4820,4848], 68–69° [4893], 67–68° [4838], 67° [4890,4894], 66° [4837,4846,4891], 65–67° [4892], 65–66° [4835]. One of the reported melting points is obviously wrong.

^{1}H NMR [4883]; TLC [4838].

1-(2-Hydroxy-5-methoxyphenyl)-2-methoxyethanone

[103323-12-2] $C_{10}H_{12}O_4$ mol.wt. 196.20



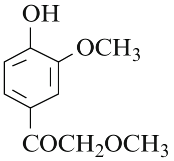
Synthesis

- Obtained by decomposition of 1-(2-acetoxy-5-methoxy-phenyl)-2-diazoethanone in methanol with copper bronze (57%). The diazoketone (dark reddish liquid) was prepared by reaction of diazomethane with 2-acetoxy-5-methoxy-benzoyl chloride in ethyl ether [4820].

reddish liquid [4820]; b.p.$_{0.3-0.4}$ 98–100° [4820].

1-(4-Hydroxy-3-methoxyphenyl)-2-methoxyethanone

[64349-38-8] $C_{10}H_{12}O_4$ mol.wt. 196.20

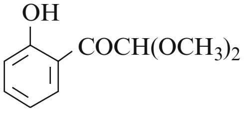


Synthesis

- Refer to: [4828] (Japanese paper).

1-(2-Hydroxyphenyl)-2,2-dimethoxyethanone

[127255-97-4] $C_{10}H_{12}O_4$ mol.wt. 196.20



Synthesis

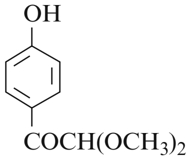
- Preparation by reaction of o-hydroxyacetophenone with catalytic amounts of diphenyl diselenide and excess of ammonium peroxydisulfate in refluxing methanol for 1.5 h (72%) [4898].

oil [4898]; TLC [4898]; GLC [4898];

^{1}H NMR [4898], ^{13}C NMR [4898], MS [4898].

1-(4-Hydroxyphenyl)-2,2-dimethoxyethanone

[144757-78-8] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

- Obtained by oxidation of p-hydroxyacetophenone with methyl nitrite gas in methanolic hydrogen chloride [4899], at 0–5° over 4 h (57%) [4900].

N.B.: The reaction involves oxidation with a source of nitrosonium ion (NO^+) in the presence of an alcohol and a source of H^+ to give a phenylglyoxal acetal.

Experimental procedure: Preparation by reaction of methyl nitrite with p-hydroxyacetophenone in 1.25 N methanolic hydrogen chloride between 0° and 5° for 4 h (72%). The methyl nitrite source was supplied by adding gradually 33% aqueous sulfuric acid to a sodium nitrite solution in aqueous methanol (1:1) under nitrogen.

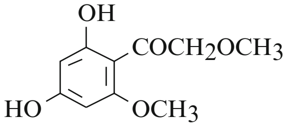
N.B.: During the course of the reaction, the bath was maintained at about −20°. The methyl nitrite generator was not cooled [4901].

- Also obtained from electrosynthesis by a selenium catalyzed transformation of p-hydroxy-acetophenone in methanol at r.t. (22%) [4902].
- Also refer to: [4899,4903].

white solid [4903];
^{1}H NMR [4901,4902], ^{13}C NMR [4901], MS [4901,4902].

1-(2,4-Dihydroxy-6-methoxyphenyl)-2-methoxyethanone

[62330-14-7] $C_{10}H_{12}O_5$ mol.wt. 212.20



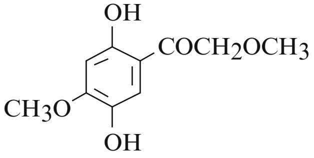
Syntheses

- Preparation by condensation of methoxyacetonitrile with phloroglucinol monomethyl ether (Hoesch reaction) (60%) [4879].
- Preparation by a three-step synthesis: first, tosylation of α-methoxyphloroacetophenone with p-toluenesulfonyl chloride in the presence of potassium carbonate in refluxing acetone for 4 h. Dimethyl sulfate and potassium carbonate were then added and the mixture refluxed for 36 h more. Finally, saponification of the residue isolated by distillation with refluxing 5% methanolic potassium hydroxide for 4 h (42%) [4861].
- Also refer to: [4839,4840,4881,4904].

m.p. 208° [4879], 190–192° [4861].

1-(2,5-Dihydroxy-4-methoxyphenyl)-2-methoxyethanone

[35930-51-9] $C_{10}H_{12}O_5$ mol.wt. 212.20



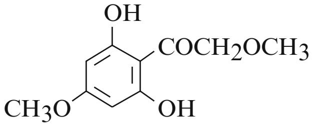
Syntheses

- Obtained on oxidation of 2-hydroxy-4,α-dimethoxy-acetophenone with potassium persulfate in aqueous sodium hydroxide at 30–40° for 90 min and at r.t. for 36 h [4905] (18%) [4848] (Elbs reaction).
- Also obtained by reaction of methoxyacetonitrile with 1,4-di-hydroxy-2-methoxybenzene (Hoesch reaction) (13%) [4906].
- Also refer to: [4907,4908].

trihydrate [4848];
m.p. 150° [4906], 148–149° [4905], 145–146° [4848].

1-(2,6-Dihydroxy-4-methoxyphenyl)-2-methoxyethanone

[70390-87-3] $C_{10}H_{12}O_5$ mol.wt. 212.20



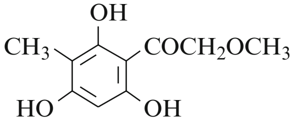
Syntheses

- Obtained by alkaline degradation of quercetin 3,7,3′,4′-tetra-methyl ether (m.p. 159–160°) (5-hydroxy-3,7,3′,4′-tetra-methoxyflavone) (SM) with potassium hydroxide. SM was isolated from *citrus reticulata* Blanco (Rutaceae) [4909].
- Also obtained by partial methylation of α-methoxy-phloroacetophenone with diazomethane in a methanol/ethyl ether mixture at 0° for 2 h (<8%) [4879].
- Also refer to: [4871,4910,4911].

m.p. 161–162° [4879]; MS [4909].

2-Methoxy-1-(2,4,6-trihydroxy-3-methylphenyl)ethanone

[110333-13-6] $C_{10}H_{12}O_5$ mol.wt. 212.20



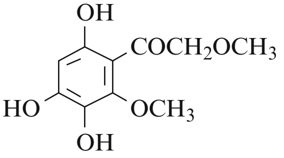
Syntheses

- Preparation by condensation of 2-methylphloroglucinol with methoxyacetic acid-boron trifluoride complex at 28–30° for 24 h (50%) [4912].
- Preparation by reaction of methoxyacetonitrile with 2-methylphloroglucinol (28%) (Hoesch reaction) [4913].
- Also refer to: [4914].

m.p. 207° [4912], 206–207° [4913]; sesquihydrate [4913].

2-Methoxy-1-(6-methoxy-2,4,5-trihydroxyphenyl)ethanone

[65039-95-4] $C_{10}H_{12}O_6$ mol.wt. 228.20



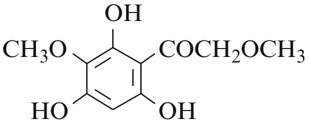
Syntheses

- Obtained by reaction of potassium persulfate with 2,4-di-hydroxy-6,α-dimethoxyacetophenone in aqueous sodium hydroxide at r.t. under nitrogen for 38 h (11%) [4904] (Elbs reaction).
- Also refer to: [4915,4916].

m.p. 163° [4904]; IR [4904], UV [4904].

2-Methoxy-1-(2,4,6-trihydroxy-3-methoxyphenyl)ethanone

[16297-02-2] $C_{10}H_{12}O_6$ mol.wt. 228.20



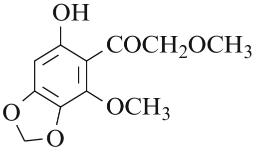
Synthesis

- Preparation by reaction of methoxyacetonitrile with iretol (Hoesch reaction) [4917], (59%) [4918].

m.p. 157–158° (anhydrous) [4917,4918], 82–84° [4919], 79–80° (dihydrate) [4918].

1-[6-Hydroxy-2-methoxy-3,4-(methylenedioxy)phenyl]-2-methoxyethanone

[91144-13-7] $C_{11}H_{12}O_6$ mol.wt. 240.21



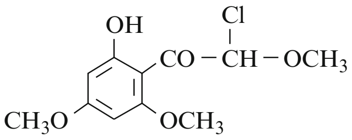
Syntheses

- Obtained by reaction of methoxyacetonitrile with 3-methoxy-4,5-(methylenedioxy)phenol (Hoesch reaction) [4905], (41%) [4920], (29%) [4904].
- Also obtained (trace) by reaction of methylene iodide with 3,4,6-trihydroxy-2,α-dimethoxyacetophenone in the presence of potassium carbonate in refluxing acetone for 30 h (1%) [4904].
- Also obtained by alkaline degradation of *meliternatin* with boiling alcoholic potassium hydroxide [4862], for 7 h (65%) [4921]. *Meliternatin*—3,5-dimethoxy-6,7,3′,4′-bis(methylenedioxy)flavone—(m.p. 198–198°5) was first isolated from *Melicope ternata* (Rutaceae) [4921], then from *Melicope mantelli* Buch [4922].

m.p. 142–144° [4862,4921], 140–142° [4904,4920]; IR [4904,4920].

2-Chloro-1-(2-hydroxy-4,6-dimethoxyphenyl)-2-methoxyethanone

[88092-53-9] $C_{11}H_{13}ClO_5$ mol.wt. 260.67



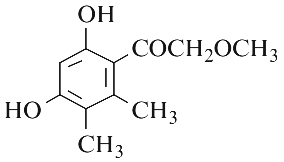
Synthesis

- Obtained (by-product) by reaction of 1-(2-hydroxy-4,6-dimethoxyphenyl)-2-methoxyethanone with iron complex $[Fe(DMF)_3Cl_2]$ $[FeCl_4]$ in refluxing dilute methanol for 4 h (<3%) [4923].

semi-solid [4923]; column chromatography [4923];
^{1}H NMR [4923], UV [4923].

1-(4,6-Dihydroxy-2,3-dimethylphenyl)-2-methoxyethanone

[132020-84-9] $C_{11}H_{14}O_4$ mol.wt. 210.23

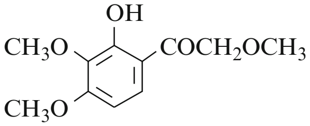


Syntheses

- Preparation by reaction of methoxyacetonitrile with 1,3-di-hydroxy-4,5-dimethylbenzene (Hoesch reaction) (71%) [4858].
- Also refer to: [4863,4864].

1-(2-Hydroxy-3,4-dimethoxyphenyl)-2-methoxyethanone

[21417-76-5] $C_{11}H_{14}O_5$ mol.wt. 226.23



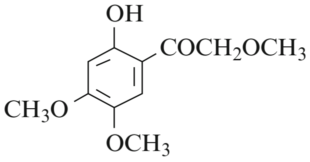
Syntheses

- Obtained by alkaline hydrolysis of 3,7,8,3′,4′-pentamethoxy-flavone (m.p. 153°) with 10% potassium hydroxide solution in boiling ethanol for 4 h (58%) [4924].
- Also obtained by alkaline hydrolysis of O-pentamethyl-dihydromelanoxetin (3,7,8,3′,4′-pentamethoxyflavanone) (m.p. 146–148°) with 8% potassium hydroxide solution in refluxing ethanol for 30 min [4925].

m.p. 85–86° [4924], 82–84° [4925]; ^{1}H NMR [4924].

1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-methoxyethanone

$C_{11}H_{14}O_5$ mol.wt. 226.23



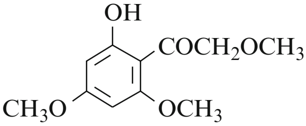
Synthesis

- Preparation by partial methylation of 4,α-dimethoxy-2,5-dihydroxyacetophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing benzene for 12 h (38%) [4848].

m.p. 90–91° [4848].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-methoxyethanone

[17874-42-9] $C_{11}H_{14}O_5$ mol.wt. 226.23



Syntheses

- Preparation by partial methylation of α-methoxyphloro-acetophenone [4874],
  - with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone/benzene mixture for 12 h (60%) [4876], (38%) [4871];
  - with methyl iodide in the presence of potassium carbonate in refluxing acetone for 3 h [4913];
  - with diazomethane (1 mol) in a methanol/ethyl ether mixture at 0° for 2 h [4879].
- Also obtained by condensation of phloroglucinol dimethyl ether with methoxyacetonitrile (Hoesch reaction) (45%) [4858], (25%) [4876].
- Also obtained by alkaline degradation of various polymethoxyflavones,
  - From *izalpinin* dimethyl ether (m.p. 194°) (3,5,7-trimethoxyflavone) on boiling with 10% ethanolic potassium hydroxide for 3 h (52%) [4926];
  - From *kaempferide* trimethyl ether (m.p. 153–154°) (SM) (3,5,7,4′-tetramethoxyflavone) with potassium hydroxide. SM was isolated from *Citrus reticulata* Blanco (Rutaceae) [4909];
  - From *populnetin* tetramethyl ether, so called *kaempferol* tetramethyl ether (m.p. 165–166°) (3,5,7,4′-tetramethoxyflavone) by refluxing with 8% ethanolic potassium hydroxide for 6 h [4874];
  - From *morin* pentamethyl ether (m.p. 154–157°) (3,5,7,2′,4′-pentamethoxyflavone) by heating at reflux with 20% ethanolic potassium hydroxide for 8–10 h [4884];
  - From *quercetin* pentamethyl ether (m.p. 148–150°) (3,5,7,3′,4′-pentamethoxyflavone) with ethanolic potassium hydroxide [4884,4889] or with boiling dilute ethanolic sodium hydroxide (54%) [4927];
  - From *oxyayanin-A* trimethyl ether (m.p. 190–193°) (3,5,7,2′,4′,5′-hexamethoxyflavone) [4928], with potassium hydroxide in boiling ethanol for 8 h (57%) [4868];
  - From *myricetin* hexamethyl ether (m.p. 153°) (3,5,7,3′,4′,5′-hexamethoxyflavone) with boiling 10% ethanolic potassium hydroxide [4889,4929].
- Also obtained by alkaline degradation of 3,4,5,7-tetramethoxycoumarin with refluxing 5% aqueous sodium hydroxide for 1 h [4838].
- Also refer to: [4640,4837,4842,4862,4865,4872,4923,4930–4935].

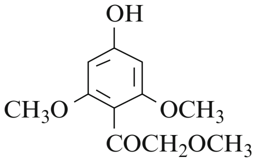
m.p. 104–106° [4874], 104–105° [4913], 103–104° [4868,4876], 102–104° [4838,4884,4889,4928], 102° [4926,4927,4929], 98–100° [4871], 98° [4879];

^{1}H NMR [4871], ^{13}C NMR [4880], IR [4871,4927], MS [4909];

TLC [4838]; GLC [4936].

1-(4-Hydroxy-2,6-dimethoxyphenyl)-2-methoxyethanone

$C_{11}H_{14}O_5$ mol.wt. 226.23



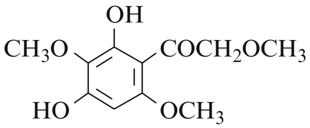
Synthesis

- Obtained (by-product) by condensation of phloroglucinol dimethyl ether with methoxyacetonitrile (Hoesch reaction) (<2%) [4876].

m.p. 259–260° [4876].

1-(2,4-Dihydroxy-3,6-dimethoxyphenyl)-2-methoxyethanone

[42923-40-0] $C_{11}H_{14}O_6$ mol.wt. 242.23



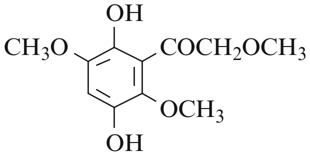
Syntheses

- Obtained by reaction of methoxyacetonitrile with 2,5-di-methoxyresorcinol (Hoesch reaction) [4937], (82%) [4938], (62%) [4939,4940].
- Also obtained by debenzylation of 4-benzyloxy-2-hydroxy-3,6,α-trimethoxyacetophenone in acetic acid in the presence of hydrochloric acid (d = 1.16) on a boiling water bath for 1 h [4941].
- Also refer to: [4942–4944].

m.p. 150–151° [4938–4940], 149–150° [4941].

1-(2,5-Dihydroxy-3,6-dimethoxyphenyl)-2-methoxyethanone

$C_{11}H_{14}O_6$ mol.wt. 242.23



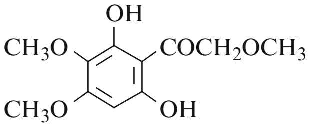
Synthesis

- Obtained by reduction of 2-(2-methoxyacetyl)-3,6-di-methoxy-1,4-benzoquinone (m.p. 222–224°) with sulfur dioxide in ethanol (40%). This quinone was prepared by oxidation of 2-hydroxy-3,5,6,α-tetramethoxyacetophenone with fuming nitric acid in ethyl ether [4945].

m.p. 175–177° [4945].

1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-2-methoxyethanone

[100059-77-6] $C_{11}H_{14}O_6$ mol.wt. 242.23



Synthesis

- Preparation by reaction of methoxyacetonitrile with 4,5-di-methoxyresorcinol (Hoesch reaction) [4938,4946,4947].

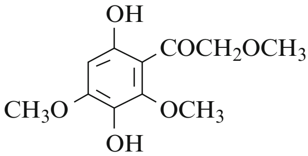
Isolation from natural sources

- Preparation by hydrolysis of *Casticin* (m.p. 186–187°) (5,3′-dihydroxy-3,6,7, 4′-tetramethoxy-flavone) with potassium hydroxide in refluxing ethanol for 4 h under nitrogen (66%) [4948].
- Also by degradation of *Gnaphaliin* monomethyl ether (SM) (m.p. 176–178°) (5-hydroxy-3,7,8-trimethoxyflavone) with 10% ethanolic potassium hydroxide for 2 h under nitrogen. SM was prepared by partial methylation of *Gnaphaliin* (m.p. 174–175°) (3,5-dihydroxy-7,8-dimethoxy-flavone), itself isolated from the aerial parts of *Gnaphalium obtusifolium* [4949].
- Also refer to: [4876].

m.p. 129–130° [4946,4948].

1-(3,6-Dihydroxy-2,4-dimethoxyphenyl)-2-methoxyethanone

[14639-73-7]



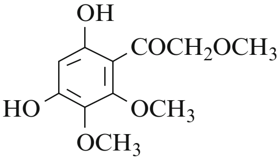
$C_{11}H_{14}O_6$ mol.wt. 242.23

Syntheses

- Preparation from 2-hydroxy-4,6,α-trimethoxyacetophenone by Elbs reaction (22%) [4858],
    - with sodium persulfate in aqueous sodium hydroxide at 15–20° for 23 h [4868,4950], (23%) [4876], (32%) [4877];
    - with potassium persulfate in aqueous sodium hydroxide at 15–20° for 20 h (26%) [4928].
- Also refer to: [4872,4907,4908,4915,4916,4951–4954].

m.p. 139–140° [4877], 135–136° [4876,4928];
sublimation at 115–130°/0.2 mm [4877]; ^{1}H NMR [4950], ^{13}C NMR [4880].

1-(4,6-Dihydroxy-2,3-dimethoxyphenyl)-2-methoxyethanone



$C_{11}H_{14}O_6$ mol.wt. 242.23

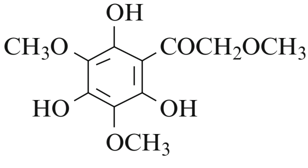
Synthesis

- Preparation by reaction of methoxyacetonitrile with 4,5-di-methoxyresorcinol, according to the Hoesch method [4946].

m.p. 129–130° [4946].

2-Methoxy-1-(2,4,6-trihydroxy-3,5-dimethoxyphenyl)ethanone

[85950-49-8]



$C_{11}H_{14}O_7$ mol.wt. 258.23

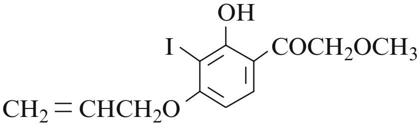
Synthesis

- Obtained by condensation of methoxyacetonitrile with 1,3,5-trihydroxy-2,4-dimethoxybenzene (m.p. 98°) (Hoesch reaction) [4955], (85%) [4956].

m.p. 152–153° [4955]; IR [4956], UV [4956], MS [4956].

1-[2-Hydroxy-3-iodo-4-(2-propenyloxy)phenyl]-2-methoxyethanone

[72511-79-6] $C_{12}H_{13}IO_4$ mol.wt. 348.14



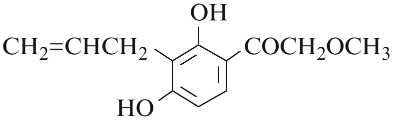
Synthesis

– Obtained by allylation of 2,4-dihydroxy-3-iodo-α-methoxyacetophenone with allyl bromide in the presence of potassium carbonate in refluxing acetone for 4–5 h (53%) [4819].

m.p. 88–90° [4819]; ^{1}H NMR [4819].

1-[2,4-Dihydroxy-3-(2-propenyl)phenyl]-2-methoxyethanone

$C_{12}H_{14}O_4$ mol.wt. 222.24



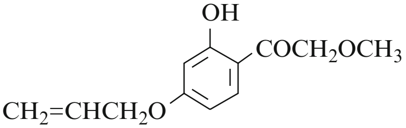
Synthesis

– Obtained by Claisen rearrangement of 2-hydroxy-4-allyloxy-α-methoxyacetophenone by heating for 2 h at 190–195° under reduced pressure (67%) [4957].

m.p. 139–139°5 [4957].

1-[2-Hydroxy-4-(2-propenyloxy)phenyl]-2-methoxyethanone

[57280-73-6] $C_{12}H_{14}O_4$ mol.wt. 222.24



Syntheses

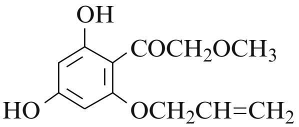
– Obtained by reaction of allyl bromide with α-methoxyresacetophenone in the presence of potassium carbonate in refluxing acetone for 5 h (52%) [4957].

– Also refer to: [4838].

pale yellow viscous liquid [4957].

1-[2,4-Dihydroxy-6-(2-propenyloxy)phenyl]-2-methoxyethanone

[62330-10-3] $C_{12}H_{14}O_5$ mol.wt. 238.24



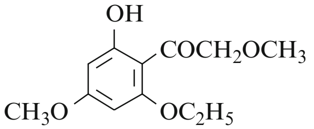
Synthesis

– Preparation by a three-step synthesis: first, tosylation of α-methoxyphloroacetophenone with p-toluenesulfonyl chloride in the presence of potassium carbonate in refluxing acetone for 4 h. Allyl bromide and potassium carbonate were then added to the reaction mixture and refluxed for 30 h. Finally, saponification of the residue, isolated by distillation, with refluxing 5% methanolic potassium hydroxide for 4 h (38%) [4861].

m.p. 184–186° [4861].

1-(2-Ethoxy-6-hydroxy-4-methoxyphenyl)-2-methoxyethanone

[21587-55-3] $C_{12}H_{16}O_5$ mol.wt. 240.26



Syntheses

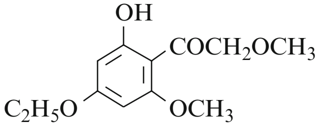
- Obtained by alkaline degradation of various flavones,
- with *potassium hydroxide* in boiling ethanol for 8 h,
  - From 5-ethoxy-3,7,3′,4′-tetramethoxyflavone (20%) [4927];
  - From 5-ethoxy-3,7,2′,4′,5′-pentamethoxyflavone [4928];
  - From 5,2′-diethoxy-3,7,4′,5′-tetramethoxyflavone [4928];
  - From 5,2′,5′-triethoxy-3,7,4′-trimethoxyflavone (*oxyayanin-A triethyl ether*) (57%) [4868];
- with *sodium hydroxide* in boiling ethanol for 1 h,
  - From 5,3′,5′-triethoxy-3,7,4′-trimethoxyflavone (33%) [4958]. This flavone (m.p. 139°) was prepared from *myricetin*, first by selective methylation, then ethylation of the obtained *myricetin 3,7,4′-trimethyl ether* (m.p. 207–208°) [4958];
  - From 5,3′-diethoxy-3,7,4′-trimethoxyflavone. This flavone was prepared from *quercetin*, first by selective methylation, then ethylation of the obtained *quercetin 3,7,4′-trimethyl ether* (m.p. 174°) [4958].

m.p. 110° [4928], 109–110° [4868], 106–107° [4927], 95° [4958];

^{1}H NMR [4958], MS [4958].

1-(4-Ethoxy-2-hydroxy-6-methoxyphenyl)-2-methoxyethanone

[91555-84-9] $C_{12}H_{16}O_5$ mol.wt. 240.26



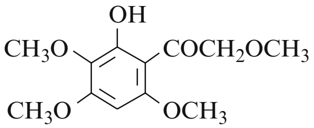
Syntheses

- Obtained by alkaline degradation of various polysubstituted flavones with potassium hydroxide in refluxing ethanol,
  - From 7-ethoxy-3,5-dimethoxyflavone (m.p. 128–129°) (81%) [4959];
  - From 7-ethoxy-3,5,4′-trimethoxyflavone [4874];
  - From 7-ethoxy-3,5,3′,4′-tetramethoxyflavone (m.p. 158–160°) (81%) [4959].

m.p. 108–110° [4874], 105–106° [4959].

1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-2-methoxyethanone

$C_{12}H_{16}O_5$ mol.wt. 240.26



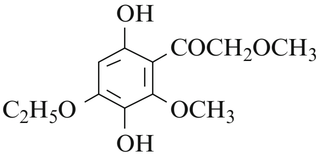
Syntheses

- Obtained by treatment of α-methoxyphloracetophenone with methyl iodide in the presence of potassium carbonate in refluxing acetone for 3 h [4913,4960], (18%) [4961].

- Also obtained by condensation of methoxyacetonitrile with 2-hydroxy-4,6-dimethoxytoluene (Hoesch reaction) [4961].
- Also obtained (by-product) by treatment of α-methoxyphloracetophenone with dimethyl sulfate in the presence of potassium carbonate by refluxing in an acetone and benzene mixture (1:3, v/v) for 12 h (<3%) [4871].
- Also obtained by alkaline degradation of 8-methylquercetin pentamethyl ether (m.p. 213–215°) with boiling ethanolic potash [4962].
- Also refer to: [4914].

m.p. 176–177° [4913,4960] (anhydrous); 148–149° [4961,4962], 141–142° [4913,4960], 140–142° [4871] (hydrate);
^{1}H NMR [4871], IR [4871].

1-(4-Ethoxy-3,6-dihydroxy-2-methoxyphenyl)-2-methoxyethanone



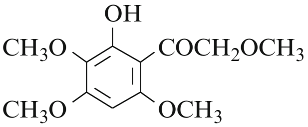
$C_{12}H_{16}O_6$ mol.wt. 256.26

Synthesis

- Obtained by reaction of potassium persulfate with 4-ethoxy-2-hydroxy-6,α-dimethoxyacetophenone in aqueous sodium hydroxide at r.t. for 24 h (23%) (Elbs reaction) [4959].

1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-2-methoxyethanone *(Gossypetol tetramethyl ether)*

[7741-43-7]



$C_{12}H_{16}O_6$ mol.wt. 256.26

Syntheses

- Preparation by partial methylation of 2,4-dihydroxy-3,6,α-trimethoxyacetophenone,
  - with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 12 h (63%) [4939];
  - with diazomethane in acetone (82%) [4942].

Isolation from natural sources

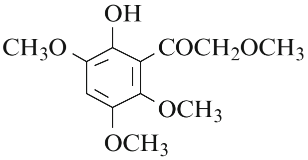
- Also obtained by alkaline degradation of various polymethoxyflavones with potassium hydroxide,
  - From *chlorflavonin dimethyl ether* (3′-chloro-3,5,7,8,2′-pentamethoxyflavone) (m.p. 114–115°) (SM), (35%). SM was obtained by methylation of *chlorflavonin* (3′-chloro-5,2′-dihydroxy-3,7,8-trimethoxyflavone) (m.p. 212°), itself isolated from cultures of *Aspergillus candidus* [4963];
  - From *Herbacetin pentamethyl ether* (3,5,7,8,4′-pentamethoxyflavone) (m.p. 156–158°) [4937];
  - From *Gossypetin hexamethyl ether* (3,5,7,8,3′,4′-hexamethoxyflavone) (m.p. 170–172°) [4964]; [4842,4939], (85%) [4965], (63%) [4939];

- From 3,5,7,8,3′,4′,5′-heptamethoxyflavone (m.p. 194–194°5) (SM), (51%) [4966]. SM was prepared according to different methods:
- by methylation of 5,7,3′-trihydroxy-3,8,4′,5′-tetramethoxyflavone (m.p. 214–216°), itself isolated from *Beyeria brevifolia* (Muell. Arg.) Benth. [4967];
- by methylation of 5,7-dihydroxy-3,8,3′,4′,5′-pentamethoxyflavone (m.p. 204–205°), itself isolated from the whole plant of *Conyza stricta* Willd. (Compositae) [4966];
- from *hibiscetin heptamethyl ether* (m.p. 194–196°). *Hibiscetin* is an aglycone of *Hibiscitrin* (3,5,7,8,3′,4′,5′-heptahydroxyflavone). It was isolated from the flowers of *Hibiscus sabdariffa* [4968].
- Also refer to: [4855].

m.p. 116–118° [4939], 115–116° [4937,4942,4963,4965–4968];
UV [4942]; GLC [4936].

1-(2-Hydroxy-3,5,6-trimethoxyphenyl)-2-methoxyethanone

[62953-05-3] $C_{12}H_{16}O_6$ mol.wt. 256.26



Syntheses

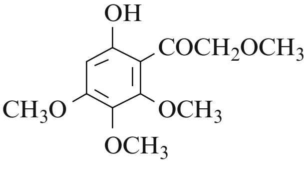
- Preparation in numerous steps starting from 2,6-dihydroxy-α-methoxyacetophenone. No data [4849].
- Also obtained by alkaline degradation of some flavones with refluxing ethanolic potassium hydroxide,

- From 3,5,6,8,4′-pentamethoxyflavone (m.p. 158–159°) (SM). SM was prepared by methylation of 5,6-dihydroxy-3,8,4′-trimethoxyflavone (m.p. 178–179°), itself isolated from the whole plant of *Conyza stricta* Willd. (Compositae) [4966];
- From *methyl gardenin* (3,5,6,8,3′,4′,5′-heptamethoxyflavone) (m.p. 116–117°) (SM) [4842], (84%) [4945]. SM was prepared by methylation of *gardenin* (m.p. 163–164°), itself isolated from Dikamali gum (gum of *Gardenia lucida*) [4945].

m.p. 110–112° [4966], 88–89° [4945]. One of the reported melting points is obviously wrong.

1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-2-methoxyethanone *(Quercetagetol tetramethyl ether)*

[14290-59-6] $C_{12}H_{16}O_6$ mol.wt. 256.26



Syntheses

- Obtained by partial methylation of 2,5-dihydroxy-α,4,6-tri-methoxyacetophenone with dimethyl sulfate [4950], in the presence of potassium carbonate,

- in boiling acetone/benzene (1:1) for 8.5 h (53%) [4877];
- in refluxing benzene for 10 h (47%) [4876].

- Also obtained by alkaline degradation of various polymethoxyflavones,
  - From *mikanin dimethyl ether* so-called *Tangeretin* [4969] (3,5,6,7,4′-pentamethoxyflavone) (SM) (m.p. 157–158°) [4867], (m.p. 155–156°) [4970] with potassium hydroxide in refluxing ethanol for 6.5 h (29%) [4867] or for 8 h [4970]; SM was isolated from oil of the bark of bitter orange [4970];
  - From *alnusin trimethyl ether* (3,5,6,7-tetramethoxyflavone) (m.p. 112–112°5) (SM) refluxing in a mixture of 50% potassium hydroxide solution and ethanol for 20 h under nitrogen (50%). SM was prepared by methylation of *alnusin* (6-methoxy-3,5,7-trihydroxyflavone) (m.p. 239–241°). *Alnusin* was the main flavonoid isolated from *Alnus sieboldiana* (Betulaceae) [4971];
  - From *vogeletin tetramethyl ether* with potassium hydroxide in refluxing ethanol for 6 h (81%) [4972], (98%) [4973];

  N.B.: The *mikanin dimethyl ether* is identical with *penduletin dimethyl ether* and *vogeletin tetramethyl ether*.

- From *apulein* (2′,5′-dihydroxy-3,5,6,7,4′-pentamethoxyflavone) (m.p. 211–213°) with 20% sodium hydroxide in refluxing dilute methanol (1:1) for 4 h [4974]. The *apulein* was isolated from the wood of *Apuleia leiocarpa* (Vog.) Macbr. (= *Apuleia praecox M*art.) (Leguminosae, subfamily Caesalpinioideae);
- From *apulein diethyl ether* (2′,5′-diethoxy-3,5,6,7,4′-pentamethoxyflavone) (m.p. 129–131°) with 10% ethanolic potassium hydroxide at reflux for 10 h under nitrogen [4974];
- From *quercetagetin* hexamethyl ether (3,5,6,7,3′,4′-hexamethoxyflavone) (m.p. 141–142°) [4876];
- From *patuletin hexamethyl ether* (3,5,6,7,3′,4′-hexamethoxyflavone) (m.p. 141–142°) [4975],
  - with refluxing (150–155°) 50% aqueous potash for 8 h(21%);
  - with refluxing 7% ethanolic potash for 6 h (94%);
- From methyl 3,5,6,7,3′,4′-hexamethoxyflavone-2′-carboxylate (m.p. 151–152°) (SM1) with potassium hydroxide in refluxing dilute ethanol for 8 h (78%) [4844]. SM1 was obtained by prolonged methylation of *distemonanthin*, itself isolated from the wood of *distemonanthus benthamianus*;
- From (3,5,6,7,2′,3′,4′-heptamethoxyflavone) (m.p. 191–192°) [4928];
- From *apulein dimethyl ether* (3,5,6,7,2′,4′,5′-heptamethoxyflavone) (m.p. 159–160°) with 50% aqueous potassium hydroxide in refluxing ethanol for 8 h [4974];
- From 3,5,6,7,3′,4′,5′-heptamethoxyflavone (m.p. 155–156°) (SM2) with potassium hydroxide in boiling ethanol for 7 h, under nitrogen [4866]. SM2 was isolated from *Eremophila fraseri* F. Muell.

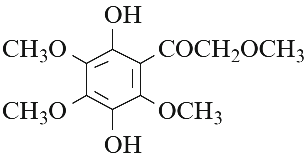
oil [4970];

m.p. 77–78° [4876], 75–76° [4928,4975], 72–73° [4973], 71–72° [4844,4877,4972,4976], 70–71° [4866,4867], 69–71° [4971,4974];

^{1}H NMR [4950,4971], IR [4877,4971,4974], UV [4971,4974], MS [4974];

TLC [4974]; GLC [4936].

1-(2,5-Dihydroxy-3,4,6-trimethoxyphenyl)-2-methoxyethanone



$C_{12}H_{16}O_7$ mol.wt. 272.25

Syntheses

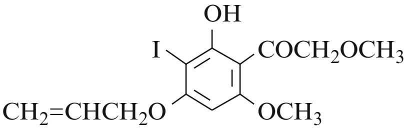
- Obtained by oxidation of 2-hydroxy-3,4,6,α-tetramethoxy-acetophenone with alkali persulfate (Elbs reaction) (14%) [4939].

- Also refer to: [4977,4978].

m.p. 102–103° [4939].

1-[2-Hydroxy-3-iodo-6-methoxy-4-(2-propenyloxy)phenyl]-2-methoxyethanone

[74047-41-9]



$C_{13}H_{15}IO_5$ mol.wt. 378.16

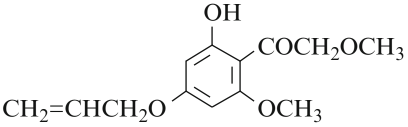
Synthesis

- Obtained by treatment of 2,4-dihydroxy-3-iodo-6,α-dimethoxyacetophenone with allyl bromide in the presence of potassium carbonate in refluxing acetone for 4–5 h (54%) [4881].

m.p. 167–168° [4881]; ^{1}H NMR [4881].

1-[2-Hydroxy-6-methoxy-4-(2-propenyloxy)phenyl]-2-methoxyethanone

[62330-15-8]



$C_{13}H_{16}O_5$ mol.wt. 238.24

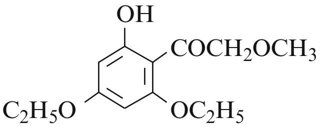
Synthesis

- Preparation by partial alkylation of 2,4-dihydroxy-6,α-dimethoxyacetophenone with allyl bromide in the presence of potassium carbonate in refluxing acetone for 4 h (80%) [4861].

m.p. 87–89° [4861].

1-(2,4-Diethoxy-6-hydroxyphenyl)-2-methoxyethanone

[2495-77-4]



$C_{13}H_{18}O_5$ mol.wt. 254.28

Syntheses

- Obtained by partial ethylation of 2,4,6-trihydroxy-α-methoxyacetophenone,
  - with diethyl sulfate in the presence of potassium carbonate in boiling acetone for 5 h (78%) [4867] or for 16 h [4854];
  - with ethyl iodide in the presence of potassium carbonate in refluxing acetone for 6 h [4874].

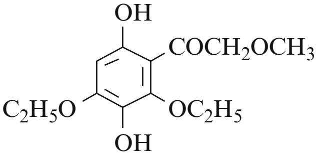
- Also obtained by alkaline degradation of some polysubstituted flavones with potassium hydroxide,
  - From 3,4′-dimethoxy-5,7,3′-triethoxyflavone (m.p. 108–109°) (SM). SM was prepared by total ethylation of 3,4′-dimethoxy-5,7,3′-trihydroxyflavone (m.p. 235–236°), itself isolated from *Baccharis sarothroides* A. Gray (Compositae) [4979];
  - From 3-methoxy-5,7,3′,4′-tetraethoxyflavone (m.p. 146–148°) (SM) in boiling ethanol for 6 h (35%). SM was prepared by total ethylation of *quercetin 3-methyl ether* (m.p. 261–263°), itself obtained by hydrolysis of its glycoside (m.p. 165–167°). This one (*stizoloside*) was isolated from the aerial parts of *Stizolophus balsamita* (Lam.) A. Takht, so-called *Centaurea balsamita* Lam. (Compositae) [4980].

m.p. 111–112° [4867], 110–112° [4874], 110–111° [4980], 109–111° [4854], 109–110° [4979];

^{1}H NMR [4979], IR [4979], UV [4979].

1-(2,4-Diethoxy-3,6-dihydroxyphenyl)-2-methoxyethanone

[4324-58-7] $C_{13}H_{18}O_6$ mol.wt. 270.28



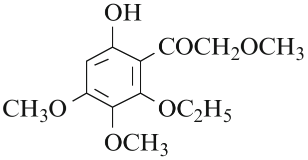
Synthesis

- Obtained by reaction of potassium persulfate with 2,4-di-ethoxy-6-hydroxy-α-methoxyacetophenone in the presence of aqueous sodium hydroxide (Elbs reaction), (33%) [4867], (25%) [4854].

m.p. 102–103° [4854], 101–103° [4867]; ^{1}H NMR [4854].

1-(2-Ethoxy-6-hydroxy-3,4-dimethoxyphenyl)-2-methoxyethanone

[14965-23-2] $C_{13}H_{18}O_6$ mol.wt. 270.28



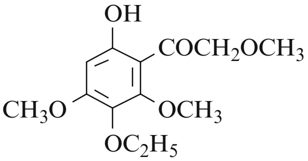
Syntheses

- Obtained by alkaline degradation of two flavones with potassium hydroxide in refluxing ethanol for 12 h under nitrogen,
  - From 5,3′,4′-triethoxy-3,6,7-trimethoxyflavone (m.p. 96–97°) (98%) [4981];
  - From 5,3′,5′-triethoxy-3,6,7,4′-tetramethoxyflavone (m.p. 120–121°) [4866].

m.p. 82–83° [4866,4981].

1-(3-Ethoxy-6-hydroxy-2,4-dimethoxyphenyl)-2-methoxyethanone

$C_{13}H_{18}O_6$ mol.wt. 270.28



Syntheses

- Obtained by alkaline degradation of two flavones with potassium hydroxide in boiling ethanol for 7 h,

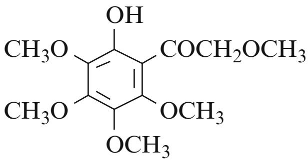
- From 6,3′-diethoxy-3,5,7,4′-tetramethoxyflavone (di-O-ethyl-O-methyl oxyay*anin-B*) (73%) [4868];
- From 6,2′-diethoxy-3,5,7,4′,5′-pentamethoxyflavone (m.p. 136–137°) [4928].

– Also obtained by reaction of ethyl iodide with 3,6-dihydroxy-2,4-dimethoxy-α-methoxy-acetophenone in the presence of potassium carbonate in refluxing acetone for 18 h [4868].

m.p. 78–79° [4868], 78° [4928].

1-(2-Hydroxy-3,4,5,6-tetramethoxyphenyl)-2-methoxyethanone *(Calycopterol pentamethyl ether)*

[5071-47-6] $C_{13}H_{18}O_7$ mol.wt. 286.28



Syntheses

– Obtained by alkaline degradation of *Calycopterin dimethyl ether* or *Thapsin dimethyl ether* (SM) both 3,5,6,7,8,4′-hexa-methoxyflavone (m.p. 133–134°) [4842,4982], (89%) [4983], (54%) [4984]. SM was prepared by total methylation of *Thapsin*, itself isolated from *Digitalis Thapsi*, L. [4983].

– Also obtained by alkaline degradation of *purpurascenin* (3,5,6,7,8,2′,4′,5′-octamethoxyflavone) (m.p. 132–133°) with refluxing ethanolic potassium hydroxide for 15 h (24%) [4985]. *Purpurascenin* was isolated from the roots, stem, leaves and flowers of *Pogostemon purpurascens* (Labiatae).

– Also obtained by alkaline degradation of *Digicitrine dimethyl ether* (3,5,6,7,8, 3′,4′,5′-octamethoxy- flavone) (m.p. 126°) with potassium hydroxide in refluxing 80% ethanol for 4 h (ca. 115°) (75%) [4986]. The *Digicitrine dimethyl ether* was prepared by methylation of *Digicitrine* (5,3′-dihydroxy-3,6,7,8,4′,5′-hexamethoxyflavone) (m.p. 178–179°), itself isolated from the leaves of *Digitalis purpurea* L.

– Also obtained by alkaline degradation of *Melibentin* with potassium hydroxide in refluxing dilute ethanol for 5 h (73%) [4987]. *Melibentin* (3,5,6,7,8-pentamethoxy-3′,4′-methylenedioxyflavone) (m.p. 134–135°) was isolated from the bark and the wood of *Melicope broadbentiana* F. M. Bail (Rutaceae).

– Also obtained by alkaline degradation of *Natsudaidain methyl ether* (3,5,6,7,8,3′,4′-heptamethoxy-flavone) (SM) (m.p. 130–131°) [4970], (m.p. 128°) [4988] with potassium hydroxide in refluxing ethanol [4970], (75%) [4988]. SM was isolated from oil of the bark of bitter orange [4970] or was prepared by methylation of *Natsudaidain* (3-hydroxy-5,6,7,8,3′,4′-hexamethoxyflavone) (m.p. 146°), itself isolated from the peel oil of *Citrus natsudaidai* HAYATA [4988].

– Also obtained by partial methylation of 2,5-dihydroxy-3,4,6,α-tetramethoxyacetophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 1 h [4978,4989], (29%) [4977].

– Also refer to: [4990–4992].

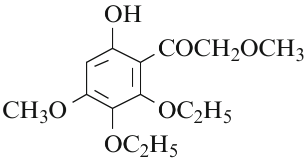
gum [4985]; sublimation at 40°/0.01 mm [4986];

m.p. 85–87° [4987], 66–67° [4983], 65–67° [4984], 65–66° [4986], 64–66° [4970], 64° [4988], 62–64° [4977]. One of the described melting points is obviously wrong.

GLC [4936]; TLC [4985,4986].

^{1}H NMR [4985,4987], IR [4987], UV [4986,4987], MS [4985].

1-(2,3-Diethoxy-6-hydroxy-4-methoxyphenyl)-2-methoxyethanone



$C_{14}H_{20}O_6$ mol.wt. 284.31

Syntheses

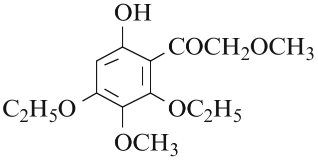
– Obtained by alkaline degradation of various polyalkylated flavones with potassium hydroxide in refluxing ethanol,

- From (5,6,3′-triethoxy-3,7,4′-trimethoxyflavone) oxyayanin-B triethyl ether (72%) [4868];
- From 5,6-diethoxy-3,7,3′,4′-tetramethoxyflavone (SM). SM was prepared by ethylation of 5,6-dihydroxy-3,7,3′,4′-tetra-methoxyflavone (m.p. 211–213°), itself isolated from the heartwood of *Distemonanthus benthamianus* Baillon [4928];
- From 5,6-diethoxy-3,7,2′,4′,5′-pentamethoxyflavone (m.p. 97–99°) (SM1). SM1 was prepared by ethylation of 5,6-dihydroxy-3,7,2′,4′,5′-pentamethoxyflavone (m.p. 142–145°), itself isolated from the heartwood of *Distemonanthus benthamianus* (Leguminosae) [4993].

m.p. 80° [4928], 79–80° [4868,4993]; ^{1}H NMR [4993].

1-(2,4-Diethoxy-6-hydroxy-3-methoxyphenyl)-2-methoxyethanone

[4324-59-8]



$C_{14}H_{20}O_6$ mol.wt. 284.31

Syntheses

– Obtained by reaction of dimethyl sulfate with 2,4-diethoxy-3,6-dihydroxy-α-methoxyacetophenone in the presence of potassium carbonate in refluxing acetone for 2.5 h [4993], (27%) [4867] or for 12 h [4854].

– Preparation by Friedel–Crafts acylation of 3,5-diethoxy-4-methoxyphenol with methoxyacetyl chloride in ethyl ether in the presence of aluminium chloride, first at 10°, then at 20° for 3 h (72%) [4918].

– Also obtained by reaction of methoxyacetonitrile with 3,5-diethoxy-4-methoxyphenol (Hoesch reaction) [4917], (7%) [4918].

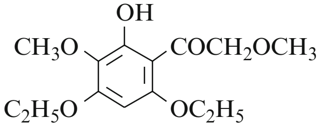
– Also obtained by alkaline degradation of 5,7-diethoxy-3,6,4′-trimethoxyflavone (SM) (m.p. 123–124°) with sodium hydroxide in refluxing dilute ethanol for

20 h under nitrogen (71%). SM was prepared by ethylation of 5,7-dihydroxy-3,6,4′-trimethoxyflavone (m.p. 164–165°), itself isolated from the leaves and terminal branches of *Dodonaea attenuata* var. linearis [4994].

m.p. 65–66° [4994], 60–62° [4854], 57–58° [4867], 56–57° [4993];
b.p.$_{0.2}$ 145–146° [4918,4917]; ^{1}H NMR [4854,4993], IR [4918].

1-(4,6-Diethoxy-2-hydroxy-3-methoxyphenyl)-2-methoxyethanone

[5128-46-1] $C_{14}H_{20}O_6$ mol.wt. 284.31



Syntheses

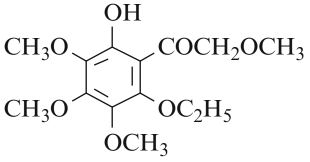
– Obtained by alkaline degradation of some polyalcoxy-flavones with potassium hydroxide,

- From 5,7-diethoxy-3,8,4′-trimethoxyflavone (m.p. 106–108°) (SM) (91%). SM was obtained by ethylation of 5,7-dihydroxy-3,8,4′-trimethoxyflavone (m.p. 173–175°), itself isolated from *Beyeria* sp [4854];
- From 5,7,3′-triethoxy-3,8,4′,5′-tetramethoxyflavone (m.p. 138–139°) (89%) [4967];
- From 5,7,4′-triethoxy-3,8-dimethoxyflavone (m.p. 128–129°) (71%) [4981];
- From 5,7,4′-triethoxy-3,8,3′-trimethoxyflavone (m.p. 110–111°) (23%) [4981].

m.p. 125–126° [4981], 124–125° [4854,4967]; ^{1}H NMR [4854].

1-(2-Ethoxy-6-hydroxy-3,4,5-trimethoxyphenyl)-2-methoxyethanone

$C_{14}H_{20}O_7$ mol.wt. 300.31



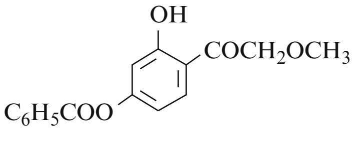
Synthesis

– Obtained by alkaline degradation of *Calycopterin diethyl ether* (m.p. 131–132°) [4982], so called *Thapsin diethyl ether* (m.p. 130°) [4983] (5,4′-diethoxy-3,6,7,8-tetramethoxyflavone) with refluxing ethanolic potash [4982], (86%) [4983].

m.p. 75–77° [4982], 63–64° [4983].

1-[(4-Benzoyloxy)-2-hydroxyphenyl]-2-methoxyethanone

$C_{16}H_{14}O_5$ mol.wt. 286.28



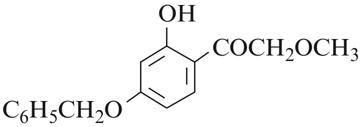
Synthesis

– Formed (by-product) by simple hydrolysis of 2,4-di-benzoyloxy-α-methoxyacetophenone with potassium ethoxide in pyridine at r.t. for 1 min (11%) [4846].

m.p. 122° [4846].

1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-2-methoxyethanone

[62952-90-3]



$C_{16}H_{16}O_4$ mol.wt. 272.30

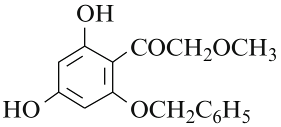
Synthesis

- Obtained by partial benzylation of α-methoxy-resacetophenone with benzyl chloride in the presence of potassium carbonate and potassium iodide in refluxing acetone for 5 h (47%) [4839].

m.p. 67–68° [4839].

1-[2,4-Dihydroxy-(6-phenylmethoxy)phenyl]-2-methoxyethanone

[62952-93-6]



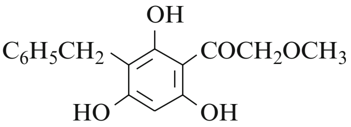
$C_{16}H_{16}O_5$ mol.wt. 288.30

Synthesis

- Preparation in one pot by tosylation of α-methoxy-phloracetophenone with 2 mol of p-toluenesulfonyl chloride, subsequent benzylation and final detosylation (35%) [4839].

m.p. 227–228° [4839].

1-[2,4,6-Trihydroxy-3-(phenylmethyl)phenyl]-2-methoxyethanone



$C_{16}H_{16}O_5$ mol.wt. 288.30

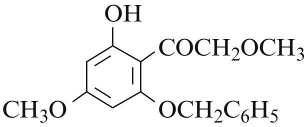
Synthesis

- Obtained by total hydrogenolysis of 1-[2-hydroxy-4,6-bis(phenylmethoxy)-3-(phenylmethyl)phenyl]-2-methoxyethanone in methanol in the presence of Pd/C under hydrogen atmosphere [4995].

m.p. 98° (monohydrate) [4995]; IR [4995], UV [4995].

1-[2-Hydroxy-4-methoxy-6-(phenylmethoxy)phenyl]-2-methoxyethanone

[62952-92-5]



$C_{17}H_{18}O_5$ mol.wt. 302.33

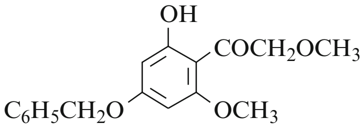
Synthesis

- Preparation by partial methylation of 6-(benzyloxy)-2,4-di-hydroxy-α-methoxyacetophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 4 h (86%) [4839].

m.p. 22–124° [4839].

1-[2-Hydroxy-6-methoxy-4-(phenylmethoxy)phenyl]-2-methoxyethanone

[62952-91-4]



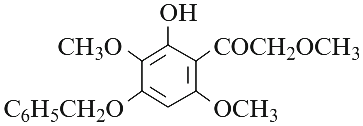
$C_{17}H_{18}O_5$ mol.wt. 302.33

Synthesis

- Obtained by reaction of benzyl chloride with 2,4-di-hydroxy-6,α-dimethoxyacetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone for 5 h [4839].

m.p. 101–102° [4839].

1-[2-Hydroxy-3,6-dimethoxy-4-(phenylmethoxy)phenyl]-2-methoxyethanone



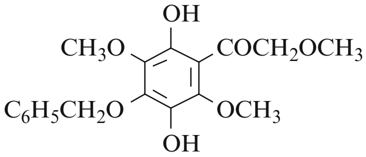
$C_{18}H_{20}O_6$ mol.wt. 332.35

Syntheses

- Obtained (by-product) during the condensation of methoxyacetonitrile with 1,3-bis(benzyloxy)-2,5-di-methoxybenzene (Hoesch reaction) (32%) [4941].
- Also refer to: [4865,4942,4996].

m.p. 109–110° [4941].

1-[2,5-Dihydroxy-3,6-dimethoxy-4-(phenylmethoxy)phenyl]-2-methoxyethanone



$C_{18}H_{20}O_7$ mol.wt. 348.35

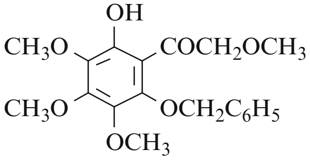
Syntheses

- Obtained by oxidation of 4-(benzyloxy)-2-hydroxy-3,6,α-trimethoxyacetophenone in alkaline solution with potassium persulfate (Elbs reaction) (10%) [4941].
- Also refer to: [4997].

deep yellow viscous oil [4941].

1-[2-Hydroxy-6-(phenylmethoxy)-3,4,5-trimethoxyphenyl]-2-methoxyethanone

[94385-86-1]



$C_{19}H_{22}O_7$ mol.wt. 362.38

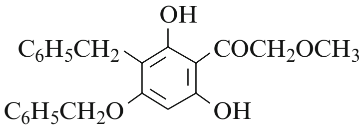
Synthesis

- Obtained by alkaline degradation of *Digicitrine dibenzylether*[5,3′-bis(benzyloxy)-3,6,7,8,4′,5′-hexamethoxyflavone] (m.p. 75–76°) with potassium hydroxide in refluxing 80% ethanol for 3 h (85%) [4986].

m.p. 64°5–65° [4986]; UV [4986].

1-[2,6-Dihydroxy-4-(phenylmethoxy)-3-(phenylmethyl)phenyl]-2-methoxyethanone

[18074-51-6]



$C_{23}H_{22}O_5$ mol.wt. 378.42

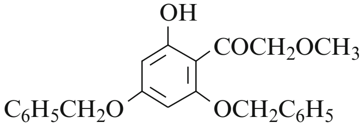
Synthesis

- Obtained from 1-[2-hydroxy-4,6-bis(phenylmethoxy)-3-(phenylmethyl)phenyl]-2-methoxyethanone by partial hydrogenolysis in methanol in the presence of Pd/C [4995].

m.p. 203–205° [4995]; IR [4995], UV [4995].

1-[2-Hydroxy-4,6-bis(phenylmethoxy)phenyl]-2-methoxyethanone

[18074-53-8]



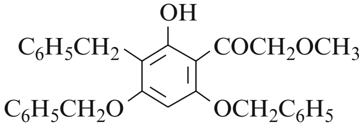
$C_{23}H_{22}O_5$ mol.wt. 378.42

Synthesis

- Obtained (poor yield) by reaction of benzyl chloride with α-methoxyphloracetophenone in the presence of potassium carbonate in refluxing acetone (5%) [4995].

m.p. 124° [4995]; IR [4995], UV [4995].

1-[2-Hydroxy-4,6-bis(phenylmethoxy)-3-(phenylmethyl)phenyl]-2-methoxyethanone



$C_{30}H_{28}O_5$ mol.wt. 468.55

Synthesis

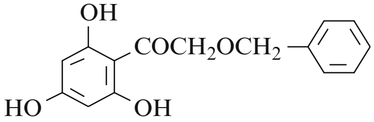
- Preparation by benzylation of α-methoxyphlor-acetophenone with benzyl chloride in the presence of sodium iodide and potassium carbonate in boiling acetone for 3 h (34%) [4995].

m.p. 147–148° [4995]; IR [4995], UV [4995].

13.2 Compounds Derived from Phenylmethoxyacetic Acids

2-(Phenylmethoxy)-1-(2,4,6-trihydroxyphenyl)ethanone

[322405-72-1]

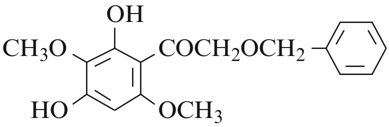


$C_{15}H_{14}O_5$ mol.wt. 274.27

Synthesis

- Preparation by reaction of benzyloxyacetonitrile with phloroglucinol (Hoesh reaction) (91%) [4998].

1-(2,4-Dihydroxy-3,6-dimethoxyphenyl)-2-(phenylmethoxy)ethanone



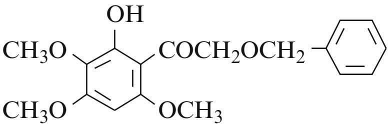
$C_{17}H_{18}O_6$ mol.wt. 318.33

Synthesis

- Obtained by hydrogenation of 2-(benzyloxy)-1-[4-(benzyloxy)-2-hydroxy-3,6-dimethoxy-phenyl]ethanone in ethyl acetate over Pd/C [4942].

m.p. 175–176° [4942].

1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-2-(phenylmethoxy)ethanone



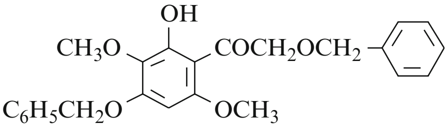
$C_{18}H_{20}O_6$ mol.wt. 332.35

Synthesis

- Obtained by partial methylation of 2-(benzyloxy)-1-(2,4-dihydroxy-3,6-dimethoxyphenyl)ethanone with diazomethane [4942].

m.p. 172–174° [4942].

1-[2-Hydroxy-3,6-dimethoxy-4-(phenylmethoxy)phenyl]-2-(phenylmethoxy) ethanone



$C_{24}H_{24}O_6$ mol.wt. 408.45

Synthesis

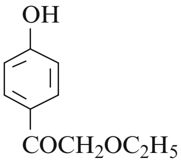
- Obtained by reaction of (benzyloxy)-acetonitrile with 2,5-dimethoxyresorcinol dibenzyl ether (Hoesch reaction) (33%) [4942].

m.p. 150–151° [4942].

13.3 Compounds Derived from Ethoxyacetic Acids

2-Ethoxy-1-(4-hydroxyphenyl)ethanone

[91061-33-5]



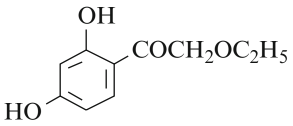
$C_{10}H_{12}O_3$ mol.wt. 180.20

Synthesis

- Obtained by adding ethanol (0.1 ml) and boron trifluoride etherate to a suspension of resin **6** (52 mg) in methylene chloride, and stirring the mixture at r.t. for 1 h. The compound was isolated by usual method and purified by preparative TLC (52%) [4814].

N.B.: Resin **6** (resin-bound α-TMS diazoketone **6**) (preparation given).
^{1}H NMR [4814], ^{13}C NMR [4814], IR [4814], MS [4814].

1-(2,4-Dihydroxyphenyl)-2-ethoxyethanone



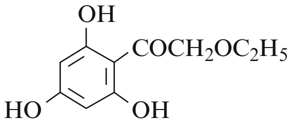
$C_{10}H_{12}O_4$ mol.wt. 196.20

Synthesis

- Obtained by reaction of ethoxyacetonitrile with resorcinol [4999], (97%) [4433], (28%) (Hoesch reaction) [5000].

m.p. 136–137° [4433], 135–136° [5000], 132–133° [4999];
b.p.$_{10}$ 195–200° [5000].

2-Ethoxy-1-(2,4,6-trihydroxyphenyl)ethanone



$C_{10}H_{12}O_5$ mol.wt. 212.201

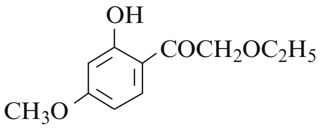
Syntheses

- Preparation by reaction of ethoxyacetonitrile with phloroglucinol (Hoesch reaction) (64%) [5001].
- Also refer to: [5000,5002].

m.p. 197–198° [5001].

2-Ethoxy-1-(2-hydroxy-4-methoxyphenyl)ethanone

[34811-99-9]



$C_{11}H_{14}O_4$ mol.wt. 210.23

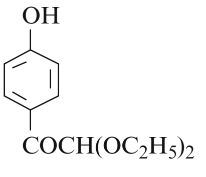
Synthesis

- Obtained by partial methylation of α-ethoxy-2,4-dihydroxy-acetophenone (SM) with diazomethane in ethyl ether [5003], (18%) [4999]. SM was prepared by reaction of ethoxyacetonitrile with resorcinol (Hoesch reaction) [5003].

oil [5003]; m.p. 30–31° [4999]; b.p.$_{0.02}$ 80–85° [4999]; ^{1}H NMR [4999], IR [4999].

2,2-Diethoxy-1-(4-hydroxyphenyl)ethanone

[200420-28-6]

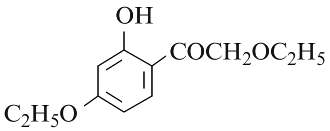


$C_{12}H_{16}O_4$ mol.wt. 224.26

Synthesis

- Refer to: [5004] (Japanese patent).

2-Ethoxy-1-(4-ethoxy-2-hydroxyphenyl)ethanone



$C_{12}H_{16}O_4$ mol.wt. 224.26

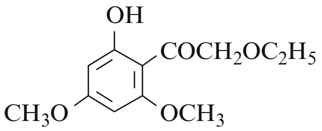
Synthesis

- Obtained by treatment of *fisetin* tetraethyl ether (3,7,3′,4′-tetraethoxyflavone) with boiling alcoholic potassium hydroxide solution [4837,4885,4886,4888].

m.p. 42–44° [4886].

2-Ethoxy-1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone

[21587-57-5]



$C_{12}H_{16}O_5$ mol.wt. 240.26

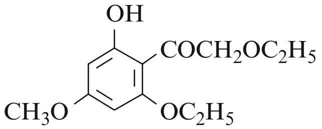
Syntheses

- Obtained by partial methylation of α-ethoxyphloro-acetophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 12 h (62%) [5000].
- Also obtained by degradation of 3-ethoxy-5,7,3′,4′-tetramethoxyflavone with sodium hydroxide in boiling dilute ethanol for 16 h (9%) [4927].

m.p. 103–104° [5000], 99–100° [4927].

2-Ethoxy-1-(2-ethoxy-6-hydroxy-4-methoxyphenyl)ethanone

[21587-58-6]



$C_{13}H_{18}O_5$ mol.wt. 254.28

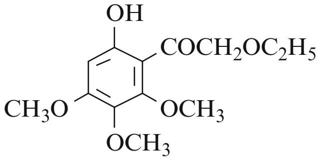
Synthesis

- Obtained (poor yield) by degradation of 3,5-diethoxy-7,3′,4′-trimethoxyflavone (m.p. 164–165°) with sodium hydroxide in refluxing ethanol (3%) [4927].

m.p. 82–83° [4927]; IR [4927].

2-Ethoxy-1-(6-hydroxy-2,3,4-trimethoxyphenyl)ethanone

[19598-24-4]



$C_{13}H_{18}O_6$ mol.wt. 270.28

Synthesis

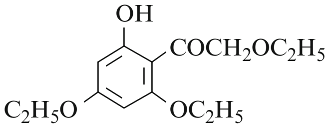
- Obtained by alkaline degradation of *Eupatoretin* diethyl ether (m.p. 119–120°) (3,3′-diethoxy-5,6,7,4′-tetramethoxy-flavone) with potassium hydroxide in refluxing ethanol under nitrogen for 17 h (46%) [5005].

m.p. 60–61° [5005]; ^{1}H NMR [5005], IR [5005], UV [5005], MS [5005].

1-(2,4-Diethoxy-6-hydroxyphenyl)-2-ethoxyethanone

[64184-96-9]

$C_{14}H_{20}O_5$ mol.wt. 268.31



Syntheses

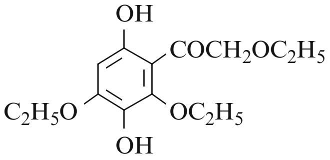
- Obtained by partial ethylation of α-ethoxyphloro-acetophenone with ethyl iodide in the presence of potassium carbonate in refluxing acetone for 12 h (53%) [5001].
- Also obtained by degradation of various polyethoxyflavones with boiling ethanolic potash,
  - From 3,5,7,3′-tetraethoxy-4′-methoxyflavone (m.p. 136–137°) (8% potassium hydroxide, reflux 6 h) [5002];
  - From 3,5,7,3′,4′-pentaethoxyflavone (*quercetin* pentaethyl ether) (7% potassium hydroxide, reflux 6 h) (good yield) [5001];
  - From 3,5,7,3′,5′-pentaethoxy-4′-methoxyflavone (m.p. 160°) (*4′-methylmyricetin pentaethyl ether*) (SM). SM was obtained by total ethylation of *4′-methylmyricetin*, itself isolated from the leaves of *Elaeocarpus lanceofolius* Roxb. (Elaeocarpaceae) [5006,5007];
  - From 3,5,7,3′,4′,5′-hexaethoxyflavone (m.p. 149–151°) (*myricetin* hexaethyl ether) [4889,5006]. *Myricetin* is the 3,5,7,3′,4′,5′-hexahydroxyflavone.

N.B.: Na salt [5001].

m.p. 97–98° [5002], 96–97° [4889,5001], 96° [5006];
^{1}H NMR [5006], UV [5006], MS [5006].

1-(2,4-Diethoxy-3,6-dihydroxyphenyl)-2-ethoxyethanone

$C_{14}H_{20}O_6$ mol.wt. 284.31



Synthesis

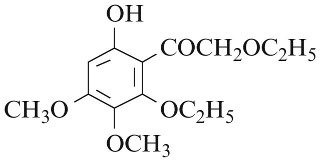
- Obtained by reaction of potassium persulfate with 2-hydroxy-4,6,α-triethoxyacetophenone in 5% aqueous sodium hydroxide, first at 15°, then at r.t. for 20 h (30%) (Elbs reaction) [5001].

m.p. 103–104° [5001].

2-Ethoxy-1-(2-ethoxy-6-hydroxy-3,4-dimethoxyphenyl)ethanone

[4324-56-5]

$C_{14}H_{20}O_6$ mol.wt. 284.31



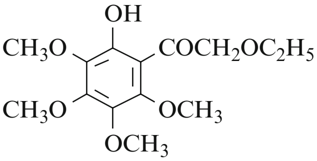
Syntheses

- Obtained by alkaline degradation of various substituted flavones with potassium hydroxide in refluxing ethanol,

- From *Mikanin* diethyl ether (m.p. 94–95°) (3,5-diethoxy-6,7,4′-trimethoxyflavone) [4867];
- From *Eupatin* triethyl ether (m.p. 105–106°) (3,5,3′-tri-ethoxy-6,7,4′-trimethoxyflavone) (26%) [5005];
- From *Eupalitin* triethyl ether (m.p. 80–81°) (3,5,4′-triethoxy-6,7-dimethoxyflavone) (88%) [5008];
- From *Eupatolitin* tetraethyl ether (m.p. 120–121°) (3,5,3′,4′-tetraethoxy-6,7-dimethoxyflavone) (97%) [5008].

m.p. 61–62° [4867], 59–60° [5005,5008];
^{1}H NMR [5005], IR [5008], UV [5005], MS [5005,5008].

2-Ethoxy-1-(2-hydroxy-3,4,5,6-tetramethoxyphenyl)ethanone



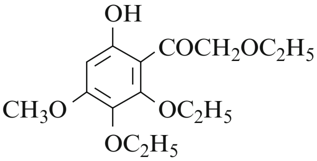
$C_{14}H_{20}O_7$ mol.wt. 300.31

Synthesis

– Obtained by alkaline degradation of *Natsudaidain ethyl ether* (3-ethoxy-5,6,7,8,3′,4′-hexamethoxyflavone) (m.p. 118°) with potassium hydroxide in refluxing ethanol [4988].

m.p. 47° [4988]; ^{1}H NMR [4988], IR [4988], MS [4988].

2-Ethoxy-1-(2,3-diethoxy-6-hydroxy-4-methoxyphenyl)ethanone

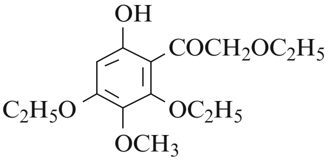


$C_{15}H_{22}O_6$ mol.wt. 298.34

Synthesis

– Refer to: [5001].

2-Ethoxy-1-(2,4-diethoxy-6-hydroxy-3-methoxyphenyl)ethanone



$C_{15}H_{22}O_6$ mol.wt. 298.34

Syntheses

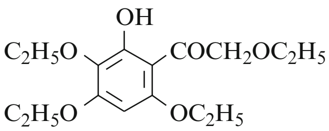
– Obtained from ethyl 3,5,7,3′,4′-pentaethoxy-6-methoxy-flavone-2′-carboxylate (m.p. 111–112°) (SM) by hydrolysis with 20% ethanolic potassium hydroxide at reflux for 8 h (73%). SM was obtained by ethylation of *distemonanthin*, itself isolated from the wood of *distemonanthus benthamianus* [4844].

– Also obtained by alkaline degradation of *patuletin* pentaethyl ether (3,5,7,3′,4′-pentaethoxy-6-methoxyflavone) (m.p. 127–128°) with refluxing 7% ethanolic potash on a water bath for 6 h (40%) [5001].

- Also obtained by partial methylation of α,4,6-triethoxy-2,5-dihydroxyacetophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing benzene for 12 h (38%) [5001].

m.p. 88–89° [4844], 86–87° [5001].

2-Ethoxy-1-(2-hydroxy-3,4,6-triethoxyphenyl)ethanone *(Gossypitol tetraethyl ether)*



$C_{16}H_{24}O_6$ mol.wt. 312.36

Synthesis

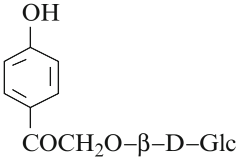
- Obtained by alkaline degradation of *Gossypetin* hexaethyl ether (m.p. 144–146°) (3,5,7,8,3′,4′-hexaethoxyflavone) with potassium hydroxide in refluxing dilute ethanol for 6 h (84%) [4964].

m.p. 110–111° [4964].

13.4 Miscellaneous

2-(β-D-Glucopyranosyloxy)-1-(4-hydroxyphenyl)ethanone

[167638-61-1]



$C_{14}H_{18}O_8$ mol.wt. 314.29

Isolation from natural sources

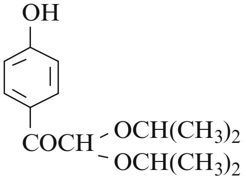
- From the fresh root bark of *Picea abies* (Pinaceae) (compound 4) [5009].

$(\alpha)_D = -33°$ (c = 0.2 methanol) [5009];

^{1}H NMR [5009], ^{13}C NMR [5009], UV [5009].

1-(4-Hydroxyphenyl)-2,2-bis(1-methylethoxy)ethanone

[144757-80-2]



$C_{14}H_{20}O_4$ mol.wt. 252.31

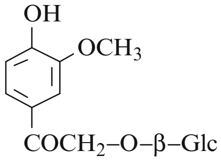
Syntheses

- Obtained by gradually adding a 33% hydrogen chloride solution in isopropanol to a solution of p-hydroxyphenyl-glyoxal and isopropyl nitrite in isopropanol cooled to 0°. Hydrogen chloride solution was added at such a speed to maintain a temperature of less than 25° [4901].
- Also refer to: [4900].

solid [4901]; ^{1}H NMR [4901], ^{13}C NMR [4901], MS [4901].

2-(β-D-Glucopyranosyloxy)-1-(4-hydroxy-3-methoxyphenyl)ethanone

[178959-37-0] $C_{15}H_{20}O_9$ mol.wt. 344.32

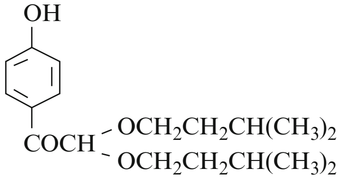


Isolation from natural sources

- From inner bark of *Pinus sylvestris* (compound 4) [5010].

1-(4-Hydroxyphenyl)-2,2-bis(3-methylbutoxy)ethanone

[144757-79-9] $C_{18}H_{28}O_4$ mol.wt. 308.42



Syntheses

- Obtained by slowly adding isoamyl nitrite to a solution of p-hydroxyacetophenone in isoamyl alcohol acidified with anhydrous hydrogen chloride at temperature <25° (62%) [4901].
- Also refer to: [4900].

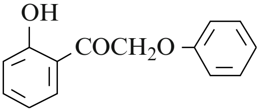
^1H NMR [4901], ^{13}C NMR [4901], MS [4901].

Chapter 14
Compounds Derived from Aryloxyacetic Acids

14.1 Compounds Derived from Phenoxyacetic Acid

1-(2-Hydroxyphenyl)-2-phenoxyethanone

[73014-18-3] $C_{14}H_{12}O_3$ mol.wt. 228.25



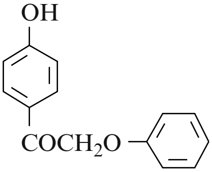
Syntheses

- Preparation by hydrogenolysis of 2-benzyloxy-α-phenoxyacetophenone in ethanol/dioxane (1:1) in the presence of Pd/C under hydrogen (85%) [5011].
- Also obtained by acidic hydrolysis of 4-hydroxy-3-phenoxycoumarin (m.p. 216°) [5012] according to [5013].
- Also prepared by reaction of phenoxyacetonitrile with phenol (Hoesch reaction) [5014].
- Also refer to: [5015].

m.p. 115–115°5 [5011], 115° [5012]; UV [5011].

1-(4-Hydroxyphenyl)-2-phenoxyethanone

[41978-29-4] $C_{14}H_{12}O_3$ mol.wt. 228.25



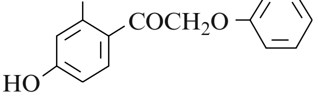
Synthesis

- Refer to: [5016,5017] (Japanese papers).

m.p. 159–160° [5017]; b.p.$_2$ 212–220° [5017].

1-(2,4-Dihydroxyphenyl)-2-phenoxyethanone

[73014-19-4] $C_{14}H_{12}O_4$ mol.wt. 244.25



Syntheses

- Obtained by reaction of phenoxyacetonitrile with resorcinol (Hoesch reaction) [4433,5018–5020], (88%) [5021], (82%) [5011].

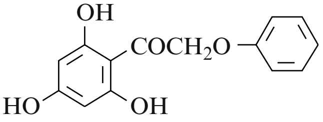
R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_14, © Springer Science+Business Media B.V. 2011

- Also refer to: [5022,5023].

m.p. 209°5–210° [5011], 207–208° [5021], 204–205° [4433];
^{1}H NMR [5021], UV [5011].

2-Phenoxy-1-(2,4,6-trihydroxyphenyl)ethanone

[72023-07-1] $C_{14}H_{12}O_5$ mol.wt. 260.25



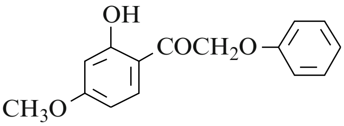
Syntheses

- Preparation by reaction of phenoxyacetonitrile with phloroglucinol (Hoesch reaction), (84%) [5011], (80%) [5024], (48%) [5025].
- Also refer to: [5026–5028].

m.p. 275° (d) [5025], 244–245° [5011], 234° [5024].
One note a very large dispersion of the various melting points.
^{1}H NMR [5024], UV [5011].

1-(2-Hydroxy-4-methoxyphenyl)-2-phenoxyethanone

[73023-08-2] $C_{15}H_{14}O_4$ mol.wt. 258.27



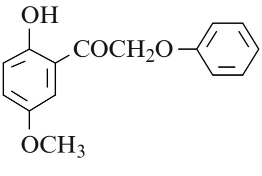
Syntheses

- Obtained by partial methylation of 2,4-dihydroxy-α-phenoxyacetophenone with dimethyl sulfate in the presence of potassium carbonate in boiling acetone for 2.5 h (83%) [5011].
- Also refer to: [5019].

m.p. 86°5–87° [5011]; UV [5011].

1-(2-Hydroxy-5-methoxyphenyl)-2-phenoxyethanone

[137612-24-9] $C_{15}H_{14}O_4$ mol.wt. 258.27

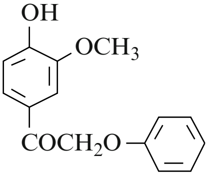


Syntheses

- Obtained by acylation of hydroquinone monomethyl ether with phenoxyacetonitrile in the presence of boron trichloride and aluminium chloride in ethylene dichloride (44%) [5029].
- Also refer to: [5014].

1-(4-Hydroxy-3-methoxyphenyl)-2-phenoxyethanone

[41978-28-3] $C_{15}H_{14}O_4$ mol.wt. 258.27



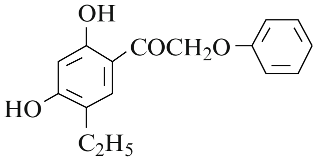
Syntheses

- Obtained (by-product, unusual result) by methylation of 2-(3-chlorophenoxy)-1-(4-hydroxy-3-methoxyphenyl)-ethanone [4429].
- Also refer to: [5017] (Japanese paper).

m.p. 94°5–95° [5017]; b.p.$_2$ 218–225° [5017].

1-(5-Ethyl-2,4-dihydroxyphenyl)-2-phenoxyethanone

[243465-56-7]



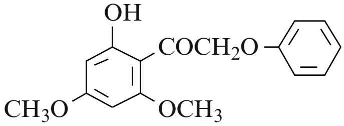
$C_{16}H_{16}O_4$ mol.wt. 272.30

Synthesis

- Obtained [5022] by previously described methods [5026].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-phenoxyethanone

[73023-09-3]



$C_{16}H_{16}O_5$ mol.wt. 288.30

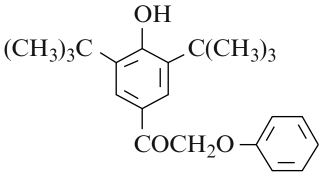
Synthesis

- Obtained by partial methylation of 2,4,6-trihydroxy-α-phenoxyacetophenone with dimethyl sulfate in the presence of potassium carbonate in boiling acetone for 8 h (63%) [5011].

m.p. 122–123° [5011]; UV [5011].

1-[3,5-Bis-(1,1-dimethylethyl)-4-hydroxyphenyl]-2-phenoxyethanone

[41978-27-2]



$C_{22}H_{28}O_3$ mol.wt. 340.46

Synthesis

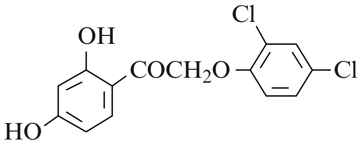
- Refer to: [5017] (Japanese paper).

m.p. 111°5–112° [5017].

14.2 Compounds Derived from Substituted Phenoxyacetic Acids

2-(2,4-Dichlorophenoxy)-1-(2,4-dihydroxyphenyl)ethanone

[137987-83-8]



$C_{14}H_{10}Cl_2O_4$ mol.wt. 313.14

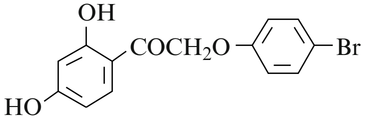
Synthesis

- Obtained by reaction of 2,4-dichlorophenoxy-acetonitrile with resorcinol (Hoesch reaction) (85%) [5021].

m.p. 192° [5021]; ^{1}H NMR [5021].

2-(4-Bromophenoxy)-1-(2,4-dihydroxyphenyl)ethanone

[243465-55-6] $C_{14}H_{11}BrO_4$ mol.wt. 323.14

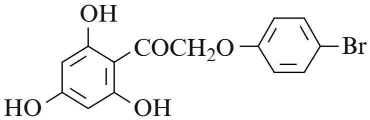


Synthesis

- Obtained [5022] by previously described method [5026].

2-(4-Bromophenoxy)-1-(2,4,6-trihydroxyphenyl)ethanone

[243657-60-5] $C_{14}H_{11}BrO_5$ mol.wt. 339.14



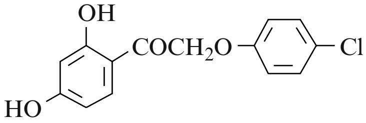
Synthesis

- Preparation by Hoesch condensation of p-bromo-phenoxyacetonitrile with phloroglucinol in benzene/ ethyl ether in the presence of zinc chloride and hydrogen chloride at 0° (82%) [5024].

m.p. 256° [5024]; ^{1}H NMR [5024].

2-(4-Chlorophenoxy)-1-(2,4-dihydroxyphenyl)ethanone

[115781-11-8] $C_{14}H_{11}ClO_4$ mol.wt. 278.69



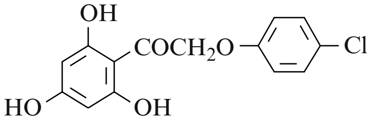
Syntheses

- Obtained by reaction of 4-chlorophenoxyacetonitrile with resorcinol (Hoesch reaction) (84%) [5021].
- Also refer to: [5030] (Japanese patent).

m.p. 187°5 [5021]; ^{1}H NMR [5021].

2-(4-Chlorophenoxy)-1-(2,4,6-trihydroxyphenyl)ethanone

[243657-59-2] $C_{14}H_{11}ClO_5$ mol.wt. 294.69



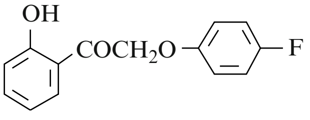
Synthesis

- Preparation by Hoesch condensation of p-chloro-phenoxyacetonitrile with phloroglucinol in benzene/ethyl ether in the presence of zinc chloride and hydrogen chloride at 0° (85%) [5024].

m.p. 251° [5024]; ^{1}H NMR [5024].

2-(4-Fluorophenoxy)-1-(2-hydroxyphenyl)ethanone

[137612-30-7] $C_{14}H_{11}FO_3$ mol.wt. 246.24

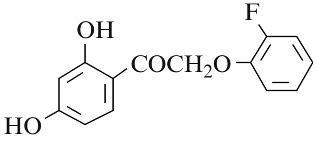


Syntheses

- Obtained by reaction of p-fluorophenoxyacetonitrile with phenol (Hoesch reaction) [5014].
- Also refer to: [5029].

1-(2,4-Dihydroxyphenyl)-2-(2-fluorophenoxy)ethanone

[137987-82-7] $C_{14}H_{11}FO_4$ mol.wt. 262.24



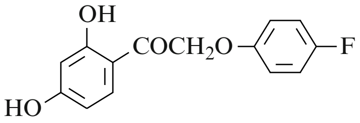
Synthesis

– Obtained by reaction of 2-fluorophenoxyacetonitrile with resorcinol (Hoesch reaction) (87%) [5021].

m.p. 163° [5021]; ^{1}H NMR [5021].

1-(2,4-Dihydroxyphenyl)-2-(4-fluorophenoxy)ethanone

[121361-56-6] $C_{14}H_{11}FO_4$ mol.wt. 262.24



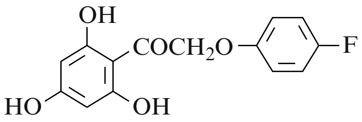
Syntheses

– Obtained by reaction of p-fluorophenoxyacetonitrile with resorcinol (Hoesch reaction), (86%) [5021], (63%) [5031].
– Also refer to: [5020,5022].

m.p. 165° [5021,5031]; ^{1}H NMR [5021,5031], IR [5031].

2-(4-Fluorophenoxy)-1-(2,4,6-trihydroxyphenyl)ethanone

[127526-42-5] $C_{14}H_{11}FO_5$ mol.wt. 278.24



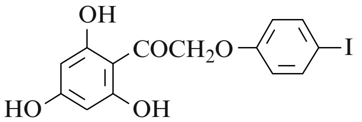
Synthesis

– Preparation by Hoesch condensation of p-fluoro-phenoxyacetonitrile with phloroglucinol in benzene/ethyl ether in the presence of zinc chloride and hydrogen chloride at 0° (81%) [5024].

m.p. 242° [5024]; ^{1}H NMR [5024].

2-(4-Iodophenoxy)-1-(2,4,6-trihydroxyphenyl)ethanone

[243657-61-6] $C_{14}H_{11}IO_5$ mol.wt. 386.14



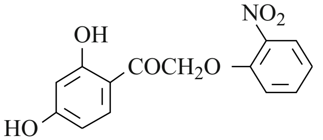
Synthesis

– Preparation by Hoesch condensation of p-iodophenoxy- acetonitrile with phloroglucinol in benzene/ethyl ether in the presence of zinc chloride and hydrogen chloride at 0° (80%) [5024].

m.p. 269° [5024]; ^{1}H NMR [5024].

1-(2,4-Dihydroxyphenyl)-2-(2-nitrophenoxy)ethanone

[137987-91-8] $C_{14}H_{11}NO_6$ mol.wt. 289.24



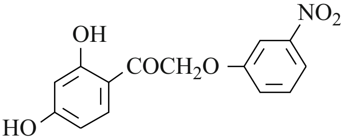
Synthesis

– Obtained by reaction of o-nitrophenoxyacetonitrile with resorcinol (Hoesch reaction) (84%) [5021].

m.p. 287° [5021]; ^{1}H NMR [5021].

1-(2,4-Dihydroxyphenyl)-2-(3-nitrophenoxy)ethanone

[137987-90-7]



$C_{14}H_{11}NO_6$ mol.wt. 289.24

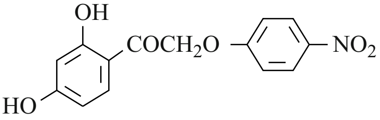
Synthesis

- Obtained by reaction of m-nitrophenoxyacetonitrile with resorcinol (Hoesch reaction) (81%) [5021].

m.p. 275° [5021]; ^{1}H NMR [5021].

1-(2,4-Dihydroxyphenyl)-2-(4-nitrophenoxy)ethanone

[137987-89-4]



$C_{14}H_{11}NO_6$ mol.wt. 289.24

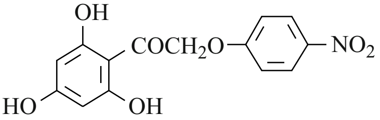
Synthesis

- Obtained by reaction of p-nitrophenoxyacetonitrile with resorcinol (Hoesch reaction) (86%) [5021].

m.p. 281° [5021]; ^{1}H NMR [5021].

2-(4-Nitrophenoxy)-1-(2,4,6-trihydroxyphenyl)ethanone

[243657-68-3]



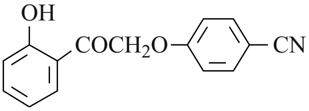
$C_{14}H_{11}NO_7$ mol.wt. 305.24

Synthesis

- Preparation by Hoesch condensation of p-nitro-phenoxyacetonitrile with phloroglucinol in benzene/ethyl ether in the presence of zinc chloride and hydrogen chloride at 0° (87%) [5024].

m.p. 295° [5024]; ^{1}H NMR [5024].

2-(4-Cyanophenoxy)-1-(2-hydroxyphenyl)ethanone



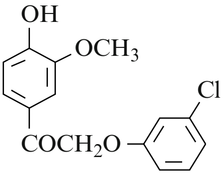
$C_{15}H_{11}NO_3$ mol.wt. 253.26

Syntheses

- Refer to: [5032,5033].

2-(3-Chlorophenoxy)-1-(4-hydroxy-3-methoxyphenyl)ethanone

[107602-85-7]



$C_{15}H_{13}ClO_4$ mol.wt. 292.72

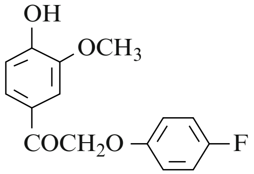
Synthesis

- Obtained by saponification of 1-(4-acetoxy-3-methoxyphenyl)-2-(3-chlorophenoxy)ethanone with sodium methoxide in refluxing methanol for 2.5 h (82%) [4429].

m.p. 118–119° [4429];
^{1}H NMR [4429], IR [4429], MS [4429].

2-(4-Fluorophenoxy)-1-(4-hydroxy-3-methoxyphenyl)ethanone

[104972-13-6] $C_{15}H_{13}FO_4$ mol.wt. 276.26



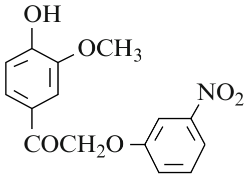
Synthesis

- Preparation by treatment of 1-(4-isopropoxy-3-methoxy-phenyl)-2-(4-fluorophenoxy)ethanone (viscous liquid) (1 mol) with aluminium chloride (4 mol) in benzene for 2 h at r.t. (95%) [5034].

^{1}H NMR [5034], ^{13}C NMR [5034],
^{19}F NMR [5034], IR [5034]; TLC [5034].

1-(4-Hydroxy-3-methoxyphenyl)-2-(3-nitrophenoxy)ethanone

[107584-70-3] $C_{15}H_{13}NO_6$ mol.wt. 303.27



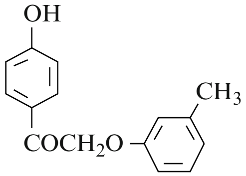
Synthesis

- Obtained by saponification of 1-(4-acetoxy-3-methoxy-phenyl)-2-(3-nitrophenoxy)ethanone (m.p. 130–133°) with sodium methoxide in refluxing methanol for 2.5 h (65%) [4429].

m.p. 187°5–189°5 [4429];
^{1}H NMR [4429], IR [4429], MS [4429].

1-(4-Hydroxyphenyl)-2-(3-methylphenoxy)ethanone

[107584-64-5] $C_{15}H_{14}O_3$ mol.wt. 242.27



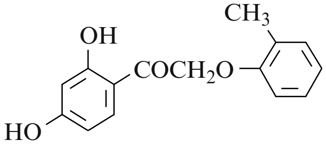
Synthesis

- Obtained by coupling 4-hydroxy-α-bromoacetophenone with m-cresol (44%) [4429].

m.p. 170°5–172°5 [4429];
^{1}H NMR [4429], IR [4429], MS [4429].

1-(2,4-Dihydroxyphenyl)-2-(2-methylphenoxy)ethanone

[137987-86-1] $C_{15}H_{14}O_4$ mol.wt. 258.27



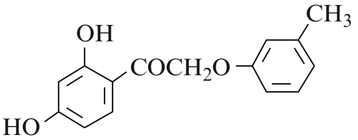
Synthesis

- Obtained by reaction of o-methylphenoxyacetonitrile with resorcinol (Hoesch reaction) (82%) [5021].

m.p. 182° [5021]; ^{1}H NMR [5021].

1-(2,4-Dihydroxyphenyl)-2-(3-methylphenoxy)ethanone

[137987-85-0]



$C_{15}H_{14}O_4$ mol.wt. 258.27

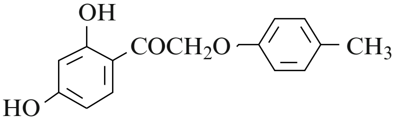
Synthesis

- Obtained by reaction of 3-methylphenoxyacetonitrile with resorcinol (Hoesch reaction) (82%) [5021].

m.p. 162° [5021]; ^{1}H NMR [5021].

1-(2,4-Dihydroxyphenyl)-2-(4-methylphenoxy)ethanone

[137987-84-9]



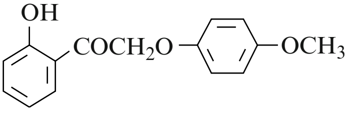
$C_{15}H_{14}O_4$ mol.wt. 258.27

Synthesis

- Obtained by condensation of (p-methylphenoxy)-acetonitrile with resorcinol in ethyl ether/benzene in the presence of zinc chloride according to Houben–Hoesch method (95%) [5018], (87%) [5021].

m.p. 171–172° [5018], 167° [5021]; ^{1}H NMR [5018,5021].

1-(2-Hydroxyphenyl)-2-(4-methoxyphenoxy)ethanone



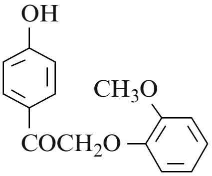
$C_{15}H_{14}O_4$ mol.wt. 258.27

Syntheses

- Refer to: [5032] (compound **1d**) and [5033].

1-(4-Hydroxyphenyl)-2-(2-methoxyphenoxy)ethanone

[143486-72-0]



$C_{15}H_{14}O_4$ mol.wt. 258.27

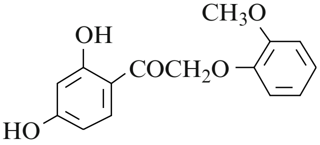
Synthesis

- Preparation by saponification of 1-(4-acetoxyphenyl)-2-(2-methoxyphenoxy)ethanone (SM) with potassium hydroxide in ethanol for 3 h at temperature <30° under nitrogen (90%) [5035]. SM was obtained by alkylation of sodium 2-methoxyphenoxide with p-acetoxy-α-bromo-acetophenone (Williamson synthesis) (96%, m.p. 67°5–68°5).

m.p. 159–160° [5035]; ^{1}H NMR [5035].

1-(2,4-Dihydroxyphenyl)-2-(2-methoxyphenoxy)ethanone

[137987-88-3]



$C_{15}H_{14}O_5$ mol.wt. 274.27

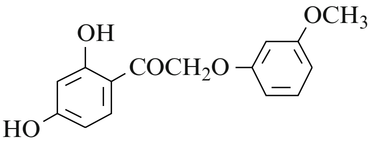
Synthesis

- Obtained by reaction of o-methoxyphenoxyacetonitrile with resorcinol (Hoesch reaction) (87%) [5021].

m.p. 193° [5021]; ^{1}H NMR [5021].

1-(2,4-Dihydroxyphenyl)-2-(3-methoxyphenoxy)ethanone

[137987-87-2]



$C_{15}H_{14}O_5$ mol.wt. 274.27

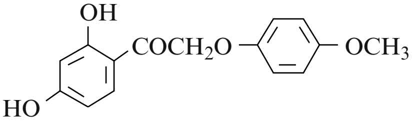
Synthesis

- Obtained by reaction of m-methoxyphenoxy-acetonitrile with resorcinol (Hoesch reaction) (85%) [5021].

m.p. 145° [5021]; ^{1}H NMR [5021].

1-(2,4-Dihydroxyphenyl)-2-(4-methoxyphenoxy)ethanone

[121361-55-5]



$C_{15}H_{14}O_5$ mol.wt. 274.27

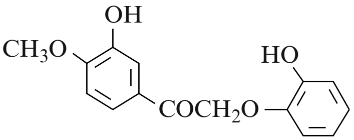
Syntheses

- Obtained by reaction of p-methoxyphenoxy-acetonitrile with resorcinol (Hoesch reaction) (89%) [5021], (73%) [5031].
- Also refer to: [4685].

m.p. 179° [4685,5031], 175° [5021];
^{1}H NMR [4685,5021,5031], IR [4685,5031].

1-(3-Hydroxy-4-methoxyphenyl)-2-(2-hydroxyphenoxy)ethanone

[99783-86-5]



$C_{15}H_{14}O_5$ mol.wt. 274.27

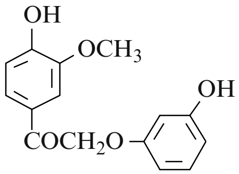
Syntheses

- Preparation by reaction of 3-hydroxy-4-methoxy-α-chloroacetophenone,
  - with pyrocatechol monosodium salt in DMF at r.t. for 3 h (51%) [4634];
  - with pyrocatechol in the presence of potassium carbonate in 2-butanone [5036].
- Also refer to: [5037].

m.p. 154° [4634]; ^{1}H NMR [4634], MS [4634].

1-(4-Hydroxy-3-methoxyphenyl)-2-(3-hydroxyphenoxy)ethanone

[107584-71-4] $C_{15}H_{14}O_5$ mol.wt. 274.27



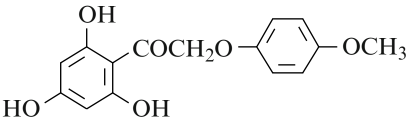
Syntheses

- Obtained by saponification of 1-(4-acetoxy-3-methoxy-phenyl)-2-(3-hydroxyphenoxy)ethanone with sodium methoxide in refluxing methanol for 2.5 h [4429].
- Also obtained by coupling reaction of 4-hydroxy-3-methoxy-α-bromoacetophenone with sodium m-acetoxyphenolate [4429].

m.p. 145–146° [4429]; ^{1}H NMR [4429], IR [4429], MS [4429].

2-(4-Methoxyphenoxy)-1-(2,4,6-trihydroxyphenyl)ethanone

[243657-65-0] $C_{15}H_{14}O_6$ mol.wt. 290.27



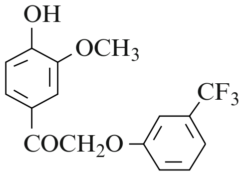
Synthesis

- Preparation by Hoesch condensation of p-methoxy-phenoxyacetonitrile with phloroglucinol in benzene/ethyl ether in the presence of zinc chloride and hydrogen chloride at 0° (78%) [5024].

m.p. 259° [5024]; ^{1}H NMR [5024].

1-(4-Hydroxy-3-methoxyphenyl)-2-[3-(trifluoromethyl)phenoxy]ethanone

[107584-69-0] $C_{16}H_{13}F_3O_4$ mol.wt. 326.27



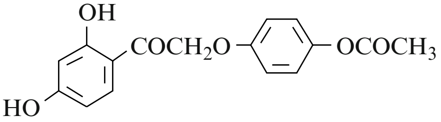
Synthesis

- Obtained by saponification of 1-(4-acetoxy-3-methoxy-phenyl)-2-[3-(trifluoromethyl)phenoxy]ethanone with sodium methoxide in refluxing methanol for 2.5 h (88%) [4429].

m.p. 107°5–108°5 [4429]; ^{1}H NMR [4429], IR [4429], MS [4429].

2-(4-Acetoxyphenoxy)-1-(2,4-dihydroxyphenyl)ethanone

[137987-93-0] $C_{16}H_{14}O_6$ mol.wt. 302.28

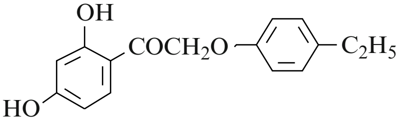


Synthesis

- Refer to: [5021].

1-(2,4-Dihydroxyphenyl)-2-(4-ethylphenoxy)ethanone

[201284-76-6] $C_{16}H_{16}O_4$ mol.wt. 272.30



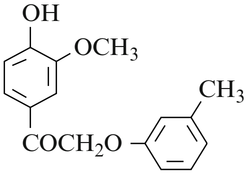
Synthesis

- Obtained by condensation of (p-ethylphenoxy)-acetonitrile with resorcinol in ethyl ether/benzene in the presence of zinc chloride according to Houben–Hoesch method (84%) [5018].

m.p. 147–148° [5018]; ^{1}H NMR [5018].

1-(4-Hydroxy-3-methoxyphenyl)-2-(3-methylphenoxy)ethanone

[107584-67-8] $C_{16}H_{16}O_4$ mol.wt. 272.30



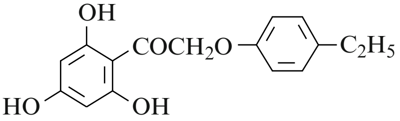
Synthesis

- Obtained by saponification of 1-(4-acetoxy-3-methoxy-phenyl)-2-(3-methylphenoxy)ethanone with sodium methoxide in refluxing methanol for 2.5 h (53%) [4429].

m.p. 170°5–172°5 [4429];
^{1}H NMR [4429], IR [4429], MS [4429].

2-(4-Ethylphenoxy)-1-(2,4,6-trihydroxyphenyl)ethanone

[243657-62-7] $C_{16}H_{16}O_5$ mol.wt. 288.30



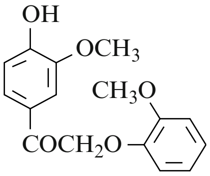
Synthesis

- Preparation by Hoesch condensation of p-ethyl-phenoxyacetonitrile with phloroglucinol in ethyl ether/benzene in the presence of zinc chloride and hydrogen chloride at 0° (88%) [5024].

m.p. 247° [5024]; ^{1}H NMR [5024].

1-(4-Hydroxy-3-methoxyphenyl)-2-(2-methoxyphenoxy)ethanone

[22317-35-7] $C_{16}H_{16}O_5$ mol.wt. 288.30



Syntheses

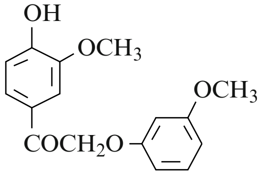
- Preparation by treatment of 1-(4-acetoxy-3-methoxy-phenyl)-2-(2-methoxyphenoxy)ethanone in chloroform solution with sodium methoxide in methanol, followed by acidification with dilute sulfuric acid (92%) [5038].
- Also obtained by hydrogenolysis of 1-[4-(benzyloxy)-3-methoxyphenyl]-2-(2-methoxyphenoxy)ethanone in methanol in the presence of 5% Pd on barium sulfate under hydrogen pressure (72%) [5039].

- Preparation by treatment of 1-(4-isopropoxy-3-methoxyphenyl)-2-(2-methoxyphenoxy)ethanone (m.p. 70°) (1 mol) with aluminium chloride (4 mol) in benzene for 2 h at r.t. (95%) [5034].
- Also obtained by oxidative degradation with 3% peracetic acid of 1-(4-hydroxy-3-methoxyphenyl)-2-(2-methoxyphenoxy)ethanol and 1-(3,4-dimethoxyphenyl)-2-(2-methoxyphenoxy)ethanol (used as softwood lignin model compounds) in 10% acetic acid or 50% ethanol for 48 h at 30° [5040].
- Also obtained by degradation of 1-(4-benzyloxy-3-methoxyphenyl)-2-(2-methoxyphenoxy)ethanol in the presence of *Aspergillus flavus*. Initial reactions in the degradation of this compound were oxidation of the α-hydroxy group to the corresponding ketone and debenzylation of the benzyloxy group [5041].
- Formation from kraft lignin in sulfate cooking [5042,5043].
- Also obtained from the cleavage of the β-ether bond in the guaiacylglycol-β-guaiacyl ether (SM) with the water solution of phthalocyanine complex trisodium tetra-4-sulfonatophthalocyanine-iron (III) (Fe(TSPc)). Fe(TSPc) catalyzed formation of oxidized products in the absence of oxygen. The radical derived from SM then undergoes further single electron oxidation and deprotonation to give the titled compound [5044].
- Also refer to: [5045–5051].

m.p. 94–95° [5039], 93° [5038], 65–67° [5043].
One the reported melting points is obviously wrong.
^{1}H NMR [5034,5039,5041], ^{13}C NMR [5034], IR [5034,5041], UV [5039], MS [5039,5041]; TLC [5034,5041]; GC [5041].

1-(4-Hydroxy-3-methoxyphenyl)-2-(3-methoxyphenoxy)ethanone

[107584-68-9] $C_{16}H_{16}O_5$ mol.wt. 288.30



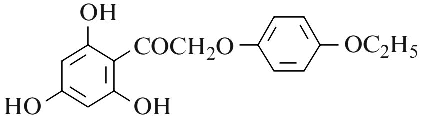
Synthesis

- Obtained by saponification of 1-(4-acetoxy-3-methoxy-phenyl)-2-(3-methoxyphenoxy)ethanone with sodium methoxide in refluxing methanol for 2.5 h (75%) [4429].

m.p. 109–110° [4429];
^{1}H NMR [4429], IR [4429], MS [4429].

2-(4-Ethoxyphenoxy)-1-(2,4,6-trihydroxyphenyl)ethanone

[243657-66-1] $C_{16}H_{16}O_6$ mol.wt. 304.30



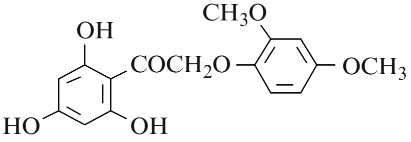
Synthesis

- Preparation by Hoesch condensation of p-ethoxyphenoxyacetonitrile with phloro-glucinol in benzene/ethyl ether in the presence of zinc chloride and hydrogen chloride at 0° (81%) [5024].

m.p. 223° [5024]; ^{1}H NMR [5024].

2-(2,4-Dimethoxyphenoxy)-1-(2,4,6-trihydroxyphenyl)ethanone

[149312-75-4]



$C_{16}H_{16}O_7$ mol.wt. 320.30

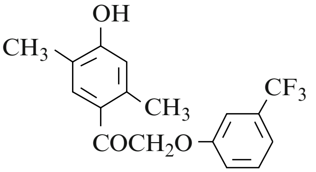
Synthesis

- Obtained by reaction of 2,4-dimethoxyphenoxy-acetonitrile with phloroglucinol in THF in the presence of zinc chloride and hydrogen chloride in an ice bath for 4 h (53%) (Hoesch reaction) [5025].

m.p. 105–107° [5025]; monohydrate [5025];
^{1}H NMR [5025], ^{13}C NMR [5025], IR [5025], UV [5025], MS [5025].

1-(4-Hydroxy-2,5-dimethylphenyl)-2-[3-(trifluoromethyl)phenoxy]ethanone

[107584-80-5]



$C_{17}H_{15}F_3O_3$ mol.wt. 324.30

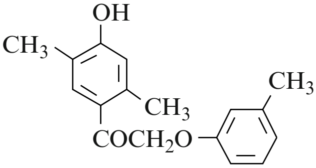
Synthesis

- Obtained by coupling 2,5-dimethyl-4-hydroxy-α-bromo-acetophenone with m-(trifluoromethyl)phenol (87%) [4429].

m.p. 191–194° [4429];
^{1}H NMR [4429], IR [4429], MS [4429].

1-(4-Hydroxy-2,5-dimethylphenyl)-2-(3-methylphenoxy)ethanone

[107584-79-2]



$C_{17}H_{18}O_3$ mol.wt. 270.33

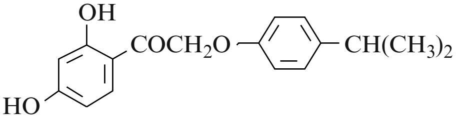
Synthesis

- Obtained by coupling 2,5-dimethyl-4-hydroxy-α-bromo-acetophenone with m-cresol (54%) [4429].

m.p. 188°5–190°5 [4429];
^{1}H NMR [4429], IR [4429], MS [4429].

1-(2,4-Dihydroxyphenyl)-2-[4-(1-methylethyl)phenoxy]ethanone

[201284-86-8]



$C_{17}H_{18}O_4$ mol.wt. 286.33

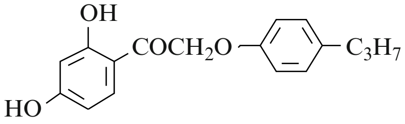
Synthesis

- Obtained by condensation of (p-isopropyl-phenoxy)acetonitrile with resorcinol in ethyl ether/benzene in the presence of zinc chloride according to Houben–Hoesch method (90%) [5018].

m.p. 167° [5018]; ^{1}H NMR [5018].

1-(2,4-Dihydroxyphenyl)-2-(4-propylphenoxy)ethanone

[201283-81-3] $C_{17}H_{18}O_4$ mol.wt. 286.33



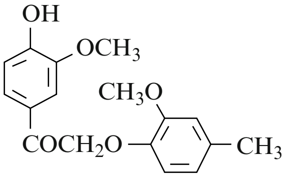
Synthesis

- Obtained by condensation of (p-propylphenoxy)-acetonitrile with resorcinol in ethyl ether/benzene in the presence of zinc chloride according to Houben–Hoesch method (88%) [5018].

m.p. 149–150° [5018]; ^{1}H NMR [5018].

1-(4-Hydroxy-3-methoxyphenyl)-2-(2-methoxy-4-methylphenoxy)ethanone

[152306-57-5]` $C_{17}H_{18}O_5$ mol.wt. 302.33



Synthesis

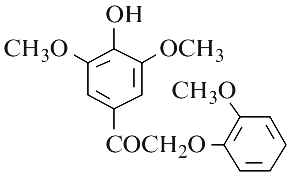
- Obtained by coupling α-bromoacetoguaiacone (1 equiv) with sodium creosolate (8 equiv) (compound 8) (19%) [5052].

m.p. 109–111° [5052];

^{1}H NMR [5052], ^{13}C NMR [5052], IR [5052], MS [5052].

1-(4-Hydroxy-3,5-dimethoxyphenyl)-2-(2-methoxyphenoxy)ethanone

[18167-90-3] $C_{17}H_{18}O_6$ mol.wt. 318.33



Syntheses

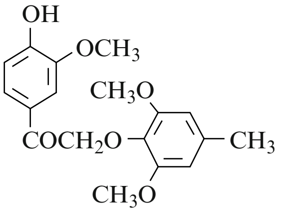
- Preparation by reaction of α-bromoacetosyringone (m.p. 130°) with guaiacol in the presence of sodium hydroxide in refluxing ethanol for 3 h (64%) [4511].
- From degradation of the lignin model com pound syringylglycol β-guaiacyl ether (m.p. 70–71°) (SM) by *Polyporus versicolor* and *Stereum frustulatum* [4511].

SM was obtained by reduction of α-guaiacoxyacetosyringone in ethyl acetate with hydrogen over 10% Pd/C or with sodium borohydride in isopropanol (almost quantitative yield).

m.p. 85–86° [4511].

2-(2,6-Dimethoxy-4-methylphenoxy)-1-(4-hydroxy-3-methoxyphenyl)ethanone

[105153-11-5] $C_{18}H_{20}O_6$ mol.wt. 332.35

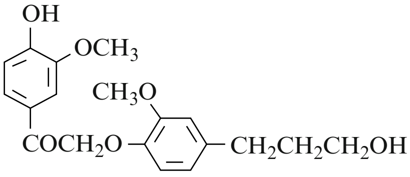


Synthesis

- Refer to: [5053].

1-(4-Hydroxy-3-methoxyphenyl)-2-[4-(3-hydroxypropyl)-2-methoxyphenoxy] ethanone

[120936-27-8]



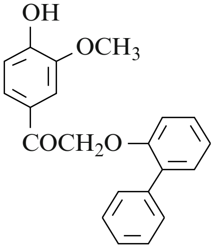
$C_{19}H_{22}O_6$ mol.wt. 346.38

Synthesis

- Refer to: [5054].

2-([1,1′-Biphenyl]-2-yloxy)-1-(4-hydroxy-3-methoxyphenyl)ethanone

[108434-12-4]



$C_{21}H_{18}O_4$ mol.wt. 334.37

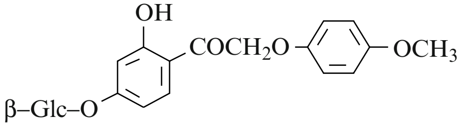
Synthesis

- Obtained by degradation of 1-(4-benzyloxy-3-methoxy-phenyl)-2-(2-phenylphenoxy)ethanol in the presence of *Aspergillus flavus*. Initial reactions in the degradation of this compound were oxidation of the α-hydroxy group to the corresponding ketone and debenzylation of the benzyloxy group [5041].

^{1}H NMR [5041], IR [5041], MS [5041];
TLC [5041]; GC [5041].

1-[4-(β-D-Glucopyranosyloxy)-2-hydroxyphenyl]-2-(4-methoxyphenoxy)ethanone

[121361-58-8]



$C_{21}H_{24}O_{10}$ mol.wt. 436.42

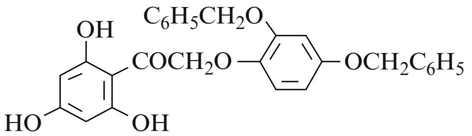
Synthesis

- Obtained by hydrolysis of 1-[2-hydroxy-4-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl) oxy]phenyl]-2-(4-methoxyphenoxy)ethanone with 2 N sodium hydroxide in refluxing dilute methanol for 30 min (93%) [5031].

m.p. 144° [5031]; $(\alpha)^{23}_{\mathrm{D}} = -38^{\circ}$ (c = 0.7 in acetone) [5031]; IR [5031].

2-[2,4-Bis(phenylmethoxy)phenoxy]-1-(2,4,6-trihydroxyphenyl)ethanone

[149312-76-5]



$C_{28}H_{24}O_7$ mol.wt. 472.49

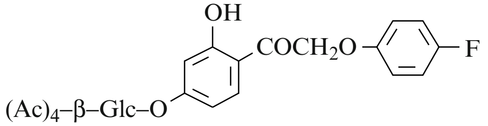
Synthesis

- Obtained by reaction of 2,4-(dibenzyl-oxy)phenoxyacetonitrile with phloro-glucinol in THF in the presence of zinc chloride and hydrogen chloride in an ice bath for 4 h (57%) (Hoesch reaction) [5025].

m.p. 91–92° [5025]; monohydrate [5025];
^{1}H NMR [5025], ^{13}C NMR [5025], IR [5025], UV [5025], MS [5025].

2-(4-Fluorophenoxy)-1-[2-hydroxy-4-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy]-phenyl]ethanone

[121377-35-3]



$C_{28}H_{29}FO_{13}$ mol.wt. 592.53

Synthesis

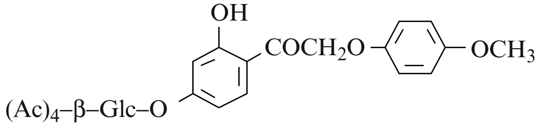
- Obtained by glycosylation of 1-(2,4-di-hydroxyphenyl)-2-(4-fluorophenoxy)ethanone with acetobromoglucose (34%) [5031].

m.p. 174° [5031]; $(\alpha)_D^{23} = -28^\circ$ (c = 1 in chloroform) [5031];

^{1}H NMR [5031], IR [5031].

1-[2-Hydroxy-4-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy]phenyl]-2-(4-methoxy-phenoxy)ethanone

[121361-57-7]



$C_{29}H_{32}O_{14}$ mol.wt. 604.56

Synthesis

- Obtained by glycosylation of 1-(2,4-di-hydroxyphenyl)-2-(4-methoxyphenoxy) ethanone with acetobromo-glucose (45%) [5031].

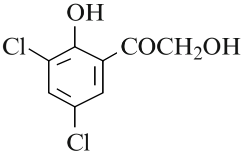
m.p. 180° [5031]; $(\alpha)_D^{23} = -27^\circ$ (c = 1 in chloroform) [5031];

^{1}H NMR [5031], IR [5031].

Chapter 15
Compounds Derived from Hydroxyacetic Acids

1-(3,5-Dichloro-2-hydroxyphenyl)-2-hydroxyethanone

[58483-53-7] $C_8H_6Cl_2O_3$ mol.wt. 221.04

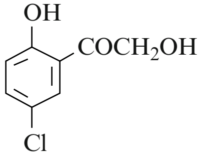


Synthesis

- Refer to: [5055] (Romanian patent).

1-(5-Chloro-2-hydroxyphenyl)-2-hydroxyethanone

[52728-05-9] $C_8H_7ClO_3$ mol.wt. 186.59



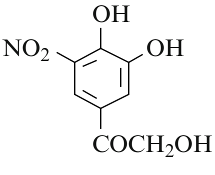
Synthesis

- Obtained by oxidative rearrangement of 5-chloro-2-hydroxy-α-bromoacetophenone in moist DMSO for 16 h at 20° (56%) [4394].

m.p. 98–99° [4394]; ^{1}H NMR [4394].

1-(3,4-Dihydroxy-5-nitrophenyl)-2-hydroxyethanone

[134612-56-9] $C_8H_7NO_6$ mol.wt. 213.15



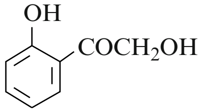
Synthesis

- Refer to: [4402].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_15,

2-Hydroxy-1-(2-hydroxyphenyl)ethanone

[17375-96-1] $C_8H_8O_3$ mol.wt. 152.15



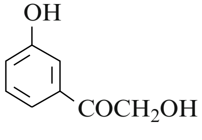
Syntheses

- Preparation by treatment of 2-hydroxy-α-bromo-acetophenone with refluxing water for 16 h (88%) [5056].
- Also obtained by hydrolysis of 2-hydroxy-α-(benzoyloxy)-acetophenone with 50% aqueous potassium hydroxide in refluxing ethanol for 34 h (73%) [4394].
- Also obtained by oxidative rearrangement of 2-hydroxy-α-bromoacetophenone in moist DMSO for 16 h at 20° (31%) [4394].
- Also obtained by action of hot aqueous sodium carbonate on 2,3-dihydro-2-hydroxybenzo[b] furan-3-one (m.p. 108°) (SM) at 100° for 1 h (33%). SM was obtained by oxidation of 2-hydroxyacetophenone with selenium dioxide [5057].
- Also obtained by hypervalent iodine oxidation of 1-(trimethylsilyloxy)-1-[2-(trimethylsilyloxy)-phenyl]ethene with iodosobenzene, boron trifluoride etherate and water. The mixture was stirred at −40° for 1 h, then the temperature was slowly (1 h) raised to r.t. and stirring was continued for 30 min (25%) [5058].
- Also obtained by a selective one-step synthesis from phenoxymagnesium bromide (1 mol) and anhydrous monomeric glyoxal (1 mol) in boiling benzene for 20 h (24%) [5059].
- Also obtained from α-chloro-o-hydroxyacetophenone by hydrolysis with boiling water for 15–20 h (20%) [4577].
- Also refer to: [4483,4879,5060–5064].

m.p. 66–67° [5057], 65° [4577,5059], 64–65° [4394,5056,5058];
^{1}H NMR [4394,5057,5059], IR [5059], UV [5059].

2-Hydroxy-1-(3-hydroxyphenyl)ethanone

[131341-58-7] $C_8H_8O_3$ mol.wt. 152.15

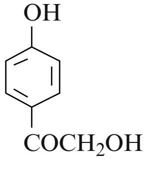


Synthesis

- Refer to: [5065].

2-Hydroxy-1-(4-hydroxyphenyl)ethanone

[5706-85-4] $C_8H_8O_3$ mol.wt. 152.15



Syntheses

- Preparation by reaction of p-hydroxy-a-bromoacetophenone with formic acid in the presence of DBU,
  - in benzene at 0°, followed by saponification of the intermediate formate ester with sodium hydroxide in methanol (99%) [5066];
  - in methylene chloride, with the same treatment (49%) [5067].

- Preparation by demethylation of α-acetoxy-p-methoxyacetophenone (SM) with aluminium chloride in refluxing benzene for 3 h (80%). In the reaction, deacetylation takes place simultaneously. SM was obtained by treatment of α-chloro-p-methoxyacetophenone with potassium acetate in ethanol [5068].
- Preparation by action of boron trifluoride etherate with p-hydroxyphenyl diazomethyl ketone (SM1) in nitromethane under nitrogen at 22° for 15 min (81%). SM1, preparation given, melted at 145–150° (d) [5069].
- Preparation from α-acetoxy-4-hydroxyacetophenone (m.p. 133°),
    - by heating with 16% aqueous sodium hydroxide for 15 min on a steam bath (quantitative yield) [5070];
    - in methanolic solution by treatment with 0.5 N aqueous sodium hydroxide at r.t. for 15 min (67%) [4523].
- Preparation by adding excess of concentrated hydrochloric acid to a warm concentrated aqueous solution of the potassium salt and cooling the solution [5071].
- Preparation by treatment of the sodium salt with aqueous hydrochloric acid [5072].
- Also obtained from p-acetoxybenzoylcarbinol (SM2) by heating with 4% ethanolic potassium hydroxide for 45 min on a water bath (20%). SM2 was prepared from p-acetoxyphenyl diazomethyl ketone (m.p. 109–110°) after treatment in dioxane with 2 N sulfuric acid at r.t. for 20 min, then at 40° until no more nitrogen evolved [5073].
- Also obtained by condensation of glyoxal with phenol,
    - in the presence of butylamine at 33° for 3 h (29%) [5074];
    - in the presence of aqueous sodium hydroxide at 33° for 6 h (25%) [5075];
    - in the presence of hydrogen chloride at 80° for 4 h (<5%) [5076].
- Also obtained by reductive condensation of p-hydroxyphenylglyoxal potassium bisulfite with diethylamine under hydrogen in the presence of Raney nickel in dilute ethanol for 1.5 h at 45° [4818].
- Also obtained from bisphenol A which is metabolized by a Gram-negative aerobic bacterium via a novel pathway involving oxidative skeletal rearrangement of the bisphenol A [5077].
- Also obtained by peroxidatic degradation of 7,4′-dihydroxyflavanone or 7,4′-dihydroxy-3′-methoxyflavanone [5078].
- Also refer to: [5064,5079].

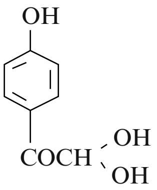
N.B.: Na salt [5070,5072], K salt [5071].

m.p. 177–178° [5068,5071], 173–174° [5073], 173° [4523], 170–177° [4818], 170–172° [5072], 170–171° [5069], 165–167° [5066,5067];

^{1}H NMR [5066,5069], IR [5066,5069], MS [5066].

2,2-Dihydroxy-1-(4-hydroxyphenyl)ethanone

[197447-05-5] $C_8H_8O_4$ mol.wt. 168.15

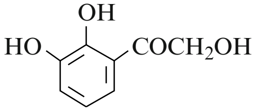


Syntheses

- Obtained by oxidation of p-hydroxyacetophenone with selenium oxide [5080].
- Also refer to: [5081] (compound 1d) and [5082,5083].

1-(2,3-Dihydroxyphenyl)-2-hydroxyethanone

[58483-49-1] $C_8H_8O_4$ mol.wt. 168.15

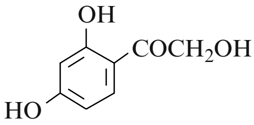


Synthesis

- Refer to: [5084].

1-(2,4-Dihydroxyphenyl)-2-hydroxyethanone *(Fisetol)*

[487-47-8] $C_8H_8O_4$ mol.wt. 168.15



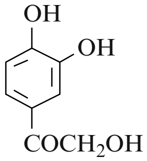
Syntheses

- Obtained by hydrolysis of a-acetoxyresacetophenone (m.p. 164°5) [5085],
    - with 5% aqueous sodium carbonate on a steam bath for 3 h (35%) [5085];
    - with 10% aqueous sodium hydroxide for 2 h at r.t. (38%) [5085].
- Also obtained by hydrolysis of 2,4,α-triacetoxyacetophenone (m.p. 94°) [5085],
    - with 2 N sodium hydroxide [5085];
    - with 5 N methanolic ammonia for 8 days in the cold [5086].
- Also obtained by demethylation of α-methoxyresacetophenone with 40% hydrobromic acid for 3 h on a boiling water bath (16%) [4640].
- Also obtained by treatment of α-[(methoxycarbonyl)oxy]resacetophenone (m.p. 157–158°) or α-[(ethoxycarbonyl)oxy]resacetophenone (m.p. 107°) with 2 N sodium hydroxide for 2 h at r.t. [4432].
- Also obtained by reaction of hydroxyacetonitrile with resorcinol [4845], (41%) (Hoesch reaction) [5087].
- Also obtained by a selective one-step synthesis from 3-hydroxyphenoxymagnesium bromide (0.1 mol) and anhydrous monomeric glyoxal (0.1 mol) in boiling benzene for 20 h (35%) [5059].
- Also obtained by treatment of *Fisetin* (3,7,3′,4′-tetrahydroxyflavone)—m.p. 330° (d)—with boiling ethanolic potassium hydroxide [4885].
- Also refer to: [4467,4835,4837,5088,5089].

m.p. 191° [4845], 189° [4432,5085–5087], 187–188° [4640], 185–186° [5059]; ^{1}H NMR [5059], IR [5059], UV [4640,5059,5090].

1-(3,4-Dihydroxyphenyl)-2-hydroxyethanone *(DOPKET)*

[29477-54-1] $C_8H_8O_4$ mol.wt. 168.15



Syntheses

- Obtained by saponification of its triacetate (SM) (m.p. 94°) with sodium hydroxide (or sodium ethoxide) in ethanol in a water bath for 20 min (33%). SM was prepared by treatment of 3,4-dihydroxy-α-chloroacetophenone with potassium acetate in refluxing acetic anhydride for 15 min (quantitative yield) [4768].
- Also obtained by heating N-acetyldopamine with 1 N hydrochloric acid [5091].
- Also obtained by hydrolysis of 2-(3′,4′-dihydroxyphenyl)-3-acetylamino-6 (or 7) -(N-acetyl-2″-aminoethyl)-2,3-dihydro-1,4-benzodioxine (SM) with refluxing 1 N hydrochloric acid for 3 h. SM was formed by incubation of N-acetyldopamine with locust cuticle [4746].
- Also obtained from the quinone of 3,4-dihydroxyphenylglycol by attack with isomerase (SM). This enzyme (SM) has been purified from the hemolymph of *Sarcophaga bullata* [5092].

Isolation from natural sources

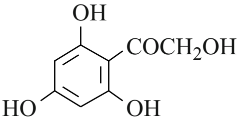
- From sclerotization of the adult cuticle *(Leucophaea maderae)* [5093].
- Also obtained by mild acid hydrolysis of sclerotized cuticles from locusts *(Schistocerca gregaria)*and beetles *(Pachynoda sinuata)* [4744].
- From acid hydrolysates of insect hard cuticle [5094].
- From acid hydrolysates of insect sclerotized cuticle in refluxing 1 N formic acid for 1 h. The cuticle used was obtained from the desert locust *(Schistocerca gregaria)* [4745].
- By acid hydrolysis from exuviae of last instar larvae of the cicada *Tibicen pruinosa* [5095].
- From the seed coat tamarind (*Tamarindus indica* L.) [5096].
- From the skins of tamarind seeds [5097].
- From mild acid hydrolysates of tanning pharate pupae cuticle from *Manduca Sexta* [4747].
- In hydrolysates of the wing-scales of butterfly (*Eurema hecabe*) in 1 N hydrochloric acid. This compound was also present in the hydrolyzate of wing-scales of *Catopsilia crocale, Appias indra and Morpho rhetenor* [5098].
- In aqueous extracts from cockroach and locust exuviae of various **Orthoptera** in refluxing water for 1 h *(Periplaneta americana, Periplaneta brunnea, Chortoicetes terminifera* and *Austracris guttulosa)* [5099].
- in acidic extracts of insect cuticles (exuviae) in refluxing 1 N hydrochloric acid for 1.5 h, i.e.:
    - **Orthoptera** *(Periplaneta americana, Periplaneta brunnea, Blattella germanica, Nauphoeta cinerea, Chortoicetes terminifera* and *Austracris guttulosa)* [5099];

    - **Hemiptera** *(Nezara viridula)* [5099];
    - **Lepidoptera** *(Papilio aegeus* and *Antheraea helena)* [5099];
    - **Coleoptera** *(Anthrenus australis)* [5099].
  - in acidic extracts of insect cuticles (preparia) in refluxing 1 N hydrochloric acid for 1.5 h, i.e.:
    - **Diptera** *(Lucilia cuprina)* [5099].
  - in acid extracts of insect cuticles (prepal cuticles):
    - **Lepidoptera** *(Papilio aegeus)* [5099].
- Also refer to: [5100–5105].

m.p. 195° [4768]; UV [4744,5094], MS [4744,4748,5094]; electrophoresis [5093]; column chromatography [4744]; TLC [4744,5093,5094]; LCEC chromatography [5093]; GC [5099]; HPLC [4747,5092]; HPLC-MS [5091].

2-Hydroxy-1-(2,4,6-trihydroxyphenyl)ethanone

[55313-03-6] $C_8H_8O_5$ mol.wt. 184.15



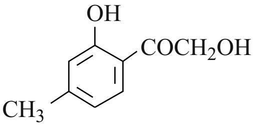
Syntheses

- Preparation according to Hoesch reaction from phloroglucinol,
  - with acetoxyacetonitrile (81%) [5106];
  - with hydroxyacetonitrile (63%) [5090].
- Also obtained from dihydrokaempferol (3,5,7,4′-tetrahydroxyflavanone) *(Aromadendrin)* by basic hydrolysis and subsequent oxidation [5107].
- Dihydrokaempferol yields kaempferol (3,5,7,4′-tetrahydroxyflavone) with peroxides and alkaline conditions; subsequent thermolysis produces the titled ketone [5107].
- Quercetin (3,5,7,3′,4′-pentahydroxyflavone) yields the same product under alkaline thermolysis (80°) [5107].
- Also refer to: [4868].

m.p. 226° [5090], 224° [5106]; UV [5090]; GC-MS [5107].

2-Hydroxy-1-(2-hydroxy-4-methylphenyl)ethanone

[55960-03-7] $C_9H_{10}O_3$ mol.wt. 166.18

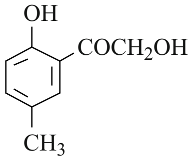


Syntheses

- Obtained by a selective one-step synthesis from 3-methyl-phenoxymagnesium bromide (1 mol) and anhydrous monomeric glyoxal (1 mol) in boiling benzene for 20 h (48%) [5059].
- Also refer to: [5084].

m.p. 68–69° [5059]; ^1H NMR [5059], IR [5059], UV [5059].

2-Hydroxy-1-(2-hydroxy-5-methylphenyl)ethanone



$C_9H_{10}O_3$ mol.wt. 166.18

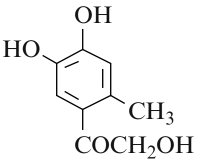
Synthesis

- Obtained by hydrolysis of 2-hydroxy-5-methyl-α-chloro-acetophenone with boiling water for 15–20 h (40%) [4577].

m.p. 76–77° [4577].

1-(4,5-Dihydroxy-2-methylphenyl)-2-hydroxyethanone

[61407-16-7]



$C_9H_{10}O_4$ mol.wt. 182.18

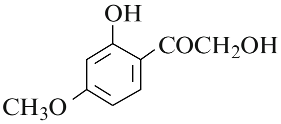
Synthesis

- Obtained (trace amounts) by heating D-fructose or D-glucose in 0.3 M acetate buffer of pH 4.5 at 96° for 48 h under nitrogen or D-fructose in 0.3 M acetate buffer of pH 4.5 in a stainless autoclave at 160° for 4 h [5108].

amorphous [5108]; ^{1}H NMR [5108], MS [5108].

2-Hydroxy-1-(2-hydroxy-4-methoxyphenyl)ethanone

[55960-07-1]



$C_9H_{10}O_4$ mol.wt. 182.18

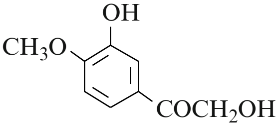
Syntheses

- Preparation from 2-hydroxy-4-methoxy-α-bromo-acetophenone with refluxing water for 20 h (76%) [4483].
- Also obtained from fisetol 4-monomethyl ether diacetate (m.p. 86°) by heating with ethanolic potassium hydroxide [4485].
- Also obtained by a selective one-step synthesis from 3-methoxyphenoxymagnesium bromide (1 mol) and anhydrous monomeric glyoxal (1 mol) in boiling benzene for 20 h (45%) [5059].
- Also obtained by action of 40% aqueous hydrobromic acid with 2-hydroxy-4,α-dimethoxy-acetophenone in acetic acid on a boiling water bath for 3 h (22%) [4640].
- Also refer to: [4433,4835,4837,5060,5088,5109].

m.p. 128° [4485], 127° [4640], 126–128° [4483], 126–127° [5059];
^{1}H NMR [4483,5059], IR [4483,5059], UV [4640,5059], MS [4483].

2-Hydroxy-1-(3-hydroxy-4-methoxyphenyl)ethanone

[90536-46-2] $C_9H_{10}O_4$ mol.wt. 182.18



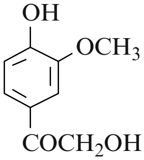
Syntheses

- Preparation by total hydrolysis of 4-methoxy-3,α-diacetoxy-acetophenone (m.p. 82–83°) [4638], (m.p. 81–82°) [4750] in methanol with concentrated hydrochloric acid,
  - for 4–5 h at r.t. (75%) [4638];
  - for 30 min at reflux (56%) [4750].

m.p. 177–178° [4750], 176–177° [4638]; IR [4750], UV [4750].

2-Hydroxy-1-(4-hydroxy-3-methoxyphenyl)ethanone

[18256-48-9] $C_9H_{10}O_4$ mol.wt. 182.18



Syntheses

- Preparation by total hydrolysis of 3-methoxy-4,α-diacetoxy-acetophenone (m.p. 77–78°) [4638], (m.p. 75–76°) [4750] in methanol with concentrated hydrochloric acid,
  - for 4–5 h at r.t. (78%) [4638], (25%) [5039];
  - during 14 h at 20°, then for 30 min at reflux (67%) [4750].
- Obtained by photorelease of l-glutamic acid from 5-[2-(4-hydroxy-3-methoxyphenyl)-2-oxoethyl] l-glutamate, mono(trifluoroacetate) [284043-07-8] with either 300 or 350 nm lamps in water or in deuterium oxide [5110].
- Also obtained by photorelease of γ-aminobutyric acid from 2-(4-hydroxy-3-methoxyphenyl)-2-oxoethyl γ-aminobutyrate [284043-11-4] with either 300 or 350 nm lamps in water or in deuterium oxide [5110].

  N.B.: Details of the synthesis and ^{1}H NMR, ^{13}C NMR, IR, UV and HRMS data are available free of charge via the Internet at http://pubs.acs.org. Complete experimental details are provided in the full paper [5110].

- Also obtained by treatment of 2-(acetoxy)-1-(4-hydroxy-3-methoxyphenyl)ethanone,
  - with boiling aqueous barium carbonate for 2 h [4637];
  - with 16% aqueous sodium hydroxide on the steam bath. The obtained sodium salt was treated with 2 N acetic acid (62%) [5111].

  N.B.: Na salt sesquihydrate (70%) [5111].

- Also refer to: [5112].

Isolation from natural sources

- From cell cultures of *Solanum khasianum* (Solanaceae) [5113,5114].

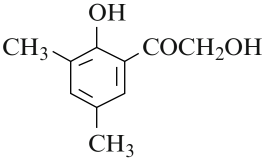
  N.B.: Microsomal preparations from heterotropic cell cultures of *Solanum khasianum* catalyse the hydroxylation of the α-methyl group of acetovanillone. The reaction requires both oxygen and NADPH [5113].

- From the Namibian shrub *Salsola tuberculatiformis* [5115].
- From the suprarenal capsules [4750].
- Also refer to: [5116].

m.p. 160–161° [4750,5039], 159–160° [4638], 158–160° (anhydrous) [5111];
^{1}H NMR [5039,5114], ^{13}C NMR [5114], IR [4750,5039],
UV [4750,5114], MS [5039,5114,5116];
fluorescence spectroscopy [5117]; HPLC [5114]; GC/MS [5114,5116].

2-Hydroxy-1-(2-hydroxy-3,5-dimethylphenyl)ethanone

[55960-05-9] $C_{10}H_{12}O_3$ mol.wt. 180.20



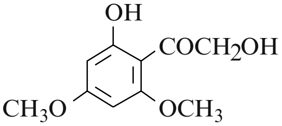
Synthesis

- Obtained by a selective one-step synthesis for 2,4-dimethyl-phenoxymagnesium bromide (1 mol) and anhydrous monomeric glyoxal (1 mol) in boiling benzene for 20 h (35%) [5059].

m.p. 100–101° [5059];
^{1}H NMR [5059], IR [5059], UV [5059].

2-Hydroxy-1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone

[83768-75-6] $C_{10}H_{12}O_5$ mol.wt. 212.20



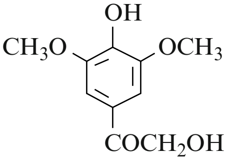
Syntheses

- Preparation by hydrolysis of 2-(2-hydroxy-4,6-dimethoxy-phenyl)-2-oxoethyl benzoate in pyridine with aqueous sodium hydroxide under nitrogen atmosphere at r.t. for 1 h (80%) [5118].
- Also obtained by degradation of 2-[2-(2-hydroxy-4,6-dimethoxyphenyl)-2-oxoethoxy]-2-methyl-propionic acid in refluxing mixture of concentrated hydrochloric acid/methanol (1 vol/5 vol) for 1 h (42%) [5119].
- Also obtained by action of 40% hydrobromic acid with 2-hydroxy-α,4,6-trimethoxyacetophenone in acetic acid by heating on a boiling water bath for 3 h (32%) [4640].
- Also refer to: [5088].

m.p. 140–142° [5119], 139–140° [4640], 131–132° [5118]; TLC [5118];
^{1}H NMR [5118,5119], IR [5118], UV [4640], MS [5118].

2-Hydroxy-1-(4-hydroxy-3,5-dimethoxyphenyl)ethanone *(Danielone)*

[90426-22-5] $C_{10}H_{12}O_5$ mol.wt. 212.20



Syntheses

- Preparation by an efficient simple three-step synthesis:
- First, slowly adding a methanolic 3,5-dimethoxy-4-(methoxymethoxy)acetophenone to a cooled

methanolic potassium hydroxide solution. Then iodosobenzene diacetate was added and the reaction mixture stirred at r.t. overnight, cooled in an ice bath and 6% hydrochloric acid was added. After refluxing at 60° for 1 h, the reaction mixture was cooled at r.t. and water was added (60%) [5120].

- Obtained by hydrolysis of its diacetate (SM) with 5% hydrochloric acid in 70% dilute ethanol at 80° for 1.5 h [5121]. SM was prepared according to [5122].
- Also obtained by photorelease of l-glutamic acid from 5-[2-(4-hydroxy-3,5-dimethoxyphenyl)-2-oxoethyl] l-glutamate, mono(trifluoroacetate) [284043-10-3] with either 300 or 350 nm lamps in water or in deuterium oxide [5110].
- Also obtained by photorelease of γ-aminobutyric acid from 2-(4-hydroxy-3,5-dimethoxyphenyl)-2-oxoethyl γ-aminobutyrate [284043-12-5] with either 300 or 350 nm lamps, in water or in deuterium oxide [5110].

N.B.: Details of the synthesis and ^{1}H NMR, ^{13}C NMR, IR, UV and HRMS data are available free of charge via the Internet at http://pubs.acs.org. Complete experimental details are provided in the full paper [5110].

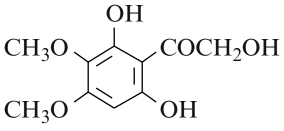
- Also obtained from 1,2-bis(4-hydroxy-3,5-dimethoxyphenyl)-propane-1,3-diol, a β-1-lignin substructure model compound, by degradation with laccase of *Coriolus versicolor* (Fr.) Quel. [5123].
- Also refer to: [5124–5126].

Isolation from natural sources

- From *Nicotiana tabacum* [5121,5127] and *Atropa belladona* root cultures [5121].
- From cell. suspension cultures of *Hyoscyamus albus* [5128].
- From *Carica papaya* fruit slices (Caricaceae) [5129].
- Isolated as virulence gene inducing compounds of *Agrobacterium* from the hairy root cultures of belladonna [5130].
- Also refer to: [5116].

m.p. 145° [5129], 109–110° [5121]. One of the reported melting points is obviously wrong.
TLC [5129]; GC/MS [5116,5127];
^{1}H NMR [5121,5129], ^{13}C NMR [5121,5129], IR [5120,5129],
UV [5121,5127,5129], MS [5116,5120,5121,5127,5129].

1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-2-hydroxyethanone *(Methyldegeranylmelicopol)*



$C_{10}H_{12}O_6$ mol.wt. 228.20

Synthesis

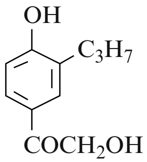
- Obtained by degradation of *methylmelicopol* (VII) (SM) with refluxing 2 N hydrochloric acid for 5 min in an atmosphere of nitrogen (16%) [4987]. SM was isolated from the leaves of *Melicope broadbentiana* F. M. Bail (Rutaceae) [4987,5131].

N.B.: In the paper [4987], the formulas (VII) as well as (XI) representing the titled compound were erroneous [5131].

m.p. 181–183° [4987];
^{1}H NMR [4987], IR [4987], UV [4987].

2-Hydroxy-1-(4-hydroxy-3-propylphenyl)ethanone

[178978-33-1] $C_{11}H_{14}O_3$ mol.wt. 194.23

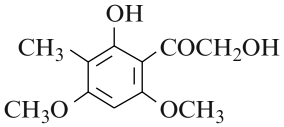


Synthesis

– Refer to: [5132].

2-Hydroxy-1-(2-hydroxy-4,6-dimethoxy-3-methylphenyl)ethanone

[184706-61-4] $C_{11}H_{14}O_5$ mol.wt. 226.23



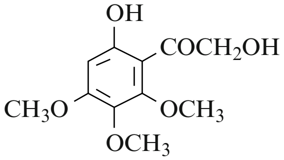
Isolation from natural sources

– From the stem wood of *Euphorbia quinquecostata* Volk. (Euphorbiaceae) [5133].

m.p. 164–166° [5133];
^{1}H NMR [5133], ^{13}C NMR [5133], IR [5133], UV [5133], MS [5133].

2-Hydroxy-1-(6-hydroxy-2,3,4-trimethoxyphenyl)ethanone *(Dimethyldegeranylmelicopol)*

[51117-08-9] $C_{11}H_{14}O_6$ mol.wt. 242.23



Syntheses

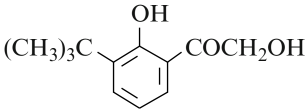
– Preparation by reaction of acetoxyacetonitrile with antiarol (Hoesch reaction), followed by heating the isolated intermediate compound in refluxing dilute ethanol for 8 h (32%) [5131].

– Also obtained by hydrogenolysis of *dimethylmelicopol* (VIII) (SM) [4987]. SM was obtained by partial methylation of *methylmelicopol* (VII) [4987], itself isolated from the leaves of *Melicope broadbentiana* F. M. Bail. (Rutaceae) [4987,5131]. In the paper [4987], the formulas (VII) and (VIII), as well as (XIX) representing the titled compound were erroneous [5131].

m.p. 87° [4987], 86–87° [5131];
^{1}H NMR [4987], IR [4987], UV [4987].

1-[3-(Dimethylethyl)-2-hydroxyphenyl]-2-hydroxyethanone

[55960-04-8] $C_{12}H_{16}O_3$ mol.wt. 208.26



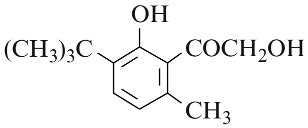
Synthesis

- Obtained by a selective one-step synthesis from 2-tert-butylphenoxymagnesium bromide (1 mol) and anhydrous monomeric glyoxal (1 mol) in boiling benzene for 20 h (45%) [5059].

oil [5059]; ^{1}H NMR [5059], IR [5059], UV [5059].

1-[3-(Dimethylethyl)-2-hydroxy-6-methylphenyl]-2-hydroxyethanone

[55960-06-0] $C_{13}H_{18}O_3$ mol.wt. 222.28



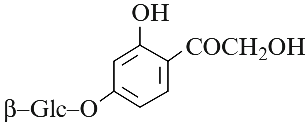
Synthesis

- Obtained by a selective one-step synthesis from 2-tert-butyl-5-methylphenoxymagnesium bromide (1 mol) and anhydrous monomeric glyoxal (1 mol) in boiling benzene for 20 h (25%) [5059].

m.p. 76–77° [5059]; ^{1}H NMR [5059], IR [5059], UV [5059].

1-[4-(β-D-Glucopyranosyloxy)-2-hydroxyphenyl]-2-hydroxyethanone

$C_{14}H_{18}O_9$ mol.wt. 330.29

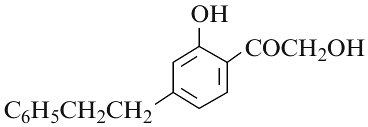


Synthesis

- Refer to: [5020].

2-Hydroxy-1-[2-hydroxy-4-(2-phenylethyl)phenyl]ethanone

[132197-47-8] $C_{16}H_{16}O_3$ mol.wt. 256.30

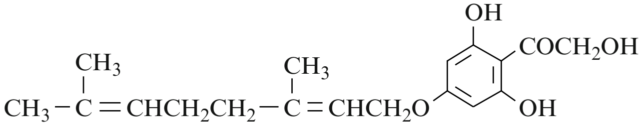


Synthesis

- Refer to: [5134] (Japanese patent).

1-[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2,6-dihydroxyphenyl]-2-hydroxyethanone

[142905-41-7] $C_{18}H_{24}O_5$ mol.wt. 320.38



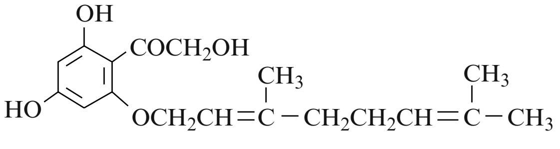
Isolation from natural source

- From the fruit of *Evodia Merrillii* Kanehira & Sasaki ex Kanehira (Rutaceae) [5135].

m.p. 106–108° [5135]; column chromatography [5135];
^{1}H NMR [5135], ^{13}C NMR [5135], IR [5135], UV [5135], MS [5135].

1-[2-[(3,7-Dimethyl-2,6-octadienyl)oxy]-4,6-dihydroxyphenyl]-2-hydroxyethanone *(E)*

[149492-42-2] $C_{18}H_{24}O_5$ mol.wt. 320.38



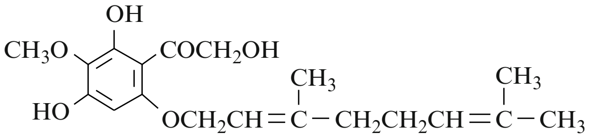
Isolation from natural source

- From the fruits of *Evodia merrillii* Kanehira & Sasaki ex Kanehira (Rutaceae) [5136].

m.p. 144°5–146° [5136];
^{1}H NMR [5136], ^{13}C NMR [5136], IR [5136], UV [5136], MS [5136].

1-[6-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2,4-dihydroxy-3-methoxyphenyl]-2-hydroxyethanone *(Melicopol)*

$C_{19}H_{26}O_6$ mol.wt. 350.41



Isolation from natural source

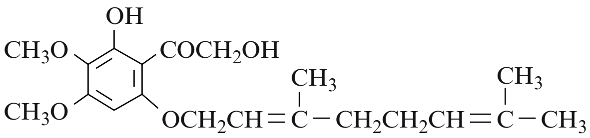
- From the leaves of *Melicope broadbentiana* F. M. Bail. (Rutaceae) [4987,5131].

N.B.: In the paper [4987], the formula (VI) representing the titled compound was erroneous [5131].

m.p. 133–134° [4987];
^{1}H NMR [4987], ^{1}H NMR NOE [5131], IR [4987], UV [4987,5131].

1-[3,4-Dimethoxy-6-[(3,7-dimethyl-2,6-octadienyl)oxy]-2-hydroxyphenyl]-2-hydroxyethanone *(Methylmelicopol)*

$C_{20}H_{28}O_6$ mol.wt. 364.44



Isolation from natural source

- From the leaves of *Melicope broadbentiana* F. M. Bail. (Rutaceae) [4987,5131].

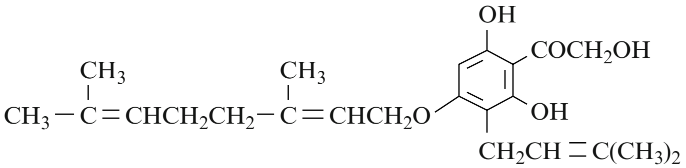
N.B.: In the paper [4987], the formula (VII) representing the titled compound was erroneous [5131].

m.p. 103° [4987];
^{1}H NMR [4987], ^{1}H NMR NOE [5131], IR [4987], UV [4987,5131].

1-[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2,6-dihydroxy-3-(3-methyl-2-butenyl) phenyl]-2-hydroxyethanone *(E)*

[149492-41-1] $C_{23}H_{32}O_5$ mol.wt. 388.26

Isolation from natural source



– From the fruits of *Evodia merrillii* Kanehira & Sasaki ex Kanehira (Rutaceae) [5136].

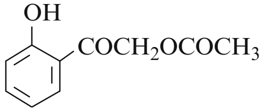
m.p. 136–137° [5136];
^{1}H NMR [5136], ^{13}C NMR [5136], IR [5136], UV [5136], MS [5136].

Chapter 16
Compounds Derived from Acyloxy- and Aroyloxyacetic Acids

16.1 Compounds Derived from Acetoxyacetic Acids

2-(Acetyloxy)-1-(2-hydroxyphenyl)ethanone

[40231-09-2] $C_{10}H_{10}O_4$ mol.wt. 194.19



Syntheses

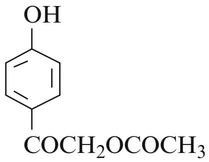
- Preparation by hydrolysis of o-acetoxy-α-acetoxy-acetophenone (SM) with aqueous potassium hydroxide solution by gently warming for 10–15 min on a water bath maintained at 80° (71%). SM was obtained by cupric chloride-catalyzed decomposition of o-acetoxy-α-diazoacetophenone in dioxane solution in the presence of acetic acid (65%, m.p. 161–162°) [5137].
- Also obtained by oxidative rearrangement of 2-acetoxy-α-bromoacetophenone in moist DMSO for 28 h at 20° (49%) [4394].
- Also obtained by reaction of potassium acetate with o-hydroxy-α-bromoaceto-phenone in acetone at r.t. for 90 min [5032].

m.p. 170–171° [5137], 58–59° [4394], 57° [5032]. One of the reported melting points is obviously wrong. Ogle and Main [5032] consider that the reported product of m.p. 171° [5137], identified by only elemental and IR analysis, is not the titled product.

^{1}H NMR [4394,5032], ^{13}C NMR [5032], IR [5032,5137].

2-(Acetyloxy)-1-(4-hydroxyphenyl)ethanone

[20816-46-0] $C_{10}H_{10}O_4$ mol.wt. 194.19



Syntheses

- Preparation by reaction of α-chloro-4-acetoxyacetophenone with potassium acetate in boiling ethanol for 4 h (quantitative yield) [5138].
- Preparation by reaction of acetic acid and potas-sium acetate with α-chloro-4-hydroxyaceto-phenone in refluxing ethanol for 1 h (80%) [5070].

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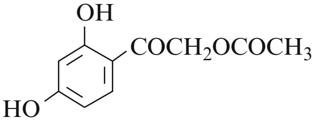
DOI 10.1007/978-1-4020-9787-4_16,

- Preparation by reaction of acetic acid with p-hydroxyphenacyl chloride in acetonitrile in the presence of triethylamine, first in an ice bath for 15 min, then at reflux for 3 h (50%) [4523].
- Also refer to: [5139,5140].

m.p. 133° [4523,5070], 127° [5138].

2-(Acetyloxy)-1-(2,4-dihydroxyphenyl)ethanone

[63124-23-2] $C_{10}H_{10}O_5$ mol.wt. 210.19



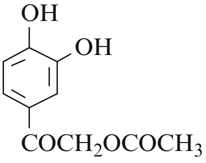
Synthesis

- Preparation by reaction of acetoxyacetonitrile with resorcinol (Hoesch reaction) (85%) [5085], (40%) [5141].

m.p. 167–168° [5141], 164°5 [5085]; ^{1}H NMR [5141], IR [5141], UV [5090].

2-(Acetyloxy)-1-(3,4-dihydroxyphenyl)ethanone

[67083-58-3] $C_{10}H_{10}O_5$ mol.wt. 210.19



Synthesis

- Preparation by adding a solution of sodium acetate in aqueous acetic acid to an ethanolic solution of 3,4-dihydroxy-α-chloroacetophenone, and heating at reflux for 24 h (83%) [5142].

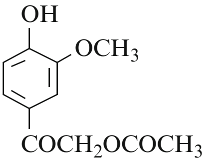
Isolation from natural sources

- Obtained by mild acid hydrolysis of sclerotized cuticles from locusts *(Schistocerca gregaria)* and beetles *(Pachynoda sinuata)* [4744].

m.p. 157–160° [5142]; UV [4744], MS [4744,4748];
column chromatography [4744]; TLC [4744].

2-(Acetyloxy)-1-(4-hydroxy-3-methoxyphenyl)ethanone

[139473-80-6] $C_{11}H_{12}O_5$ mol.wt. 224.21



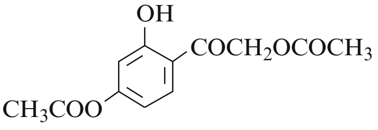
Syntheses

- Obtained by reaction of α-chloroacetovanillone with potassium acetate,
  - in refluxing ethanol for 2 h (56%) [4637] or for 4 h (67%) [5138];
  - in refluxing acetic acid and ethanol mixture for 1 h (57%) [5111].
- Also obtained by partial hydrolysis of 4,α-diacetoxy-3-methoxyacetophenone (m.p. 75–76°) in the presence of potassium bicarbonate in methanol for 16 h at 20° (96%) [4750].

- Also obtained from 4-hydroxy-3-methoxy-α-diazoacetophenone by slowly heating in acetic acid at 110° (29%) [4750].
- Also refer to: [5143].

m.p. 113–114° [4750], 110° [5111,5138].

2-(Acetyloxy)-1-[4-(acetyloxy)-2-hydroxyphenyl]ethanone



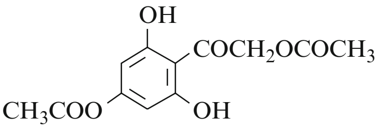
$C_{12}H_{12}O_6$ mol.wt. 252.22

Syntheses

- Preparation by reaction of ammonia with fisetol triacetate (m.p. 94°) in ethanol for 30 min at r.t. (93%) [5090].
- Also obtained by adding acetic anhydride to a suspension of fisetol sodium salt in a water/ethyl ether mixture (27%) [5090].
- Also obtained by adding, with stirring and cooling, a solution of fisetol (m.p. 189–190°) in aqueous sodium hydroxide to a solution of acetic anhydride in chloroform (or benzene). Then, the mixture was maintained for 10 min at r.t. (19%) [5090].

m.p. 99–100° [5090]; UV [5090].

2-(Acetyloxy)-1-[4-(acetyloxy)-2,6-dihydroxyphenyl]ethanone



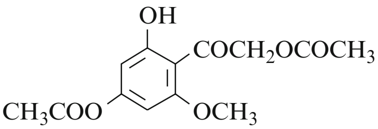
$C_{12}H_{12}O_7$ mol.wt. 268.22

Syntheses

- Obtained by partial acetylation of α-hydroxy-phloracetophenone with acetic anhydride in chloroform in the presence of aqueous sodium hydroxide, for 24 h at r.t. (31%) [5090].
- Also obtained by partial deacetylation of α-hydroxyphloracetophenone tetraacetate (m.p. 109–110°) in ethanol with ammonia, for 1.5 h at r.t. (30%) [5090].

m.p. 167–169° [5090]; UV [5090].

2-(Acetyloxy)-1-[4-(acetyloxy)-2-hydroxy-6-methoxyphenyl]ethanone



$C_{13}H_{14}O_7$ mol.wt. 282.25

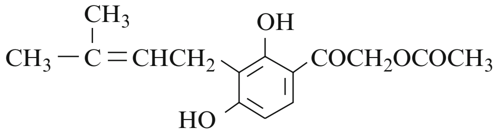
Synthesis

- Obtained by reaction of 4,α-diacetoxy-2,6-di-hydroxyacetophenone with diazomethane in ethyl ether at 0° (37%) [5090].

m.p. 121° [5090].

2-(Acetyloxy)-1-[2,4-dihydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[63124-25-4] $C_{15}H_{18}O_5$ mol.wt. 278.30



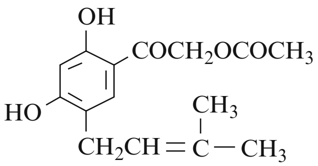
Synthesis

- Obtained (poor yield) by reaction of 2-methyl-3-buten-2-ol with 2,4-di-hydroxy-α-(acetoxy)acetophenone in dioxane in the presence of boron trifluoride etherate at 50–60° for 3 h (6%) [5141].

m.p. 147–148° [5141]; ^{1}H NMR [5141], IR [5141].

2-(Acetyloxy)-1-[2,4-dihydroxy-5-(3-methyl-2-butenyl)phenyl]ethanone

[63124-24-3] $C_{15}H_{18}O_5$ mol.wt. 278.30



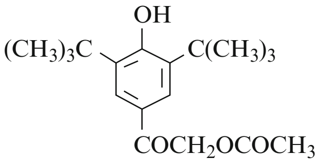
Synthesis

- Obtained by reaction of 2-methyl-3-buten-2-ol with 2,4-di-hydroxy-α-(acetoxy)acetophenone in dioxane in the presence of boron trifluoride etherate at 50–60° for 3 h (20%) [5141].

m.p. 129–130° [5141]; ^{1}H NMR [5141], IR [5141].

2-(Acetyloxy)-1-[3,5-bis(1,1-dimethylethyl)-4-hydroxyphenyl]ethanone

[28441-16-9] $C_{18}H_{26}O_4$ mol.wt. 306.40



Syntheses

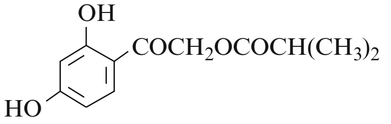
- Preparation by reaction of acetic acid with 3,5-di-tert-butyl-4-hydroxy-α-bromoacetophenone in toluene in the presence of DBU, first at 0° for 1 h, then at r.t. overnight (79%) [4523].
- Also refer to: [5144].

m.p. 103–105° [4523,5144].

16.2 Compounds Derived from Other Acyloxy- and Phenacyloxyacetic Acids

2-(2,4-Dihydroxyphenyl)-2-oxoethyl 2-methylpropanoate

[63124-27-6] $C_{12}H_{14}O_5$ mol.wt. 238.24



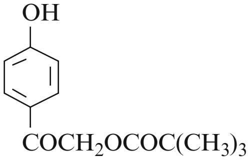
Synthesis

- Obtained by reaction of isobutyryloxy-acetonitrile with resorcinol (Hoesch reaction) (40%) [5141].

m.p. 116–117° [5141]; ^{1}H NMR [5141], IR [5141].

2-(4-Hydroxyphenyl)-2-oxoethyl 2,2-dimethylpropanoate

[230310-21-1] $C_{13}H_{16}O_4$ mol.wt. 236.27



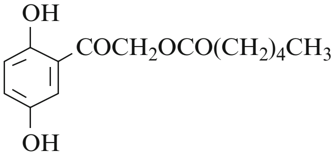
Synthesis

– Obtained by adding dropwise triethylamine over 15 min to a cooled solution of p-hydroxyphenacyl chloride and pivalic acid in acetonitrile in an ice bath and then refluxing for 3 h [4523].

m.p. 178°5 [4523]; ^{1}H NMR [4523].

2-(2,5-Dihydroxyphenyl)-2-oxoethyl hexanoate

[216301-66-5] $C_{14}H_{18}O_5$ mol.wt. 266.29



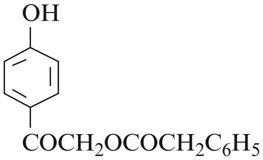
Synthesis

– Obtained by reaction of hexanoic acid with 2,5-di-hydroxy-α-bromoacetophenone in acetonitrile in the presence of triethylamine at 70° for 4 h [4441].

flash chromatography [4441]; ^{1}H NMR [4441], IR [4441], MS [4441].

2-(4-Hydroxyphenyl)-2-oxoethyl benzeneacetate

[230310-20-0] $C_{16}H_{14}O_4$ mol.wt. 270.28



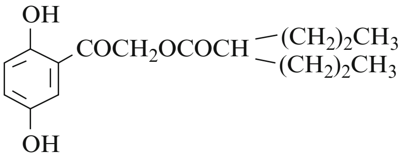
Synthesis

– Obtained by adding dropwise triethylamine over 15 min to a cooled solution of p-hydroxyphenacyl chloride and phenylacetic acid in acetonitrile in an ice bath and then refluxing for 3 h [4523].

m.p. 106–107° [4523]; ^{1}H NMR [4523].

2-(2,5-Dihydroxyphenyl)-2-oxoethyl 2-propylpentanoate *(so called Valproate)*

[216301-65-4] $C_{16}H_{22}O_5$ mol.wt. 294.35



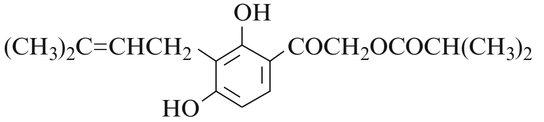
Synthesis

– Obtained by reaction of valproic acid with 2,5-dihydroxy-α-bromoacetophenone in acetonitrile in the presence of triethylamine at 70° for 4 h [4441].

flash chromatography [4441]; ^{1}H NMR [4441], IR [4441], MS [4441].

2-[2,4-Dihydroxy-3-(3-methyl-2-butenyl)phenyl]-2-oxoethyl 2-methylpropanoate

[63124-29-8] $C_{17}H_{22}O_5$ mol.wt. 306.36



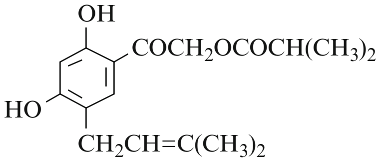
Synthesis

– Obtained (poor yield) by reaction of 2-methyl-3-buten-2-ol with 2,4-dihydroxy-α-(isobutyryloxy)-acetophenone in dioxane in the presence of boron trifluoride etherate at 50–60° for 3 h (5%) [5141].

m.p. 147–148° [5141]; ^{1}H NMR [5141], IR [5141].

2-[2,4-Dihydroxy-5-(3-methyl-2-butenyl)phenyl]-2-oxoethyl 2-methylpropanoate

[63124-28-7] $C_{17}H_{22}O_5$ mol.wt. 306.36



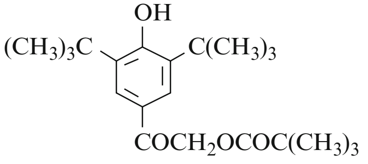
Synthesis

– Obtained by reaction of 2-methyl-3-buten-2-ol with 2,4-dihydroxy-α-(isobutyryloxy)acetophenone in dioxane in the presence of boron trifluoride etherate at 50–60° for 3 h (20%) [5141].

m.p. 122–123° [5141]; ^{1}H NMR [5141], IR [5141].

2-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-2-oxoethyl 2,2-dimethylpropanoate

[230310-24-4] $C_{21}H_{32}O_4$ mol.wt. 348.48



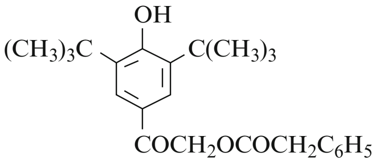
Synthesis

– Preparation by adding in one portion DBU to a solution of 2-bromo-1-(3,5-di-tert-butyl-4-hydroxyphenyl)ethanone and pivalic acid in toluene. The solution was stirred in ice for 1 h and then overnight at r.t. (70%) [4523].

m.p. 134–135°5 [4523]; ^{1}H NMR [4523].

2-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-2-oxoethyl benzeneacetate

[230310-23-3] $C_{24}H_{30}O_4$ mol.wt. 382.50



Synthesis

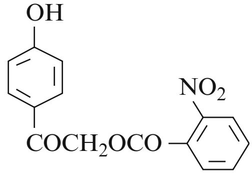
– Preparation by adding in one portion DBU to a solution of 2-bromo-1-(3,5-di-tert-butyl-4-hydroxy-phenyl)ethanone and phenylacetic acid in toluene. The solution was stirred in an ice bath for 1 h and then at r.t. overnight (58%) [4523].

m.p. 63–63°5 [4523]; ^{1}H NMR [4523].

16.3 Compounds Derived from Benzoyloxyacetic Acids

1-(4-Hydroxyphenyl)-2-[(2-nitrobenzoyl)oxy]ethanone

[130627-04-2] $C_{15}H_{11}NO_6$ mol.wt. 301.26

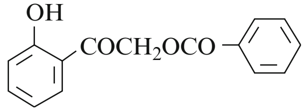


Syntheses

- Refer to: [5145,5146].

2-(Benzoyloxy)-1-(2-hydroxyphenyl)ethanone

[52728-02-6] $C_{15}H_{12}O_4$ mol.wt. 256.26



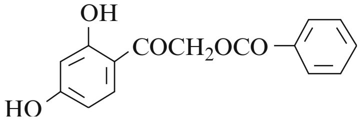
Syntheses

- Preparation by hydrolysis of 2-(benzoyloxy)-1-(2-acetoxy-phenyl)ethanone (SM) with aqueous potassium hydroxide solution by gently warming for 10–15 min on a water bath maintained at 80° (75%) [5137]. SM was obtained by cupric chloride-catalyzed decomposition of o-acetoxy-α-diazoacetophenone in dioxane solution in the presence of benzoic acid (86%, m.p. 110–111°).
- Also obtained by oxidative rearrangement of 2-benzoyloxy-α-bromoacetophenone in moist DMSO for 24 h at 20° (23%) [4394].

m.p. 123–124° [5137], 104–105° [4394]. One of the reported melting points is obviously wrong. ^1H NMR [4394].

2-(Benzoyloxy)-1-(2,4-dihydroxyphenyl)ethanone

[143091-87-6] $C_{15}H_{12}O_5$ mol.wt. 272.26



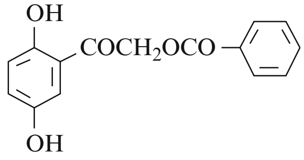
Syntheses

- Preparation by reaction of benzoyloxyacetonitrile (so called benzoylglycollonitrile) with resorcinol (Hoesch reaction), (79%) [5147], (63%) [5148].
- Also refer to: [5062,5149,5150].

m.p. 202–203° [5147], 200° [5148].

2-(Benzoyloxy)-1-(2,5-dihydroxyphenyl)ethanone

[117421-24-6] $C_{15}H_{12}O_5$ mol.wt. 272.26



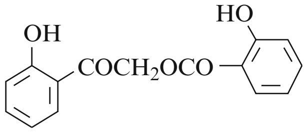
Synthesis

- Obtained by reaction of 2,5-dihydroxy-α-bromoacetophenone with benzoic acid in the presence of triethylamine in acetonitrile at 45° for 4 h [4421].

MS [4421]; HPLC [4421].

2-(2-Hydroxyphenyl)-2-oxoethyl 2-hydroxybenzoate

[68176-44-3] $C_{15}H_{12}O_5$ mol.wt. 272.26



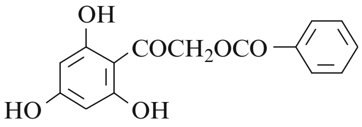
Synthesis

- Preparation by hydrolysis of 1-(2-acetoxyphenyl)-2-[(2-methoxybenzoyl)oxy]ethanone (SM) with aqueous potassium hydroxide solution by gently warming for 10–15 min on a water bath maintained at 80° (70%) [5137]. SM was obtained by cupric chloride-catalyzed decomposition of o-acetoxy-α-diazoacetophenone in dioxane solution in the presence of o-acetoxybenzoic acid (35%, m.p. 124–125°).

m.p. 139–140° [5137].

2-(Benzoyloxy)-1-(2,4,6-trihydroxyphenyl)ethanone

[65982-77-6] $C_{15}H_{12}O_6$ mol.wt. 288.26



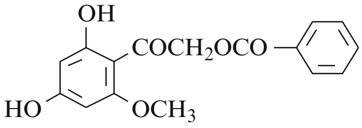
Syntheses

- Obtained by reaction of benzoyloxyacetonitrile with phloroglucinol (Hoesch reaction) [4870,4877,4879,5000], (89%) [5106], (67%) [5147].
- Also refer to: [4873,5062,5151–5153].

m.p. 235° [5106], 234–235° [5147], 220–225° [5062].

2-(Benzoyloxy)-1-(2,4-dihydroxy-6-methoxyphenyl)ethanone

$C_{16}H_{14}O_6$ mol.wt. 302.28



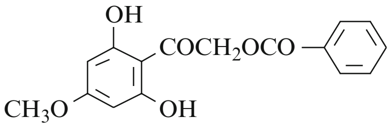
Synthesis

- Obtained by reaction of benzoyloxyacetonitrile with phloroglucinol monomethyl ether (Hoesch reaction) (68%) [4879].

m.p. 145° [4879].

2-(Benzoyloxy)-1-(2,6-dihydroxy-4-methoxyphenyl)ethanone

$C_{16}H_{14}O_6$ mol.wt. 302.28



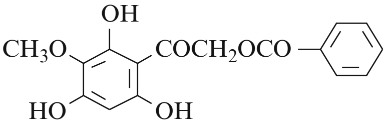
Syntheses

- Obtained by partial methylation of α-(benzoyl-oxy)phloroacetophenone with diazomethane in ethyl ether for 4 h at 5° (37%) [5062] or in a methanol/ethyl ether mixture for 1 h at 0° (27%) [4879].
- Also refer to: [4877].

m.p. 215–217° [5062], 211° [4879].

2-(Benzoyloxy)-1-(2,4,6-trihydroxy-3-methoxyphenyl)ethanone

[1162-73-8] $C_{16}H_{14}O_7$ mol.wt. 318.28



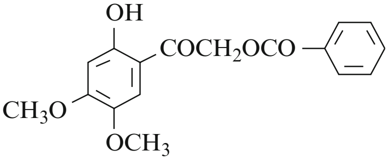
Syntheses

- Obtained by reaction of benzoyloxyacetonitrile with iretol (Hoesch reaction) [5154], (28%) [5155].
- Also refer to: [5156].

m.p. 227–229° [5155]; ^{1}H NMR [5155], IR [5155], UV [5155].

2-(Benzoyloxy)-1-(2-hydroxy-4,5-dimethoxyphenyl)ethanone

$C_{17}H_{16}O_6$ mol.wt. 316.31



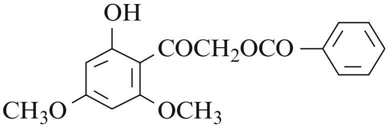
Synthesis

- Obtained by reaction of benzoyloxyacetonitrile with 3,4-dimethoxyphenol (Hoesch reaction) (17%) [4655].

m.p. 128° [4655].

2-(Benzoyloxy)-1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone

[147437-71-6] $C_{17}H_{16}O_6$ mol.wt. 316.31



Syntheses

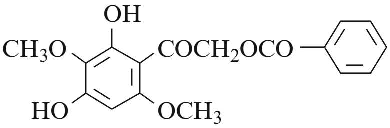
- Preparation by reaction of benzoyloxyacetonitrile with phloroglucinol dimethyl ether (Hoesch reaction) (56%) [4879].
- Preparation by reaction of benzoyloxyacetonitrile with phloroglucinol (Hoesh reaction), followed by partial methylation of the obtained ketone with dimethyl sulfate [5118].
- Preparation by partial methylation of α-(benzoyloxy)phloroacetophenone with excess diazomethane in ethyl ether for 2 h (50%) [4879] or for 4 h at 5° (6%) [5062].
- Also refer to: [5157].

m.p. 135° [4879], 132° [5062], 120–122° [5157]. One of the reported melting points is obviously wrong.

^{1}H NMR [5157], ^{13}C NMR [5157], IR [5157], UV [5157], MS [5157].

2-(Benzoyloxy)-1-(2,4-dihydroxy-3,6-dimethoxyphenyl)ethanone

[7741-48-2] $C_{17}H_{16}O_7$ mol.wt. 332.31



Syntheses

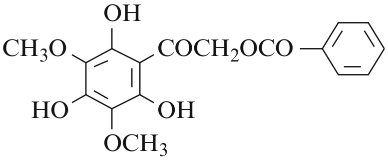
- Preparation by hydrogenation of 2-(benzoyloxy)-1-[4-(benzyloxy)-2-hydroxy-3,6-dimethoxy-phenyl]ethanone in ethyl acetate in the presence of Pd/C (86%) [4942].

- Also obtained by reaction of benzoyloxyacetonitrile with 2,5-dimethoxyresorcinol (Hoesch reaction) (51%) [5158].

m.p. 177° [5158], 175–176° [4942]; UV [4942,5158].

2-(Benzoyloxy)-1-(2,4,6-trihydroxy-3,5-dimethoxyphenyl)ethanone

[1167-74-4] $C_{17}H_{16}O_8$ mol.wt. 348.31



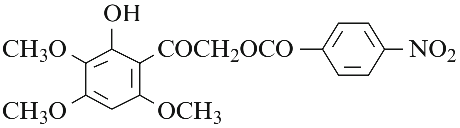
Syntheses

- Obtained by reaction of benzoyloxyacetonitrile with 2,4-dimethoxyphloroglucinol (Hoesch reaction) (12–15%) [5155].
- Also refer to: [5159].

m.p. 139–142° [5155]; ^{1}H NMR [5155], IR [5155], UV [5155].

1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-2-[(4-nitrobenzoyl)oxy]ethanone

[116512-01-7] $C_{18}H_{17}NO_9$ mol.wt. 391.33



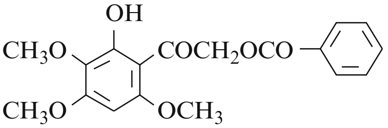
Synthesis

- Preparation by treatment of 2,3,4,6-tetra-methoxy-α-(p-nitrobenzoyloxy)acetophenone with aluminium chloride in acetonitrile at 60° for 2 h (90%) [5160].

m.p. 173–174° [5160]; ^{1}H NMR [5160].

2-(Benzoyloxy)-1-(2-hydroxy-3,4,6-trimethoxyphenyl)ethanone

[7741-49-3] $C_{18}H_{18}O_7$ mol.wt. 346.34



Synthesis

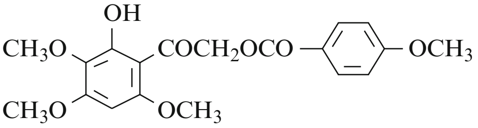
- Preparation by partial methylation of 2-(benzoyl-oxy)-1-(2,4-dihydroxy-3,6-dimethoxyphenyl)ethanone with diazomethane (71%) [4942].

m.p. 172–174° [4942]; UV [4942].

2-(2-Hydroxy-3,4,6-trimethoxyphenyl)-2-oxoethyl 4-methoxybenzoate

[116512-00-6]

$C_{19}H_{20}O_8$ mol.wt. 376.36



Synthesis

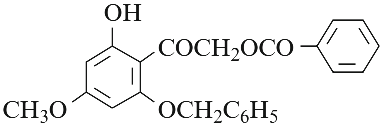
- Preparation by treatment of 2,3,4,6-tetra-methoxy-α-(p-methoxybenzoyloxy)-acetophenone with aluminium chloride in acetonitrile at 60° for 2 h (90%) [5160].

m.p. 151–152° [5160]; ^{1}H NMR [5160].

2-(Benzoyloxy)-1-[2-hydroxy-4-methoxy-6-(phenylmethoxy)phenyl]ethanone

[14585-08-1]

$C_{23}H_{20}O_6$ mol.wt. 392.41



Synthesis

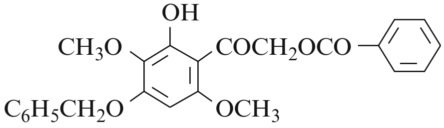
- Obtained by reaction of benzyl chloride with α- benzoyloxy-2,6-dihydroxy-4-methoxy-acetophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone for 3.5 h (17%) [4877].

m.p. 151–153° [4877].

2-(Benzoyloxy)-1-[2-hydroxy-3,6-dimethoxy-4-(phenylmethoxy)phenyl] ethanone

[10048-37-0]

$C_{24}H_{22}O_7$ mol.wt. 422.43



Synthesis

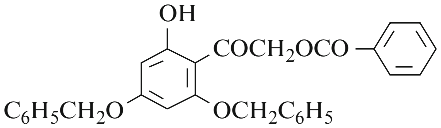
- Obtained by reaction of benzoyloxyacetonitrile with 1,3-bis(benzyloxy)-2,4-di-methoxy-benzene (Hoesch reaction) (32%) [4942].

m.p. 150–151° [4942]; UV [4942].

2-(Benzoyloxy)-1-[2-hydroxy-4,6-bis(phenylmethoxy)phenyl]ethanone

[14585-09-2]

$C_{29}H_{24}O_6$ mol.wt. 468.51



Synthesis

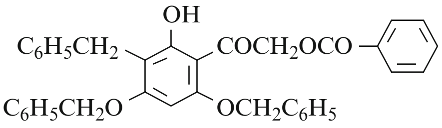
- Obtained by reaction of benzyl chloride with α-(benzoyloxy)phloroacetophenone in the presence of potassium carbonate in refluxing acetone for 2 h (12%) [4877] or for 26 h (13%) [5151].

m.p. 140° [5151], 136° [4877]; UV [5151].

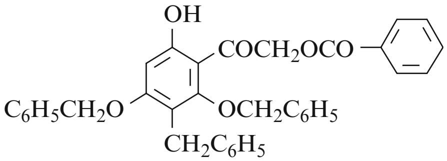
2-(Benzoyloxy)-1-[2-hydroxy-4,6-bis(phenylmethoxy)-3-(phenylmethyl)phenyl]ethanone

or

2-(Benzoyloxy)-1-[6-hydroxy-2,4-bis(phenylmethoxy)-3-(phenylmethyl)phenyl]ethanone



or



$C_{36}H_{30}O_6$ mol.wt. 558.63

Synthesis

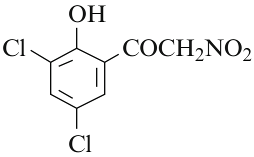
- Obtained by reaction of benzyl chloride with α-(benzoyloxy)phloracetophenone in refluxing acetone for 26 h in the presence of potassium carbonate (7%) or in the presence of potassium carbonate and sodium iodide in the same time (12%). The same result was obtained using only benzyl bromide [5151].

m.p. 182° [5151]; UV [5151].

Chapter 17
Compounds Derived from Nitroacetic Acids

1-(3,5-Dichloro-2-hydroxyphenyl)-2-nitroethanone

[60795-15-5] $C_8H_5Cl_2NO_4$ mol.wt. 250.04



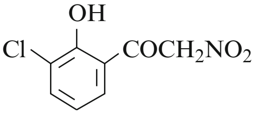
Synthesis

– Preparation by treatment of 6,8-dichloro-4-hydroxy-3-nitro-coumarin with 4% potassium hydroxide solution at r.t. for 24 h (85%) [5161].

m.p. 137° [5161]; IR [5161].

1-(3-Chloro-2-hydroxyphenyl)-2-nitroethanone

[60795-09-7] $C_8H_6ClNO_4$ mol.wt. 215.59



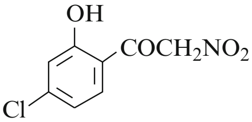
Synthesis

– Preparation by treatment of 8-chloro-4-hydroxy-3-nitro-coumarin with 4% potassium hydroxide solution at r.t. for 24 h (83%) [5161].

m.p. 103° [5161]; IR [5161].

1-(4-Chloro-2-hydroxyphenyl)-2-nitroethanone

[60795-11-1] $C_8H_6ClNO_4$ mol.wt. 215.59



Synthesis

– Preparation by treatment of 7-chloro-4-hydroxy-3-nitro-coumarin with 4% potassium hydroxide solution at r.t. for 24 h (73%) [5161].

m.p. 117–118° [5161]; IR [5161].

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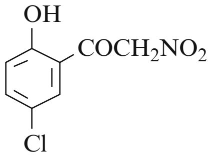
DOI 10.1007/978-1-4020-9787-4_17,

1-(5-Chloro-2-hydroxyphenyl)-2-nitroethanone

[60795-14-4] $C_8H_6ClNO_4$ mol.wt. 215.59



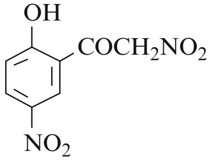
Syntheses

- Preparation by treatment of 6-chloro-4-hydroxy-3-nitro-coumarin with 4% potassium hydroxide solution at r.t. for 24 h (65%) [5161].
- Also refer to: [5162].

m.p. 112° [5161]; IR [5161].

1-(2-Hydroxy-5-nitrophenyl)-2-nitroethanone

[59507-91-4] $C_8H_6N_2O_6$ mol.wt. 226.15



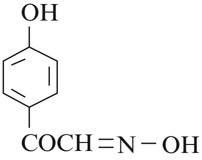
Synthesis

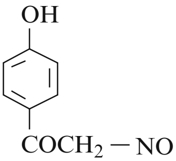
- Preparation by heating a solution of 3,6-dinitro-4-hydroxy-coumarin (m.p. 188°) in 10% aqueous sodium hydroxide at 65° for 2 h, then cooling and acidification with hydrochloric acid (91%) [5056].

m.p. 160° [5056]; ^{1}H NMR [5056].

1-(4-Hydroxyphenyl)-2-nitrosoethanone

[143527-88-2] $C_8H_7NO_3$ mol.wt. 165.15



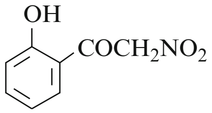


Synthesis

- Refer to: [5163].

1-(2-Hydroxyphenyl)-2-nitroethanone

[29378-60-7] $C_8H_7NO_4$ mol.wt. 181.15



Syntheses

- Preparation by alkaline degradation of 4-hydroxy-3-nitro-coumarin [5164]—m.p. 177° (d)—with 5% sodium hydroxide,
  - for 1.5 h at 50–60° (95%) [5056];
  - for 24 h at 20°, (79%) [4712], (75%) [5161].
- The same compound was isolated in reactions of either some *coumarins* or some *chromenes* with 5% sodium hydroxide for 1 h at r.t. or by heating at 90–95° (70–90%) [5165]:

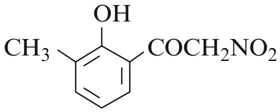
Coumarins	
3-Nitro-4-(pyridylamino)coumarin	m.p. 224–225°
3-Nitro-4-(3-methyl-2-pyridylamino)coumarin	m.p. 227–229°
3-Nitro-4-(4-methyl-2-pyridylamino)coumarin	m.p. 243–244°
3-Nitro-4-(5-methyl-2-pyridylamino)coumarin	m.p. 225–226°
3-Nitro-4-(6-methyl-2-pyridylamino)coumarin	m.p. 250–252°
Chromenes	
2-Hydroxy-3-nitro-4-(3-methyl-2-pyridylimino)-4*H*-chromene	m.p. 245–246°
2-Hydroxy-3-nitro-4-(4-methyl-2-pyridylimino)-4*H*-chromene	m.p. 250–252°
2-Hydroxy-3-nitro-4-(5-methyl-2-pyridylimino)-4*H*-chromene	m.p. 233–234°
2-Hydroxy-3-nitro-4-(6-methyl-2-pyridylimino)-4*H*-chromene	m.p. 274–275°

- Also refer to: [5142,5162,5166–5169].

m.p. 106–107° [4712], 106° [5056], 105–106° [5161], 96–97° [5165]; ^{1}H NMR [5056,5161,5165], IR [5056,5161,5165].

1-(2-Hydroxy-3-methylphenyl)-2-nitroethanone

[60795-08-6] $C_9H_9NO_4$ mol.wt. 195.17



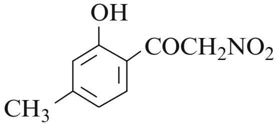
Synthesis

- Preparation by treatment of 4-hydroxy-8-methyl-3-nitro-coumarin with 4% potassium hydroxide solution at r.t. for 24 h (80%) [5161].

m.p. 126° [5161]; ^{1}H NMR [5161], IR [5161].

1-(2-Hydroxy-4-methylphenyl)-2-nitroethanone

[60795-10-0] $C_9H_9NO_4$ mol.wt. 195.17



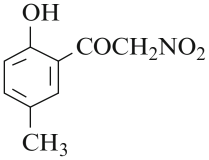
Synthesis

- Preparation by treatment of 4-hydroxy-7-methyl-3-nitro-coumarin with 4% potassium hydroxide solution at r.t. for 24 h (72%) [5161].

m.p. 114° [5161]; IR [5161].

1-(2-Hydroxy-5-methylphenyl)-2-nitroethanone

[60795-13-3] $C_9H_9NO_4$ mol.wt. 195.17



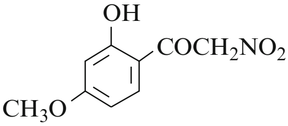
Syntheses

- Preparation by treatment of 4-hydroxy-6-methyl-3-nitro-coumarin with 4% potassium hydroxide solution at r.t. for 24 h (88%) [5161].
- Also refer to: [5162,5168,5170].

m.p. 134° [5161]; ^{1}H NMR [5161], IR [5161].

1-(2-Hydroxy-4-methoxyphenyl)-2-nitroethanone

[60795-12-2] $C_9H_9NO_5$ mol.wt. 211.17



Syntheses

- Preparation by treatment of 4-hydroxy-7-methoxy-3-nitro-coumarin with 4% potassium hydroxide solution at r.t. for 24 h (76%) [5161].
- Also refer to: [5142,5171].

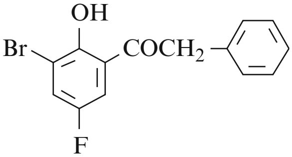
m.p. 140° [5161]; IR [5161].

Chapter 18
Compounds Derived from Arylacetic Acids

18.1 Compounds Derived from Phenylacetic Acid

1-(3-Bromo-5-fluoro-2-hydroxyphenyl)-2-phenylethanone

[4108-04-7] $C_{14}H_{10}BrFO_2$ mol.wt. 309.13



Syntheses

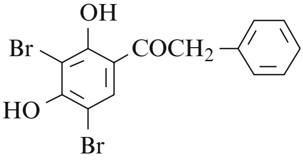
- Preparation by Fries rearrangement of 2-bromo-4-fluoro-phenyl phenylacetate with aluminium chloride, according to the methods [5172],
  - without solvent at 130° for 2 h [5173];
  - in nitrobenzene at 25° for 6 h [5173].

- Also refer to: [5174].

m.p. 130° [5174]; b.p.$_{1.5}$ 154–155° [5173].

1-(3,5-Dibromo-2,4-dihydroxyphenyl)-2-phenylethanone

[19816-40-1] $C_{14}H_{10}Br_2O_3$ mol.wt. 386.04



Syntheses

- Obtained by reaction of bromine (2 mol) with 4-phenyl-acetylresorcinol in acetic acid for 48 h [5175].
- Also obtained by Friedel–Crafts acylation of 2,4-dibromo-resorcinol with phenylacetyl chloride in nitrobenzene in the presence of aluminium chloride, first at r.t. overnight, then heating on a steam bath for 4 h (44%) [5176].

m.p. 190° [5176], 180° [5175].

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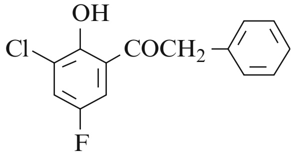
1-(3-Chloro-5-fluoro-2-hydroxyphenyl)-2-phenylethanone

[4108-05-8] $C_{14}H_{10}ClFO_2$ mol.wt. 264.68



Syntheses

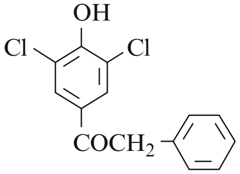
– Preparation by Fries rearrangement of 2-chloro-4-fluoro-phenyl phenylacetate with aluminium chloride, according to the methods [5172],
  - without solvent at 130° for 2 h [5173];
  - in nitrobenzene at 25° for 6 h [5173].

– Also obtained by Friedel–Crafts acylation of p-fluoroanisole, followed by demethylation and chlorination of the obtained ketone [5174].

m.p. 122° [5174]; b.p.$_{1-1.5}$ 209–210° [5173].

1-(3,5-Dichloro-4-hydroxyphenyl)-2-phenylethanone

[73048-86-9] $C_{14}H_{10}Cl_2O_2$ mol.wt. 281.11



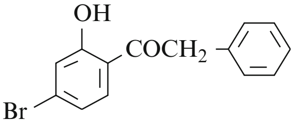
Synthesis

– Obtained by DDQ oxidation of 1-(3,5-dichloro-4-hydroxy-phenyl)-2-phenylethanol in dioxane at r.t. for 16 h (82%) [5177].

m.p. 132–135° [5177]; ^{1}H NMR [5177].

1-(4-Bromo-2-hydroxyphenyl)-2-phenylethanone

[54981-35-0] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



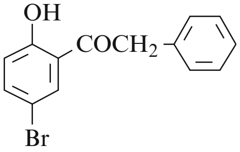
Synthesis

– Obtained by Fries rearrangement of 3-bromophenyl phenylacetate with aluminium chloride in carbon disulfide for 1 h at r.t. [5178].

m.p. 68° [5178].

1-(5-Bromo-2-hydroxyphenyl)-2-phenylethanone

[54981-34-9] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



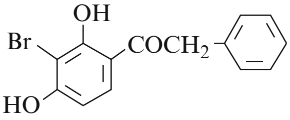
Synthesis

– Obtained by Fries rearrangement of 4-bromophenyl phenylacetate with aluminium chloride in carbon disulfide for 1 h at r.t. [5178].

m.p. 70° [5178].

1-(3-Bromo-2,4-dihydroxyphenyl)-2-phenylethanone

[19816-35-4]



$C_{14}H_{11}BrO_3$ mol.wt. 307.14

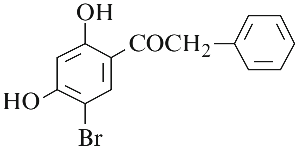
Synthesis

- Obtained by Friedel–Crafts acylation of 2-bromoresorcinol with phenylacetyl chloride in nitrobenzene in the presence of aluminium chloride, first at r.t. overnight, then heating on a steam bath for 4 h (50%) [5176].

m.p. 195° [5176].

1-(5-Bromo-2,4-dihydroxyphenyl)-2-phenylethanone

[92152-59-5]



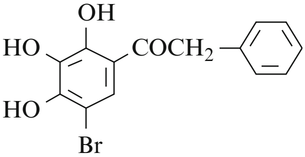
$C_{14}H_{11}BrO_3$ mol.wt. 307.14

Syntheses

- Obtained by reaction of bromine (1 mol) with 4-phenyl-acetylresorcinol in acetic acid at r.t. for 24 h [5175].
- Also obtained by Friedel–Crafts acylation [5179] of 4-bromoresorcinol with phenylacetyl chloride in nitrobenzene in the presence of aluminium chloride [5180].
- Also refer to: [5181].

m.p. 112° [5179,5180], 103° [5175].

1-(5-Bromo-2,3,4-trihydroxyphenyl)-2-phenylethanone



$C_{14}H_{11}BrO_4$ mol.wt. 323.14

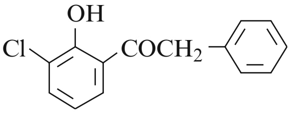
Synthesis

- Obtained by reaction of bromine with 4-phenylacetyl-pyrogallol in acetic acid [5182], at r.t. for 24 h [5175].

m.p. 164° [5182], 155–156° [5175].

1-(3-Chloro-2-hydroxyphenyl)-2-phenylethanone

[70331-83-8]



$C_{14}H_{11}ClO_2$ mol.wt. 246.69

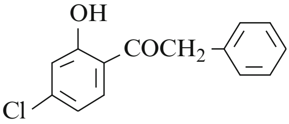
Syntheses

- Preparation by Friedel–Crafts reaction [5183].
- Also refer to: [5184].

protonation constants [5185];
complexes with Cu (II), Ni (II) and Co (II) [5185].

1-(4-Chloro-2-hydroxyphenyl)-2-phenylethanone

[107410-55-9] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



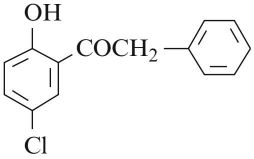
Synthesis

- Preparation by Fries rearrangement of m-chlorophenyl phenylacetate with aluminium chloride, first in carbon disulfide for 1 h, then, after elimination of the solvent, at 100° for 5 h (58%) [5186].

m.p. 62–64° [5186].

1-(5-Chloro-2-hydroxyphenyl)-2-phenylethanone

[126260-45-5] $C_{14}H_{11}ClO_2$ mol.wt. 246.69



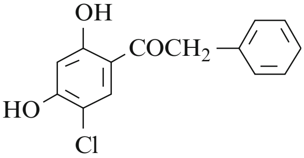
Syntheses

- Obtained by Friedel–Crafts acylation of p-chlorophenol with phenylacetic acid in the presence of boron trifluoride at 160° for 4 h in a sealed tube (68%) [5187].
- Also obtained by Fries rearrangement of p-chlorophenyl phenylacetate with aluminium chloride in refluxing chlorobenzene for 4 h (25%) [5188].
- Also obtained by Friedel–Crafts acylation of p-chloroanisole with phenylacetyl chloride in the presence of aluminium chloride in refluxing carbon disulfide for 5 h (25%) [4471].

m.p. 69° [5187], 66–67° [4471], 64–65° [5188];
$b.p._{0.3}$ 153–154° [5187], $b.p._{30}$ 280–285° [4471].

1-(5-Chloro-2,4-dihydroxyphenyl)-2-phenylethanone

[92103-22-5] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



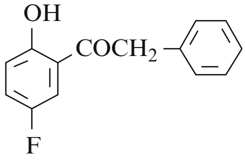
Syntheses

- Preparation by Friedel–Crafts acylation [5179] of 4-chlororesorcinol with phenylacetyl chloride in nitrobenzene in the presence of aluminium chloride, first at 10°, then at r.t. for 36 h (73%) [5180].
- Also refer to: [5181].

m.p. 121° [5180], 120° [5179].

1-(5-Fluoro-2-hydroxyphenyl)-2-phenylethanone

[343-59-9] $C_{14}H_{11}FO_2$ mol.wt. 230.24



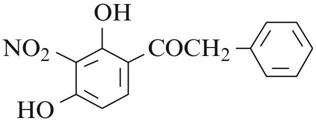
Syntheses

- Preparation by Fries rearrangement of p-fluorophenyl phenylacetate with aluminium chloride,
  - at 150–180° for 20 min (85%) [5189];
  - at 130° for 2 h (77%) [5190], according to the method [5191];
  - at 130° for 2 h [5173], according to the method [5172];
  - in nitrobenzene at 25° for 6 h [5173], according to the method [5172].

m.p. 85° [5189]; b.p.$_{1.5}$ 175–179° [5173], b.p.$_{1-2}$ 200–205° [5190].

1-(2,4-Dihydroxy-3-nitrophenyl)-2-phenylethanone

[19816-52-5] $C_{14}H_{11}NO_5$ mol.wt. 273.25



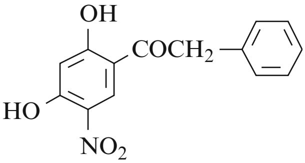
Synthesis

- Preparation by Fries rearrangement of 2,6-di(phenyl-acetoxy)nitrobenzene (m.p. 124°) with aluminium chloride in nitrobenzene, first at r.t. overnight, then at 70–80° for 30 min (72%) [5176].

m.p. 109° [5176].

1-(2,4-Dihydroxy-5-nitrophenyl)-2-phenylethanone

$C_{14}H_{11}NO_5$ mol.wt. 273.25



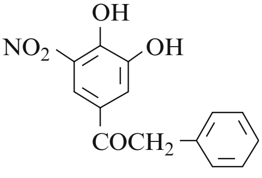
Synthesis

- Obtained by reaction of fuming nitric acid (d = 1.5) with 4-phenylacetylresorcinol in acetic acid in an ice bath for 48 h [5175].

m.p. 156–157° [5175].

1-(3,4-Dihydroxy-5-nitrophenyl)-2-phenylethanone

[274925-86-9] $C_{14}H_{11}NO_5$ mol.wt. 273.25

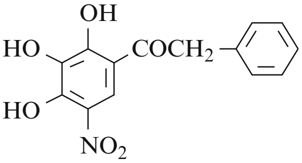


Synthesis

- Preparation by treatment of 1-(4-hydroxy-3-methoxy-5-nitrophenyl)-2-phenylethanone with aluminium chloride in refluxing ethyl acetate/ pyridine mixture for 2 h (99%) [5192,5193].

m.p. 178–179° [5193], 177°6–178°8 [5192];
^{1}H NMR [5192,5193], ^{13}C NMR [5192,5193], IR [5192,5193];
HPLC [5192], IR [5192,5193].

1-(2,3,4-Trihydroxy-5-nitrophenyl)-2-phenylethanone



$C_{14}H_{11}NO_6$ mol.wt. 289.24

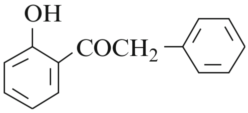
Synthesis

- Obtained by reaction of fuming nitric acid (d = 1.5) with 4-phenylacetylpyrogallol in acetic acid in an ice bath for 48 h [5175].

m.p. 179–180° [5175].

1-(2-Hydroxyphenyl)-2-phenylethanone

[2491-31-8] $C_{14}H_{12}O_2$ mol.wt. 212.25



Syntheses

- Obtained by Fries rearrangement of phenyl phenylacetate,
  - with aluminium chloride,
    - without solvent, between 80° and 130° for 1 h (72%) [5194], at 140° for 3 h (60%) [5195–5197], at 120° for 4 h [5198,5199] or at 60° for 4 h (26%) [5200];
    - in nitrobenzene at 60° for 4 h (14%) [5201], at r.t. for 24 h (4%) [5195,5196];
    - in nitroethane at r.t. for 24 h (<13%) [5202];
    - in chlorobenzene at 50° for 4 h (21%) [5200];
  - with titanium tetrachloride in chlorobenzene at 50° for 4 h (<5%) [5200];
  - with polyphosphoric acid at 100° (1%) [5203];
  - with or without 20% *Bleicherde* at 200° for 9 h (poor yields) [5204].
- Also obtained by stirring a mixture of S-[3-hydroxy-4-(phenylacetyl)phenyl] dimethylcarbamothioate, Raney nickel and ethanol at r.t. for 1 h (67%) [5205].
- Also obtained by photo-Fries rearrangement of phenyl phenylacetate,
  - in the presence of α- or β-cyclodextrin in organic solvents [5206];
  - included in a Nafion membrane, at r.t. for 7 h (quantitative yield) [5207].
- Also obtained by acylation of phenol with phenylacetic acid,
  - in the presence of boron trifluoride etherate under argon on a water bath for 1.5 h (23%) [5208];
  - in the presence of zinc chloride and phosphorous oxychloride for 24 h at r.t. (21%) [5209];
  - in the presence of polyphosphoric acid at 100° (4%) [5203].
- Also obtained by degradation of 3-phenyl-4-hydroxycoumarin in refluxing 30% ethanolic hydrogen chloride for 1 h (56%) [5210].
- Also obtained by demethylation of 2-methoxyphenyl benzyl ketone (oil, $b.p._{0.001}$ 130–140°),

  - with 47% hydrobromic acid (d = 1.5) in acetic acid for 5 h at reflux (87%) [5199] or for 10 h on a steam bath (61%) [5211];
  - with aluminium chloride in nitrobenzene on a steam bath for 1 h (36%) [5211].

- Also obtained by hydrolysis of (2-methoxybenzoyl)phenylacetonitrile (m.p. 108–109°) in acetic acid,
  - with concentrated hydrochloric acid on a steam bath for 20 h (48%) [5211];
  - with 47% hydrobromic acid on a steam bath for 10 h (34%) [5211].
- Also obtained from ethyl (2-methoxybenzoyl)phenylacetate (m.p. 67–68°),
  - with boiling pyridinium chloride for 20 min (ca. 220°) (47%) [5212];
  - in acetic acid with concentrated hydrochloric acid for 15 h on a steam bath (35%) [5211].
- Also obtained by heating under reflux flavone with 5% aqueous sodium hydroxide [5213].
- Also refer to: [5214–5223].

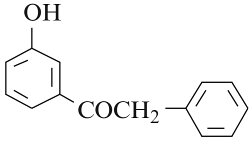
N.B.: Complexes with Mn (II) [5224], Ni (II) [5224], Hg (II) [5224] and Co (II) [5224,5225].

m.p. 60–61° [5205], 60° [5198,5199], 59° [5214], 58–60° [5200], 57–58° [5212], 56–57° [5211], 55° [5195–5197,5204,5210].

b.p.$_{0.004}$ 150–155° [5211], b.p.$_{23}$ 165° [5204];
^{1}H NMR [5205], IR [5195–5197], UV [5195–5197],
MS [5205]; GC [5207]; GC-MS [5207].

1-(3-Hydroxyphenyl)-2-phenylethanone

[332072-68-1] $C_{14}H_{12}O_2$ mol.wt. 212.25



Synthesis

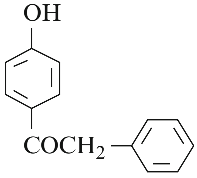
- Obtained by electrolysing in an undivided cell a DMF solution containing 3-iodophenol, chloromethylbenzene, iron pentacarbonyl and a catalytic amount of a nickel-2,2′-bi-pyridine complex (57%) [5226].
- This compound seems to have not been described previously.

It is not mentioned in the ***Chemical Abstracts*** between 1907 (volume 1) and 2000 (volume 133) under the various denominations, namely: m-Hydroxy-α-phenylacetophenone, 3′-Hydroxy-2-phenyl-acetophenone and actually 1-(3-Hydroxyphenyl)-2-phenylethanone, neither in the ***Beilsteins Handbuch der Organischen Chemie*** under the denomination [3-Oxy-phenyl]-benzyl-keton. This ketone might very likely be prepared more simply by diazotization of the 1-(3-aminophenyl)-2-phenylethanone [55251-36-0], an amino ketone known for a long time [5227].

^{1}H NMR [5226], ^{13}C NMR [5226], IR [5226], MS [5226].

1-(4-Hydroxyphenyl)-2-phenylethanone

[2491-32-9] $C_{14}H_{12}O_2$ mol.wt. 212.25



Syntheses

- Obtained by reaction of phenylacetic acid with phenol,
  - in the presence of zinc chloride (Nencki reaction),
    - at 170–200° (reflux) for 1.5 h (20%) [5228,5229];
    - at 170–180° [5230], for 2 h (15%) [5231];
  - in the presence of zinc chloride and phosphorous oxychloride for 24 h at r.t. (75%) [5209];
  - in the presence of polyphosphoric acid in a boiling water bath for 15 min (28%) [5232] or at 100° (19%) [5203];
  - in the presence of boron trifluoride at 80° for 2 h (87%) [5233];
  - in the presence of boron trifluoride etherate under argon on a water bath for 1.5 h (75%) [5208].
- Also obtained by Friedel–Crafts acylation of phenol with phenylacetyl chloride in nitrobenzene in the presence of aluminium chloride (60%) [5231], at 80–90° for 1.5 h (61%) [5234] or at ≤80° for 0.75 h (60–70%) [5229].
- Also obtained (by-product) by reaction of phenylacetyl chloride with anisole in benzene in the presence of stannic chloride between 55° and 75° for 1 h [5228].
- Also obtained by Fries rearrangement of phenyl phenylacetate,
  - with aluminium chloride,
    - without solvent at 50° for 4 h (25%) [5200], at 80° for 4 h (72%) [5200], first in a water bath for 1 h, then at 120° for 4 h [5198,5199] or at 140° for 3 h (10%) [5195–5197];
    - in nitrobenzene at r.t. for 12 h (35%) [5202] or for 24 h (65%) [5195,5196], at 50° for 4 h (64%) [5200], at 60° for 4 h (64%) [5201];
    - in nitroethane at r.t. for 24 h (70%) [5202];
    - in nitropropane at 70° for 6 h (30%) [5202];
  - with polyphosphoric acid at 100° (8%) [5203];
  - with titanium tetrachloride in chlorobenzene at 50° for 4 h (19%) [5200].
- Also obtained by diazotization of p-aminodeoxybenzoin [5235].
- Also obtained (poor yield) by heating phenyl phenylacetate with or without 20% of *Bleicherde* for 9 h at 200° [5204].
- Also obtained by photo-Fries rearrangement of phenyl phenylacetate in the presence of α- or β-cyclodextrin in organic solvents [5206].
- Also refer to: [5236–5247].

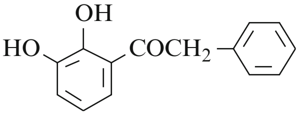
m.p. 151° [5198,5199], 149° [5231], 148° [5209], 146–147° [5228], 145–147° [5200], 144° [5230,5248], 143° [5232], 142° [5195,5196,5229,5233], 141° [5204], 139–142° [5234], 129° [5235].

There is discrepancy between the different melting points.

b.p.$_1$ 220–230° [5231]; IR [5195,5199], UV [5195,5199,5230,5248].

1-(2,3-Dihydroxyphenyl)-2-phenylethanone

[107410-01-5] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

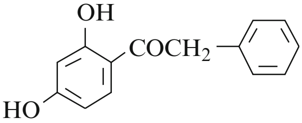
– Preparation by total demethylation of 2,3-dimethoxy-desoxybenzoin (yellow oil, b.p.$_3$ 170–173°) with hydrobromic acid (d = 1.5) in refluxing acetic acid for 5 h (74%) [5199].

– Also obtained by alkaline degradation of 8-hydroxyisoflavone (m.p. 222–224°) with sodium hydroxide in refluxing methanol for 1.5 h [5199].

m.p. 79–81° [5199]; paper chromatography [5199].

1-(2,4-Dihydroxyphenyl)-2-phenylethanone

[3669-41-8] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

– Preparation by Friedel–Crafts acylation of resorcinol with phenylacetyl chloride in the presence of aluminium chloride,

- in methylene chloride (85%) [5184];
- in nitrobenzene at 70–80° for 15 min (<80%) [5249], at r.t. for 24 h (63%) [5250] or for 2 days (60%) [5175];
- in ethyl ether at r.t. for 24 h (43%) [5251].

– Preparation by acylation of resorcinol with phenylacetyl chloride in boiling ethylene dichloride (84°), using a series of clay based catalysts (KSF, KSF/0, KP10, K10, K0, KS) (65–81%) [5252], (60%) [5253].

– Preparation by reaction of phenylacetic anhydride with resorcinol,

- in the presence of concentrated sulfuric acid as catalyst at 130° for some minutes (60%) [5254];
- in the presence of boron trifluoride etherate for 2.5 h at 70–75° (48%) [5255].

– Preparation by reaction of phenylacetic acid with resorcinol,

- in the presence of boron trifluoride etherate under argon on a water bath for 1 h (89%) [5208];

- in the presence of boron trifluoride at 105–108° for 15 min, followed by hydrolysis of the obtained boron difluoride chelate (m.p. 154–155°) (78%) [5256] or at 90° for 1 h (66%) [5257];
- in the presence of boron trifluoride in chloroform (87%) [5258];
- in the presence of zinc chloride (Nencki reaction) at 120° for 2.5 h (70%) [5175], at 125–135° [5259], at 140° for 15 min (10%) [5249] or at 145–150° for 2 h [5230];
- in the presence of zinc chloride and phosphorous oxychloride for 24 h at r.t. (50%) [5209];
- in the presence of 70% perchloric acid at 150° for 30 min (30%) [5260];
- in the presence of Amberlite IR-120, a cation exchange resin sulfonic acid type, at 160° for 2–3 h (41%) [5261].

N.B.: Zeokarb 225 was found to be as effective.

- Preparation by reaction of phenylacetonitrile with resorcinol (Hoesch reaction) [5262–5265], (64%) [5266], (58%) [5267], (40%) [5268].
- Also obtained by heating 2-phenyl-4-benzylidene-7-hydroxy-[4*H*]-1-benzopyran (SM) with refluxing aqueous sodium hydroxide for 1 h. SM was obtained by condensation of 1,4-diphenyl-1,3-butanediol with resorcinol in acetic acid [5269].
- Also obtained by degradation of 7-hydroxy-2-methyl-3-phenylchromone (m.p. 244–246°) with refluxing 5% aqueous sodium hydroxide for 3 h [5175].
- Also obtained by treatment of ethyl (2,4-dimethoxybenzoyl)phenylacetate with boiling pyridinium chloride (ca. 220°) for 20 min (48%) [5212].
- Also refer to: [4685,4935,5181,5205,5216,5217,5220,5270–5282].

N.B.: Complexes with Mn (II), Co (II), Ni (II) and Hg (II) [5224].

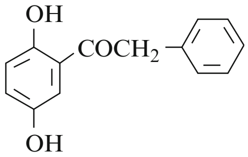
m.p. 116° [5230,5248], 115–116° [5175,5283], 115° [5250,5257,5258,5260,5262,5269], 114–116° [5261], 114–115° [5254,5264,5275], 114° [5249,5284], 113–115° [5256], 113–114° [5251], 113° [5209], 110–113° [5212];

b.p.$_{10}$ 220–225° [5259];

^{1}H NMR [5268], IR [5256], UV [5230,5248,5256,5264], MS [5268].

1-(2,5-Dihydroxyphenyl)-2-phenylethanone

[52122-86-8] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

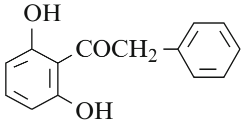
- Obtained by partial demethylation of 2,5-dimethoxyphenyl benzyl ketone,
  - with hydriodic acid in acetic acid on a water bath for 2 h [4989];
  - with hydrobromic acid in acetic acid, first at 0°, then at reflux for 6 h [5285].

- Also obtained by Friedel–Crafts acylation of hydroquinone with phenylacetyl chloride in the presence of aluminium chloride in nitrobenzene, keeping overnight, then on a water bath for 3 h [5285] or at 70–80° for 15 min [5249].
- Also obtained by acylation of hydroquinone with phenylacetic acid,
  - in the presence of boron trifluoride etherate under argon on a water bath for 6 h (68%) [5208];
  - in the presence of boron trifluoride (saturation) at 125° for 1.5 h (56%) [5257];
  - in the presence of zinc chloride at 150° [5249] (Nencki reaction).
- Also refer to: [5286].

m.p. 170° [5249], 113° [5285], 112° [5257], 109°5 [4989].
One of the reported melting points is obviously wrong.

1-(2,6-Dihydroxyphenyl)-2-phenylethanone

[13936-92-0] $C_{14}H_{12}O_3$ mol.wt. 228.25



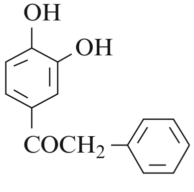
Syntheses

- Obtained by treatment of 3,5-dicarbomethoxy-2,6-di-hydroxyphenyl benzyl ketone (m.p. 129–131°),
  - with boiling 10% alcoholic caustic soda for 3 h. The obtained dicarboxylic acid was decarboxylated by boiling with water for 3 h (75%) [5287];
  - with refluxing 4% methanolic potassium hydroxide for 4 h, followed by refluxing 12 h in water (35%) [5288].
- Also refer to: [5217,5258].

m.p. 170° [5287], 166–167° [5288].

1-(3,4-Dihydroxyphenyl)-2-phenylethanone

[107410-02-6] $C_{14}H_{12}O_3$ mol.wt. 228.25



Syntheses

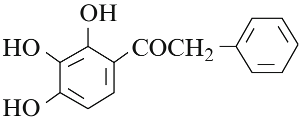
- Preparation by reaction of phenylacetic acid with pyrocatechol,
  - in the presence of zinc chloride and phosphorous oxychloride for 24 h at r.t. (60%) [5209];
  - in the presence of phosphorous oxychloride for 2 h at 90–100° (59%) [5234];
  - in the presence of boron trifluoride in chloroform, first at 10°, then at r.t. overnight (36%) [5289];
  - in the presence of zinc chloride at 140–150° (Hoesch reaction) [5249].

- Also obtained by total demethylation of 3,4-dimethoxydesoxybenzoin (m.p. 87–88°) with hydrobromic acid (d = 1.5) in refluxing acetic acid for 5 h [5289].

m.p. 173–174° [5289], 173° [5209,5249], 168–170° [5234].

2-Phenyl-1-(2,3,4-trihydroxyphenyl)ethanone

[22761-00-8] $C_{14}H_{12}O_4$ mol.wt. 244.25



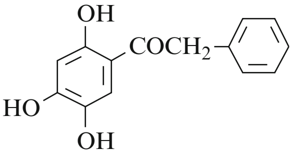
Syntheses

- Obtained by reaction of phenylacetic acid with pyrogallol,
  - in the presence of zinc chloride at 150° for 30 min (Nencki reaction) [5230], (52%) [5290] or at 120° for 2.5 h (60%) [5175];
  - in the presence of Amberlite IR-120 cation exchange resin (sulfonic acid type) at 160° for 2–3 h (34%) [5261]. **N.B.**: Zeokarb 225 was found to be as effective;
  - in the presence of 70% perchloric acid at 150° for 30 min (25%) [5260];
  - in the presence of boron trifluoride in chloroform, first in ice cooling, then at r.t. overnight (96%) [5258].
- Also obtained by Friedel–Crafts acylation of pyrogallol,
  - with phenylacetyl chloride in nitrobenzene in the presence of aluminium chloride at r.t. for 2 days (50%) [5175];
  - with phenylacetic anhydride in the presence of boron trifluoride etherate for 2.5 h at 75–80° (26%) [5255].
- Also refer to: [5220,5272,5277].

m.p. 147° [5260], 144–145° [5175], 141–142° [5290],
140–141° [5230,5248,5258], 135–136° [5261].

2-Phenyl-1-(2,4,5-trihydroxyphenyl)ethanone

[787-06-4] $C_{14}H_{12}O_4$ mol.wt. 244.25



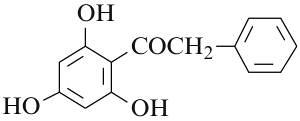
Syntheses

- Obtained by reaction of phenylacetonitrile with hydroxy-hydroquinone (Hoesch reaction) (43%) [5291].
- Also refer to: [5292].

m.p. 208–210° [5291]; ^{13}C NMR [5293].

2-Phenyl-1-(2,4,6-trihydroxyphenyl)ethanone

[727-71-9] $C_{14}H_{12}O_4$ mol.wt. 244.25



Syntheses

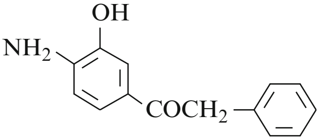
- Preparation by reaction of phenylacetonitrile with phloroglucinol (Hoesch reaction),
  - in the presence of zinc chloride [4852,5262,5264,5265], (44–45%) [5250,5294], (39%) [5255];
  - in the presence of boron trifluoride etherate (50%) [5255].
- Also obtained by reaction of phenylacetic acid with phloroglucinol in the presence of zinc chloride and phosphorous oxychloride for 24 h at r.t. (50%) [5209].
- Also refer to: [5217,5220,5263,5272,5273,5295,5296].

N.B.: Complexes with Mn (II), Co (II) Ni (II) and Hg (II) [5224].
The monohydrate of this ketone was at first obtained [5250,5262]. The water of crystallisation is lost on heating the crystals at 90°.

m.p. 164–165° [5264], 163° [5209], 162° [5250,5262,5294];
IR [5294], UV [5264,5294]; TLC [5294].

1-(4-Amino-3-hydroxyphenyl)-2-phenylethanone

[54903-53-6] $C_{14}H_{13}NO_2$ mol.wt. 227.26



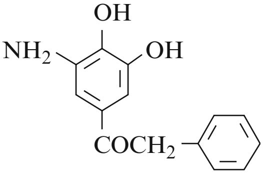
Syntheses

- Preparation from 6-phenylacetyl-benzoxazolinone by alkaline hydrolysis with boiling 10% aqueous sodium hydroxide solution for 4 h (90–100%) [5297].
- Also refer to: [5298].

m.p. 141–142° [5297].

1-(3-Amino-4,5-dihydroxyphenyl)-2-phenylethanone

[473790-02-2] $C_{14}H_{13}NO_3$ mol.wt. 243.26



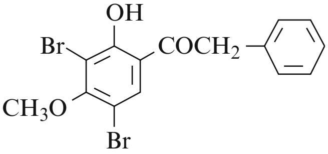
Synthesis

- Preparation by hydrogenation of 1-(3,4-dihydroxy-5-nitro-phenyl)-2-phenylethanone in methanolic suspension in the presence of 10% Pd/C at r.t. for 2 h (91%) [5299].

m.p. 234–237° [5299];
^{1}H NMR [5299], ^{13}C NMR [5299], IR [5299].

1-(3,5-Dibromo-2-hydroxy-4-methoxyphenyl)-2-phenylethanone

[19816-38-7] $C_{15}H_{12}Br_2O_3$ mol.wt. 400.07



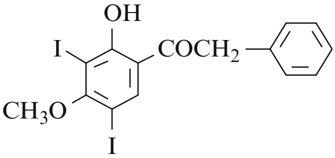
Syntheses

– Obtained by alkaline degradation of two substituted isoflavones with 10% sodium hydroxide in refluxing ethanol for 4 h,
  - from 6,8-dibromo-7-methoxyisoflavone (m.p. 139°) (92%) [5176];
  - from 2,6,8-tribromo-7-methoxyisoflavone (m.p. 218°) [5176].

m.p. 120° [5176].

1-(2-Hydroxy-3,5-diiodo-4-methoxyphenyl)-2-phenylethanone

[19816-44-5] $C_{15}H_{12}I_2O_3$ mol.wt. 494.07



Syntheses

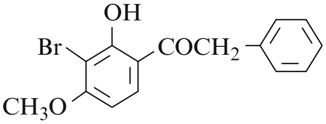
– Obtained by alkaline degradation of 6,8-diiodo-7-methoxyisoflavone (m.p. 210°) with 10% sodium hydroxide in refluxing ethanol for 4 h [5176].

– Also obtained by iodination of benzyl 2-hydroxy-3-iodo-4-methoxyphenyl ketone with iodine and iodic acid in ethanol at 60–70° overnight [5176].

m.p. 140° [5176].

1-(3-Bromo-2-hydroxy-4-methoxyphenyl)-2-phenylethanone

[19816-33-2] $C_{15}H_{13}BrO_3$ mol.wt. 321.17



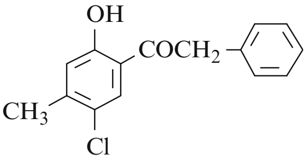
Synthesis

– Obtained by alkaline degradation of 8-bromo-7-methoxy-isoflavone (m.p. 178°) wit 10% sodium hydroxide in refluxing ethanol for 4 h (83%) [5176].

m.p. 198° [5176].

1-(5-Chloro-2-hydroxy-4-methylphenyl)-2-phenylethanone

[92435-54-6] $C_{15}H_{13}ClO_2$ mol.wt. 260.72



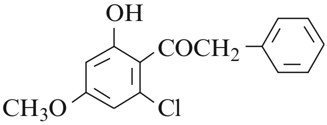
Synthesis

– Preparation by Friedel–Crafts acylation of 4-chloro-3-methylphenol with phenylacetyl chloride in nitrobenzene in the presence of aluminium chloride [5180].

m.p. 112° [5180].

1-(2-Chloro-6-hydroxy-4-methoxyphenyl)-2-phenylethanone

[116475-72-0] $C_{15}H_{13}ClO_3$ mol.wt. 276.72



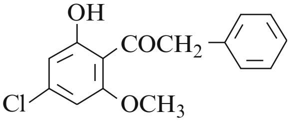
Syntheses

- Obtained by Friedel–Crafts reaction of 1-chloro-3,5-di-methoxybenzene with phenylacetyl chloride in the presence of aluminium chloride and zinc chloride in ethylene dichloride between −10° and −7°, then at r.t. for 1 h and subsequent demethylation at 70° for 1 h (47%) [5300].
- Also refer to: [5301,5302].

m.p. 123–125° [5300]; ^{1}H NMR [5300], IR [5300].

1-(4-Chloro-2-hydroxy-6-methoxyphenyl)-2-phenylethanone

[137986-09-5] $C_{15}H_{13}ClO_3$ mol.wt. 276.72



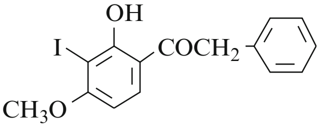
Syntheses

- Obtained by Friedel–Crafts reaction of phenylacetyl chloride with 1-chloro-3,5-dimethoxybenzene in the presence of aluminium chloride and zinc chloride in ethylene dichloride between 5° and 10°, then at r.t. for 1 h and subsequent demethylation at 65–70° for 3 h (54%) [5300].
- Also refer to: [5302].

m.p. 74–75° [5300]; ^{1}H NMR [5300], IR [5300].

1-(2-Hydroxy-3-iodo-4-methoxyphenyl)-2-phenylethanone

$C_{15}H_{13}IO_3$ mol.wt. 368.17



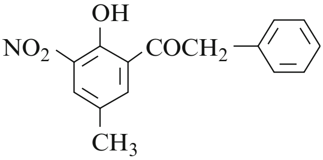
Syntheses

- Obtained by alkaline degradation of 8-iodo-7-methoxy-isoflavone (m.p. 169°) with 10% sodium hydroxide in refluxing ethanol for 4 h (81%) [5176].
- Also obtained by Friedel–Crafts acylation of 2-iodoresorcinol dimethyl ether with phenylacetyl chloride in nitrobenzene in the presence of aluminium chloride and heating for 2 h [5176].

m.p. 217° [5176].

1-(2-Hydroxy-5-methyl-3-nitrophenyl)-2-phenylethanone

[70978-50-6] $C_{15}H_{13}NO_4$ mol.wt. 271.27



Synthesis

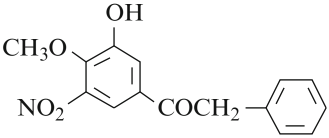
– Obtained by nitration of 2-hydroxy-5-methylphenyl benzyl ketone,

- using standard reagents at −20°(81%) [4699];
- with fuming nitric acid in acetic acid/methylene chloride at r.t. [4700].

m.p. 80–82° [4699,4700].

1-(3-Hydroxy-4-methoxy-5-nitrophenyl)-2-phenylethanone

[473789-93-4] $C_{15}H_{13}NO_5$ mol.wt. 287.27



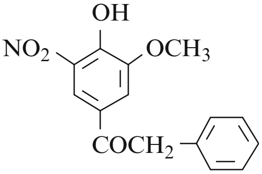
Synthesis

– Obtained by partial methylation of 1-(3,4-dihydroxy-5-nitrophenyl)-2-phenylethanone with dimethyl sulfate in the presence of potassium carbonate in DMF for 1 h at 80° (25%) [5299].

m.p. 121–123° [5299]; ^{1}H NMR [5299], ^{13}C NMR [5299], IR [5299].

1-(4-Hydroxy-3-methoxy-5-nitrophenyl)-2-phenylethanone

[274925-97-2] $C_{15}H_{13}NO_5$ mol.wt. 287.27



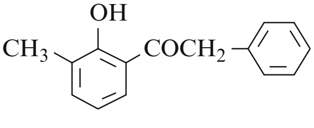
Synthesis

– Preparation by treatment of 1-(4-hydroxy-3-methoxy-phenyl)-2-phenylethanone with 70% nitric acid in acetic acid at r.t. for 30 min (72%) [5192], (71%) [5193].

m.p. 129–130° [5193];
^{1}H NMR [5193], ^{13}C NMR [5193].

1-(2-Hydroxy-3-methylphenyl)-2-phenylethanone

[7294-92-0] $C_{15}H_{14}O_2$ mol.wt. 226.27



Syntheses

– Obtained by Fries rearrangement of o-cresyl phenylacetate,

- in the presence of aluminium chloride,
  – without solvent at 160–180° for 30 min (53%) [5303], at 140° for 3 h (45%) [5196,5304] or at 130° for 4 h (12%) [5305];
  – in nitrobenzene at 60° for 4 h (10%) [5201];

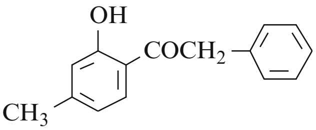
  - with alumina in methanesulfonic acid at 160° for 10 min (15%) [5306].

- Also obtained by Friedel–Crafts acylation of o-cresol with phenylacetic acid in the presence of alumina in methanesulfonic acid at 140° for 5 min (12%) [5306].
- Also obtained by photo-Fries rearrangement of o-tolyl phenylacetate,
  - in the presence of α- or β-cyclodextrin in organic solvents [5206];
  - included in a Nafion membrane at r.t. for 7 h (quantitative yield) [5207].
- Also refer to: [5307].

m.p. 44° [5196,5304]; b.p.$_8$ 176–180° [5303];
IR [5196,5304], UV [5196,5304]; GC [5207]; GC-MS [5207].

1-(2-Hydroxy-4-methylphenyl)-2-phenylethanone

[2491-34-1] $C_{15}H_{14}O_2$ mol.wt. 226.27



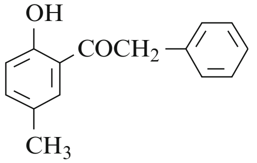
Syntheses

- Preparation by Fries rearrangement of m-cresyl phenylacetate,
  - with aluminium chloride,
    - without solvent, for 3 h at 140° (50%) [5197,5304];
    - starting in carbon disulfide and, after solvent elimination, for 1 h at 60–70°, then 24 h at r.t. (77%) [5308];
    - in nitrobenzene for 4 h at 60° (65%) [5201] or in refluxing nitromethane for 3 h (49%) [5305];
  - with alumina in methanesulfonic acid for 15 min at 160° (90%) [5306].
- Preparation by direct acylation of m-cresol with phenylacetic acid,
  - with boron trifluoride for 2 h at 90° (93%) [5233];
  - with alumina in methanesulfonic acid for 5 min at 120° (83%) [5306].
- Also obtained by hydrolysis of 2-difluoroboryloxy-4-methylphenyl benzyl ketone (SM) (m.p. 125–126°) with refluxing dilute ethanol for 15–20 min. SM was prepared by action of phenylacetic acid with m-cresol in the presence of boron trifluoride etherate for 30 min at 125–130° (50%) [5309].

m.p. 52–53° [5309], 49° [5308], 32–33° [5233].
One of the reported melting points is obviously wrong.
b.p.$_5$ 164° [5197,5304], b.p.$_4$ 170–175° [5233], b.p.$_{17}$ 218° [5308];
IR [5309], UV [5309].

1-(2-Hydroxy-5-methylphenyl)-2-phenylethanone

[24258-63-7] $C_{15}H_{14}O_2$ mol.wt. 226.27



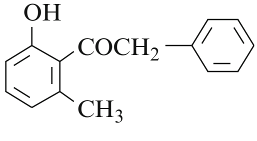
Syntheses

- Preparation by Fries rearrangement of p-cresyl phenylacetate with aluminium chloride (68%) [5310],
  - without solvent at 120–140° for 20 min (82%) [5303], at 130–135° for 30 min (80%) [5311] or at 130–140° for 3 h (14%) [5312];
  - in nitrobenzene at r.t. for 48 h (80%) [5195–5197] or at 60° for 4 h (64%) [5201];
  - in 1,2,4-trichlorobenzene at reflux for 30 min (78%) [5188];
  - in chlorobenzene at reflux for 4 h (23%) [5305] or for 30 min (86%) [5188].
- Preparation by Friedel–Crafts acylation of p-cresol with phenylacetic acid,
  - in the presence of boron trifluoride at 80° for 2 h (89%) [5233];
  - in the presence of zinc chloride [5313].
- Also obtained by Friedel–Crafts acylation of p-cresol methyl ether with phenylacetyl chloride in the presence of aluminium chloride, first in refluxing carbon disulfide for 5 h, then, after solvent elimination, at 120–130° for 5 h (54–61%) [5314].
- Also obtained by photo-Fries rearrangement of p-tolyl phenylacetate,
  - in the presence of α- or β-cyclodextrin in organic solvents [5206];
  - included in a Nafion membrane, at r.t. for 7 h (quantitative yield) [5207].
- Also obtained by reaction of N-diethylaniline with α-bromo-2-hydroxy-5-methyldeoxybenzoin [5314].
- Also refer to: [5315].

m.p. 65–66° [5188,5312], 65° [5195–5197,5314], 64° [5233], 63°5–65° [5310], 63° [5311,5313], 54–58° [5303];
$b.p._7$ 169–174° [5303], $b.p._{0.6}$ 170° [5233], $b.p._6$ 195–199° [5311],
$b.p._{13-14}$ 210–213° [5314]; GC [5207]; GC-MS [5207];
IR [5195–5197], UV [5195–5197].

1-(2-Hydroxy-6-methylphenyl)-2-phenylethanone

[137937-39-4] $C_{15}H_{14}O_2$ mol.wt. 226.27



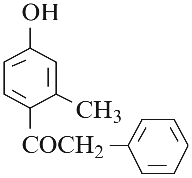
Synthesis

- Obtained (by-product) by Fries rearrangement of m-tolyl phenylacetate with aluminium chloride in chlorobenzene at 140° for 4 h (5%) [5307].

m.p. 85° [5307];
^1H NMR (Sadtler: standard n° 57893 M) [5307],
IR (Sadtler: standard n° 84941 K) [5307], UV [5307], MS [5307].

1-(4-Hydroxy-2-methylphenyl)-2-phenylethanone

[3669-50-9] $C_{15}H_{14}O_2$ mol.wt. 226.27



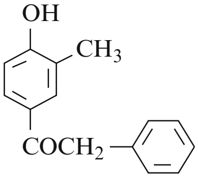
Syntheses

- Obtained by reaction of phenylacetyl chloride with m-cresol in the presence of aluminium chloride in nitrobenzene for 30 min in a boiling water bath (26%) [5316].
- Also obtained by reaction of phenylacetic acid with m-cresol,
  - in the presence of boron trifluoride at 90° for 2 h (3%) [5233];
  - in the presence of zinc chloride at reflux (200°) for 1 h [5316,5317].
- Also obtained by Fries rearrangement of m-cresyl phenylacetate with aluminium chloride,
  - in refluxing chlorobenzene for 4 h (24%) [5305];
  - in nitromethane or in nitroethane at r.t. for 12 h (18–21%) [5202];
  - in nitrobenzene at 60° for 4 h (10%) [5202], (8%) [5201] or at r.t. for 10 h (10%) [5202];
  - without solvent at 140° for 3 h (10%) [5304] or first in carbon disulfide, then after elimination of the solvent, at 60–70° for 1 h and at r.t. for 24 h (2%) [5308].

m.p. 142° [5316], 138–139° [5233], 138° [5304,5308].

1-(4-Hydroxy-3-methylphenyl)-2-phenylethanone

[7354-81-6] $C_{15}H_{14}O_2$ mol.wt. 226.27



Syntheses

- Preparation by Fries rearrangement of o-cresyl phenylacetate with aluminium chloride,
  - in nitroethane at r.t. for 12 h (73%) [5202];
  - in nitrobenzene at 60° for 4 h (72%) [5201] or at 50° for 4 h (60%) [5305];
  - in refluxing chlorobenzene for 4 h (58%) [5305];
  - in refluxing nitromethane for 4 h (21%) [5305];
  - without solvent at 130° for 4 h (49%) [5305] or at 140° for 3 h (30%) [5196,5304].
- Preparation by Fries rearrangement of o-cresyl phenylacetate with alumina in methanesulfonic acid for 10 min at 160° (85%) [5306].
- Also obtained by photo-Fries rearrangement of o-cresyl phenylacetate in the presence of α- or β-cyclodextrin in organic solvents [5206].
- Also obtained by reaction of phenylacetic acid with o-cresol,

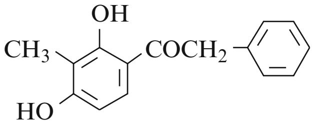
- in the presence of alumina in methanesulfonic acid at 140° for 5 min (88%) [5306];
- in the presence of aluminium chloride in nitrobenzene in a water bath for 1–5 h–1.25 h (60–70%) [5317];
- in the presence of zinc chloride at reflux (180–200°) [5318], (<20%) [5317] (Nencki reaction).

– Also refer to: [5307].

m.p. 156° [5196,5304], 152° [5317];
IR [5196,5304], UV [5196,5304].

1-(2,4-Dihydroxy-3-methylphenyl)-2-phenylethanone

[39581-98-1] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

– Preparation by demethylation of 2-hydroxy-4-methoxy-3-methylphenyl benzyl ketone with hydriodic acid in refluxing acetic anhydride (125–135°) for 2 h (64%) [5319].

– Also obtained by reaction of phenylacetic acid with 2-methylresorcinol in the presence of phosphorous oxychloride and zinc chloride, heating on a water bath for 3 h (45%) [5320].

– Also obtained by reaction of phenylacetonitrile with 2-methylresorcinol (Hoesch reaction) [5321].

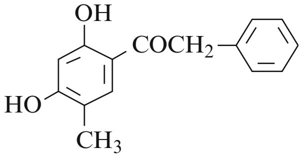
– Also refer to: [5279,5322].

m.p. 178° [5320], 157–159° [5319,5321].

One of the reported melting points is obviously wrong.

1-(2,4-Dihydroxy-5-methylphenyl)-2-phenylethanone

[106737-29-5] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

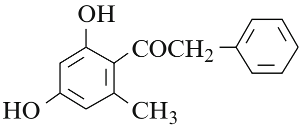
– Preparation by reaction of phenylacetonitrile with 4-methyl-resorcinol (Hoesch reaction) [5323].

– Also obtained by demethylation of 4-hydroxy-2-methoxy-5-methylphenyl benzyl ketone with aluminium chloride in refluxing benzene for 4 h (74%) [5324].

m.p. 98–99° [5323], 96° [5324].

1-(2,4-Dihydroxy-6-methylphenyl)-2-phenylethanone

[55338-29-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



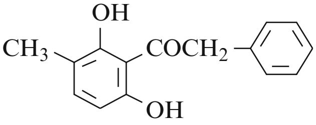
Syntheses

- Preparation by Friedel–Crafts acylation of orcinol with phenylacetyl chloride in nitrobenzene in the presence of aluminium chloride [5180].
- Also refer to: [5325,5326].

m.p. 148° [5180].

1-(2,6-Dihydroxy-3-methylphenyl)-2-phenylethanone

[15578-06-0] $C_{15}H_{14}O_3$ mol.wt. 242.27



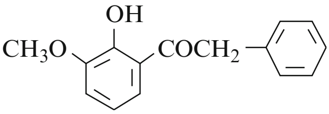
Synthesis

- Obtained by treatment of 5-carbomethoxy-2,6-dihydroxy-3-methyldeoxybenzoin (m.p. 168–170°) with potassium hydroxide in refluxing dilute ethanol for 4 h (38%) [5327].

m.p. 135–136° [5327].

1-(2-Hydroxy-3-methoxyphenyl)-2-phenylethanone

[93899-00-4] $C_{15}H_{14}O_3$ mol.wt. 242.27



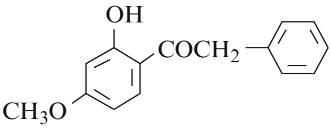
Syntheses

- Preparation from 2-acetoxy-3-methoxybenzonitrile and benzylmagnesium chloride (75–90%) [5328].
- Also obtained (by-product) by reaction of phenylacetic acid with guaiacol in the presence of zinc chloride and phosphorous oxychloride for 24 h at r.t. (5%) [5209].

m.p. 165–168° [5209], 78–79° [5328]. One of the reported melting points is obviously wrong. UV [5209].

1-(2-Hydroxy-4-methoxyphenyl)-2-phenylethanone

[18439-96-8] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

- Preparation by partial methylation of 2,4-dihydroxyphenyl benzyl ketone [5265],
  - with methyl iodide in the presence of potassium carbonate in refluxing acetone for 3 h (85%) [5250] or for 12 h (91%) [5284];
  - with methyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [5329];

- with dimethyl sulfate,
  - in the presence of potassium carbonate in boiling benzene for 90 min (51%) [5330] or in boiling acetone [4935];
  - in the presence of alkali in boiling ethanol [5331].

- Also obtained by Friedel–Crafts acylation of resorcinol dimethyl ether with phenylacetyl chloride in the presence of aluminium chloride in boiling carbon disulfide for 1 h (50%) [5332] or in boiling ethyl ether (33%) [5333].
- Also obtained by acylation of resorcinol monomethyl ether with phenylacetic acid in the presence of polyphosphoric acid at 95° for 30 min (40%) [5334].
- Also obtained by degradation of 4-hydroxy-7-methoxy-3-phenylcoumarin with refluxing 30% ethanolic hydrogen chloride (45%) [5210].
- Also obtained from 4,7-dimethoxy-3-phenylcoumarin on heating with 5% aqueous sodium hydroxide for 2 h on a water bath (71%) [5335].
- Also obtained by hydrolysis of ethyl 2,4-dimethoxybenzoyl-phenylacetate (m.p. 76–77°) in acetic acid with concentrated hydrochloric acid on a steam bath for 15 h (68%) [5211].
- Also obtained by hydrolysis of 2,4-dimethoxybenzoyl-phenylacetonitrile (m.p. 108–109°) in acetic acid with concentrated hydrochloric acid on a steam bath for 15 h (47%) [5211].
- Also obtained by condensation of phenylacetonitrile with resorcinol monomethyl ether (Hoesch reaction) (23%) [5336].
- Also refer to: [4935,5215,5216,5222,5270,5273,5277,5278,5337–5340].

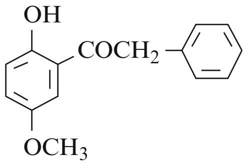
m.p. 92° [5332], 90° [5284,5331,5336], 88–89° [5211],
88° [5250,5330], 87–88° [5335], 86–87° [5329], 86° [5210], 75° [5334].

One of the reported melting points is obviously wrong.

b.p.$_{0.001}$ 155–165° [5211]; ^{1}H NMR [5341,5337].

1-(2-Hydroxy-5-methoxyphenyl)-2-phenylethanone

[80427-38-9] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

- Preparation by Fries rearrangement of p-methoxyphenyl phenylacetate with titanium tetrachloride at 120° for 1 h (60–74%) [5342].
- Also obtained by Friedel–Crafts acylation of hydroquinone dimethyl ether with phenylacetyl chloride in the presence of aluminium chloride,

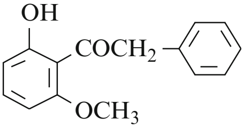
- in carbon disulfide at r.t. for 1 h (by-product) [5343];
- in refluxing ethyl ether for 8 h [5344], (43%) [5217].

- Also obtained (poor yield) by partial degradation of 6-methoxy-3-phenyl-4-hydroxycoumarin with 30% ethanolic hydrogen chloride at reflux for 1 h [5210].
- Also refer to: [5345,5346].

m.p. 45° [5217], 44° [5342]; IR [5342], UV [5342].

1-(2-Hydroxy-6-methoxyphenyl)-2-phenylethanone

[40584-06-3] $C_{15}H_{14}O_3$ mol.wt. 242.27



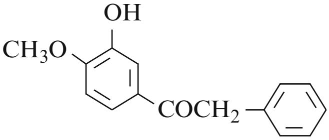
Syntheses

- Obtained by partial methylation of benzyl 2,6-dihydroxy-phenyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone [5217].
- Also obtained on heating 3-(6-methoxy-2-tosyloxyphenyl)-3-oxo-2-phenylpropanal (m.p. 137–138°) with ethanolic potassium hydroxide (2N) at reflux for 2 h (88%) [5347].

m.p. 71° [5347], 66° [5217]; IR [5347].

1-(3-Hydroxy-4-methoxyphenyl)-2-phenylethanone

[58451-99-3] $C_{15}H_{14}O_3$ mol.wt. 242.27



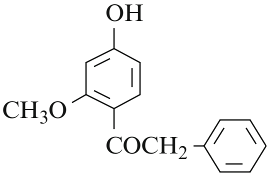
Syntheses

- Preparation from 2-methoxy-5-(phenylacetyl)phenyl phenylacetate (SM) with refluxing methanolic potassium hydroxide (91%) [5348]. SM was obtained by acylation of 2-methoxyphenyl phenylacetate with phenylacetyl chloride in the presence of stannic chloride in nitromethane for 1 h at 20° (76%, m.p. 95°).
- Preparation from 3-benzyloxy-4-methoxybenzonitrile and benzylmagnesium chloride (75–90%) [5328].
- Preparation by treatment of 3-benzyloxy-4-methoxyphenyl benzyl ketone (SM) with a mixture of concentrated hydrochloric acid and acetic acid (1:2 v/v) and heating at 70° for 1 h (69%) [5349]. SM was obtained by oxidation of 1-(3-benzyloxy-4-methoxyphenyl)-2-phenylethanol (m.p. 79–82°) with potassium dichromate in dilute sulfuric acid at 50° for 1 h (80%, m.p. 105–106°).

m.p. 106–107° [5328,5349], 101° [5348];
^{1}H NMR (Sadtler: standard n° 28214 M) [5349],
IR (Sadtler: standard n° 55286) [5348], UV [5348], MS [5349].

1-(4-Hydroxy-2-methoxyphenyl)-2-phenylethanone

[85288-47-7] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

- Obtained by acylation of resorcinol monomethyl ether with phenylacetic acid in the presence of polyphosphoric acid at 95° for 30 min (30%) [5334].

- Also obtained (by-product) by condensation of phenylacetonitrile with resorcinol monomethyl ether (Hoesch reaction) (7%) [5336].

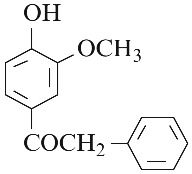
m.p. 113° [5336], 86° [5334].

One of the reported melting points is obviously wrong.

b.p.$_{13}$ 260–265° [5336]; ^{1}H NMR [5334,5341].

1-(4-Hydroxy-3-methoxyphenyl)-2-phenylethanone

[66476-02-6] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

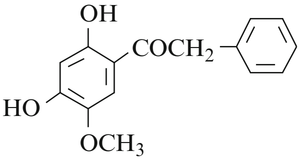
- Preparation by oxidation of 1-(4-hydroxy-3-methoxyphenyl)-2-phenylethanol with DDQ in dioxane at r.t. for 16 h (89%) [5177].
- Preparation by reaction of benzylmagnesium chloride with 4-acetoxy-3-methoxybenzonitrile (72%) [5350].
- Preparation by reaction of phenylacetic acid with guaiacol in the presence of zinc chloride and phosphorous oxychloride for 24 h at r.t. (60%) [5209].
- Preparation from 1-(4-benzyloxy-3-methoxyphenyl)-2-phenylethanone (m.p. 136–138°) by catalytic hydrogen transfer using ammonium formate as hydrogen donor and 10% Pd/C catalysis in refluxing methanol for 30 min (94%) [5193].

N.B.: Na salt [5350].

m.p. 110–111° [5350], 108–110° [5177], 108° [5209], 106–108° [5193]; ^{1}H NMR [5177,5193], ^{13}C NMR [5193], UV [5209].

1-(2,4-Dihydroxy-5-methoxyphenyl)-2-phenylethanone

[79744-57-3] $C_{15}H_{14}O_4$ mol.wt. 258.27



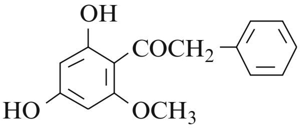
Syntheses

- Refer to: [5293,5351].

^{13}C NMR [5293].

1-(2,4-Dihydroxy-6-methoxyphenyl)-2-phenylethanone

[55607-18-6] $C_{15}H_{14}O_4$ mol.wt. 258.27



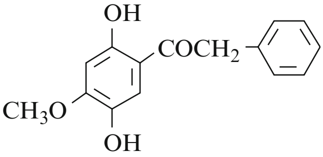
Syntheses

- Obtained by reaction of phenylacetonitrile with phloro-glucinol monomethyl ether (Hoesch reaction) (38%) [5295].
- Preparation by tosylation of 2,4,6-trihydroxyphenyl benzyl ketone with p-toluenesulfonyl chloride in the presence of potassium carbonate in refluxing acetone for 4 h, followed by methylation with dimethyl sulfate (reflux 30 h) and final detosylation with refluxing ethanolic sodium hydroxide for 45 min (19%) [5335].
- Also refer to: [5352].

m.p. 146° [5335], 145–146° [5295].

1-(2,5-Dihydroxy-4-methoxyphenyl)-2-phenylethanone

[789-80-0] $C_{15}H_{14}O_4$ mol.wt. 258.27



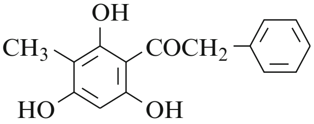
Syntheses

- Obtained by partial methylation of 2,4,5-trihydroxyphenyl benzyl ketone with excess diazomethane in ethyl ether at r.t. overnight (47%) [5291].
- Also obtained by alkaline oxidation of 2-hydroxy-4-methoxyphenyl benzyl ketone with potassium persulfate in aqueous potassium hydroxide/pyridine mixture (Elbs reaction) [5344].
- Also refer to: [5217].

m.p. 153–154° [5344], 150–152° [5291]; ^{13}C NMR [5293].

2-Phenyl-1-(2,4,6-trihydroxy-3-methylphenyl)ethanone

[3136-47-8] $C_{15}H_{14}O_4$ mol.wt. 258.27



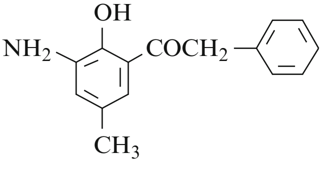
Syntheses

- Obtained by reaction of phenylacetonitrile with 2-methyl-phloroglucinol (Hoesch reaction) [5353].
- Also obtained by reduction of 2,4,6-trihydroxy-3-formyl-phenyl benzyl ketone with hydrogen in acetic acid using 5% Pd/C as catalyst [5354].
- Also refer to: [5296,5355,5356].

m.p. 200° [5353], 198–199° [5354].

1-(3-Amino-2-hydroxy-5-methylphenyl)-2-phenylethanone

[70977-87-6] $C_{15}H_{15}NO_2$ mol.wt. 241.29



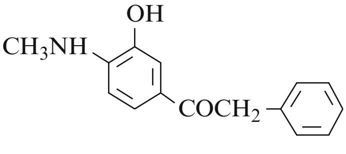
Synthesis

- Preparation by hydrogenation of 2-hydroxy-5-methyl-3-nitrophenyl benzyl ketone in ethanol using 5% Pd/C as catalyst at atmospheric pressure [4700], (74%) [4699].

m.p. 74–76° [4699,4700].

1-[3-Hydroxy-4-(methylamino)phenyl]-2-phenylethanone

[54943-18-9] $C_{15}H_{15}NO_2$ mol.wt. 241.29

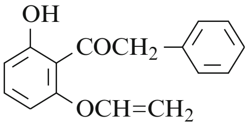


Synthesis

- Preparation from 3-methyl-6-phenylacetylbenzoxazolinone by alkaline hydrolysis with boiling 10% aqueous sodium hydroxide for 4 h (90–100%) [5297].

m.p. 164–165° [5297].

1-[2-(Ethenyloxy)-6-hydroxyphenyl]-2-phenylethanone



$C_{16}H_{14}O_3$ mol.wt. 254.29

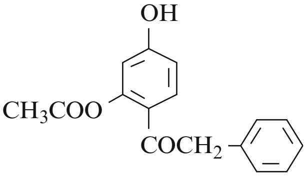
Synthesis

- Obtained (by-product) by reaction of diethylaminochloro-ethane with 2,6-dihydroxydesoxybenzoin in the presence of sodium ethoxide in refluxing ethanol for 4 h (6%) [5357].

m.p. 85° [5357].

1-[2-(Acetyloxy)-4-hydroxyphenyl]-2-phenylethanone

[145747-27-9]



$C_{16}H_{14}O_4$ mol.wt. 270.28

Syntheses

- Obtained by selective deacetylation catalyzed by porcine pancreas lipase in THF at 42–45° of,
  - 1-acetoxy-1-(2,4-diacetoxyphenyl)-2-phenylethene during 72 h (20%) [5265];
  - 2,4-diacetoxyphenyl benzyl ketone during 48 h (65%) [5265] or [5358,5359] (in the table below):

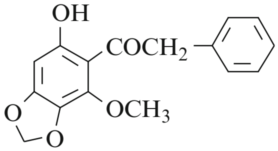
Lipase	Solvent	Time (h)	Yields (%)
PPL	Acetone/n-BuOH	40	35
PPL	CH_3CN/n-BuOH	50	35
PPL	THF/n-BuOH	42	65
CCL	DIPE/n-BuOH	45	60

PPL = porcine pancreas lipase; CCL = candida cylindracea lipase; DIPE = diisopropyl ether

m.p. 140° [5265]; TLC [5265];
^{1}H NMR [5265], ^{13}C NMR [5265], IR [5265], UV [5265], MS [5265].

1-(6-Hydroxy-4-methoxy-1,3-benzodioxol-5-yl)-2-phenylethanone

[2652-17-7]



$C_{16}H_{14}O_5$ mol.wt. 286.28

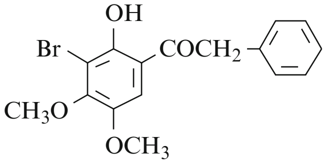
Synthesis

- Obtained by reaction of phenylacetonitrile with 3-methoxy-4,5-methylenedioxyphenol (Hoesch reaction) (24%) [5360].

m.p. 162–164° [5360]; UV [5360].

1-(3-Bromo-2-hydroxy-4,5-dimethoxyphenyl)-2-phenylethanone

[54921-24-3] $C_{16}H_{15}BrO_4$ mol.wt. 351.20



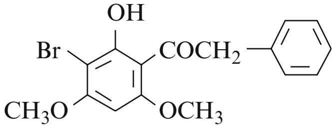
Synthesis

- Obtained by alkaline degradation of 6,7-dimethoxy-8-bromoisoflavone with 3% alcoholic potassium hydroxide at reflux for 30 min (41%) [5361].

m.p. 146° [5361].

1-(3-Bromo-2-hydroxy-4,6-dimethoxyphenyl)-2-phenylethanone

[28750-55-2] $C_{16}H_{15}BrO_4$ mol.wt. 351.20



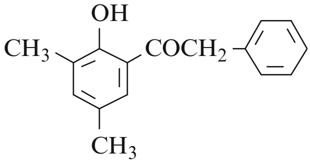
Syntheses

- Preparation by bromination of 2-acetoxy-4,6-dimethoxyphenyl benzyl ketone with bromine in acetic acid for 1 h at r.t. (69%) [5215].
- Also obtained by bromination of 2-hydroxy-4,6-dimethoxyphenyl benzyl ketone with bromine in chloroform under UV light at r.t. overnight (55%) [5294].

m.p 205–206° [5215], 200–202° [5294]; TLC [5294];
^{1}H NMR [5215,5297], IR [5294], UV [5294].

1-(2-Hydroxy-3,5-dimethylphenyl)-2-phenylethanone

[93433-76-2] $C_{16}H_{16}O_2$ mol.wt. 240.30



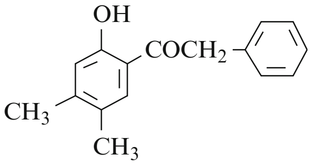
Syntheses

- Preparation by Fries rearrangement of 2,4-dimethylphenyl phenylacetate with aluminium chloride,
  - without solvent for 15 min at 140–145° (85%) [5362] or for 1 h at 120° (56%) [5313];
  - in refluxing chlorobenzene for 4 h (18%) [5305].

b.p.$_{0.01}$ 180–190° [5362], b.p.$_{10}$ 201–203° [5313].

1-(2-Hydroxy-4,5-dimethylphenyl)-2-phenylethanone

[18439-99-1] $C_{16}H_{16}O_2$ mol.wt. 240.30



Syntheses

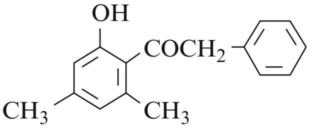
- Preparation by Fries rearrangement of 3,4-dimethylphenyl phenylacetate with aluminium chloride at 130° for 25 min (72%) [5363].

- Also obtained (poor yield) by treatment of 6,7-dimethyl-3-phenyl-4-hydroxycoumarin with refluxing 30% ethanolic hydrogen chloride for 1 h [5210].

m.p. 69–70° [5363], 68° [5210]; ^{1}H NMR [5363], IR [5363].

1-(2-Hydroxy-4,6-dimethylphenyl)-2-phenylethanone

[38319-83-4] $C_{16}H_{16}O_2$ mol.wt. 240.30



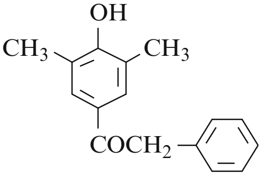
Syntheses

- Preparation by Friedel–Crafts acylation of 3,5-dimethyl-anisole with phenylacetyl chloride in the presence of aluminium chloride, first for 1 h at r.t., then for 1 h at reflux (43%) [5364].
- Also obtained by Fries rearrangement of 3,5-dimethylphenyl phenylacetate with aluminium chloride for 30 min on a water bath (10%) [5313].

b.p.$_{1}$ 168–173° [5364], b.p.$_{20}$ 220–225° [5313];
^{1}H NMR [5364], IR [5364]; $n_D^{23.2} = 1.5921$ [5364].

1-(4-Hydroxy-3,5-dimethylphenyl)-2-phenylethanone

[73049-13-5] $C_{16}H_{16}O_2$ mol.wt. 240.30



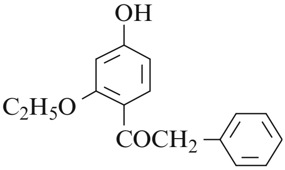
Syntheses

- Preparation by oxidation of 1-(4-hydroxy-3,5-dimethyl-phenyl)-2-phenylethanol with DDQ in dioxane at r.t. for 16 h (83%) [5177].
- Preparation by Fries rearrangement of 2,6-dimethylphenyl phenylacetate with aluminium chloride in refluxing chlorobenzene for 4 h (74%) [5305].

m.p. 117–118° [5177]; ^{1}H NMR [5177].

1-(2-Ethoxy-4-hydroxyphenyl)-2-phenylethanone

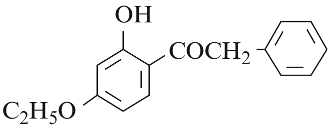
[50775-90-1] $C_{16}H_{16}O_3$ mol.wt. 256.30



Syntheses

- Refer to: [5365,5366].

1-(4-Ethoxy-2-hydroxyphenyl)-2-phenylethanone



$C_{16}H_{16}O_3$ mol.wt. 256.30

Syntheses

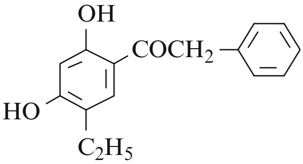
- Obtained by partial ethylation of 2,4-dihydroxydeoxy-benzoin with ethyl iodide in the presence of potassium carbonate in boiling acetone during 3 h (68%) [5250].

- Also refer to: [5357].

m.p. 86° [5250].

1-(5-Ethyl-2,4-dihydroxyphenyl)-2-phenylethanone

[96643-95-7]



$C_{16}H_{16}O_3$ mol.wt. 256.30

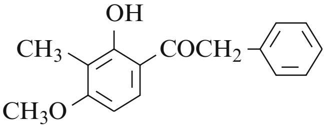
Syntheses

- Preparation by reaction of phenylacetonitrile with 4-ethyl-resorcinol,
- in the presence of boron trifluoride etherate (96%) [5367,5371];
- in the presence of zinc chloride (Hoesch reaction) [5323].

m.p. 105–105°5 [5323], 100–102° [5367,5371]; ^{1}H NMR [5367].

1-(2-Hydroxy-4-methoxy-3-methylphenyl)-2-phenylethanone

[87538-40-7]



$C_{16}H_{16}O_3$ mol.wt. 256.30

Synthesis

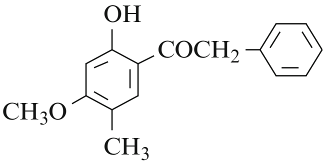
- Obtained by reaction of methyl iodide with benzyl 2,4-di-hydroxyphenyl ketone in methanol in the presence of potassium hydroxide, first at 0°, then standing overnight and refluxing for 6 h (45%) [5319].

- Also refer to: [5278,5338].

m.p. 110–111° [5319].

1-(2-Hydroxy-4-methoxy-5-methylphenyl)-2-phenylethanone

[87538-41-8]

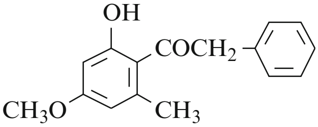


$C_{16}H_{16}O_3$ mol.wt. 256.30

Syntheses

- Refer to: [5278,5338].

1-(2-Hydroxy-4-methoxy-6-methylphenyl)-2-phenylethanone



$C_{16}H_{16}O_3$ mol.wt. 256.30

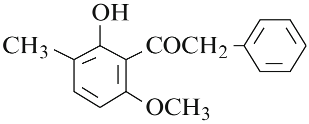
Syntheses

- Obtained by condensation of phenylacetonitrile with orcinol monomethyl ether (1%) (Hoesch reaction) [5336].
- Also obtained by reaction of phenylacetyl chloride with orcinol dimethyl ether in the presence of aluminium chloride [5369].

m.p. 110° [5336].

1-(2-Hydroxy-6-methoxy-3-methylphenyl)-2-phenylethanone

[15578-05-9]



$C_{16}H_{16}O_3$ mol.wt. 256.30

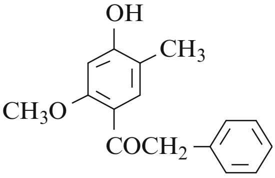
Synthesis

- Obtained by partial methylation of 2,6-dihydroxy-3-methyldeoxybenzoin with dimethyl sulfate in the presence of potassium carbonate in boiling acetone (74%) [5327].

m.p. 80–82° [5327].

1-(4-Hydroxy-2-methoxy-5-methylphenyl)-2-phenylethanone

[101169-10-2]



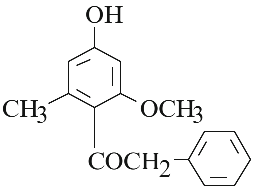
$C_{16}H_{16}O_3$ mol.wt. 256.30

Synthesis

- Obtained by reaction of phenylacetonitrile with 4-methyl-resorcinol dimethyl ether (Hoesch reaction) [5324].

m.p. 129° [5324].

1-(4-Hydroxy-2-methoxy-6-methylphenyl)-2-phenylethanone



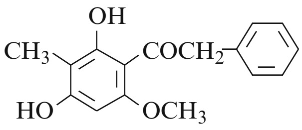
$C_{16}H_{16}O_3$ mol.wt. 256.30

Synthesis

- Obtained (poor yield) by condensation of phenylacetonitrile with orcinol monomethyl ether (7%) (Hoesch reaction) [5336].

m.p. 93° [5336].

1-(2,4-Dihydroxy-6-methoxy-3-methylphenyl)-2-phenylethanone



$C_{16}H_{16}O_4$ mol.wt. 272.30

Synthesis

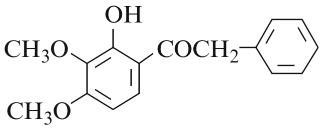
- Obtained by reaction of phenylacetonitrile with 2,6-di-hydroxy-4-methoxytoluene (Hoe-sch reaction) (52%) [5353].

m.p. 141–143° [5353].

1-(2-Hydroxy-3,4-dimethoxyphenyl)-2-phenylethanone

[24852-33-3]

$C_{16}H_{16}O_4$ mol.wt. 272.30



Syntheses

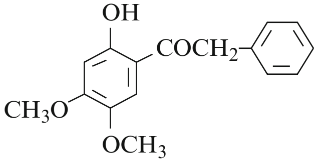
- Obtained by Friedel–Crafts acylation of pyrogallol trimethyl ether with phenylacetyl chloride in the presence of aluminium chloride,
  - in boiling carbon disulfide for 30 min (39%) [5370] or for 12 h [5344];
  - in ice-cold ethyl ether, then at r.t. overnight (57%) [5371].
- Also refer to: [5277,5293,5351,5372,5375].

m.p. 113–114° [5344], 112–113° [5370], 106–107° [5371]; ^{13}C NMR [5293].

1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-phenylethanone

[24195-31-1]

$C_{16}H_{16}O_4$ mol.wt. 272.30



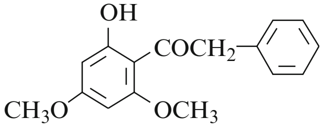
Syntheses

- Obtained by partial methylation of benzyl 2,5-dihydroxy-4-methoxyphenyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone [5217].
- Also obtained (by-product) by reaction of phenylacetyl chloride with hydroxyhydroquinone trimethyl ether in the presence of aluminium chloride [5344,5377,5378].
- Also obtained by reaction of phenylacetonitrile with 3,4-dimethoxyphenol (Hoesch reaction) (56%) [5379].
- Also obtained (compound **7c**) [5380] according to the procedure [5291].

m.p. 94–95° [5379], 94° [5377,5378], 93° [5217];
^{13}C NMR [5293], IR [5379], UV [5379], MS [5379].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-phenylethanone

[39604-66-5] $C_{16}H_{16}O_4$ mol.wt. 272.30



Syntheses

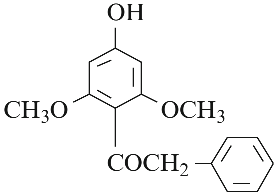
- Obtained by partial methylation of benzyl 2,4,6-tri-hydroxyphenyl ketone [5265],
  - with dimethyl sulfate in the presence of potassium carbonate in boiling acetone [5294], for 1 h [5381], (69%) [5250] or for 14 h (68%) [5382];
  - with methyl iodide in the presence of potassium carbonate in refluxing acetone for 6 h [5353].
- Also obtained by condensation of phenylacetonitrile with phloroglucinol dimethyl ether (Hoesch reaction) (28%) [5383], (19%) [5384].
- Also obtained by Friedel–Crafts acylation of phloroglucinol trimethyl ether with phenylacetyl chloride in ethyl ether in the presence of aluminium chloride, first in an ice bath for 30 min and then at r.t. for 24 h [5385].
- Also obtained from 4,5,7-trimethoxy-3-phenylcoumarin on heating with 5% aqueous sodium hydroxide [5335].
- Also refer to: [4935,5215,5220,5273,5278,5295,5337,5338,5339,5340,5375,5380,5386].

m.p. 118° [5384], 117–118° [5385], 117° [5250,5294,5383], 116° [5353,5382], 115–116° [5335];

^{1}H NMR [5341], IR [5294], UV [5294]; TLC [5294].

1-(4-Hydroxy-2,6-dimethoxyphenyl)-2-phenylethanone

[131196-74-2] $C_{16}H_{16}O_4$ mol.wt. 272.30



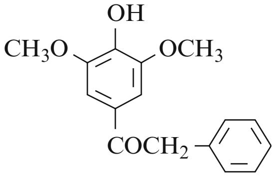
Syntheses

- Obtained (by-product) by condensation of phenylacetonitrile with phloroglucinol dimethyl ether (Hoesch reaction) [5383], (26%) [5384].
- Also obtained by saponification of (4-acetoxy-2,6-di-methoxyphenyl) benzyl ketone (m.p. 108–110°) in ethanol with 3% aqueous sodium hydroxide on a water bath (77%) [5384].

m.p. 77° [5384].

1-(4-Hydroxy-3,5-dimethoxyphenyl)-2-phenylethanone

[73049-12-4] $C_{16}H_{16}O_4$ mol.wt. 272.30

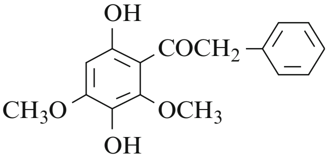


Synthesis

- Preparation by oxidation of 1-(3,5-dimethoxy-4-hydroxy-phenyl)-2-phenylethanol with DDQ in dioxane at r.t. for 16 h (92%) [5177].

m.p. 117–118° [5177]; ^{1}H NMR [5177].

1-(3,6-Dihydroxy-2,4-dimethoxyphenyl)-2-phenylethanone



$C_{16}H_{16}O_5$ mol.wt. 288.30

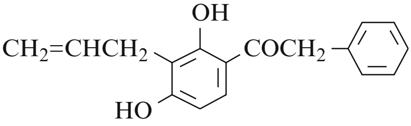
Syntheses

- Preparation by condensation of 2,6-dimethoxy-hydroquinone with the complex phenylacetic acid and boron trifluoride (83%) [5387], (quantitative yield) [5388].
- Also obtained by saponification of 6-hydroxy-2,4-di-methoxy-3-(phenylacetoxy) phenyl benzyl ketone with 10% alcoholic potassium hydroxide for 2 h on a water bath [5387].
- Also obtained (poor yield) by persulfate oxidation of 2-hydroxy-4,6-dimethoxyphenyl benzyl ketone (Elbs reaction) (8%) [5387].

yellow oil [5388]; m.p. 108° [5387]; b.p.$_1$ 220–240° [5387], b.p.$_1$ 230–250° [5388].

1-[2,4-Dihydroxy-3-(2-propenyl)phenyl]-2-phenylethanone

[38987-02-9]



$C_{17}H_{16}O_3$ mol.wt. 268.31

Syntheses

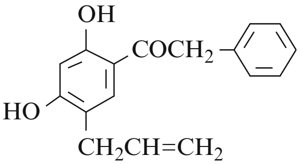
- Obtained by Claisen rearrangement of 4-allyloxy-2-hydroxyphenyl benzyl ketone either using boiling dimethylaniline or heating up to 185–190° under reduced pressure [5276].
- Also obtained by reaction of allyl bromide with 2,4-dihydroxydesoxybenzoin in the presence of methanolic potassium hydroxide (22%) [5283].
- Also refer to: [5277].

m.p. 162–163° [5283], 126° [5276]. One of the reported melting points is obviously wrong.

^{1}H NMR [5283], IR [5283], UV [5283,5279].

1-[2,4-Dihydroxy-5-(2-propenyl)phenyl]-2-phenylethanone

[38987-03-0]



$C_{17}H_{16}O_3$ mol.wt. 268.31

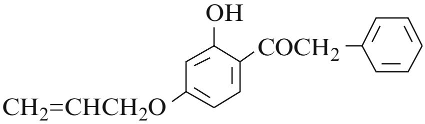
Synthesis

- Obtained by demethylation of 5-allyl-4-hydroxy-2-methoxy-phenyl benzyl ketone with aluminium chloride in ethyl ether or acetonitrile [5276].

m.p. 99–100° [5276]; UV [5276].

1-[2-Hydroxy-4-(2-propenyloxy)phenyl]-2-phenylethanone

[78660-73-8]



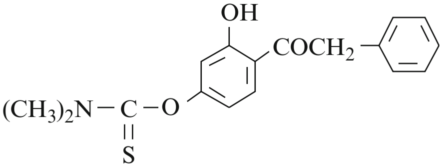
$C_{17}H_{16}O_3$ mol.wt. 268.31

Syntheses

- Refer to: [5276,5279].

O-[3-Hydroxy-4-(phenylacetyl)phenyl] dimethylcarbamothioate

[142751-36-8]



$C_{17}H_{17}NO_3S$ mol.wt. 315.39

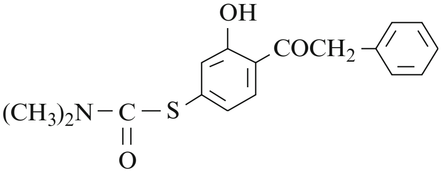
Synthesis

- Obtained by stirring a mixture of 2,4-di-hydroxyphenyl benzyl ketone (1 mol), dimethylthiocarbamoyl chloride (2 mol), 1,4-diazabicyclo[2,2,2]octane (2 mol) and DMF at r.t. for 2 h (95%) [5205].

m.p. 94–95° [5205]; ^{1}H NMR [5205], MS [5205].

S-[3-Hydroxy-4-(phenylacetyl)phenyl] dimethylcarbamothioate

[142751-40-4]



$C_{17}H_{17}NO_3S$ mol.wt. 315.39

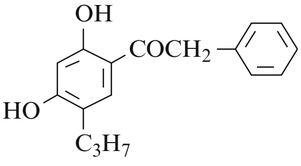
Synthesis

- Obtained by refluxing a solution of O-[3-hydroxy-4-(phenylacetyl) phenyl] dimethylcarbamothioate [142751-36-8] in N,N-dimethylaniline for 1 h (87%) (Newman-Kwart rearrangement) [5205].

m.p. 100–101° [5205]; ^{1}H NMR [5205], MS [5205].

1-(2,4-Dihydroxy-5-propylphenyl)-2-phenylethanone

[96661-12-0]



$C_{17}H_{18}O_3$ mol.wt. 270.33

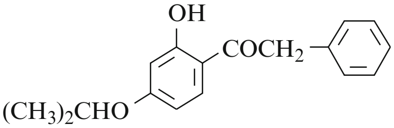
Syntheses

- Preparation by reaction of phenylacetonitrile with 4-propyl-resorcinol,
  - in the presence of boron trifluoride etherate (88%) [5367,5368];
  - in the presence of zinc chloride (Hoesch reaction) [5323].

m.p. 95–96° [5323], 92–93° [5367,5368]; ^{1}H NMR [5367].

1-[2-Hydroxy-4-(1-methylethoxy)phenyl]-2-phenylethanone

[50561-04-1]



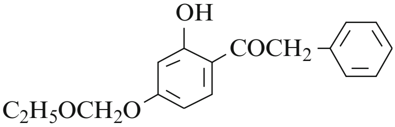
$C_{17}H_{18}O_3$ mol.wt. 270.33

Syntheses

- Obtained by partial alkylation of benzyl 2,4-di-hydroxyphenyl ketone with isopropyl bromide in DMF in the presence of potassium carbonate (79%) [5266].
- Also obtained by alkali degradation of *ipriflavone* (7-(1-methylethoxy)-3-phenyl-[4*H*]-1-benzo-pyran-4-one) (m.p. 115–117°) at high pH (pH >9) (main degradation product) [5389].
- Also refer to: [5365,5366,5390,5391,5392].

1-[4-(Ethoxymethoxy)-2-hydroxyphenyl]-2-phenylethanone

[97714-79-9]



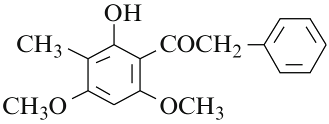
$C_{17}H_{18}O_4$ mol.wt. 286.33

Syntheses

- Obtained by reaction of chloromethyl ethyl ether with benzyl 2,4-dihydroxyphenyl ketone in acetone in the presence of potassium carbonate at r.t. for 15–45 min [5393].
- Also refer to: [5273].

1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-2-phenylethanone

[39604-67-6]



$C_{17}H_{18}O_4$ mol.wt. 286.33

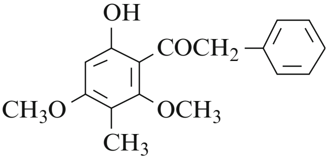
Syntheses

- Preparation by partial methylation of 2,4-dihydroxy-3-methyl-6-methoxyphenyl benzyl ketone with methyl iodide in the presence of potassium carbonate in boiling acetone for 4 h (82%) [5353].
- Also obtained by O and nuclear methylations of 2,4,6-trihydroxyphenyl benzyl ketone with methyl iodide in the presence of potassium carbonate in refluxing acetone for 6 h (11%) [5353].
- Also obtained by partial methylation of 2,4,6-trihydroxy-3-methylphenyl benzyl ketone with dimethyl sulfate or with an excess methyl iodide in the presence of potassium carbonate in refluxing acetone for 3 h [5353].
- Also obtained by reduction of 2-hydroxy-3-formyl-4,6-dimethoxyphenyl benzyl ketone with hydrogen in acetic acid using 5% Pd/C as catalyst (90%) [5354].
- Also refer to: [5381].

m.p. 153–155° [5353], 153–154° [5354].

1-(6-Hydroxy-2,4-dimethoxy-3-methylphenyl)-2-phenylethanone

[22080-99-5]



$C_{17}H_{18}O_4$ mol.wt. 286.33

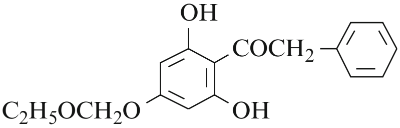
Syntheses

- Obtained (poor yield) by reaction of phenylacetyl chloride with 4-hydroxy-2,6-dimethoxytoluene in ethyl ether in the presence of aluminium chloride for 3 days at r.t. (8%) [5394].
- Also refer to: [5381].

m.p. 47–48° [5394]; IR [5394].

1-[4-(Ethoxymethoxy)-2,6-dihydroxyphenyl]-2-phenylethanone

[97714-81-3]



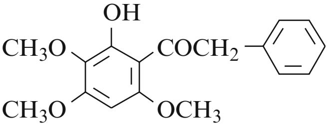
$C_{17}H_{18}O_5$ mol.wt. 302.33

Synthesis

- Obtained by reaction of chloromethyl ethyl ether with benzyl 2,4,6-trihydroxyphenyl ketone in acetone in the presence of potassium carbonate at r.t. for 15–45 min [5393].

1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-2-phenylethanone

[55742-64-8]



$C_{17}H_{18}O_5$ mol.wt. 302.33

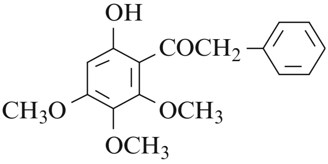
Syntheses

- Obtained by reaction of phenylacetyl chloride with 1,2,3,5-tetramethoxybenzene in the presence of aluminium chloride,
  - in nitrobenzene on a water bath for 6 h (13%) [5395].
  - in ethyl ether for 8 h on a water bath [5344].

m.p. 89° [5395], 85–86° [5344].

1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-2-phenylethanone

[22137-59-3]



$C_{17}H_{18}O_5$ mol.wt. 302.33

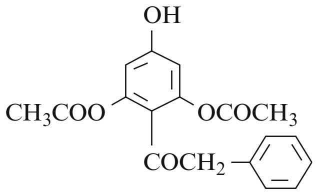
Synthesis

- Preparation by Friedel–Crafts acylation of antiarol with phenylacetyl chloride in ethyl ether in the presence of aluminium chloride at r.t. for 12 h (45%) [5396] or for 24 h (59%) [5251].

m.p. 64° [5396], 63° [5251].

1-[2,6-Bis(acetyloxy)-4-hydroxyphenyl]-2-phenylethanone

[145747-29-1]



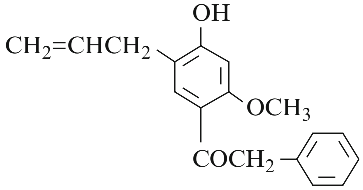
$C_{18}H_{16}O_6$ mol.wt. 328.32

Syntheses

- Obtained by regioselective enzyme-catalyzed deacetylation of benzyl 2,4,6-triacetoxyphenyl ketone in various solvents containing n-butanol with two different lipases at 42–45° for 40 h,
  - using porcine pancreas lipase in acetone or in acetonitrile (40%), in THF (70%) [5358,5359], in diisopropyl ether (65%) [5358];
  - using candida cylindracea lipase in diisopropyl ether (40%) [5359].
- Also obtained (small amount) by selective deacetylation of 1-acetoxy-1-(2,4,6-triacetoxyphenyl)-2-phenylethene (m.p. 67°) using porcine pancreas lipase in THF at 42–45° for 72 h [5265].

1-[4-Hydroxy-2-methoxy-5-(2-propenyl)phenyl]-2-phenylethanone

[39022-25-8]



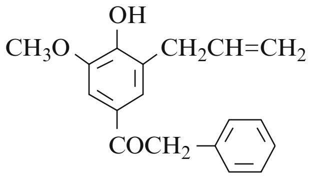
$C_{18}H_{18}O_3$ mol.wt. 282.34

Synthesis

- Obtained by Claisen rearrangement of 4-allyloxy-2-methoxyphenyl benzyl ketone (m.p. 126–127°) either using boiling dimethylaniline or heating up to 185–190° under reduced pressure [5276].

m.p. 127–128° [5276]; UV [5276].

1-[4-Hydroxy-3-methoxy-5-(2-propenyl)phenyl]-2-phenylethanone



$C_{18}H_{18}O_3$ mol.wt. 282.34

Synthesis

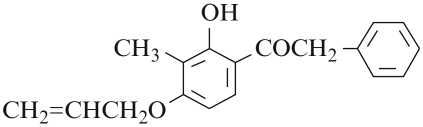
- Obtained by DDQ oxidation of 1-(3-allyl-4-hydroxy-5-methoxyphenyl)-2-phenylethanol in dioxane at r.t. for 16 h (88%) [5177].

m.p. 140–142° [5177]; ^{1}H NMR [5177].

N.B.: In the original paper—page 1600—[5177], the authors point out a registry N° [73049-14-6] for the title compound (**4e**) (benzyl 3-allyl-4-hydroxy-5-methoxyphenyl ketone) $C_{18}H_{18}O_3$. Nevertheless, the same registry number was assigned, undoubtedly by mistake, to 1-[4-hydroxy-3-methoxy-5-(2-propenyloxy)phenyl]-2-phenylethanone $C_{18}H_{18}O_4$, Chem. Abstr., Formula Index **92**, 215017w (1980). Actually, this ketone is not represented in [5177]. The assigning of this registry number for the ketone $C_{18}H_{18}O_4$ by Chem. Abstr. is definitive.

1-[2-Hydroxy-3-methyl-4-(2-propenyloxy)phenyl]-2-phenylethanone

[57097-17-3]



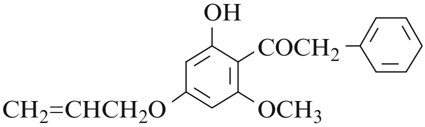
$C_{18}H_{18}O_3$ mol.wt. 282.34

Syntheses

- Refer to: [5279,5322].

1-[2-Hydroxy-6-methoxy-4-(2-propenyloxy)phenyl]-2-phenylethanone

[66541-26-2]



$C_{18}H_{18}O_4$ mol.wt. 298.34

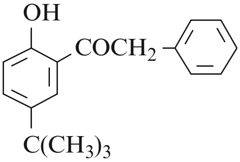
Synthesis

- Obtained by treatment of 2,4-dihydroxy-6-methoxyphenyl benzyl ketone with allyl bromide in the presence of potassium carbonate in refluxing acetone for 4 h [5335].

m.p. 81–82° [5335].

1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-2-phenylethanone

[75060-51-4]



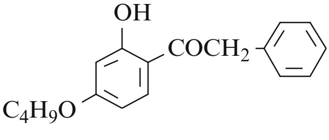
$C_{18}H_{20}O_2$ mol.wt. 268.36

Synthesis

- Obtained by demethylation of 2-phenylacetyl-4-tert-butyl-anisole with a 47% hydrobromic acid/57% hydriodic acid mixture in refluxing acetic acid (63%) [4661].

1-(4-Butoxy-2-hydroxyphenyl)-2-phenylethanone

[50775-75-2]



$C_{18}H_{20}O_3$ mol.wt. 284.36

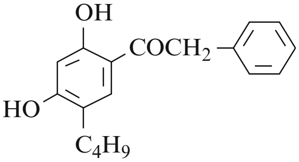
Syntheses

- Preparation by partial alkylation of 2,4-dihydroxyphenyl benzyl ketone with butyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [5329].
- Also refer to: [5365,5366,5390,5392].

m.p. 72–75° [5329].

1-(5-Butyl-2,4-dihydroxyphenyl)-2-phenylethanone

[96643-96-8]



$C_{18}H_{20}O_3$ mol.wt. 284.36

Syntheses

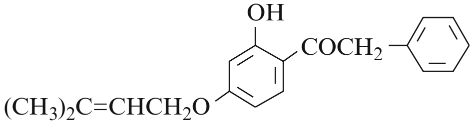
- Preparation by reaction of phenylacetonitrile with 4-butyl-resorcinol,
  - in the presence of boron trifluoride etherate (92%) [5367,5368];

- in the presence of zinc chloride (Hoesch reaction) [5323].

m.p. 91° [5323], 79–80° [5367,5368]; ^{1}H NMR [5367].

1-[2-Hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-2-phenylethanone

[35486-77-2]



$C_{19}H_{20}O_3$ mol.wt. 296.37

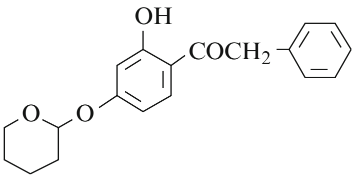
Synthesis

– Refer to: [5397].

paper chromatography [5397].

1-[2-Hydroxy-4-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]-2-phenylethanone

[130064-20-9]



$C_{19}H_{20}O_4$ mol.wt. 312.37

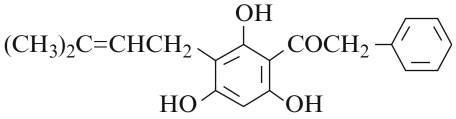
Synthesis

– Preparation by reaction of 3,4-dihydro-2*H*-pyran with 1-(2,4-dihydroxyphenyl)-2-phenylethanone in dioxane in the presence of PTSA (p-toluenesulfonic acid) at r.t. for 4 h (80%) [5280].

m.p. 89° [5280];
^{1}H NMR [5280], IR [5280], MS [5280].

2-Phenyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[85602-17-1]



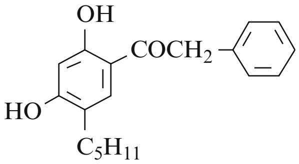
$C_{19}H_{20}O_4$ mol.wt. 312.37

Synthesis

– Refer to: [5398].

1-(2,4-Dihydroxy-5-pentylphenyl)-2-phenylethanone

[96643-97-9]



$C_{19}H_{22}O_3$ mol.wt. 298.38

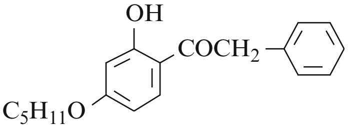
Syntheses

– Preparation by reaction of phenylacetonitrile with 4-pentyl-resorcinol,
  - in the presence of boron trifluoride etherate (81%) [5367,5371];
  - in the presence of zinc chloride (Hoesch reaction) [5323].

m.p. 94–95° [5367,5371], 89–90° [5323]; ^{1}H NMR [5367].

1-[2-Hydroxy-4-(pentyloxy)phenyl]-2-phenylethanone

[50775-76-3]



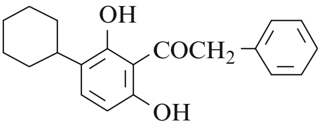
$C_{19}H_{22}O_3$ mol.wt. 298.38

Synthesis

- Preparation by partial alkylation of benzyl 2,4-di-hydroxyphenyl ketone with pentyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [5329].
- Also refer to: [5365,5366,5390,5392].

m.p. 70–73° [5329].

1-(3-Cyclohexyl-2,6-dihydroxyphenyl)-2-phenylethanone



$C_{20}H_{22}O_3$ mol.wt. 310.39

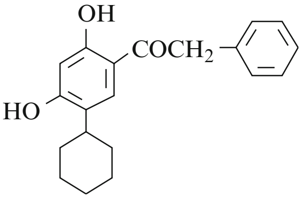
Synthesis

- Obtained by reaction of phenylacetonitrile with 4-cyclo-hexylresorcinol (Hoesch reaction) [5357].

m.p. 221° [5357].

1-(5-Cyclohexyl-2,4-dihydroxyphenyl)-2-phenylethanone

[159977-40-9]



$C_{20}H_{22}O_3$ mol.wt. 310.39

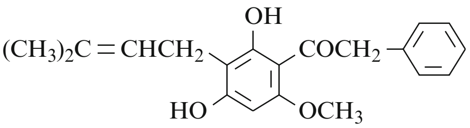
Syntheses

- Preparation by reaction of phenylacetic acid with 4-cyclohexylresorcinol in the presence of boron trifluoride etherate at 125° for 30 min, followed by hydrolysis of the complex obtained (m.p. 165–166°) with boiling dilute ethanol for 15–20 min (39%) [5256].
- Also obtained by reaction of phenylacetonitrile with 4-cyclohexylresorcinol (Hoesch reaction) [5357].

m.p. 133° [5357], 132–133° [5256]; IR [5256], UV [5256].

1-[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-phenylethanone

[55607-21-1]



$C_{20}H_{22}O_4$ mol.wt. 326.39

Syntheses

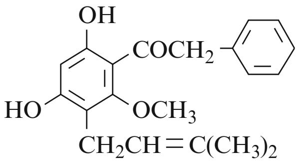
- Obtained by reaction of 2-hydroxy-2-methyl-3-butene with 2,4-dihydroxy-6-methoxyphenyl benzyl ketone in dioxane in the presence of boron trifluoride etherate for 1 h at r.t. (14%) [5352].

- Also obtained by reaction of prenyl bromide with 2,4-dihydroxy-6-methoxyphenyl benzyl ketone in methanolic potassium hydroxide for 20 h at r.t. (20%) [5352].

m.p. 133–134° [5352]; ^{1}H NMR [5352]; TLC [5352].

1-[4,6-Dihydroxy-2-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-phenylethanone

[55607-22-2] $C_{20}H_{22}O_4$ mol.wt. 326.39



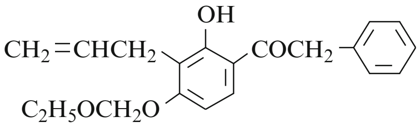
Syntheses

- Obtained by reaction of 2-hydroxy-2-methyl-3-butene with 2,4-dihydroxy-6-methoxyphenyl benzyl ketone in dioxane in the presence of boron trifluoride etherate for 1 h at r.t. (19%) [5352].
- Also obtained by reaction of prenyl bromide with 2,4-di-hydroxy-6-methoxyphenyl benzyl ketone in methanolic potassium hydroxide for 20 h at r.t. (13%) [5352].

m.p. 93–94° [5352]; ^{1}H NMR [5352]; TLC [5352].

1-[4-(Ethoxymethoxy)-2-hydroxy-3-(2-propenyl)phenyl]-2-phenylethanone

[117951-95-8] $C_{20}H_{22}O_4$ mol.wt. 326.39



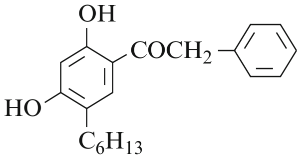
Synthesis

- Obtained (poor yield) by reaction of ethoxymethyl chloride with 3-allyl-2,4-di-hydroxydesoxybenzoin in the presence of potassium carbonate in acetone (9%) [5283].

light brown oil [5283]; ^{1}H NMR [5283], IR [5283], UV [5283].

1-(5-Hexyl-2,4-dihydroxyphenyl)-2-phenylethanone

[96643-98-0] $C_{20}H_{24}O_3$ mol.wt. 312.41



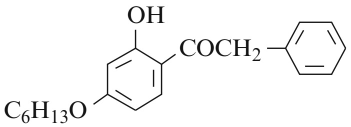
Syntheses

- Preparation by reaction of phenylacetonitrile with 4-hexyl-resorcinol,
  - in the presence of boron trifluoride etherate under hydrogen chloride atmosphere (8–10 h) and at r.t. overnight (83%) [5367,5371];
  - in the presence of zinc chloride (Hoesch reaction) [5323].
- Also obtained (poor yield) by Friedel–Crafts acylation of 4-hexylresorcinol with phenylacetyl chloride in the presence of aluminium chloride in nitrobenzene at 80° for 2 days [5399].

m.p. 90° [5399], 86–87° [5323], 83–84° [5367,5371]; ^{1}H NMR [5367].

1-[4-(Hexyloxy)-2-hydroxyphenyl]-2-phenylethanone

[50776-01-7]



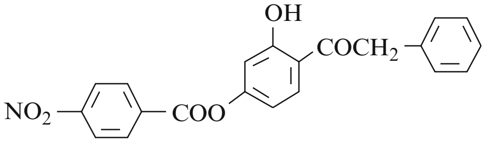
$C_{20}H_{24}O_3$ mol.wt. 312.41

Syntheses

- Preparation by partial alkylation of benzyl 2,4-di-hydroxyphenyl ketone with hexyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [5329].
- Also refer to: [5365,5366].

m.p. 60–62° [5329].

1-[2-Hydroxy-4-(4-nitrobenzoyloxy)phenyl]-2-phenylethanone



$C_{21}H_{15}NO_6$ mol.wt. 377.35

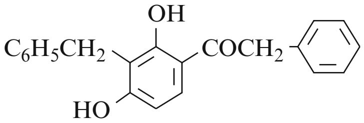
Synthesis

- Obtained by partial esterification of benzyl 2,4-dihydroxyphenyl ketone [5250] with p-nitrobenzoyl chloride in the presence of pyridine [5217].

m.p. 178–180° [5217].

1-[2,4-Dihydroxy-3-(phenylmethyl)phenyl]-2-phenylethanone

[95832-51-2]



$C_{21}H_{18}O_3$ mol.wt. 318.37

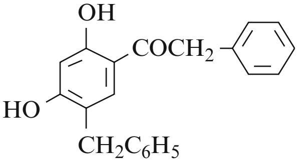
Syntheses

- Obtained by reaction of benzyl alcohol with 2,4-di-hydroxydesoxybenzoin in dioxane in the presence of boron trifluoride etherate at 60–70° for 7 h (21%) [5400].
- Also obtained by rearrangement of 2-hydroxy-4-(benzyloxy)desoxybenzoin in TFA at r.t. for 70 h (16%) [5400].
- Obtained by reaction of benzyl bromide with 2,4-dihydroxydesoxybenzoin in methanol in the presence of potassium hydroxide at r.t. for 24 h (11%) [5275].

m.p. 122–124° [5275], 121–122° [5400];
column chromatography [5400]; TLC [5400];
^{1}H NMR [5400], IR [5400], UV [5400].

1-[2,4-Dihydroxy-5-(phenylmethyl)phenyl]-2-phenylethanone

[95832-52-3] $C_{21}H_{18}O_3$ mol.wt. 318.37



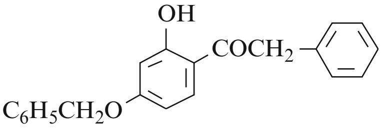
Syntheses

- Obtained by reaction of benzyl alcohol with 2,4-dihydroxy-desoxybenzoin in dioxane in the presence of boron trifluoride etherate at 60–70° for 7 h (31%) [5400].
- Also obtained by rearrangement of 2-hydroxy-4-(benzyl-oxy)desoxybenzoin in TFA at r.t. for 70 h (31%) [5400].
- Also obtained by reaction of benzyl bromide with 2,4-dihydroxydesoxybenzoin in methanol in the presence of potassium hydroxide at r.t. for 24 h (<3%) [5275].

m.p. 128–129° [5400], 126–128° [5275];
column chromatography [5400]; TLC [5400];
^{1}H NMR [5400], IR [5400], UV [5400].

1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-2-phenylethanone

[39604-80-3] $C_{21}H_{18}O_3$ mol.wt. 318.37



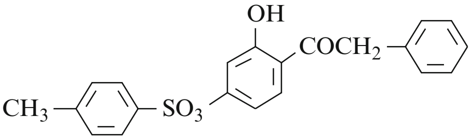
Syntheses

- Preparation by partial benzylation of 2,4-dihydroxy-phenyl benzyl ketone,
  - with benzyl chloride in the presence of potassium carbonate in refluxing acetone [5265], (72%) [5381];
  - with benzyl bromide in the presence of potassium carbonate in refluxing acetone [5329] or in the presence of potassium hydroxide at r.t. for 24 h (29%) [5275].
  - Also refer to: [5337,5404].

m.p. 105–106° [5275], 104–108° [5329], 104–105° [5381].

1-[2-Hydroxy-4-[[(4-methylphenyl)sulfonyl]oxy]phenyl]-2-phenylethanone

[102478-26-2] $C_{21}H_{18}O_5S$ mol.wt. 382.44



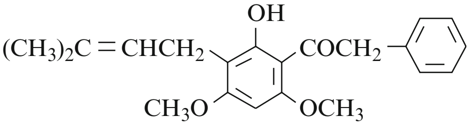
Synthesis

- Obtained by partial esterification of benzyl 2,4-dihydroxyphenyl ketone with p-toluenesulfonyl chloride in acetone in the presence of potassium carbonate [5217].

m.p. 117° [5217].

1-[2-Hydroxy-4,6-dimethoxy-3-(3-methyl-2-butenyl)phenyl]-2-phenylethanone

[55607-23-3] $C_{21}H_{24}O_4$ mol.wt. 340.42



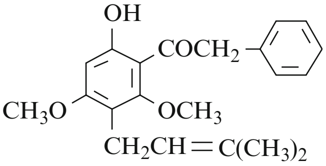
Synthesis

- Obtained by partial methylation of 2,4-di-hydroxy-6-methoxy-3-prenylphenyl benzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 3.5 h (96%) [5352].

m.p. 113–114° [5352]; ^{1}H NMR [5352]; TLC [5352].

1-[6-Hydroxy-2,4-dimethoxy-3-(3-methyl-2-butenyl)phenyl]-2-phenylethanone

[55607-25-5] $C_{21}H_{24}O_4$ mol.wt. 340.42



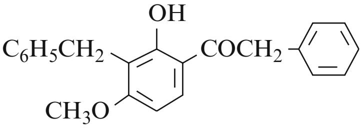
Synthesis

- Obtained by partial methylation of 2,4-dihydroxy-6-methoxy-5-prenylphenyl benzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 4 h [5352].

m.p. 80–81° [5352]; ^{1}H NMR [5352]; TLC [5352].

1-[2-Hydroxy-4-methoxy-3-(phenylmethyl)phenyl]-2-phenylethanone

[95832-54-5] $C_{22}H_{20}O_3$ mol.wt. 332.40



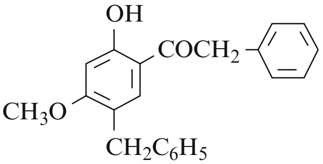
Synthesis

- Preparation by reaction of dimethyl sulfate with 3-benzyl-2,4-dihydroxydeoxybenzoin in the presence of potassium carbonate in refluxing acetone for 3 h [5400].

oil [5400]; TLC [5400]; ^{1}H NMR [5400].

1-[2-Hydroxy-4-methoxy-5-(phenylmethyl)phenyl]-2-phenylethanone

[95832-53-4] $C_{22}H_{20}O_3$ mol.wt. 332.40



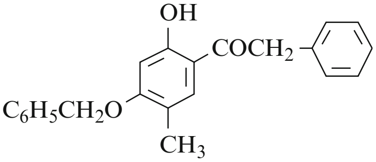
Synthesis

- Preparation by reaction of dimethyl sulfate with 5-benzyl-2,4-dihydroxydeoxybenzoin in the presence of potassium carbonate in refluxing acetone for 3 h (84%) [5400].

m.p. 80–81° [5400]; TLC [5400]; ^{1}H NMR [5400].

1-[2-Hydroxy-5-methyl-4-(phenylmethoxy)phenyl]-2-phenylethanone

[112198-28-4]



$C_{22}H_{20}O_3$ mol.wt. 332.40

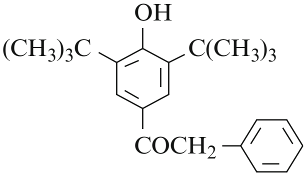
Synthesis

- Preparation by reaction of benzyl chloride with 2,4-dihydroxy-5-methylphenyl benzyl ketone in the presence of potassium carbonate in refluxing acetone for 7 h (73%) [5324].

m.p. 108° [5324].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-2-phenylethanone

[14035-39-3]



$C_{22}H_{28}O_2$ mol.wt. 324.46

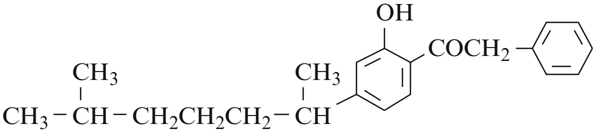
Syntheses

- Preparation by Friedel–Crafts acylation of 2,6-di-tert-butylphenol with phenylacetyl chloride in the presence of aluminium chloride for 15 min at −10° (84%) [5402], (75%) [5403].
- Preparation by oxidation of 1-(3,5-di-tert-butyl-4-hydroxyphenyl)-2-phenylethanol with DDQ in dioxane at r.t. for 16 h (90%) [5177].
- Also refer to: [5404,5405].

m.p. 129–130° [5177], 120–122° [5402,5403]; ^{1}H NMR [5177].

1-[4-(1,5-Dimethylhexyl)-2-hydroxyphenyl]-2-phenylethanone

[146935-09-3]

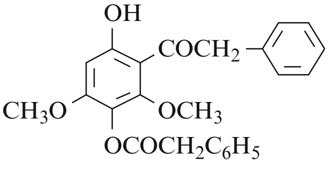


$C_{22}H_{28}O_2$ mol.wt. 324.46

Synthesis

- Refer to: [5406] (Japanese patent).

1-[6-Hydroxy-2,4-dimethoxy-3-[(phenylacetyl)oxy]phenyl]-2-phenylethanone



$C_{24}H_{22}O_6$ mol.wt. 406.44

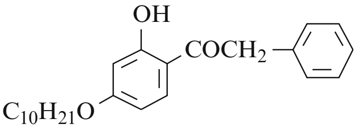
Synthesis

- Obtained (by-product) by condensation of 2,6-dimethoxy-hydroquinone with the complex phenylacetic acid and boron trifluoride [5387].

m.p. 105° [5387].

1-[4-(Decyloxy)-2-hydroxyphenyl]-2-phenylethanone

[143287-02-9] $C_{24}H_{32}O_3$ mol.wt. 368.52



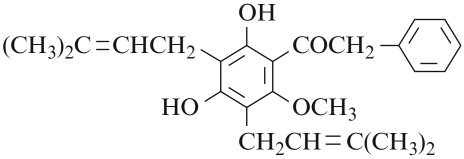
Synthesis

- Preparation by partial alkylation of benzyl 2,4-di-hydroxyphenyl ketone with decyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [5329].

m.p. 66–69° [5329].

1-[2,4-Dihydroxy-6-methoxy-3,5-bis(3-methyl-2-butenyl)phenyl]-2-phenylethanone

[55607-20-0] $C_{25}H_{30}O_4$ mol.wt. 394.51



Syntheses

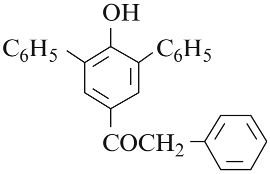
- Obtained (poor yield) by reaction of 2-hydroxy-2-methyl-3-butene with 2,4-di-hydroxy-6-methoxyphenyl benzyl ketone in dioxane in the presence of boron trifluoride etherate for 1 h at r.t. (4%) [5352].
- Also obtained (poor yield) by reaction of prenyl bromide with 2,4-dihydroxy-6-methoxyphenyl benzyl ketone in methanolic potassium hydroxide for 20 h at r.t. (5%) [5352].

oil [5352]; TLC [5352].

1-[(4-Hydroxy-3,5-diphenyl)phenyl]-2-phenylethanone

1-(2′-Hydroxy[1,1′:3′,1″-terphenyl]-5′-yl)-2-phenylethanone

[73048-87-0] $C_{26}H_{20}O_2$ mol.wt. 364.45



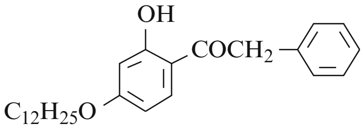
Synthesis

- Obtained by DDQ oxidation of 1-[4-hydroxy-3, 5-(di-phenyl)phenyl]-2-phenylethanol in dioxane at r.t. for 76 h (89%) [5177].

m.p. 155–156° [5177]; ^{1}H NMR [5177].

1-[4-(Dodecyloxy)-2-hydroxyphenyl]-2-phenylethanone

[143287-03-0]



$C_{26}H_{36}O_3$ mol.wt. 396.57

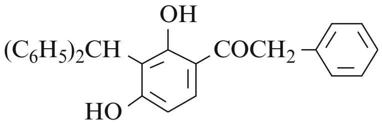
Synthesis

- Preparation by partial alkylation of 2,4-dihydroxy-phenyl benzyl ketone with dodecyl bromide in the presence of potassium carbonate in refluxing acetone for 20 h [5329].

m.p. 68–71° [5329].

1-[3-(Diphenylmethyl)-2,4-dihydroxyphenyl]-2-phenylethanone

[98497-96-2]



$C_{27}H_{22}O_3$ mol.wt. 394.47

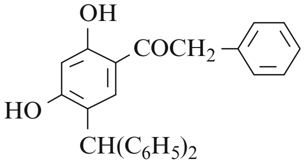
Synthesis

- Obtained by reaction of 2,4-dihydroxydesoxybenzoin with diphenylcarbinol in dioxane in the presence of boron trifluoride etherate at 60–70° for 3 h (17%) [5274].

m.p. 133–134° [5274]; column chromatography [5274]; TLC [5274];
^{1}H NMR [5274], IR [5274], UV [5274].

1-[5-(Diphenylmethyl)-2,4-dihydroxyphenyl]-2-phenylethanone

[98497-97-3]



$C_{27}H_{22}O_3$ mol.wt. 394.47

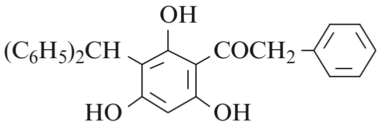
Synthesis

- Obtained by reaction of 2,4-dihydroxydesoxybenzoin with diphenylcarbinol in dioxane in the presence of boron trifluoride etherate at 60–70° for 3 h (24%) [5274].

m.p. 142–143° [5274];
column chromatography [5274]; TLC [5274];
^{1}H NMR [5274], IR [5274], UV [5274].

1-[3-(Diphenylmethyl)-2,4,6-trihydroxyphenyl]-2-phenylethanone

[104310-95-4]



$C_{27}H_{22}O_4$ mol.wt. 410.47

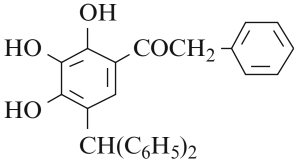
Synthesis

- Obtained by reaction of diphenylcarbinol with 2,4,6-trihydroxyphenyl benzyl ketone in dioxane in the presence of boron trifluoride etherate at 60–70° for 3.5 h (24%) [5407].

m.p. 160–162° [5407]; TLC [5407];
^{1}H NMR [5407], IR [5407], UV [5407].

1-[5-(Diphenylmethyl)-2,3,4-trihydroxyphenyl]-2-phenylethanone

[106556-47-2] $C_{27}H_{22}O_4$ mol.wt. 410.47



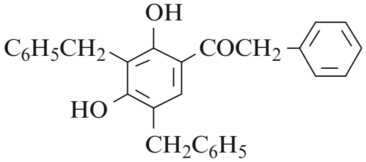
Synthesis

- Obtained by reaction of diphenylcarbinol with 2,3,4-tri-hydroxyphenyl benzyl ketone in dioxane in the presence of boron trifluoride etherate for 4 h at r.t. (39%) [5272].

m.p. 177–178° [5272]; TLC [5272];
^{1}H NMR [5272], IR [5272], UV [5272].

1-[2,4-Dihydroxy-3,5-bis(phenylmethyl)phenyl]-2-phenylethanone

[95832-50-1] $C_{28}H_{24}O_3$ mol.wt. 408.50



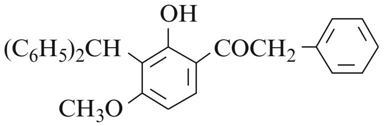
Syntheses

- Obtained by reaction of benzyl alcohol with 2,4-di-hydroxydesoxybenzoin in dioxane in the presence of boron trifluoride etherate at 60–70° for 7 h (10%) [5400].
- Also obtained (trace) by reaction of benzyl bromide with 2,4-dihydroxydesoxybenzoin in methanol in the presence of potassium hydroxide at r.t. for 24 h (<2%) [5275].

m.p. 111–112° [5400], 110–112° [5275]; column chromatography [5400];
^{1}H NMR [5400], ^{13}C NMR [5275], IR [5400], UV [5400].

1-[3-(Diphenylmethyl)-2-hydroxy-4-methoxyphenyl]-2-phenylethanone

[98498-01-2] $C_{28}H_{24}O_3$ mol.wt. 408.50



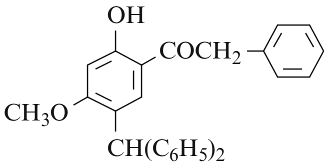
Synthesis

- Preparation by reaction of dimethyl sulfate with 2,4-dihydroxy-3-(diphenylmethyl)desoxybenzoin in the presence of potassium carbonate in refluxing acetone for 3 h (93%) [5274].

m.p. 126–127° [5274]; TLC [5274]; ^{1}H NMR [5274].

1-[5-(Diphenylmethyl)-2-hydroxy-4-methoxyphenyl]-2-phenylethanone

[98498-02-3] $C_{28}H_{24}O_3$ mol.wt. 408.50



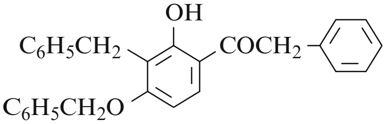
Synthesis

- Preparation by reaction of dimethyl sulfate with 2,4-di-hydroxy-5-(diphenyl-methyl)desoxybenzoin in the presence of potassium carbonate in refluxing acetone for 3 h (93%) [5274].

m.p. 110–111° [5274]; TLC [5274]; ^{1}H NMR [5274].

1-[2-Hydroxy-4-(phenylmethoxy)-3-(phenylmethyl)phenyl]-2-phenylethanone

[107044-42-8] $C_{28}H_{24}O_3$ mol.wt. 408.50



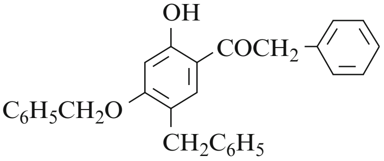
Syntheses

- Obtained by reaction of benzyl chloride (1 mol) with 3-benzyl-2,4-dihydroxydesoxybenzoin in the presence of potassium carbonate (4 mol) in boiling acetone for 1.5 h [5275].
- Also obtained (trace) by reaction of benzyl bromide with 2,4-dihydroxydesoxybenzoin in methanol in the presence of potassium hydroxide at r.t. for 24 h (<1%) [5275].

m.p. 97–98° [5275]; column chromatography [5275];
^{1}H NMR [5275], IR [5275], UV [5275].

1-[2-Hydroxy-4-(phenylmethoxy)-5-(phenylmethyl)phenyl]-2-phenylethanone

[107044-44-0] $C_{28}H_{24}O_3$ mol.wt. 408.50



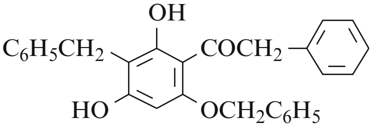
Synthesis

- Obtained by reaction of benzyl bromide (1 mol) with 5-benzyl-2,4-dihydroxydesoxybenzoin in the presence of potassium carbonate (4 mol) in boiling acetone for 1.5 h (19%) [5275].

m.p. 90–92° [5275]; column chromatography [5275];
^{1}H NMR [5275], IR [5275].

1-[2,4-Dihydroxy-6-(phenylmethoxy)-3-(phenylmethyl)phenyl]-2-phenylethanone

[39548-97-5] $C_{28}H_{24}O_4$ mol.wt. 424.50



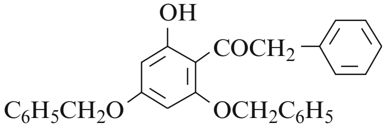
Synthesis

- Obtained (by-product) by benzylation of 2,4,6-tri-hydroxyphenyl benzyl ketone with benzyl chloride in the presence of potassium carbonate in refluxing acetone for 7 h (9%) [5408].

m.p. 170–171° [5408]; ^{1}H NMR [5408], UV [5408].

1-[2-Hydroxy-4,6-bis(phenylmethoxy)phenyl]-2-phenylethanone

[39548-96-4] $C_{28}H_{24}O_4$ mol.wt. 424.50



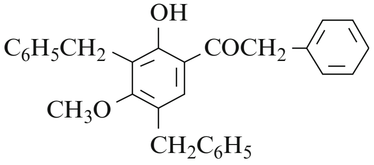
Syntheses

- Obtained by reaction of benzyl chloride with 2,4,6-trihydroxyphenyl benzyl ketone in the presence of potassium carbonate in refluxing acetone for 5 h (34%) [5381] or for 7 h (21%) [5408].
- Also refer to: [5401].

m.p. 99–100° [5381], 95–96° [5408]; ^{1}H NMR [5408], UV [5408].

1-[2-Hydroxy-4-methoxy-3,5-bis(phenylmethyl)phenyl]-2-phenylethanone

[95832-55-6] $C_{29}H_{26}O_3$ mol.wt. 422.52



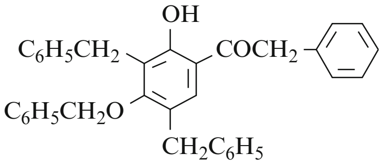
Synthesis

- Obtained by reaction of dimethyl sulfate with 3,5-di-benzyl-2,4-dihydroxyphenyl benzyl ketone in the presence of potassium carbonate in refluxing acetone for 3 h (23%) [5400].

oil [5400]; TLC [5400]; ^{1}H NMR [5400].

1-[2-Hydroxy-4-(phenylmethoxy)-3,5-bis(phenylmethyl)phenyl]-2-phenylethanone

[107044-43-9] $C_{35}H_{30}O_3$ mol.wt. 498.62



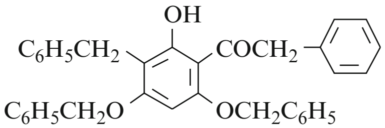
Synthesis

- Obtained by reaction of benzyl chloride (1 mol) with 3,5-dibenzyl-2, 4-dihydroxydesoxybenzoin in the presence of potassium carbonate (4 mol) in boiling acetone for 1.5 h (33%) [5275].

m.p. 51–52° [5275]; TLC [5275];
^{1}H NMR [5275], IR [5275], UV [5275].

1-[2-Hydroxy-4,6-bis(phenylmethoxy)-3-(phenylmethyl)phenyl]-2-phenylethanone

[39548-95-3] $C_{35}H_{30}O_4$ mol.wt. 514.62



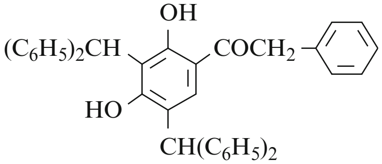
Synthesis

- Obtained (by-product) by benzylation of 2,4,6-tri-hydroxyphenyl benzyl ketone with benzyl chloride in the presence of potassium carbonate in refluxing acetone for 7 h (6%) [5408].

m.p. 131–132° [5408]; ^{1}H NMR [5408], UV [5408].

1-[3,5-Bis(diphenylmethyl)-2,4-dihydroxyphenyl]-2-phenylethanone

[98497-95-1]



$C_{40}H_{32}O_3$ mol.wt. 560.69

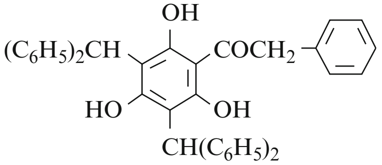
Synthesis

- Obtained by reaction of 2,4-dihydroxydesoxy-benzoin with diphenylcarbinol in dioxane in the presence of boron trifluoride etherate at 60–70° for 3 h (15%) [5274].

m.p. 136–137° [5274]; column chromatography [5274]; TLC [5274]; ^{1}H NMR [5274], IR [5274], UV [5274].

1-[3,5-Bis(diphenylmethyl)-2,4,6-trihydroxyphenyl]-2-phenylethanone

[104310-93-2]



$C_{40}H_{32}O_4$ mol.wt. 576.69

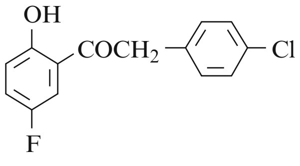
Synthesis

- Obtained by reaction of diphenylcarbinol with 2,4,6-trihydroxyphenyl benzyl ketone in dioxane in the presence of boron trifluoride etherate at 60–70° for 3.5 h (21%) [5407].

m.p. 122–124° [5407]; TLC [5407]; ^{1}H NMR [5407], IR [5407], UV [5407].

18.2 Compounds Derived from Substituted Phenylacetic Acids

2-(4-Chlorophenyl)-1-(5-fluoro-2-hydroxyphenyl)ethanone



$C_{14}H_{10}ClFO_2$ mol.wt. 264.68

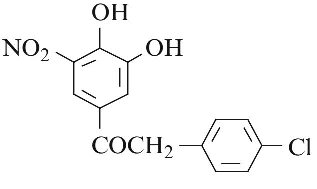
Synthesis

- Preparation by Fries rearrangement of p-fluorophenyl p-chlorophenylacetate with aluminium chloride at 150–180° for 20 min (32%) [5189].

m.p. 124–126° [5189]; ^{1}H NMR [5189], MS [5189].

2-(4-Chlorophenyl)-1-(3,4-dihydroxy-5-nitrophenyl)ethanone

[274925-89-2] $C_{14}H_{10}ClNO_5$ mol.wt. 307.69



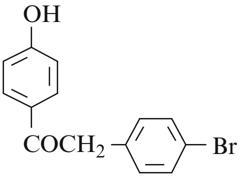
Synthesis

- Preparation by treatment of 2-(4-chlorophenyl)-1-(4-hydroxy-3-methoxy-5-nitrophenyl)ethanone with aluminium chloride in refluxing ethyl acetate/pyridine mixture for 2 h (94%) [5192], (90–96%) [5193].

m.p. 162–164° [5192,5193]; HPLC [5192];
^{1}H NMR [5192,5193], ^{13}C NMR [5192,5193], IR [5192,5193].

2-(4-Bromophenyl)-1-(4-hydroxyphenyl)ethanone

[63186-92-5] $C_{14}H_{11}BrO_2$ mol.wt. 291.14



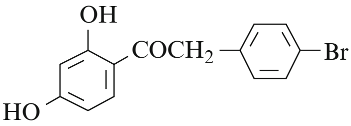
Synthesis

- Preparation by demethylation of 2-(4-bromophenyl)-1-(4-methoxyphenyl)ethanone with 48% hydrobromic acid in refluxing acetic acid for 7 h (89%) [5409], (82%) [5410].

m.p. 186° [5409,5410];
^{1}H NMR [5409], IR [5409].

2-(4-Bromophenyl)-1-(2,4-dihydroxyphenyl)ethanone

[92152-60-8] $C_{14}H_{11}BrO_3$ mol.wt. 307.14



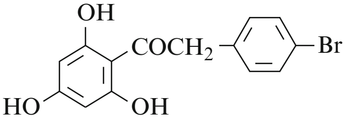
Syntheses

- Preparation by reaction of p-bromophenylacetonitrile with resorcinol,
  - in the presence of boron trifluoride etherate under hydrogen chloride atmosphere (8–10 h) and at r.t. overnight (90%) [5367,5368];
  - in the presence of zinc chloride and hydrogen chloride (Hoesch reaction) [5181].
- Preparation by Friedel–Crafts acylation of resorcinol with p-bromophenylacetyl chloride in nitrobenzene in the presence of aluminium chloride at 50° (54%) [5357].
- Also refer to: [4685,5411].

m.p. 176–177° [5367,5368], 176° [5357], 100–101° [5181]. One of the reported melting points is obviously wrong. ^{1}H NMR [5367].

2-(4-Bromophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[147220-80-2] $C_{14}H_{11}BrO_4$ mol.wt. 323.15



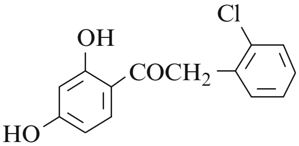
Synthesis

- Refer to: [4685] (compound **IIg**), preparation according to reported procedures [5412,5413].

m.p. 231° [4685]; ^{1}H NMR [4685], IR [4685].

2-(2-Chlorophenyl)-1-(2,4-dihydroxyphenyl)ethanone

$C_{14}H_{11}ClO_3$ mol.wt. 262.69



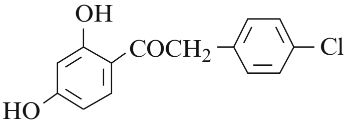
Synthesis

- Obtained by reaction of o-chlorophenylacetonitrile with resorcinol (Hoesch reaction) (20%) [5414].

m.p. 142° [5414].

2-(4-Chlorophenyl)-1-(2,4-dihydroxyphenyl)ethanone

[15485-64-0] $C_{14}H_{11}ClO_3$ mol.wt. 262.69



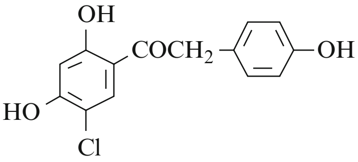
Syntheses

- Preparation by Friedel–Crafts acylation of resorcinol with p-chlorophenylacetyl chloride in nitrobenzene in the presence of aluminium chloride for some hours at 40–50° (64%) [5357].
- Obtained by reaction of p-chlorophenylacetonitrile with resorcinol (Hoesch reaction) [5262,5264].
- Also refer to: [4685,5271,5411,5415].

m.p. 159°5–160° [5264], 156° [5357], 153–154° [5262]; UV [5264].

1-(5-Chloro-2,4-dihydroxyphenyl)-2-(4-hydroxyphenyl)ethanone

[139256-02-3] $C_{14}H_{11}ClO_4$ mol.wt. 278.69



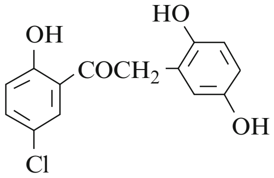
Synthesis

- Obtained by reaction of p-hydroxyphenylacetic acid with 4-chlororesorcinol in the presence of boron trifluoride etherate under argon, on a water bath for 1 h (67%) [5208].

m.p. 196–197° [5208]; ^{1}H NMR [5208], ^{13}C NMR [5208], MS [5208].

1-(5-Chloro-2-hydroxyphenyl)-2-(2,5-dihydroxyphenyl)ethanone

[115781-55-0] $C_{14}H_{11}ClO_4$ mol.wt. 278.69



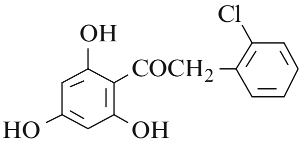
Synthesis

- Obtained by alkali cleavage of 6-chloro-3-(2′,5′-dihydroxy-phenyl)-4-hydroxycoumarin with refluxing 2% methanolic potassium hydroxide for 4 h (73%) [5416].

m.p. 219° [5416]; IR [5416], UV [5416].

2-(2-Chlorophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

$C_{14}H_{11}ClO_4$ mol.wt. 278.69



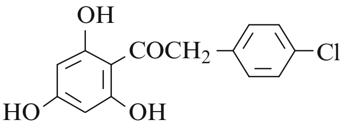
Synthesis

- Obtained by reaction of o-chlorophenylacetonitrile with phloroglucinol (20%) (Hoesch reaction) [5414].

m.p. 172–172°5 [5414].

2-(4-Chlorophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[15485-68-4] $C_{14}H_{11}ClO_4$ mol.wt. 278.69



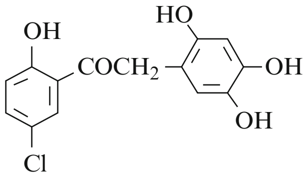
Syntheses

- Obtained by reaction of p-chloropheny-lacetonitrile with phloroglucinol (Hoesch reaction) [5262,5264].
- Also refer to: [4685,5415,5417].

m.p. 224–225° [5264], 221–222° [5262]; UV [5264].

1-(5-Chloro-2-hydroxyphenyl)-2-(2,4,5-trihydroxyphenyl)ethanone

[115781-51-6] $C_{14}H_{11}ClO_5$ mol.wt. 294.69



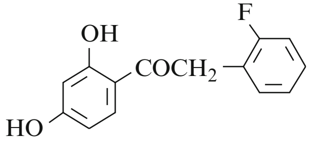
Synthesis

- Obtained from 6-chloro-4-hydroxy-3-(2′,4′,5′-trihydroxy-phenyl)coumarin with refluxing 2% methanolic potassium hydroxide for 4 h (72%) [5416].

m.p. 189° [5416]; IR [5416], UV [5416].

1-(2,4-Dihydroxyphenyl)-2-(2-fluorophenyl)ethanone

[121060-02-4]



$C_{14}H_{11}FO_3$ mol.wt. 246.24

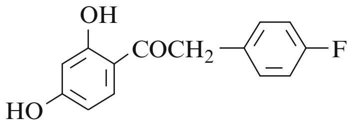
Syntheses

- Refer to: [4685] (compound **Id**), preparation according to reported procedures [5412,5413].
- Also refer to: [5026,5418,5419].

m.p. 138° [4685]; ^{1}H NMR [4685], IR [4685].

1-(2,4-Dihydroxyphenyl)-2-(4-fluorophenyl)ethanone

[15485-70-8]



$C_{14}H_{11}FwO_3$ mol.wt. 246.24

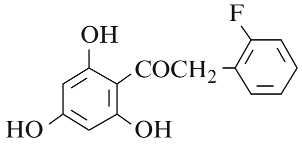
Syntheses

- Preparation by reaction of p-fluorophenylacetonitrile with resorcinol (Hoesch reaction) [5264], (70%) [5420], (45%) [5421].
- Also refer to: [4685,5026,5415,5418,5419].

m.p. 149–150° [5264], 144–145° [5421], 143–144° [5420]; ^{1}H NMR [5420,5421], UV [5264], MS [5421].

2-(2-Fluorophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[101068-28-4]



$C_{14}H_{11}FO_4$ mol.wt. 262.24

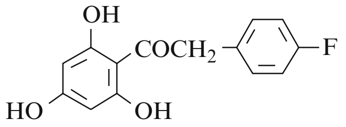
Synthesis

- Refer to: [4685] (compound **IId**), preparation according to reported procedures [5412,5413].

m.p. 182° [4685]; ^{1}H NMR [4685], IR [4685].

2-(4-Fluorophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[15485-69-5]



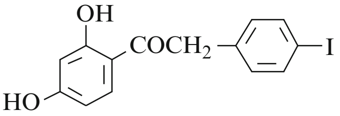
$C_{14}H_{11}FO_4$ mol.wt. 262.24

Syntheses

- Obtained by reaction of p-fluorophenylacetonitrile with phloroglucinol (Hoesch reaction) [4852,5264].
- Also refer to: [4685,5415].

m.p. 199–200° [5264]; UV [5264].

1-(2,4-Dihydroxyphenyl)-2-(4-iodophenyl)ethanone



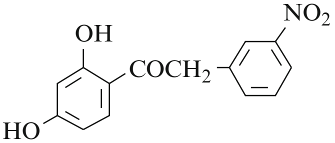
$C_{14}H_{11}IO_3$ mol.wt. 354.14

Syntheses

- Obtained by reaction of resorcinol with p-iodophenyl-acetyl chloride in nitrobenzene in the presence of aluminium chloride at 50–60° (27%) [5357].
- Also refer to: [5422].

m.p. 186° [5357,5422].

1-(2,4-Dihydroxyphenyl)-2-(3-nitrophenyl)ethanone



$C_{14}H_{11}NO_5$ mol.wt. 273.25

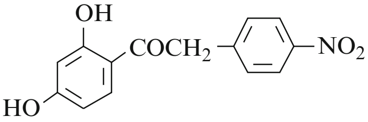
Synthesis

- Preparation by reaction of m-nitrophenylacetonitrile with resorcinol (Hoesch reaction) (46%) [5423].

m.p. 156°5 [5423].

1-(2,4-Dihydroxyphenyl)-2-(4-nitrophenyl)ethanone

[15485-63-9]



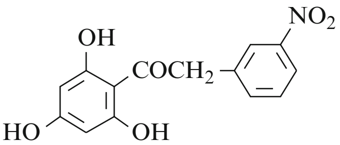
$C_{14}H_{11}NO_5$ mol.wt. 273.25

Syntheses

- Obtained by reaction of p-nitrophenylacetonitrile with resorcinol (Hoesch reaction) [5264], (65%) [5399], (60%) [5268], (40%) [5424], (35%) [5425], (27%) [5423].
- Also obtained by reaction of p-nitrophenylacetic acid with resorcinol in the presence of boron trifluoride in chloroform, first cooling in ice, then at r.t. overnight (12%) [5258].
- Also refer to: [4685,5217,5271].

m.p. 295–297° [5264], 210° [5258,5425], 205° [5399], 204° [5424], 202° [5423]. One of the reported melting points is obviously wrong. ^{1}H NMR [5268], UV [5264], MS [5268].

2-(3-Nitrophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone



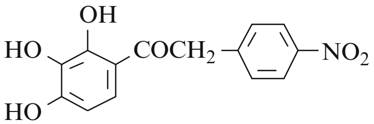
$C_{14}H_{11}NO_6$ mol.wt. 289.25

Synthesis

- Preparation by reaction of m-nitrophenylacetonitrile with phloroglucinol (Hoesch reaction) (63%) [5423].

m.p. 211–212° [5423].

2-(4-Nitrophenyl)-1-(2,3,4-trihydroxyphenyl)ethanone



$C_{14}H_{11}NO_6$ mol.wt. 289.25

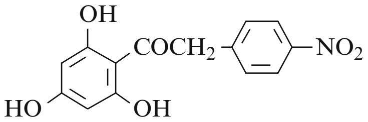
Synthesis

- Obtained by reaction of p-nitrophenylacetic acid with pyrogallol in the presence of boron trifluoride in chloroform, first cooling in ice, then at r.t. overnight (93%) [5258].

m.p. 227–228° [5258].

2-(4-Nitrophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[15485-67-3]



$C_{14}H_{11}NO_6$ mol.wt. 289.25

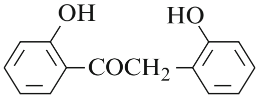
Syntheses

- Obtained by reaction of p-nitrophenylacetonitrile with phloroglucinol (Hoesch reaction) [5264], (quantitative yield) [5424], (56%) [5423].
- Also refer to: [5217,5246,5258].

m.p. 249–250° [5264], 247° [5423], 245° [5424]; UV [5264].

1,2-Bis(2-hydroxyphenyl)ethanone

[7622-42-6]



$C_{14}H_{12}O_3$ mol.wt. 228.25

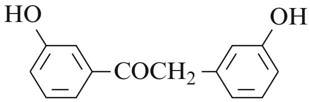
Syntheses

- Preparation by reduction of 2,2′-dihydroxybenzoin with zinc dust and 15% potassium hydroxide in boiling ethanol for 8 h [5426], (70–75%) [5427].
- Also refer to: [5428].

m.p. 104° [5427].

1,2-Bis(3-hydroxyphenyl)ethanone

[63192-59-6]



$C_{14}H_{12}O_3$ mol.wt. 228.25

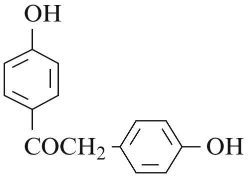
Syntheses

- Obtained by reductive coupling of methyl m-hydroxy-benzoate using $TiCl_3/LiAlH_4$ in refluxing tetrahydrofuran for 3 h under nitrogen (20%) [5429].
- Also refer to: [5430].

GC [5429]; GC-MS [5429];
^{1}H NMR [5429], ^{13}C NMR [5429], IR [5429], MS [5429].

1,2-Bis(4-hydroxyphenyl)ethanone

[3669-47-4] $C_{14}H_{12}O_3$ mol.wt. 228.25



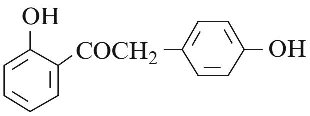
Syntheses

- Preparation by total demethylation of 4-methoxyphenyl 4-methoxybenzyl ketone (4,4′-dimethoxydeoxybenzoin),
  - with refluxing pyridinium chloride (4 equiv) for 1 h (80–85%) [5431];
  - with boiling a mixture of 50% aqueous hydriodic acid and phenol for 30 min (94%) [5432];
  - with hydriodic acid (d = 1.7) in acetic acid at 135–140° for 10 min (quantitative yield) [5432];
  - with aluminium chloride in refluxing benzene for 1.5 h (25%) [5433].
- Also obtained by diazotization of 4,4′-diaminodeoxybenzoin, followed by hydrolysis of the diazonium salt formed [5434].
- Also obtained (by-product) by Fries rearrangement of phenyl p-methoxyphenylacetate with aluminium chloride for 1.5 h at 145° (31%) [5280].
- Also refer to: [5435].

m.p. 217° [5432], 215–219° [5433], 215° [5431], 214–215° [5434], 212° [5280].

1-(2-Hydroxyphenyl)-2-(4-hydroxyphenyl)ethanone

[109561-92-4] $C_{14}H_{12}O_3$ mol.wt. 228.25



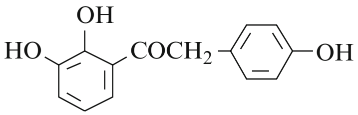
Syntheses

- Obtained by treatment of ethyl [(2-methoxybenzoyl)-(4-methoxyphenyl)]acetate with boiling pyridinium chloride for 20 min (ca. 220°) (48%) [5212].
- Also obtained (by-product) by Fries rearrangement of phenyl p-methoxyphenylacetate with aluminium chloride for 1.5 h at 145° (12%) [5280].
- Also obtained by demethylation of 2-hydroxyphenyl 4-methoxybenzyl ketone with pyridinium chloride at 220° for 1 h (79%) [5280].
- Also refer to: [5436,5437].

m.p. 140° [5280], 106–107° [5212]. One of the reported melting points is obviously wrong.

1-(2,3-Dihydroxyphenyl)-2-(4-hydroxyphenyl)ethanone

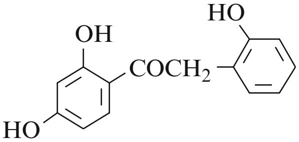
[139256-01-2] $C_{14}H_{12}O_4$ mol.wt. 244.25



Synthesis

- Refer to: [5208].

1-(2,4-Dihydroxyphenyl)-2-(2-hydroxyphenyl)ethanone



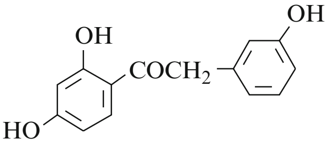
$C_{14}H_{12}O_4$ mol.wt. 244.25

Synthesis not yet described

N.B.: This ketone (XIII) cannot be prepared by condensation of o-hydroxyphenylacetonitrile with resorcinol (Hoesch reaction) [5438].

1-(2,4-Dihydroxyphenyl)-2-(3-hydroxyphenyl)ethanone

[89019-84-1]



$C_{14}H_{12}O_4$ mol.wt. 244.25

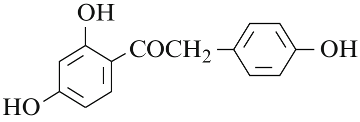
Syntheses

- Preparation by reaction of m-hydroxyphenylacetic acid with resorcinol in the presence of boron trifluoride etherate under argon on a water bath for 1 h (93%) [5208].
- Also obtained by demethylation of 1-(2,4-dihydroxy-phenyl)-2-(3-methoxyphenyl)ethanone with concentrated hydrobromic acid in refluxing acetic acid for 4 h under an argon atmosphere (89%) [5439].

m.p. 214–216° [5439]; ^{1}H NMR [5439], IR [5439], UV [5439], MS [5439].

1-(2,4-Dihydroxyphenyl)-2-(4-hydroxyphenyl)ethanone

[17720-60-4]



$C_{14}H_{12}O_4$ mol.wt. 244.25

Syntheses

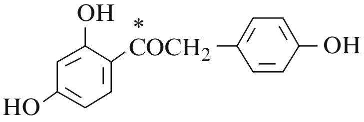
- Preparation by reaction of p-hydroxyphenylacetic acid with resorcinol [5440] in the presence of boron trifluoride etherate under argon on a water bath for 1 h (98%) [5208], at 100° for 1 h (70%) [5441] or for 15 min (40%) [5268].
- Preparation by demethylation of 2,4-dihydroxy-4′-methoxydesoxybenzoin with pyridinium bromide, kept at the melting stage for 1 min (quantitative yield) [5442].
- Preparation by catalytic hydrogenation of 2,4-dihydroxy-4′-(benzyloxy)desoxybenzoin [5443].
- Also obtained by treatment of ethyl 2,4-dimethoxybenzoyl-4-methoxyphenylacetate with boiling pyridinium chloride for 20 min (ca. 220°) (49%) [5212].
- Also obtained by alkaline degradation of *daidzein* (m.p. 315–320°) (7,4′-dihydroxyisoflavone) with refluxing 30% potassium hydroxide for 5 min (99%) [5444].

- Also obtained by reaction of p-hydroxyphenylacetonitrile with resorcinol (Hoesch reaction) (26%) [5444].
- Also refer to: [4685,5216,5270,5280,5292,5436,5445,5446,5447,5448,5449].

m.p. 192° [5443,5444], 190–191° [5442], 183–184° [5212];
^{1}H NMR [5268], ^{13}C NMR [5293,5450], MS [5268].

1-(2,4-Dihydroxyphenyl)-2-(4-hydroxyphenyl)ethanone-*1*-^{13}*C*

[215653-80-8] $C_{14}H_{12}O_4$ mol.wt. 245.25

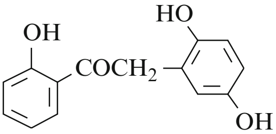


Synthesis

- Preparation by reaction of 4-benzyloxyphenyl-acetonitrile [1-^{13}C] with resorcinol (Hoesch reaction) (96%) [5451].

2-(2,5-Dihydroxyphenyl)-1-(2-hydroxyphenyl)ethanone

[115781-54-9] $C_{14}H_{12}O_4$ mol.wt. 244.25



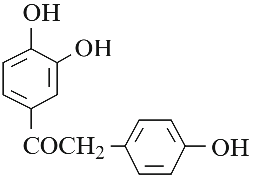
Synthesis

- Obtained by alkali cleavage of 3-(2′,5′-dihydroxyphenyl)-4-hydroxycoumarin with refluxing 2% methanolic potassium hydroxide for 4 h (62%) [5416].

m.p. 203° [5416]; IR [5416], UV [5416].

1-(3,4-Dihydroxyphenyl)-2-(4-hydroxyphenyl)ethanone

[150295-88-8] $C_{14}H_{12}O_4$ mol.wt. 244.25



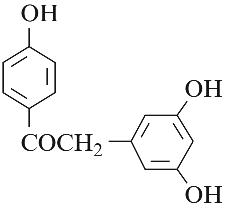
Synthesis

- Obtained by reaction of p-hydroxyphenylacetic acid with pyrocatechol in the presence of boron trifluoride etherate under argon on a water bath for 2 h (78%) [5208].

m.p. 211° [5208];
^{1}H NMR [5208], ^{13}C NMR [5208], MS [5208].

2-(3,5-Dihydroxyphenyl)-1-(4-hydroxyphenyl)ethanone

[402490-73-7] $C_{14}H_{12}O_4$ mol.wt. 244.25

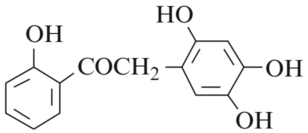


Synthesis

- Obtained by treatment of fluororesveratrol blocked by three MOM groups using trifluoroacetic acid in methylene chloride at r.t. [5452].
MS [5452].

1-(2-Hydroxyphenyl)-2-(2,4,5-trihydroxyphenyl)ethanone

[115781-50-5]



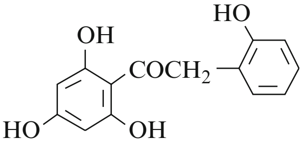
$C_{14}H_{12}O_5$ mol.wt. 260.25

Synthesis

- Obtained from 4-hydroxy-3-(2′,4′,5′-trihydroxyphenyl)-coumarin with refluxing 2% methanolic potassium hydroxide for 4 h (75%) [5416].

m.p. 185° [5416]; IR [5416], UV [5416].

2-(2-Hydroxyphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone



$C_{14}H_{12}O_5$ mol.wt. 260.25

Syntheses

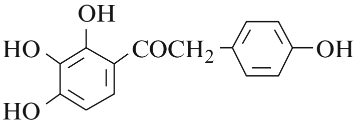
- Obtained by alkaline degradation of *isogenistein* (5,7,2′-trihydroxyisoflvone) (SM) (m.p. 302°) [5453] with potassium hydroxide [5453,5454,5455,5456]. SM was obtained by hydrolysis of *isogenistin*, its glycoside, (m.p. 265°) [5455], isolated from soya bean [5453,5454,5455].
- Also obtained by partial demethylation of 2-hydroxy-4,6-dimethoxyphenyl 2-methoxybenzyl ketone (m.p. 116–118°) with aluminium chloride in refluxing benzene for 2 h (35%) [5456].
- Also refer to: [5457].

m.p. 217–220° [5456], 182–183° [5453].

One of the reported melting points is obviously wrong.

2-(4-Hydroxyphenyl)-1-(2,3,4-trihydroxyphenyl)ethanone

[77316-95-1]



$C_{14}H_{12}O_5$ mol.wt. 260.25

Syntheses

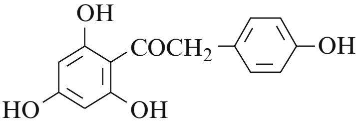
- Obtained by reaction of p-hydroxyphenylacetic acid with pyrogallol in the presence of boron trifluoride etherate under argon on a water bath for 1 h (92%) [5208] or at 100° for 15 min (40%) [5268].
- Also refer to: [5458] (Chinese paper).

m.p. 208–209° [5208];

^{1}H NMR [5208,5268], ^{13}C NMR [5208], MS [5208,5268].

2-(4-Hydroxyphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[15485-65-1] $C_{14}H_{12}O_5$ mol.wt. 260.25



Syntheses

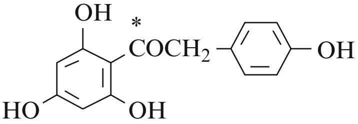
- Preparation by reaction of p-hydroxyphenyl-acetonitrile with phloroglucinol (Hoesch reaction) (58%) [5459].
- Also obtained by alkaline degradation of *genistein* (5,7,4′-trihydroxyisoflavone) (m.p. 296–298°) with refluxing 5% potassium hydroxide for 30 min [5444].
- Also obtained by reaction of p-hydroxyphenylacetic with phloroglucinol in the presence of boron trifluoride etherate under argon for 5 h at 0° (83%) [5208].
- Also refer to: [5277,5295,5460,5461,5462].

monohydrate [5459];

m.p. 259° (d) [5459], 253–257° [5444]; ^{13}C NMR [5293].

2-(4-Hydroxyphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone-*1-^{13}C*

[262591-28-6] $C_{14}H_{12}O_5$ mol.wt. 261.25

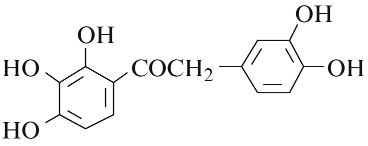


Synthesis

- Preparation by reaction of 4-benzyloxyphenylacetonitrile [1-13C] with phloroglucinol (Hoesch reaction) (75%) [5451].

2-(3,4-Dihydroxyphenyl)-1-(2,3,4-trihydroxyphenyl)ethanone

[57165-58-9] $C_{14}H_{12}O_6$ mol.wt. 276.25



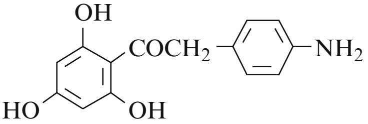
Synthesis

- Preparation in two steps: First, reaction of 3,4-di-methoxyphenylacetyl chloride with 1,2,3-trimethoxy-benzene in the presence of aluminium chloride at 30–40° for 16 h. Then, the formed 2,3,4,3′,4′-penta-methoxydeoxybenzoin was demethylated by heating at reflux with pyridinium chloride [5463].

m.p. 155–156° [5463].

2-(4-Aminophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[64225-20-3] $C_{14}H_{13}NO_4$ mol.wt. 259.26



Syntheses

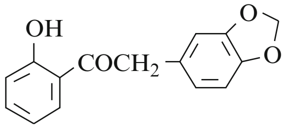
- Preparation by reaction of p-acetamidophenyl-acetonitrile with phloroglucinol in ethyl ether in the presence of zinc chloride under hydrogen chloride atmosphere for 4 h. Then, hydrolysis of the obtained ketimine hydrochloride in boiling water for 2 h (Hoesch reaction) [5382].

- Preparation by hydrogenation of 2,4,6-trihydroxyphenyl 4-nitrobenzyl ketone in ethanol in the presence of Raney nickel as catalyst with hydrogen at 40 lb pressure for 6 h (67%) [5382].
- Also refer to: [5464].

m.p. 240–242° [5464], 197° [5382]. One of the reported melting points is obviously wrong. IR [5464].

2-(1,3-Benzodioxol-5-yl)-1-(2-hydroxyphenyl)ethanone

[142751-44-8] $C_{15}H_{12}O_4$ mol.wt. 256.26



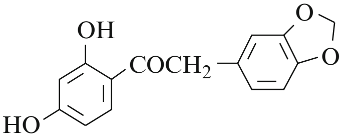
Synthesis

- Preparation by stirring a mixture of S-[4-(1,3-benzodioxol-5-ylacetyl)-3-hydroxyphenyl] dimethylcarbamothioate [142751-43-7], Raney nickel and ethanol at r.t. for 1 h (71%) [5205].

m.p. 62–63° [5205]; ^{1}H NMR [5205], MS [5205].

2-(1,3-Benzodioxol-5-yl)-1-(2,4-dihydroxyphenyl)ethanone
(Pseudo-baptigenetin)

[5653-25-8] $C_{15}H_{12}O_5$ mol.wt. 272.26



Syntheses

- Preparation by reaction of (3,4-methylenedioxy)-phenylacetyl chloride with resorcinol in nitromethane in the presence of aluminium chloride, under nitrogen, first at 0° for 3 h and at r.t. for 20 h (59%) [5465].
- Also obtained by reaction of (3,4-methylenedioxy)phenylacetonitrile with resorcinol (Hoesch reaction) [5466].
- Also obtained by alkaline degradation of *pseudo-baptigenin* (7-hydroxy-3′,4′-methylenedioxy-isoflavone) (SM) (m.p. 298–299° [5466], 298° [5467], 296–298° [5468], 293–295° [5469]) with 12% sodium hydroxide in refluxing dilute ethanol for 15 min [5469] or with refluxing 5% potassium hydroxide for 2 h (78%) [5468]. SM was obtained from *pseudo-baptisin* (isolated from *Baptisia tinctoria* RBr) whether by heating at 280° or by hydrolysis with various acids or emulsin [5467].
- Also refer to: [5205,5271,5277,5380,5470].

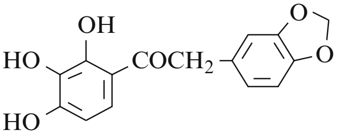
m.p. 151° [5468], 148–149° [5283], 146–148° [5469], 87–89° [5465]. One of the reported melting points is obviously wrong.

b.p.$_{0.03}$ 210–220° [5468]; TLC [5465];
^{1}H NMR [5465], IR [5465].

2-(1,3-Benzodioxol-5-yl)-1-(2,3,4-trihydroxyphenyl)ethanone

[84018-72-4]

$C_{15}H_{12}O_6$ mol.wt. 288.26



Synthesis

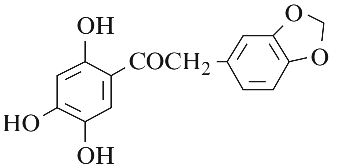
– Obtained by reaction of 3,4-(methylenedioxy)phenyl-acetonitrile with pyrogallol (Hoesch reaction) (21%) [5470].

m.p. 185° [5470].

2-(1,3-Benzodioxol-5-yl)-1-(2,4,5-trihydroxyphenyl)ethanone

[2828-14-0]

$C_{15}H_{12}O_6$ mol.wt. 288.26



Synthesis

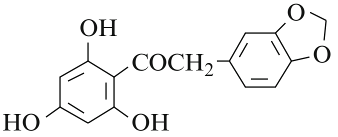
– Obtained by reaction of 3,4-(methylenedioxy)phenyl-acetonitrile with hydroxyhydroquinone (Hoesch reaction) (73%) [5471], (42%) [5472].

m.p. 206–208° [5472], 202–203° [5471];
^{1}H NMR [5471]· ^{13}C NMR [5293], UV [5472];
TLC [5471].

2-(1,3-Benzodioxol-5-yl)-1-(2,4,6-trihydroxyphenyl)ethanone

[39548-98-6]

$C_{15}H_{12}O_6$ mol.wt. 288.26



Syntheses

– Preparation by reaction of 3,4-(methylenedioxy)phenyl-acetonitrile with phloroglucinol (Hoesch reaction) (65–66%) [5330,5473].

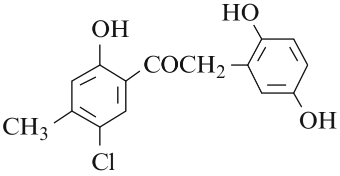
– Also refer to: [5295].

m.p. 202° [5330,5473].

1-(5-Chloro-2-hydroxy-4-methylphenyl)-2-(2,5-dihydroxyphenyl)ethanone

[115781-56-1]

$C_{15}H_{13}ClO_4$ mol.wt. 292.74



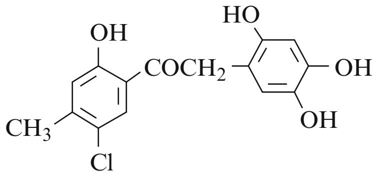
Synthesis

– Obtained by alkali cleavage of 6-chloro-3-(2′,5′-di-hydroxyphenyl)-4-hydroxy-7-methylcoumarin with refluxing 2% methanolic potassium hydroxide for 4 h (65%) [5416].

m.p. 222° [5416]; IR [5416], UV [5416].

1-(5-Chloro-2-hydroxy-4-methylphenyl)-2-(2,4,5-trihydroxyphenyl)ethanone

[115781-52-7] $C_{15}H_{13}ClO_5$ mol.wt. 308.72



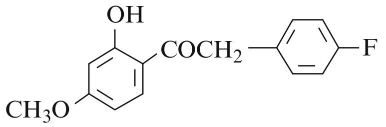
Synthesis

- Obtained from 6-chloro-4-hydroxy-7-methyl-3-(2′,4′,5′-trihydroxyphenyl) coumarin with refluxing 2% methanolic potassium hydroxide for 4 h (78%) [5416].

m.p. 201° [5416]; IR [5416], UV [5416].

1-(2-Hydroxy-4-methoxyphenyl)-2-(4-fluorophenyl)ethanone

[128040-46-0] $C_{15}H_{13}FO_3$ mol.wt. 260.26



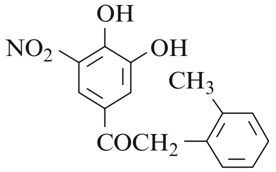
Synthesis

- Preparation by partial methylation of 4′-fluoro-2,4-dihydroxydeoxybenzoin with dimethyl sulfate in refluxing acetone for 6 h (90%) [5420] or in the presence of potassium carbonate in refluxing acetone for 4 h (60%) [5421].

m.p. 90–92° [5421], 78–80° [5420]; ^{1}H NMR [5420,5421], MS [5421].

1-(3,4-Dihydroxy-5-nitrophenyl)-2-(2-methylphenyl)ethanone

[274925-87-0] $C_{15}H_{13}NO_5$ mol.wt. 287.27



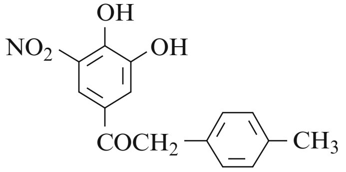
Synthesis

- Preparation by treatment of 1-(4-hydroxy-3-methoxy-5-nitrophenyl)-2-(2-methylphenyl)ethanone with aluminium chloride in refluxing ethyl acetate/pyridine mixture for 2 h (90–96%) [5193].

m.p. 163–165° [5193];
^{1}H NMR [5193], ^{13}C NMR [5193], IR [5193].

1-(3,4-Dihydroxy-5-nitrophenyl)-2-(4-methylphenyl)ethanone

[400871-10-5] $C_{15}H_{13}NO_5$ mol.wt. 287.27



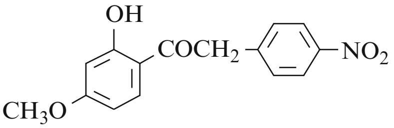
Synthesis

- Preparation by treatment of 1-(4-hydroxy-3-methoxy-5-nitrophenyl)-2-(4-methylphenyl) ethanone with aluminium chloride in refluxing ethyl acetate/pyridine mixture for 2 h (90–96%) [5193].

m.p. 189–190° [5193];
^{1}H NMR [5193], ^{13}C NMR [5193], IR [5193].

1-(2-Hydroxy-4-methoxyphenyl)-2-(4-nitrophenyl)ethanone

[57272-98-7]



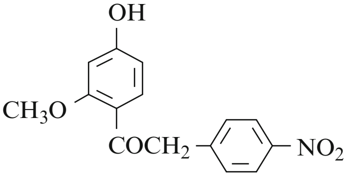
$C_{15}H_{13}NO_5$ mol.wt. 287.27

Syntheses

- Obtained (poor yield) by reaction of p-nitrophenyl-acetonitrile with resorcinol monomethyl ether (9%) (Hoesch reaction) [5425].
- Also obtained by partial methylation of 2,4-di-hydroxyphenyl 4-nitrobenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone [5217].

m.p. 136° [5425], 134–136° [5217].

1-(4-Hydroxy-2-methoxyphenyl)-2-(4-nitrophenyl)ethanone



$C_{15}H_{13}NO_5$ mol.wt. 287.27

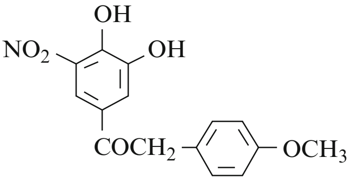
Synthesis

- Obtained by reaction of p-nitrophenylacetonitrile with resorcinol monomethyl ether (Hoesch reaction) [5425].

m.p. 149–150° [5425].

1-(3,4-Dihydroxy-5-nitrophenyl)-2-(4-methoxyphenyl)ethanone

[440362-23-2]



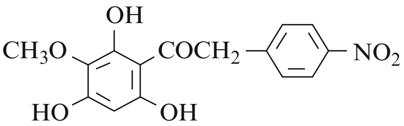
$C_{15}H_{13}NO_6$ mol.wt. 303.27

Synthesis

- Preparation by partial demethylation of 1-(4-hydroxy-3-methoxy-5-nitrophenyl)-2-(4-methoxyphenyl)ethanone using aluminium chloride and pyridine in ethyl acetate at reflux for 2 h (91%) [5192].

diacetate m.p. 88–89° [5192].

2-(4-Nitrophenyl)-1-(2,4,6-trihydroxy-3-methoxyphenyl)ethanone



$C_{15}H_{13}NO_7$ mol.wt. 319.27

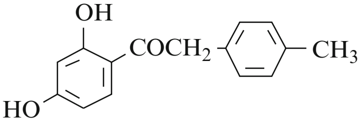
Synthesis

- Obtained by reaction of p-nitrophenylacetonitrile with iretol (Hoesch reaction) (50%) [5474].

m.p. 220° [5474].

1-(2,4-Dihydroxyphenyl)-2-(4-methylphenyl)ethanone

[59208-55-8] $C_{15}H_{14}O_3$ mol.wt. 242.27



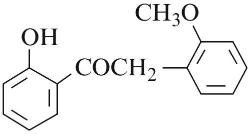
Synthesis

– Obtained by reaction of p-tolylacetonitrile with resorcinol (Hoesch reaction) [5262].

m.p. 114° [5262].

1-(2-Hydroxyphenyl)-2-(2-methoxyphenyl)ethanone

[92549-19-4] $C_{15}H_{14}O_3$ mol.wt. 242.27



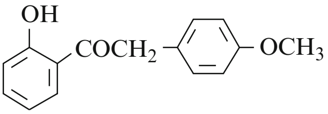
Syntheses

– Preparation by stirring a mixture of S-[3-hydroxy-4-[(2-methoxyphenyl)acetyl]phenyl] dimethylcarbamothioate [142751-41-5], Raney nickel and ethanol at r.t. for 1 h (71%) [5205].
– Also obtained by heating 2-methoxybenzoyl-2-methoxy-phenylacetonitrile (m.p. 107–108°) in acetic acid with concentrated hydrochloric acid on a steam bath for 15 h (47%) [5475].
– Also obtained by alkaline degradation of 2′-methoxyisoflavone (m.p. 184°) with potassium hydroxide in boiling aqueous methanol for 1.5 h (almost quantitative yield) [5476].
– Also obtained by heating ethyl 2-methoxybenzoyl-2-methoxyphenylacetate (m.p. 76–77°) in acetic acid with concentrated hydrochloric acid on a steam bath for 15 h (54%) [5475].
– Also refer to: [5477].

colourless oil [5475]; b.p.$_{0.004}$ 140–150° [5475];
m.p. 64° [5476], 59–60° [5205]; ^{1}H NMR [5205], MS [5205].

1-(2-Hydroxyphenyl)-2-(4-methoxyphenyl)ethanone

[79744-47-1] $C_{15}H_{14}O_3$ mol.wt. 242.27



Syntheses

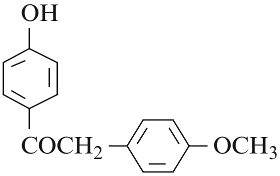
– Preparation by addition of p-methoxybenzylmagnesium chloride to a solution of 2-hydroxybenzonitrile in THF at r.t. overnight, then refluxing with aqueous hydrochloric acid for 4 h (90%) [5478].
– Also obtained by heating a solution of 2-methoxy-benzoyl-4-methoxyphenylacetonitrile (m.p. 109–110°5) in acetic acid containing hydrochloric acid on a steam bath for 15 h (47%) [5479].
– Also obtained by stirring a mixture of S-[3-hydroxy-4-[(4-methoxyphenyl) acetyl]phenyl] dimethylcarbamothioate [142751-42-6], Raney nickel and ethanol at r.t. for 1 h (73%) [5205].

- Also obtained by heating a solution of ethyl 2-methoxybenzoyl-4-methoxyphenylacetate (b.p.$_{0.004}$ 180–200°) in acetic acid containing hydrochloric acid on a steam bath for 15 h (54%) [5479].
- Also obtained by Fries rearrangement of phenyl p-methoxyphenylacetate with aluminium chloride for 1.5 h at 145° (13%) [5280].

m.p. 86° [5280], 85–86° [5205], 79–81° [5478]; b.p.$_{0.003}$ 160–180° [5479];
^{1}H NMR [5205,5280], ^{13}C NMR [5293], IR [5280], MS [5205,5280,5478];
TLC [5478].

1-(4-Hydroxyphenyl)-2-(4-methoxyphenyl)ethanone

[3669-46-3] $C_{15}H_{14}O_3$ mol.wt. 242.27



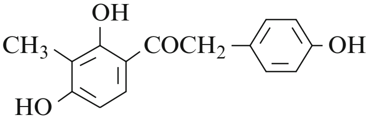
Syntheses

- Obtained by Fries rearrangement of phenyl p-methoxy-phenylacetate in nitromethane,
  - in the presence of aluminium chloride for 25 h at 20° (48%) [5480] or for 1.5 h at 145° (12%) [5280];
  - in the presence of titanium tetrachloride for 6 h at 20° (26%) [5480].
- Also obtained by partial demethylation of 4,4′-dimethoxydeoxybenzoin,
  - with aluminium chloride in refluxing benzene for 1.5 h (33%) [5433];
  - with sodium in refluxing ethylene glycol for 3 h (12%) [5481].

m.p. 175–178° [5433], 175° [5280,5481], 171° [5480];
^{1}H NMR (Sadtler: standard n° 44611 M),
IR (Sadtler: standard n° 71639 K) [5480], UV [5480], MS [5480].

1-(2,4-Dihydroxy-3-methylphenyl)-2-(4-hydroxyphenyl)ethanone

[139256-03-4] $C_{15}H_{14}O_4$ mol.wt. 258.27



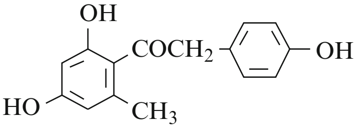
Synthesis

- Obtained by reaction of p-hydroxyphenylacetic acid with 2-methylresorcinol in the presence of boron trifluoride etherate under argon, on a water bath for 5 h (97%) [5208].

m.p. 187–188° [5208]; ^{1}H NMR [5208], ^{13}C NMR [5208], MS [5208].

1-(2,4-Dihydroxy-6-methylphenyl)-2-(4-hydroxyphenyl)ethanone

[139256-04-5] $C_{15}H_{14}O_4$ mol.wt. 258.27



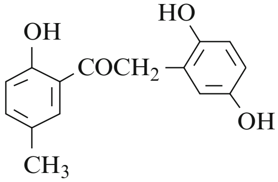
Synthesis

- Obtained by reaction of p-hydroxyphenylacetic acid with orcinol in the presence of boron trifluoride etherate under argon on a water bath for 2 h (86%) [5208].

m.p. 186–187° [5208]; ^{1}H NMR [5208], ^{13}C NMR [5208], MS [5208].

2-(2,5-Dihydroxyphenyl)-1-(2-hydroxy-5-methylphenyl)ethanone

[115781-53-8] $C_{15}H_{14}O_4$ mol.wt. 258.27



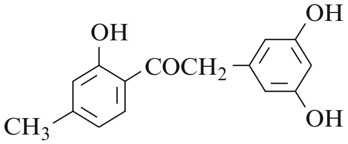
Synthesis

- Obtained by alkali cleavage of 3-(2′,5′-dihydroxyphenyl)-4-hydroxy-6-methylcoumarin with refluxing 2% methanolic potassium hydroxide for 4 h (79%) [5416].

m.p. 223° [5416]; IR [5416], UV [5416].

2-(3,5-Dihydroxyphenyl)-1-(2-hydroxy-4-methylphenyl)ethanone

[111192-02-0] $C_{15}H_{14}O_4$ mol.wt. 258.27



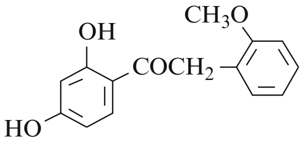
Synthesis

- Obtained by decarboxylation of 6,8-dihydroxy-3-(2-hydroxy-4-methylphenyl) isocoumarin (m.p. 201–202°) with refluxing 10% aqueous potassium hydroxide solution for 6 h (90%) [5482].

m.p. 89° [5482]; ^{1}H NMR [5482], IR [5482], MS [5482].

1-(2,4-Dihydroxyphenyl)-2-(2-methoxyphenyl)ethanone

[92549-46-7] $C_{15}H_{14}O_4$ mol.wt. 258.27



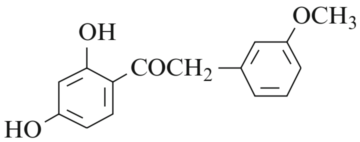
Syntheses

- Preparation by reaction of o-methoxyphenylacetic acid with resorcinol in the presence of boron trifluoride etherate under argon on a water bath for 1 h (98%) [5208].
- Also obtained by reaction of o-methoxyphenylacetonitrile with resorcinol (Hoesch reaction) (25%) [5438], (23%) [5483].
- Also refer to: [5263,5479].

m.p. 164° [5483], 159–160° [5438].

1-(2,4-Dihydroxyphenyl)-2-(3-methoxyphenyl)ethanone

[89019-83-0] $C_{15}H_{14}O_4$ mol.wt. 258.27



Syntheses

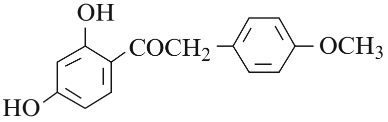
- Preparation by reaction of m-methoxyphenylacetic acid with resorcinol under argon atmosphere,
  - in the presence of boron trifluoride etherate on a water bath for 1 h (96%) [5208];

- in ethylene dichloride in the presence of boron trifluoride, first at r.t., then at 60° for 2 h (63%) [5439].

m.p. 109–110° [5439]; ^{1}H NMR [5439], IR [5439], UV [5439], MS [5439].

1-(2,4-Dihydroxyphenyl)-2-(4-methoxyphenyl)ethanone *(Ononetin)*

[487-49-0] $C_{15}H_{14}O_4$ mol.wt. 258.27



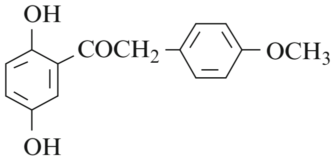
Syntheses

- Preparation by condensation of p-methoxyphenyl-acetonitrile with resorcinol (Hoesch reaction) [4989,5264,5265,5385], (64%) [5484], (50%) [5268], (48%) [5444].
- Preparation by Friedel–Crafts acylation of resorcinol with p-methoxyphenylacetyl chloride in the presence of aluminium chloride for 24 h at r.t., in nitrobenzene (50%) [5250] or in ethyl ether (36%) [5251].
- Also obtained by reaction of p-methoxyphenylacetic anhydride with resorcinol in the presence of boron trifluoride etherate for 3.5 h at 75° (67%) [5255].
- Also obtained by reaction of p-methoxyphenylacetic acid with resorcinol,
  - in the presence of boron trifluoride etherate under argon on a water bath for 1.5 h (98%) [5208] or at 100° for 1 h (77%) [5439];
  - in the presence of boron trifluoride in chloroform (51%) [5258].
- Also obtained by alkaline degradation of *formononetin* (7-hydroxy-4′-methoxyisoflavone) (SM) (m.p. 265°) [5444,5484,5485,5486], (95%) [5487], (51%) [5484]. SM was prepared by hydrolysis of *ononin* with 4% sulfuric acid [5484].
- Also obtained by degradation of *onospin* (m.p. 172°) (SM1) by heating with dilute sulfuric acid or by treatment with emulsin. SM1 was prepared from *ononin* by heating with 10% sodium hydroxide for 2 min [5484,5486].
- Also obtained by decarboxylation of 5-carboxy-2,4-dihydroxy-4′-methoxydeoxybenzoin (m.p. 200°) in boiling quinoline containing copper bronze during 5 min (26%) [5488].
- Also refer to: [4685,4983,5181,5205,5217,5220,5263,5271,5273,5277,5279, 5281,5415,5417,5442,5448,5449].

m.p. 161°5–162°5 [5264], 159°5 [5484], 159° [4989,5258], 158–159° [5251], 158° [5250,5444,5487,5488], 156–157° [5283], 153–155° [5439];
^{1}H NMR [5268,5439], ^{13}C NMR [5293,5450], IR [5439],
UV [5264,5439], MS [5268,5439].

1-(2,5-Dihydroxyphenyl)-2-(4-methoxyphenyl)ethanone

[56308-07-7] $C_{15}H_{14}O_4$ mol.wt. 258.27

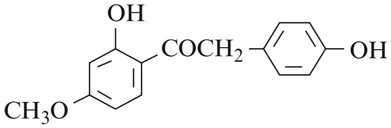


Synthesis

– Refer to: [5281] (Hungarian paper).

1-(2-Hydroxy-4-methoxyphenyl)-2-(4-hydroxyphenyl)ethanone

[60278-33-3] $C_{15}H_{14}O_4$ mol.wt. 258.27



Syntheses

– Obtained by reaction of p-hydroxyphenylacetic acid with m-methoxyphenol,

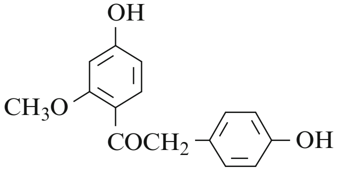
- in the presence of boron trifluoride in ethylene dichloride at 80° for 2 h under an argon atmosphere (47%) [5439];
- in the presence of polyphosphoric acid at 95° for 30 min (57%) [5334].

– Also refer to: [5270,5448,5489].

gum [5334]; m.p. 151–154° [5439];
^{1}H NMR [5439], ^{13}C NMR [5293], IR [5439], UV [5439], MS [5439].

1-(4-Hydroxy-2-methoxyphenyl)-2-(4-hydroxyphenyl)ethanone

[89019-88-5] $C_{15}H_{14}O_4$ mol.wt. 258.27



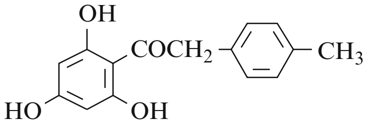
Synthesis

– Obtained by reaction of p-hydroxyphenylacetic acid with m-methoxyphenol in ethylene dichloride in the presence of boron trifluoride at 80° for 2 h under an argon atmosphere (23%) [5439].

m.p. 147–151° [5439]; ^{1}H NMR [5439],
IR [5439], UV [5439], MS [5439].

2-(4-Methylphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[59108-68-8] $C_{15}H_{14}O_4$ mol.wt. 258.27



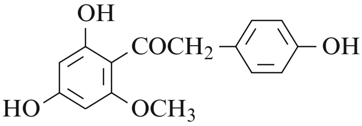
Synthesis

– Preparation by reaction of p-tolylacetonitrile with phloroglucinol (Hoesch reaction) [4852,5265].

m.p. 205–206° (anhydrous) [5262];
sesquihydrate [5262].

1-(2,4-Dihydroxy-6-methoxyphenyl)-2-(4-hydroxyphenyl)ethanone

[56308-11-3]



$C_{15}H_{14}O_5$ mol.wt. 274.27

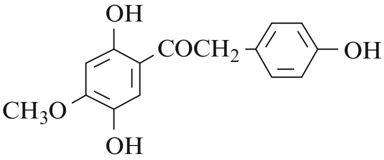
Syntheses

- Preparation by reaction of p-hydroxyphenyl-acetonitrile with phloroglucinol monomethyl ether (57%) (Hoesch reaction) [5330].
- Also refer to: [5281,5295].

m.p. 186–188° [5330].

1-(2,5-Dihydroxy-4-methoxyphenyl)-2-(4-hydroxyphenyl)ethanone

[79744-54-0]



$C_{15}H_{14}O_5$ mol.wt. 274.27

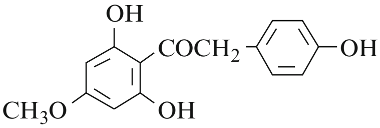
Synthesis

- Obtained by reaction of p-hydroxyphenylacetonitrile with 2-methoxyhydroquinone (Hoesch reaction) [5293,5351].

^{13}C NMR [5293].

1-(2,6-Dihydroxy-4-methoxyphenyl)-2-(4-hydroxyphenyl)ethanone

[101094-12-6]



$C_{15}H_{14}O_5$ mol.wt. 274.27

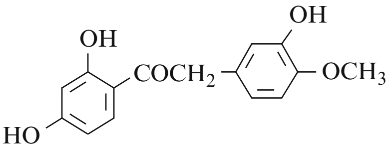
Synthesis

- Obtained by heating a mixture of 5,4′-dihydroxy-7-methoxyisoflavone and tribasic sodium phosphate in water at reflux for 1 h (83%) [5295].

m.p. 247–249° [5295].

1-(2,4-Dihydroxyphenyl)-2-(3-hydroxy-4-methoxyphenyl)ethanone

[36754-72-0]



$C_{15}H_{14}O_5$ mol.wt. 274.27

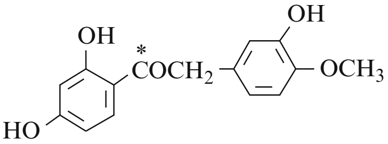
Syntheses

- Obtained by reaction of (3-hydroxy-4-methoxy-phenyl)acetonitrile with resorcinol (Hoesch reaction) (41%) [5490,5491].
- Also refer to: [5380,5492].

m.p. 161–162° [5490,5491].

1-(2,4-Dihydroxyphenyl)-2-(3-hydroxy-4-methoxyphenyl)ethanone-*1*-^{14}*C*

[142050-40-6] $C_{15}H_{14}O_5$ mol.wt. 274.27



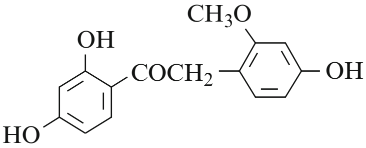
Synthesis

- Obtained by hydrolysis of 2,4-dihydroxyphenyl 3-benzoyloxy-4-methoxybenzyl [^{14}C] ketone (SM) with sodium hydroxide in dilute methanol (31–35%). SM was obtained by reaction of 3-benzoyloxy-4-methoxybenzyl [^{14}C] nitrile with resorcinol (Hoesch reaction) [5492].

m.p. 166–168° [5492].

1-(2,4-Dihydroxyphenyl)-2-(4-hydroxy-2-methoxyphenyl)ethanone

[175546-62-0] $C_{15}H_{14}O_5$ mol.wt. 274.27

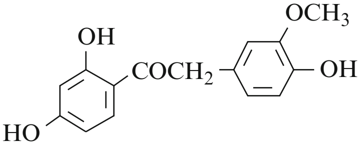


Synthesis

- Refer to: [5277].

1-(2,4-Dihydroxyphenyl)-2-(4-hydroxy-3-methoxyphenyl)ethanone

[40456-49-3] $C_{15}H_{14}O_5$ mol.wt. 274.27



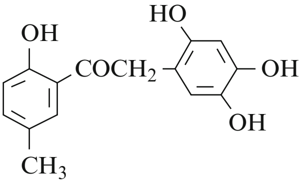
Syntheses

- Preparation by reaction of 4-hydroxy-3-methoxyphenyl-acetic acid with resorcinol in the presence of boron trifluoride etherate under argon on a water bath for 1 h (99%) [5208].
- Also obtained by reaction of (4-acetoxy-3-methoxy-phenyl)acetonitrile with resorcinol (Hoesch reaction) [5493].

colourless granules [5493]; ^{1}H NMR [5493], IR [5493].

1-(2-Hydroxy-5-methylphenyl)-2-(2,4,5-trihydroxyphenyl)ethanone

[115781-49-2] $C_{15}H_{14}O_5$ mol.wt. 274.27

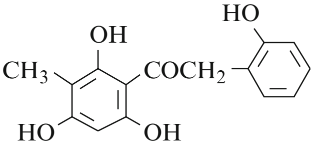


Synthesis

- Obtained from 4-hydroxy-6-methyl-3-(2′,4′, 5′-trihydroxy-phenyl)coumarin with refluxing 2% methanolic potassium hydroxide for 4 h (67%) [5416].

m.p. 193° [5416]; IR [5416], UV [5416].

2-(2-Hydroxyphenyl)-1-(2,4,6-trihydroxy-3-methylphenyl)ethanone



$C_{15}H_{14}O_5$ mol.wt. 274.27

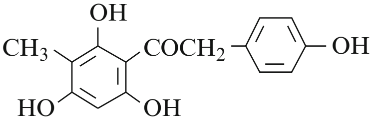
Synthesis

- Obtained by alkaline degradation of methyl*isogenistein* (8-methyl-5,7,2′-trihydroxyisoflavone) (SM) (m.p. 255°) with potassium hydroxide [5453,5455]. SM was obtained by hydrolysis of *methylisogenistin*, its glycoside, (m.p. 301–302°) [5455], isolated from soya bean [5453,5455].

N.B.: This ketone cannot be prepared by condensation of o-hydroxyphenylacetonitrile with 2-methylphloroglucinol (Hoesch reaction) [5438].

m.p. 186° [5455].

2-(4-Hydroxyphenyl)-1-(2,4,6-trihydroxy-3-methylphenyl)ethanone



$C_{15}H_{14}O_5$ mol.wt. 274.27

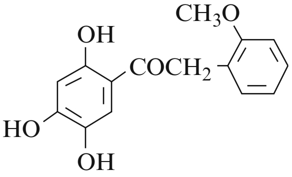
Syntheses

- Obtained by partial demethylation of 2-hydroxy-4,6-dimethoxy-3-methylphenyl 4-methoxybenzyl ketone with aluminium chloride in refluxing benzene for 2 h (58%) [5494].
- Also obtained by alkaline degradation of *methylgenistein* (8-methyl-5,7,4′-trihydroxyisoflavone) (m.p. 298°) with potash [5453,5455].

m.p. 235–237° [5494], 190° [5455]. One of the reported melting points is obviously wrong.

2-(2-Methoxyphenyl)-1-(2,4,5-trihydroxyphenyl)ethanone

[79744-49-3]



$C_{15}H_{14}O_5$ mol.wt. 274.27

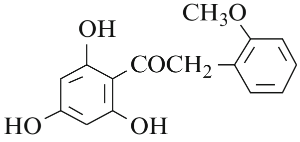
Synthesis

- Obtained by reaction of 2-methoxyphenylacetonitrile with hydroxyhydroquinone (Hoesch reaction) [5293,5351].

^{13}C NMR [5293].

2-(2-Methoxyphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[116854-95-6] $C_{15}H_{14}O_5$ mol.wt. 274.27



Syntheses

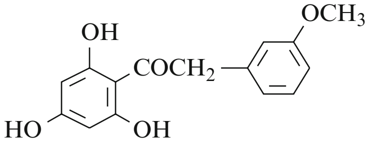
- Preparation by reaction of 2-methoxyphenylacetonitrile with phloroglucinol (Hoesch reaction),
  - in the presence of zinc chloride [5287,5457], (74%) [5454], (48%) [5456], (42%) [5255], (37%) [5495];
  - in the presence of boron trifluoride etherate (45%) [5255].
- Also refer to: [5295,5496,5497,5498].

monohydrate [5456,5495]; sublimation at 120°/0.04 mm [5495];

m.p. 170° [5287], 169° [5495], 168–170° [5456], 167–169° [5454,5457].

2-(3-Methoxyphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[111474-27-2] $C_{15}H_{14}O_5$ mol.wt. 274.27



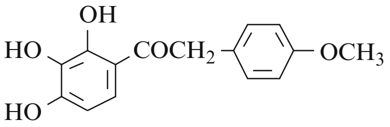
Synthesis

- Preparation by reaction of 3-methoxyphenyl-acetonitrile with phloroglucinol (Hoesch reaction) (75%) [5496], (30%) [5295].

m.p. 168–169° [5295], 165–166° [5496]; ^{1}H NMR [5496], IR [5496].

2-(4-Methoxyphenyl)-1-(2,3,4-trihydroxyphenyl)ethanone

[38412-59-8] $C_{15}H_{14}O_5$ mol.wt. 274.27



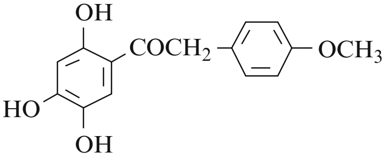
Syntheses

- Obtained by acid hydrolysis of 4-methoxybenzyl 2-hydroxy-3,4-diphenylmethylenedioxyphenyl ketone (m.p. 146°) in acetic acid in the presence of 2 drops of concentrated hydrochloric acid at 100° for 5 min (48%) [5499].
- Also obtained by reaction of 4-methoxyphenylacetic acid with pyrogallol in chloroform in the presence of excess boron trifluoride, first at 0°, then at r.t. overnight (77%) [5258] or for 2 days (54%) [5499].
- Also refer to: [5351,5296,5500].

m.p. 157° [5499], 145–146° [5258];
^{1}H NMR [5499], ^{13}C NMR [5293]; TLC [5499].

2-(4-Methoxyphenyl)-1-(2,4,5-trihydroxyphenyl)ethanone

[76095-38-0] $C_{15}H_{14}O_5$ mol.wt. 274.27



Syntheses

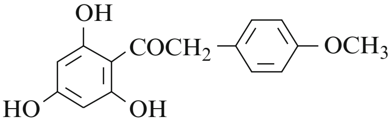
– Preparation by reaction of p-methoxyphenyl-acetonitrile with hydroxyhydroquinone (Hoesch reaction) [5291,5293,5351,5501], (80%) [5471].

– Also refer to: [5502,5503,5504,5505].

m.p. 180–181° [5471,5501]; TLC [5471];
^{1}H NMR [5471], ^{13}C NMR [5293], UV [5501].

2-(4-Methoxyphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[15485-66-2] $C_{15}H_{14}O_5$ mol.wt. 274.27



Syntheses

– Preparation by reaction of p-methoxyphenyl-acetonitrile with phloroglucinol (Hoesch reaction),
  - in the presence of zinc chloride [4852,5264,5265], (91–92%) [5461,5462,5506], (80%) [5459], (66%) [5294], (57%) [5250], (55%) [5255];
  - in the presence of boron trifluoride etherate (82%) [5255].

– Preparation by Fries rearrangement of 3,5-dihydroxyphenyl p-methoxyphenylacetate with aluminium chloride in nitrobenzene, first at 60°, then at 150° for 2 h (75%) [5506].

– Preparation by Friedel–Crafts acylation of phloroglucinol with p-methoxyphenylacetyl chloride in nitrobenzene at 100° for 2 h (50%) [5506].

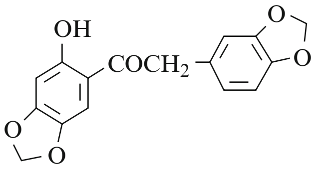
– Preparation by reaction of p-methoxyphenylacetic acid with phloroglucinol in the presence of zinc chloride and phosphorous oxychloride for 24 h at r.t. (68%) [5209].

– Also refer to: [4685,5263,5273,5281,5295,5415,5417,5448,5460].

m.p. 198–200° [5462], 198–199° [5264], 195° [5250], 194° [5294],
192–193° [5459,5506], 192° [5209]; monohydrate [5459];
^{1}H NMR [5462], ^{13}C NMR [5293,5450,5462], IR [5294],
UV [5264,5294,5462]; TLC [5294].

2-(1,3-Benzodioxol-5-yl)-1-(6-hydroxy-1,3-benzodioxol-5-yl)ethanone

[2746-90-9] $C_{16}H_{12}O_6$ mol.wt. 300.27



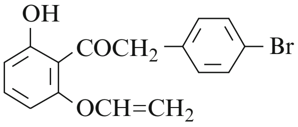
Syntheses

– Obtained by reaction of (3,4-methylenedioxy)phenyl-acetonitrile with *sesamol* (5-hydroxy-1,3-benzodioxol) (Hoesch reaction) (13%) [5472].

- Also obtained by reaction of methylene sulfate with 2,4,5-trihydroxyphenyl (3,4-methylenedioxy)benzyl ketone in the presence of potassium hydroxide in dilute acetone for 70 min at 45–50° (9%) [5472].

m.p. 172–173° [5472]; IR [5472], UV [5472].

2-(4-Bromophenyl)-1-[2-(ethenyloxy)-6-hydroxyphenyl]ethanone



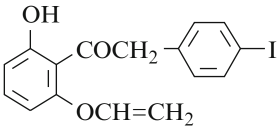
$C_{16}H_{13}BrO_3$ mol.wt. 333.18

Synthesis

- Obtained (by-product) by reaction of diethylaminochloro-ethane with 2,6-dihydroxy-4′-bromodesoxybenzoin in the presence of sodium ethoxide in refluxing ethanol for 4 h [5357].

m.p. 103° [5357].

1-[2-(Ethenyloxy)-6-hydroxyphenyl]-2-(4-iodophenyl)ethanone



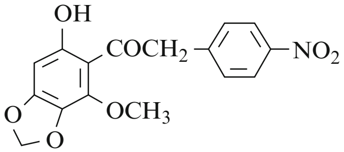
$C_{16}H_{13}IO_3$ mol.wt. 380.18

Synthesis

- Obtained (by-product) by reaction of diethylaminochloro-ethane with 2,6-dihydroxy-4′-iododesoxybenzoin in the presence of sodium ethoxide in refluxing ethanol for 4 h [5357].

m.p. 131° [5357].

1-(6-Hydroxy-4-methoxy-1,3-benzodioxol-5-yl)-2-(4-nitrophenyl)ethanone



$C_{16}H_{13}NO_7$ mol.wt. 331.28

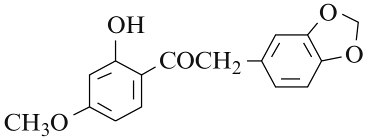
Synthesis

- Obtained by condensation of 3-methoxy-4,5-methylenedioxyphenol with 4-nitrophenyl-acetontrile (Hoesch reaction) (37%) [5507].

m.p. 165–167° [5507]; IR [5507].

2-(1,3-Benzodioxol-5-yl)-1-(2-hydroxy-4-methoxyphenyl)ethanone

[5128-56-3]



$C_{16}H_{14}O_5$ mol.wt. 286.28

Syntheses

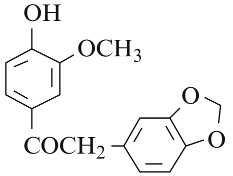
- Obtained by partial methylation of *pseudo-baptigenetin* in ethanol with diazomethane in ethyl ether for 24 h (82%) [5468].

- Also refer to: [5380,5508].

m.p. 145° [5468].

2-(1,3-Benzodioxol-5-yl)-1-(4-hydroxy-3-methoxyphenyl)ethanone

[56766-87-1] $C_{16}H_{14}O_5$ mol.wt. 286.28



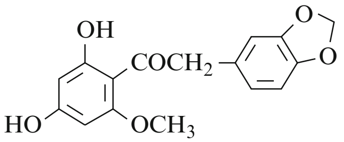
Synthesis

– Obtained by hydrogenolysis of 4-benzyloxy-3-methoxy-phenyl 3,4-methylenedioxybenzyl ketone (oil) with hydrogen in the presence of 10% Pd/C in ethanol at r.t. for 1.5 h (87%) [5509].

m.p. 132–133° [5509];
^{1}H NMR [5509], IR [5509], UV [5509], MS [5509].

2-(1,3-Benzodioxol-5-yl)-1-(2,4-dihydroxy-6-methoxyphenyl)ethanone

[55607-36-8] $C_{16}H_{14}O_6$ mol.wt. 302.28



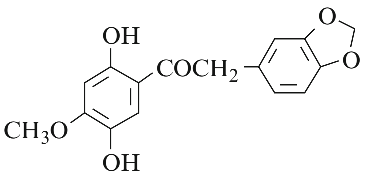
Synthesis

– Obtained by reaction of 3,4-methylenedioxyphenyl-acetonitrile with phloroglucinol monomethyl ether (Hoesch reaction) [5295], (27%) [5352].

m.p. 143–144° [5295], 138–139° [5352];
^{1}H NMR [5352]; TLC [5352].

2-(1,3-Benzodioxol-5-yl)-1-(2,5-dihydroxy-4-methoxyphenyl)ethanone

[2746-89-6] $C_{16}H_{14}O_6$ mol.wt. 302.28



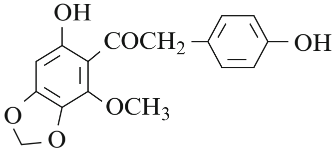
Synthesis

– Obtained by treatment of 2,4,5-trihydroxyphenyl (3,4-methylenedioxy)benzyl ketone in acetone with an ethereal diazomethane solution at r.t. overnight (76%) [5472].

m.p. 194–196° [5472]; UV [5472].

1-(6-Hydroxy-4-methoxy-1,3-benzodioxol-5-yl)-2-(4-hydroxyphenyl)ethanone

[3207-38-3] $C_{16}H_{14}O_6$ mol.wt. 302.28



Syntheses

– Obtained by alkaline degradation of *irisolone* (4′-hydroxy-5-methoxy-6,7-methylenedioxyisoflavone) (m.p. 258–265°) (SM) with refluxing aqueous sodium hydroxide for 1.5 h. SM was isolated from the rhizomes of *iris nepalensis* D. DON (Iridaceae) [5510].

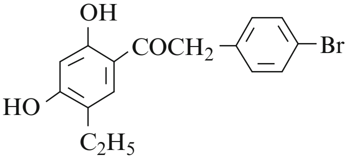
– Also obtained by reaction of 4-hydroxyphenylacetonitrile with 3-methoxy-4,5-methylenedioxy-phenol (Hoesch reaction) (7%) [5507].

- Also obtained by diazotization of 6-hydroxy-2-methoxy-3,4-methylenedioxyphenyl 4-aminobenzyl ketone, followed by hydrolysis of the diazonium salt obtained (10%) [5507].

m.p. 159–160° [5510], 152–153° [5507]; IR [5507], UV [5507,5510].

2-(4-Bromophenyl)-1-(5-ethyl-2,4-dihydroxyphenyl)ethanone

[96643-99-1] $C_{16}H_{15}BrO_3$ mol.wt. 335.20



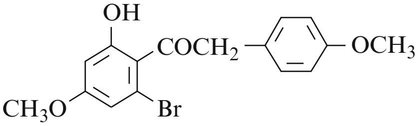
Synthesis

- Preparation by reaction of p-bromophenylacetonitrile with 4-ethylresorcinol in the presence of boron trifluoride etherate under hydrogen chloride atmosphere at r.t. overnight (80%) [5368], (70%) [5367].

m.p. 124–125° [5367,5371]; ^{1}H NMR [5367].

1-(2-Bromo-6-hydroxy-4-methoxyphenyl)-2-(4-methoxyphenyl)ethanone

[191847-25-3] $C_{16}H_{15}BrO_4$ mol.wt. 351.20



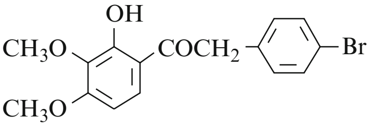
Synthesis

- Obtained by Friedel–Crafts acylation of 3,5-di-methoxybromobenzene with 4-methoxyphenyl-acetyl chloride in the presence of aluminium chloride (12%) [5511].

m.p. 180–182° [5511]; ^{1}H NMR [5511], MS [5511].

2-(4-Bromophenyl)-1-(2-hydroxy-3,4-dimethoxyphenyl)ethanone

$C_{16}H_{15}BrO_4$ mol.wt. 351.20



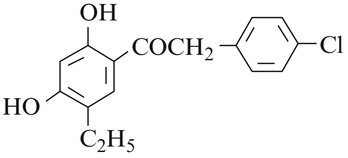
Synthesis

- Refer to: [5370] (Japanese paper).

m.p. 134–135° [5370].

2-(4-Chlorophenyl)-1-(5-ethyl-2,4-dihydroxyphenyl)ethanone

[96644-00-7] $C_{16}H_{15}ClO_3$ mol.wt. 290.75



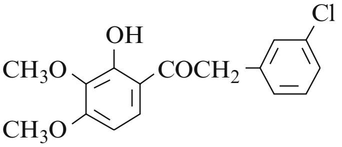
Synthesis

- Preparation by reaction of p-chlorophenylacetonitrile with 4-ethylresorcinol in the presence of boron trifluoride etherate under hydrogen chloride atmosphere at r.t. overnight (80%) [5368], (70%) [5367].

m.p. 130–131° [5367,5368]; ^{1}H NMR [5367].

2-(3-Chlorophenyl)-1-(2-hydroxy-3,4-dimethoxyphenyl)ethanone

[24863-50-1] $C_{16}H_{15}ClO_4$ mol.wt. 306.75



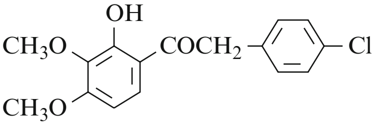
Synthesis

– Obtained by Friedel–Crafts acylation of pyrogallol tri-methyl ether with m-chlorophenylacetyl chloride in the presence of aluminium chloride in carbon disulfide for 30 min (54%) [5370].

m.p. 111–112° [5370]; ^{1}H NMR [5370].

2-(4-Chlorophenyl)-1-(2-hydroxy-3,4-dimethoxyphenyl)ethanone

[24852-34-4] $C_{16}H_{15}ClO_4$ mol.wt. 306.75



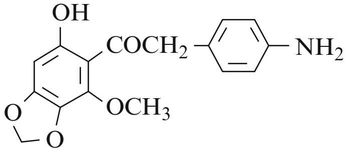
Synthesis

– Obtained by Friedel–Crafts acylation of pyrogallol trimethyl ether with p-chlorophenylacetyl chloride in carbon disulfide in the presence of aluminium chloride for 30 min (59%) [5370].

m.p. 113–114° [5370].

2-(4-Aminophenyl)-1-(6-hydroxy-4-methoxy-1,3-benzodioxol-5-yl)ethanone

$C_{16}H_{15}NO_5$ mol.wt. 301.30



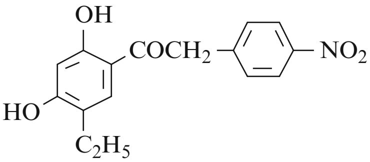
Synthesis

– Obtained by catalytic reduction of 6-hydroxy-2-methoxy-3,4-methylenedioxyphenyl 4-nitrobenzyl ketone in ethyl acetate with hydrogen in the presence of 10% Pd/C at r.t. (82%) [5507].

m.p. 170–172° [5507]; IR [5507].

1-(5-Ethyl-2,4-dihydroxyphenyl)-2-(4-nitrophenyl)ethanone

[96644-02-9] $C_{16}H_{15}NO_5$ mol.wt. 301.30

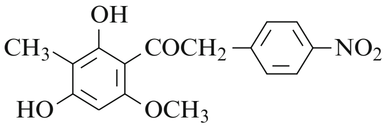


Synthesis

– Obtained by reaction of p-nitrophenylacetonitrile with 4-ethylresorcinol in the presence of boron trifluoride etherate under hydrogen chloride atmosphere (8–10 h), then at r.t. overnight (94%) [5367].

m.p. 159–160° [5367]; ^{1}H NMR [5367].

1-(2,4-Dihydroxy-6-methoxy-3-methylphenyl)-2-(4-nitrophenyl)ethanone



$C_{16}H_{15}NO_6$ mol.wt. 317.30

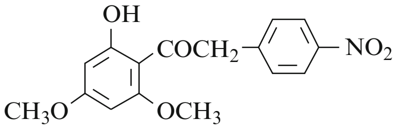
Synthesis

– Obtained by reaction of p-nitrophenylacetonitrile with 2,6-dihydroxy-4-methoxytoluene (Hoesch reaction) [5258].

m.p. 201° [5258].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-(4-nitrophenyl)ethanone

[56982-36-6]



$C_{16}H_{15}NO_6$ mol.wt. 317.30

Syntheses

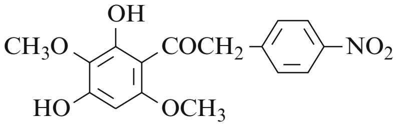
– Obtained by partial methylation of 4-nitrobenzyl 2,4,6-trihydroxyphenyl ketone,

- with diazomethane in ethyl ether at 0° for 48 h (57%) [5382];
- with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone [5217].

– Also refer to: [5512].

m.p. 150° [5217], 148–149° [5512], 148° [5382].

1-(2,4-dihydroxy-3,6-dimethoxyphenyl)-2-(4-Nitrophenyl)ethanone



$C_{16}H_{15}NO_7$ mol.wt. 333.30

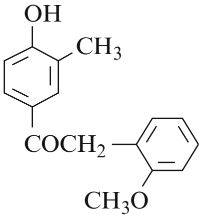
Synthesis

– Obtained by reaction of p-nitrophenylacetonitrile with 2,5-dimethoxyresorcinol (Hoesch reaction) [5474].

m.p. 174° [5474].

1-(2-Hydroxy-3-methylphenyl)-2-(2-methoxyphenyl)ethanone

[74384-36-4]



$C_{16}H_{16}O_3$ mol.wt. 256.30

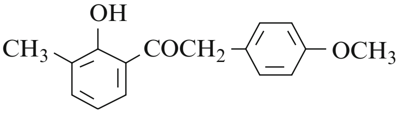
Synthesis

– Preparation by Fries rearrangement of o-cresyl o-methoxy-phenylacetate with aluminium chloride in nitromethane for 170 h at 20° (60%) [5480].

m.p. 167° [5480];
^{1}H NMR (Sadtler: standard n° 44610 M);
IR (Sadtler: standard n° 71638 K) [5480],
UV [5480], MS [5480].

1-(2-Hydroxy-3-methylphenyl)-2-(4-methoxyphenyl)ethanone

[74384-33-1] $C_{16}H_{16}O_3$ mol.wt. 256.30



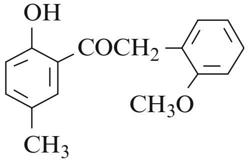
Synthesis

– Obtained (by-product) by Fries rearrangement of o-cresyl p-methoxyphenylacetate with aluminium chloride in nitromethane for 25 h at 20° (11%) [5480].

oil [5480]; IR [5480], UV [5480], MS [5480].

1-(2-Hydroxy-5-methylphenyl)-2-(2-methoxyphenyl)ethanone

[74384-38-6] $C_{16}H_{16}O_3$ mol.wt. 256.30



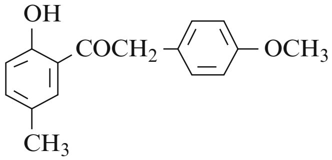
Synthesis

– Obtained by Fries rearrangement of p-cresyl o-methoxy-phenylacetate with aluminium chloride in nitromethane for 170 h at 20° (41%) [5480].

m.p. 95° [5480];
^{1}H NMR (Sadtler: standard n° 44606 M), IR (Sadtler: standard n°71634 K) [5480], UV [5480], MS [5480].

1-(2-Hydroxy-5-methylphenyl)-2-(4-methoxyphenyl)ethanone

[74384-34-2] $C_{16}H_{16}O_3$ mol.wt. 256.30



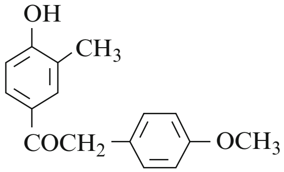
Synthesis

– Obtained by Fries rearrangement of p-cresyl p-methoxyphenylacetate with aluminium chloride in nitromethane for 25 h at 20° (22%) [5480].

m.p. 55° [5480];
IR [5480], UV [5480], MS [5480].

1-(4-Hydroxy-3-methylphenyl)-2-(4-methoxyphenyl)ethanone

[74384-31-9] $C_{16}H_{16}O_3$ mol.wt. 256.30



Synthesis

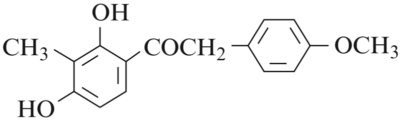
– Preparation by Fries rearrangement of o-cresyl p-methoxy-phenylacetate with aluminium chloride in nitromethane for 25 h at 20° (71%) [5480].

m.p. 160° [5480];

^{1}H NMR (Sadtler: standard n° 44612 M),
IR (Sadtler: standard n° 71640 K) [5480], UV [5480], MS [5480].

1-(2,4-Dihydroxy-3-methylphenyl)-2-(4-methoxyphenyl)ethanone

[93434-89-0] $C_{16}H_{16}O_4$ mol.wt. 272.30



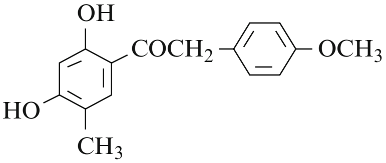
Syntheses

- Preparation by hydrogenation of 2,4-dihydroxy-3-formylphenyl 4-methoxybenzyl ketone (m.p. 114–115°) in acetic acid in the presence of 5% Pd/C in an atmosphere of hydrogen at r.t. and at atmospheric pressure (79%) [5354].
- Preparation by reaction of p-methoxyphenylacetonitrile with 2-methylresorcinol (48%) (Hoesch reaction) [5513].

Also refer to: [5514].
m.p. 175° [5513], 172–173° [5354].

1-(2,4-Dihydroxy-5-methylphenyl)-2-(4-methoxyphenyl)ethanone

[56308-10-2] $C_{16}H_{16}O_4$ mol.wt. 272.30



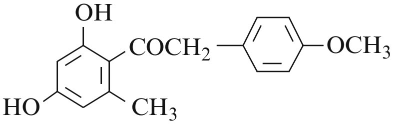
Syntheses

- Obtained by reaction of 4-methoxyphenylacetonitrile with 4-methylresorcinol (Hoesch reaction) (44%) [5515], (36%) [5488].
- Also refer to: [5281] (Hungarian paper).

m.p. 155° [5488], 139–140° [5515]. One of the reported melting points is obviously wrong.

1-(2,4-Dihydroxy-6-methylphenyl)-2-(4-methoxyphenyl)ethanone

[15485-71-9] $C_{16}H_{16}O_4$ mol.wt. 272.30



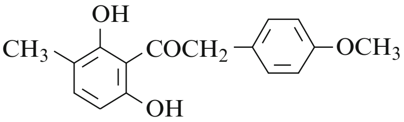
Synthesis

- Obtained by treatment of orcinol with p-methoxyphenylacetonitrile in ethyl ether in the presence of zinc chloride and hydrogen chloride (Hoesch reaction) [5264].

m.p. 109–110° [5264]; UV [5264].

1-(2,6-Dihydroxy-3-methylphenyl)-2-(4-methoxyphenyl)ethanone

[131196-70-8]



$C_{16}H_{16}O_4$ mol.wt. 272.30

Synthesis

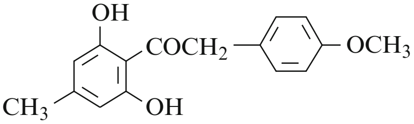
- Obtained from 2,6-dihydroxy-4′-methoxy-5-methoxycarbonyl-3-methyldeoxybenzoin

(m.p. 116°) which was simultaneously hydrolysed and decarboxylated by treatment with potassium hydroxide in refluxing dilute ethanol for 1.5 h (84%) [5488].

m.p. 164° [5488].

1-(2,6-Dihydroxy-4-methylphenyl)-2-(4-methoxyphenyl)ethanone

[128672-42-4]



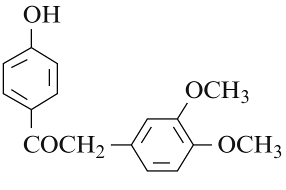
$C_{16}H_{16}O_4$ mol.wt. 272.30

Synthesis

- Refer to: [5516] (Chinese paper).

2-(3,4-Dimethoxyphenyl)-1-(4-hydroxyphenyl)ethanone

[183054-34-4]



$C_{16}H_{16}O_4$ mol.wt. 272.30

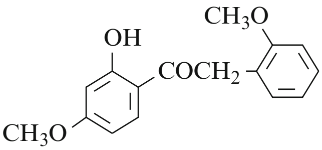
Synthesis

- Obtained by Friedel–Crafts acylation of isopropyl phenyl ether with homoveratroyl chloride in ethylene dichloride in the presence of aluminium chloride, first at 20°, then at 40° for 2–3 h and at r.t. overnight (31%) [5517].

m.p. 165–167° [5517];
^{1}H NMR [5517], ^{13}C NMR [5517], IR [5517], MS [5517].

1-(2-Hydroxy-4-methoxyphenyl)-2-(2-methoxyphenyl)ethanone

[18440-00-1]



$C_{16}H_{16}O_4$ mol.wt. 272.30

Syntheses

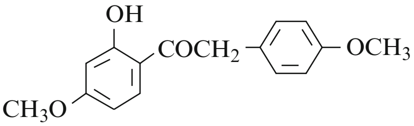
- Preparation by partial methylation of 2,4-dihydroxy-2′-methoxydeoxybenzoin,
  - with methyl iodide in the presence of potassium carbonate in boiling acetone during 1.5 h (quantitative yield) [5483];
  - with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 5 h (83%) [5518].

- Also obtained by Friedel–Crafts acylation of resorcinol dimethyl ether with o-methoxyphenylacetyl chloride in the presence of aluminium chloride, first in carbon disulfide, then on steam bath for 2.5 h after solvent elimination (40%) [5519].
- Also obtained by hydrolysis of 2,4-dimethoxybenzoyl-2-methoxyphenylacetonitrile (m.p. 114–115°) in acetic acid with concentrated hydrochloric acid on a steam bath for 15 h (43%) [5211].
- Also obtained by hydrolysis of ethyl 2,4-dimethoxybenzoyl-2-methoxyphenylacetate (m.p. 94–96°) in acetic acid with concentrated hydrochloric acid on a steam bath for 15 h (40%) [5211].
- Also obtained by degradation of 7,2′-dimethoxy-3-phenyl-4-hydroxycoumarin with refluxing 30% alcoholic hydrogen chloride for 1 h (36%) [5210].
- Also refer to: [5278,5338,5340,5448,5476].

m.p. 94° [5483], 93–95° [5519], 93–94° [5518], 92° [5210], 90–91° [5211]; b.p.$_{0.001}$ 180° [5211].

1-(2-Hydroxy-4-methoxyphenyl)-2-(4-methoxyphenyl)ethanone

[39604-64-3] $C_{16}H_{16}O_4$ mol.wt. 272.30

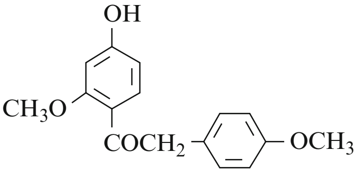


Syntheses

- Preparation by partial methylation of 2,4-di-hydroxyphenyl 4-methoxybenzyl ketone,
  - with methyl iodide in the presence of potassium carbonate in boiling acetone during 3 h (83%) [5250];
  - with dimethyl sulfate [5520], in the presence of potassium carbonate in boiling acetone [5385];
  - with diazomethane in ethyl ether [5484,5487].
- Also obtained by alkaline degradation of *formononetin methyl ether* (7,4′-dimethoxyisoflavone) [5487], (m.p. 156°) with boiling 10% sodium hydroxide for 1 h [5484].
- Also obtained by hydrolysis of 2,4-dimethoxybenzoyl-4-methoxyphenyl-acetonitrile (m.p. 105–106°) in acetic acid with concentrated hydrochloric acid on a steam bath for 15 h (38%) [5211].
- Also obtained by hydrolysis of ethyl 2,4-dimethoxybenzoyl-4-methoxyphenylacetate (m.p. 48–50°) in acetic acid with concentrated hydrochloric acid on a steam bath for 15 h (33%) [5211].
- Also obtained by reaction of p-methoxyphenylacetonitrile with resorcinol monomethyl ether (Hoesch reaction) [5486].
- Also refer to: [4935,5273,5278,5330,5337,5338,5340,5381,5420,5421,5447, 5448,5461,5462].

m.p. 104° [5250,5484], 102° [5487], 100–100°5 [5211], 92–93° [5486]; b.p.$_{0.001}$ 190° [5211]; ^{13}C NMR [5293,5450].

1-(4-Hydroxy-2-methoxyphenyl)-2-(4-methoxyphenyl)ethanone



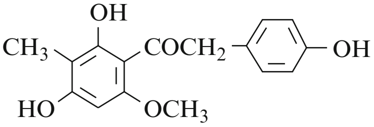
$C_{16}H_{16}O_4$ mol.wt. 272.30

Syntheses

- Obtained by heating 1-[4-(glucopyranosyloxy)-2-methoxyphenyl]-2-(4-methoxyphenyl)ethanone (SM) with concentrated sulfuric acid for 20 min. SM was obtained by methylation of synthetic *onospin* (m.p. 179°5) with excess methyl iodide in the presence of potassium carbonate in boiling methanol for 2 h. This same methylation can be realized by using diazomethane [5486].
- Also obtained by reaction of p-methoxyphenylacetonitrile with resorcinol monomethyl ether (Hoesch reaction) [5489].

m.p. 173–175° [5486].

1-(2,4-Dihydroxy-6-methoxy-3-methylphenyl)-2-(4-hydroxyphenyl)ethanone



$C_{16}H_{16}O_5$ mol.wt. 288.30

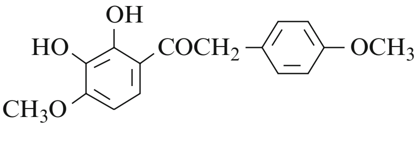
Syntheses

- Obtained by reaction of p-hydroxyphenylacetonitrile with 2,6-dihydroxy-4-methoxytoluene (m.p. 124°) (Hoesch reaction) (50%) [5495].
- Also refer to: [5488].

m.p. 207° [5495].

1-(2,3-Dihydroxy-4-methoxyphenyl)-2-(4-methoxyphenyl)ethanone

[38412-65-6]



$C_{16}H_{16}O_5$ mol.wt. 288.30

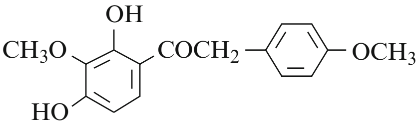
Synthesis

- Obtained by reaction of 4-methoxyphenylacetic acid with 3-methoxycatechol in chloroform in the presence of excess boron trifluoride, first at 0°, then at r.t. for 2 days (97%) [5499].

m.p. 137° [5499].

1-(2,4-Dihydroxy-3-methoxyphenyl)-2-(4-methoxyphenyl)ethanone

[61243-80-9]



$C_{16}H_{16}O_5$ mol.wt. 288.30

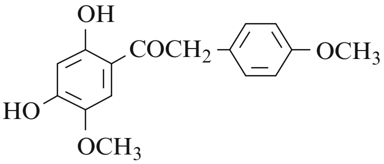
Synthesis

- Obtained by mild base hydrolysis of 8-O-methyl-retusin (7-hydroxy-8,4′-dimethoxyisoflavone) (SM) (m.p. 229–232°), itself isolated from heartwood of *Xanthocercis zambesiaca* (Bak.) (Leguminosae) [5522].

m.p. 140–142° [5522]; ^{1}H NMR [5522], IR [5522], UV [5522], MS [5522].

1-(2,4-Dihydroxy-5-methoxyphenyl)-2-(4-methoxyphenyl)ethanone

[5128-54-1]



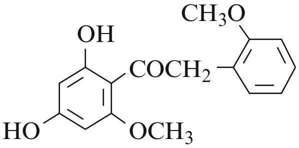
$C_{16}H_{16}O_5$ mol.wt. 288.30

Syntheses

- Obtained by alkaline degradation of *afromosin* (7-hydroxy-6,4′-dimethoxyisoflavone), (69%) [5501], (64%) [5523]. *Afromosin* (m.p. 228–229°), was isolated from *afromosia elata* Harms [5523]. *Afromosin* is the aglycone of *wistin* (m.p. 209–210°), itself isolated from the bark of *wistaria floribunda* DC [5501].
- Also obtained by reaction of 4-methoxyphenyl-acetonitrile with 4-methoxyresorcinol (Hoesch reaction) (20%) [5523].

m.p. 128–129° [5523], 127° [5501]; ^{13}C NMR [5293], IR [5523], UV [5501,5523].

1-(2,4-Dihydroxy-6-methoxyphenyl)-2-(2-methoxyphenyl)ethanone



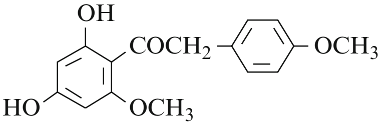
$C_{16}H_{16}O_5$ mol.wt. 288.30

Synthesis

- Refer to: [5295].

1-(2,4-Dihydroxy-6-methoxyphenyl)-2-(4-methoxyphenyl)ethanone

[13539-34-9] $C_{16}H_{16}O_5$ mol.wt. 288.30



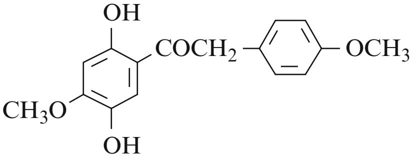
Syntheses

- Preparation by reaction of p-methoxyphenyl-acetonitrile with phloroglucinol monomethyl ether (Hoesch reaction) [5295,5524], (39%) [5525], (31%) [5495].
- Also obtained by alkaline degradation of *genistein 5,4′-dimethyl ether* (7-hydroxy-5,4′-dimethoxyisoflavone) (m.p. 290–293°) in boiling 30% potassium hydroxide for 15 min (93%) [5444].

crystals [5444]; m.p. 166–167° [5295], 129–130° [5495], 126–127° [5525]. One of the reported melting points is obviously wrong. UV [5525].

1-(2,5-Dihydroxy-4-methoxyphenyl)-2-(4-methoxyphenyl)ethanone

[79744-55-1] $C_{16}H_{16}O_5$ mol.wt. 288.30



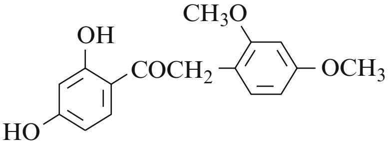
Syntheses

- Obtained by alkaline persulfate oxidation of 2-hydroxy-4-methoxyphenyl 4-methoxybenzyl ketone (Elbs reaction) (21%) [5520].
- Also obtained by partial methylation of 2,4,5-tri-hydroxyphenyl 4-methoxybenzyl ketone with excess diazomethane in ethyl ether at r.t. overnight (14%) [5291].
- Also obtained by reaction of 4-methoxyphenylacetonitrile with 2-methoxyhydroquinone so called methoxyquinol (24%) (Hoesch reaction) [5523].

m.p. 150° [5523], 148–149° [5291], 133–134° [5520].
One of the reported melting points is obviously wrong.
^{13}C NMR [5293], IR [5523], UV [5520,5526].

1-(2,4-Dihydroxyphenyl)-2-(2,4-dimethoxyphenyl)ethanone

[1855-30-7] $C_{16}H_{16}O_5$ mol.wt. 288.30



Syntheses

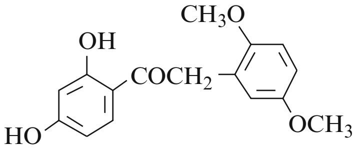
- Obtained by reaction of 2,4-dimethoxyphenyl-acetonitrile with resorcinol (Hoesch reaction) [5265], (55%) [5526], (37%) [5527,5531], (5%) [5529].
- Also refer to: [5530,5445].

m.p. 158–159° [5526], 155–156° [5529], 154° [5528], 152° [5527];
b.p.$_{0.02}$ 200–210° [5529]; IR [5528].

1-(2,4-Dihydroxyphenyl)-2-(2,5-dimethoxyphenyl)ethanone

[18086-25-4]

$C_{16}H_{16}O_5$ mol.wt. 288.30



Synthesis

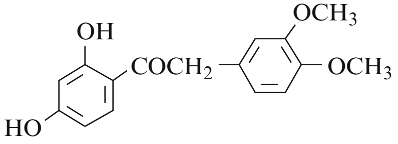
- Obtained by reaction of 2,5-dimethoxyphenylacetonitrile (m.p. 56–57°) with resorcinol (40%) (Hoesch reaction) [5531].

m.p. 144–145° [5531].

1-(2,4-Dihydroxyphenyl)-2-(3,4-dimethoxyphenyl)ethanone

[24126-98-5]

$C_{16}H_{16}O_5$ mol.wt. 288.30



Syntheses

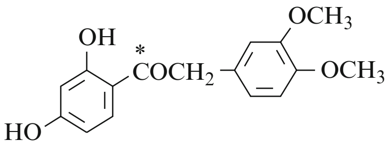
- Preparation by Friedel–Crafts acylation of resorcinol with 3,4-dimethoxyphenylacetyl chloride in nitro- benzene in the presence of aluminium chloride for 24 h at r.t. (56%) [5250].
- Preparation by reaction of 3,4-dimethoxyphenyl-acetonitrile with resorcinol (Hoesch reaction) [5332], (60%) [5532], (49%) [5527].
- Obtained by heating *Cladrin* (7-hydroxy-3′,4′-dimethoxyisoflavone) (m.p. 257–258°) with 10% aqueous barium hydroxide at reflux for 2 h (73%). *Cladrin* was isolated from *Cladrastis lutea* (Mich. f.) K. Koch (Leguminosae) [5533].
- Also refer to: [5271,5537,5280].

m.p. 182–183° [5533], 180° [5527], 177°5 [5250], 177–178° [5532], 176°5 [5332].

1-(2,4-Dihydroxyphenyl)-2-(3,4-dimethoxyphenyl)ethanone-*1*-^{14}C

[142050-41-7]

$C_{16}H_{16}O_5$ mol.wt. 290.29



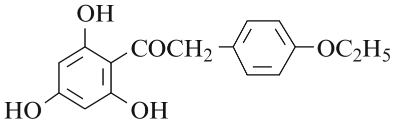
Synthesis

- Obtained by reaction of 3,4-dimethoxybenzyl [^{14}C] nitrile with resorcinol (Hoesch reaction) (38%) [5492].

m.p. 183–184° [5492]; TLC [5492].

2-(4-Ethoxyphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

$C_{16}H_{16}O_5$ mol.wt. 288.30



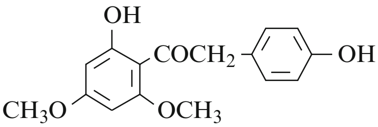
Synthesis

- Preparation by condensation of p-ethoxyphenyl-acetonitrile with phloroglucinol (Hoesch reaction) [5535].

m.p. 208–210° [5535].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-(4-hydroxyphenyl)ethanone

[69127-79-3]



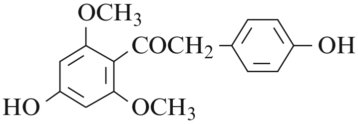
$C_{16}H_{16}O_5$ mol.wt. 288.30

Syntheses

- Obtained by alkaline degradation of *genistein 5,7-di-methyl ether* (5,7-dimethoxy-4′-hydroxyisoflavone) (m.p. 266°) with 40% potassium hydroxide in a water bath for 15 min (41%) [5536].
- Also obtained by diazotization of 4-aminobenzyl 2-hydroxy-4,6-dimethoxyphenyl ketone hydrochloride, followed by hydrolysis of the diazonium salt obtained (33%) [5382].
- Also obtained by reaction of p-hydroxyphenylacetonitrile with phloroglucinol dimethyl ether (Hoesch reaction) (25%) [5537], (19%) [5536].
- Also refer to: [5295,5463].

m.p. 112° [5537,5539], 110° [5382].

1-(4-Hydroxy-2,6-dimethoxyphenyl)-2-(4-hydroxyphenyl)ethanone



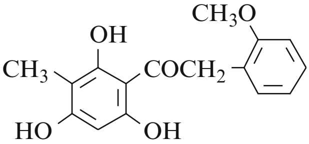
$C_{16}H_{16}O_5$ mol.wt. 288.30

Synthesis

- Obtained by reaction of p-hydroxyphenylacetonitrile with phloroglucinol dimethyl ether (Hoesch reaction) [5537,5539].

m.p. 182° [5536], 181° [5537].

2-(2-Methoxyphenyl)-1-(2,4,6-trihydroxy-3-methylphenyl)ethanone



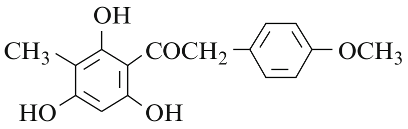
$C_{16}H_{16}O_5$ mol.wt. 288.30

Syntheses

- Preparation by reaction of o-methoxyphenylacetonitrile with 2-methylphloroglucinol (m.p. 215°) (Hoesch reaction), (45%) [5495], (35%) [5497].
- Also refer to: [5355].

m.p. 206° [5495], 198–200° [5497]; sublimation at 160°/0.01 mm [5495].

2-(4-Methoxyphenyl)-1-(2,4,6-trihydroxy-3-methylphenyl)ethanone



$C_{16}H_{16}O_5$ mol.wt. 288.30

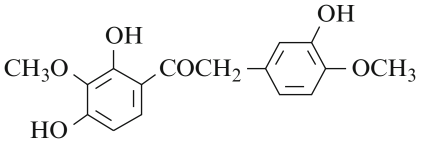
Syntheses

- Obtained by reaction of p-methoxyphenyl-acetonitrile with 2-methylphloroglucinol (m.p. 215°) [5495] (Hoesch reaction), (54%) [5538], (26%) [5495].
- Also refer to: [5296,5355,5359].

m.p. 228° [5495], 220–221° [5538]; sublimation at 160°/ 0.01 mm [5495].

1-(2,4-Dihydroxy-3-methoxyphenyl)-2-(3-hydroxy-4-methoxyphenyl)ethanone

[61243-85-4]



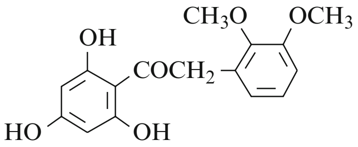
$C_{16}H_{16}O_6$ mol.wt. 304.

Syntheses

- Obtained by mild base hydrolysis of 7,3′-di-hydroxy-8,4′-dimethoxy-isoflavone (m.p. 212–213°), (71%) [5522].
- Also refer to: [5539].

m.p. 127–129° [5522]; ^{1}H NMR [5522], IR [5522], UV [5522], MS [5522].

2-(2,3-Dimethoxyphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone



$C_{16}H_{16}O_6$ mol.wt. 304.30

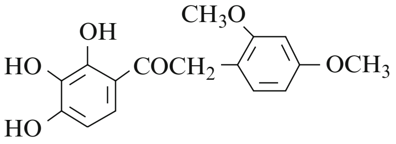
Syntheses

- Obtained by reaction of 2,3-dimethoxyphenylacetonitrile with phloroglucinol (Hoesch reaction) (44%) [5483].
- Also refer to: [5476].

m.p. 193° [5483].

2-(2,4-Dimethoxyphenyl)-1-(2,3,4-trihydroxyphenyl)ethanone

[14756-83-3]



$C_{16}H_{16}O_6$ mol.wt. 304.30

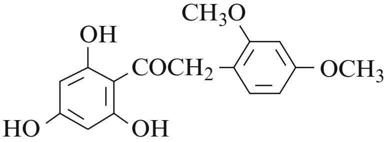
Syntheses

- Obtained by reaction of phenylacetic acid with pyrogallol in the presence of zinc chloride at 130° for 2 h (Nencki reaction) (39%) [5540].
- Also obtained (poor yield) by reaction of 2,4-di-methoxyphenylacetonitrile with pyrogallol (Hoesch reaction) (4%) [5540].

m.p. 134–135° [5540].

2-(2,4-Dimethoxyphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[65568-08-3] $C_{16}H_{16}O_6$ mol.wt. 304.30



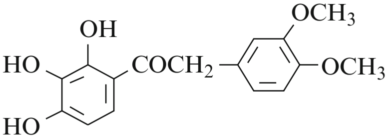
Syntheses

- Obtained by condensation of 2,4-dimethoxyphenyl-acetonitrile (m.p. 76°) with phloroglucinol (Hoesch reaction) [5276,5381,5541,5542].
- Also refer to: [5277,5295,5476,5534,5543].

m.p. 178° [5542], 175° [5541]; paper chromatography [5397].

2-(3,4-Dimethoxyphenyl)-1-(2,3,4-trihydroxyphenyl)ethanone

[93435-58-6] $C_{16}H_{16}O_6$ mol.wt. 304.30



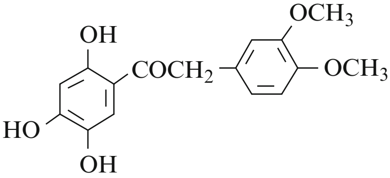
Synthesis

- Obtained by condensation of 3,4-dimethoxyphenyl-acetonitrile (m.p. 45–47°) [5544] with pyrogallol (Hoesch reaction) [5545], (23%) [5470].

m.p. 174° [5470].

2-(3,4-Dimethoxyphenyl)-1-(2,4,5-trihydroxyphenyl)ethanone

[66116-74-3] $C_{16}H_{16}O_6$ mol.wt. 304.30



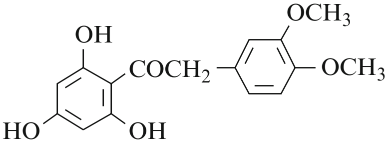
Syntheses

- Preparation by reaction of 3,4-dimethoxyphenyl-acetonitrile with hydroxyhydroquinone (Hoesch reaction) (81%) [5471].
- Also refer to: [5351].

m.p. 193–194° [5471]; TLC [5471];
^1H NMR [5471], ^{13}C NMR [5293].

2-(3,4-Dimethoxyphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[53084-06-3] $C_{16}H_{16}O_6$ mol.wt. 304.30



Syntheses

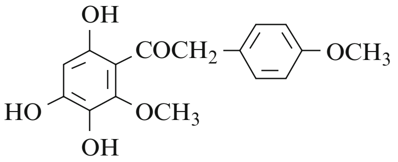
- Preparation by reaction of 3,4-dimethoxyphenyl-acetonitrile with phloroglucinol (Hoesch reaction),
  - in the presence of zinc chloride [5295,5549], (47%) [5250], (42%) [5294], (38%) [5255];
  - in the presence of boron trifluoride etherate (59%) [5255].
- Also refer to: [5263].

N.B.: The monohydrate of this ketone was at first obtained [5250]. The water of crystallisation is lost on heating the crystals at 90°.

m.p. 208–210° [5546], 184–186° [5295], 182–184° [5255], 181° [5250], 180–181° [5294]. One of the reported melting points is obviously wrong.
^{13}C NMR [5293], IR [5294], UV [5294]; TLC [5294].

2-(4-Methoxyphenyl)-1-(3,4,6-trihydroxy-2-methoxyphenyl)ethanone

[14701-83-8]



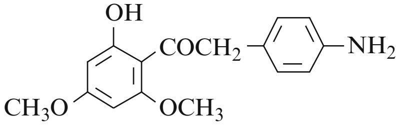
$C_{16}H_{16}O_6$ mol.wt. 304.30

Synthesis

- Obtained by persulfate oxidation of 2,4-dihydroxy-6-methoxyphenyl 4-methoxybenzyl ketone (Elbs reaction) [5525].

m.p. 144° [5525]; UV [5525].

2-(4-Aminophenyl)-1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone



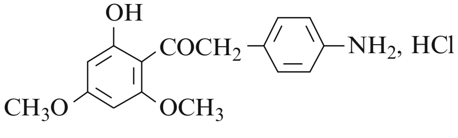
$C_{16}H_{17}NO_4$ mol.wt. 287.32

Synthesis

- Preparation by hydrogenation of 2-hydroxy-4,6-dimethoxyphenyl 4- itrobenzyl ketone in ethanol in the presence of Raney nickel as catalyst with hydrogen for 8 h [5382].

m.p. 103–104° [5382].

2-(4-Aminophenyl)-1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone *(Hydrochloride)*



$C_{16}H_{17}NO_4$, HCl mol.wt. 323.78

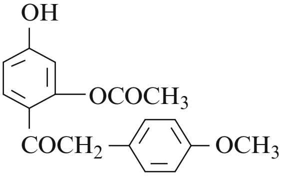
Synthesis

- Obtained by treatment of the above base with hot dilute hydrochloric acid [5382].

m.p. 198–200° [5382].

1-[2-(Acetyloxy)-4-hydroxyphenyl]-2-(4-methoxyphenyl)ethanone

[145747-28-0]



$C_{17}H_{16}O_5$ mol.wt. 300.31

Syntheses

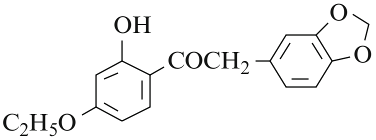
- Obtained by selective deacetylation catalyzed by porcine pancreas lipase in THF at 42–45° of,
  - 2,4-diacetoxyphenyl 4-methoxybenzyl ketone during 48 h (73%) [5265,5358,5359].

- 1-acetoxy-1-(2,4-diacetoxyphenyl)-2-(4-methoxyphenyl)-ethene during 72 h (18%) [5265].

semi solid [5265]; TLC [5265];
^{1}H NMR [5265], IR [5265], UV [5265], MS [5265].

2-(1,3-Benzodioxol-5-yl)-1-(4-ethoxy-2-hydroxyphenyl)ethanone

(Pseudo-baptigenetin monoethyl ether)



$C_{17}H_{16}O_5$ mol.wt. 300.31

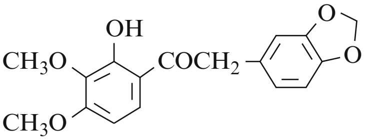
Syntheses

- Obtained by partial ethylation of pseudo-baptigenetin in ethanol with diazoethane in ethyl ether for 1.25 h [5468].
- Also obtained by alkaline degradation of *pseudo-baptigenin monoethyl ether* (m.p. 172°) with potassium hydroxide in boiling dilute ethanol [5468].

m.p. 129° [5468].

2-(1,3-Benzodioxol-5-yl)-1-(2-hydroxy-3,4-dimethoxyphenyl)ethanone

[84018-73-5]



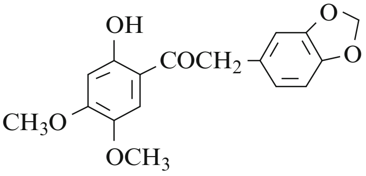
$C_{17}H_{16}O_6$ mol.wt. 316.31

Synthesis

- Obtained by reaction of 3,4-(methylenedioxy)phenyl-acetonitrile with pyrogallol (Hoesch reaction) and subsequent partial methylation of the 2,3,4-trihydroxy-phenyl 3,4-methylenedioxybenzyl ketone so obtained with dimethyl sulfate [5374] according to the method [5472].

2-(1,3-Benzodioxol-5-yl)-1-(2-hydroxy-4,5-dimethoxyphenyl)ethanone

[2746-88-5]



$C_{17}H_{16}O_6$ mol.wt. 316.31

Syntheses

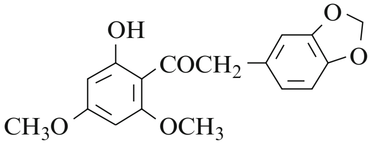
- Obtained by partial methylation of 3,4-(methylene-dioxy)benzyl 2,4,5-trihydroxyphenyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 6 h (40%) [5472] or according to [5273], (90%) [5471].
- Also obtained by partial demethylation of 3,4-(methylenedioxy)benzyl 2,4,5-trimethoxyphenyl ketone (m.p. 153–154°) with aluminium chloride in refluxing acetonitrile for 45 min (43%) [5379].

- Also obtained (trace) by reaction of 3,4-(methylenedioxy)phenylacetyl chloride with 1,2,4-tri-methoxybenzene in the presence of aluminium chloride in ethyl ether at 0° overnight (<1%) [5379].
- Also refer to: [5381].

m.p. 153–154° [5472], 119° [5471], 118–119° [5379]. One of the reported melting points is obviously wrong.
IR [5379,5475], UV [5379,5475], MS [5379].

2-(1,3-Benzodioxol-5-yl)-1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone

[22044-73-1] $C_{17}H_{16}O_6$ mol.wt. 316.31



Syntheses

- Preparation by partial methylation of 3,4-(methylene-dioxy)benzyl 2,4,6-trihydroxyphenyl ketone with dimethyl sulfate in the presence of potassium carbonate in boiling acetone [5295], for 14 h (73%) [5473].
- Also refer to: [5281,5380].

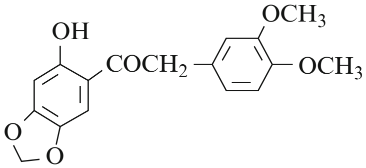
Isolation from natural sources

- Obtained (major product) by alkaline hydrolysis of *Derrustone* (5,7-dimethoxy-3′,4′-methylene-dioxyisoflavone) (m.p. 153–154°) (SM) with 25% aqueous potassium hydroxide in refluxing methanol for 2 h (69%). SM was isolated from the root material of *Derris robusta* (Roxb.) Benth [5547].

m.p. 102–103° [5547], 98–99° [5295], 97° [5473]; IR [5547].

2-(3,4-Dimethoxyphenyl)-1-(6-hydroxy-1,3-benzodioxol-5-yl)ethanone

[61243-78-5] $C_{17}H_{16}O_6$ mol.wt. 316.31

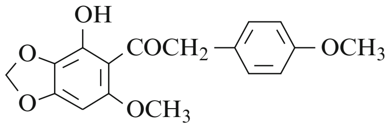


Synthesis

- Obtained by mild base hydrolysis of 3′,4′-dimethoxy-6,7-methylenedioxyisoflavone [5522].

m.p. 148–151° [5522];
^{1}H NMR [5522], IR [5522], UV [5522], MS [5522];
TLC [5522].

1-(4-Hydroxy-6-methoxy-1,3-benzodioxol-5-yl)-2-(4-methoxyphenyl)ethanone



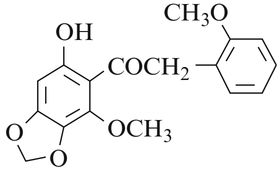
$C_{17}H_{16}O_6$ mol.wt. 316.31

Synthesis

- Obtained by alkali degradation of 5,4′-dimethoxy-7,8-methylenedioxy-2-methylisoflavone (m.p. 214–215°) with potassium hydroxide in refluxing dilute ethanol for 2 h under a stream of nitrogen gas (88%) [5507].

m.p. 133–134° [5507]; IR [5507], UV [5507].

1-(6-Hydroxy-4-methoxy-1,3-benzodioxol-5-yl)-2-(2-methoxyphenyl)ethanone



$C_{17}H_{16}O_6$ mol.wt. 316.31

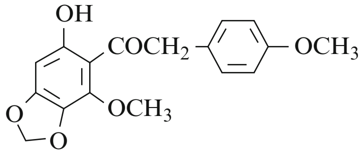
Synthesis

- Obtained by alkaline degradation of *tlatlancuayin* (5,2′-di-methoxy-6,7-methylenedioxyisoflavone) (SM) with refluxing aqueous sodium hydroxide for 1.5 h (quantitative yield). SM was isolated from *Iresine celosioides* L. (Amarantaceae) [5213].

m.p. 115–116° [5213]; IR [5213], UV [5213].

1-(6-Hydroxy-4-methoxy-1,3-benzodioxol-5-yl)-2-(4-methoxyphenyl)ethanone

[3207-42-9]



$C_{17}H_{16}O_6$ mol.wt. 316.31

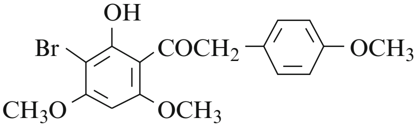
Syntheses

- Obtained by methylenation of 2,4,5-trihydroxy-6-methoxyphenyl 4-methoxybenzyl ketone with methylene iodide in the presence of potassium carbonate in refluxing acetone for 40 h [5525].
- Also obtained by alkaline degradation of *irisolone methyl ether* (4′,5-dimethoxy-6,7-methylenedioxy-isoflavone) (m.p. 184–185°) with refluxing aqueous sodium hydroxide for 1.5 h [5510].
- Also obtained by reaction of 4-methoxyphenylacetonitrile with 3-methoxy-4,5-methylenedioxy-phenol (Hoesch reaction) (12%) [5507].
- Also obtained in two steps: First, methylation of 6-benzyloxy-2-hydroxy-3,4-methylenedioxy-phenyl 4-methoxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 27 h. The obtained methyl ether was debenzylated with hydrogen in ethanol in the presence of 10% Pd/C for 5 h at r.t. (31%) [5507].

m.p. 114–115° [5507], 113–114° [5510,5525];
IR [5507], UV [5507,5510,5525].

1-(3-Bromo-2-hydroxy-4,6-dimethoxyphenyl)-2-(4-methoxyphenyl)ethanone

[28750-74-5] $C_{17}H_{17}BrO_5$ mol.wt. 381.22



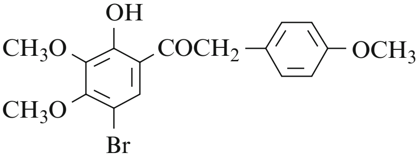
Synthesis

– Preparation by bromination of 2-hydroxy-4,6,4′-trimethoxydeoxybenzoin with bromine in chloroform under UV light at r.t. overnight (52%) [5294].

m.p. 158–159° [5294]; TLC [5294]; ^{1}H NMR [5294], IR [5294], UV [5294].

1-(5-Bromo-2-hydroxy-3,4-dimethoxyphenyl)-2-(4-methoxyphenyl)ethanone

[24852-43-5] $C_{17}H_{17}BrO_5$ mol.wt. 381.22



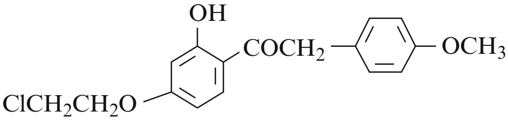
Synthesis

– Obtained by bromination of 2-hydroxy-3,4-di-methoxyphenyl 4-methoxybenzyl ketone with bromine in the presence of sodium acetate in chloroform for 5 h (70%) [5370].

m.p. 81–82° [5370]; ^{1}H NMR [5370].

1-[4-(2-Chloroethoxy)-2-hydroxyphenyl]-2-(4-methoxyphenyl)ethanone

[247931-29-9] $C_{17}H_{17}ClO_4$ mol.wt. 320.77



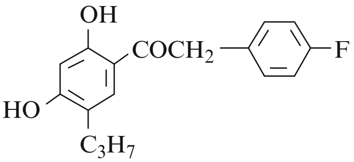
Synthesis

– Obtained by partial alkylation of 1-(2,4-dihydroxyphenyl)-2-(4-methoxyphenyl)ethanone with 1-bromo-2-chloroethane in the presence of potassium carbonate in refluxing acetone for 24 h (70%) [5420].

m.p. 87–89° [5420]; ^{1}H NMR [5420].

1-(2,4-Dihydroxy-5-propylphenyl)-2-(4-fluorophenyl)ethanone

[96644-01-8] $C_{17}H_{17}FO_3$ mol.wt. 288.32



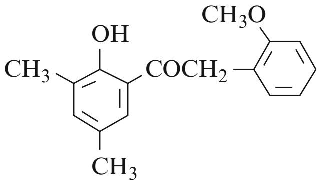
Synthesis

– Obtained by reaction of p-fluorophenylacetonitrile with 4-propylresorcinol in the presence of boron trifluoride etherate under hydrogen chloride at r.t. for 8–10 h, then at r.t. overnight (63%) [5367].

m.p. 100–101° [5367]; ^{1}H NMR [5367].

1-(2-Hydroxy-3,5-dimethylphenyl)-2-(2-methoxyphenyl)ethanone

[74384-39-7] $C_{17}H_{18}O_3$ mol.wt. 270.33



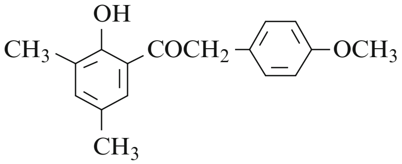
Synthesis

– Obtained by Fries rearrangement of 2,4-dimethylphenyl 2-methoxyphenylacetate with aluminium chloride in nitromethane at 20° for 170 h (39%) [5480].

m.p. 85° [5480];
^{1}H NMR (Sadtler: standard n° 44603 M), [5480],
IR (Sadtler: standard n° 71631 K), [5480], UV [5480],
MS [5480].

1-(2-Hydroxy-3,5-dimethylphenyl)-2-(4-methoxyphenyl)ethanone

[74384-35-3] $C_{17}H_{18}O_3$ mol.wt. 270.33



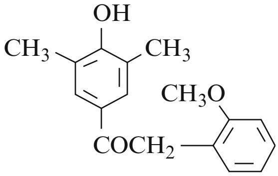
Synthesis

– Preparation by Fries rearrangement of 2,4-di-methylphenyl 4-methoxyphenylacetate with aluminium chloride in nitromethane at 20° for 170 h (59%) [5480].

m.p. 23° [5480]; IR [5480], UV [5480], MS [5480].

1-(4-Hydroxy-3,5-dimethylphenyl)-2-(2-methoxyphenyl)ethanone

[74384-37-5] $C_{17}H_{18}O_3$ mol.wt. 270.33



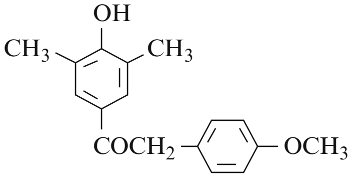
Synthesis

– Obtained by Fries rearrangement of 2,6-dimethylphenyl 2-methoxyphenylacetate in the presence of ferric chloride or aluminium chloride in nitromethane at 20° for 50 h (29% and 6% yields, respectively) [5480].

m.p. 160° [5480];
^{1}H NMR (Sadtler: standard n° 44607 M), [5480],
IR (Sadtler: standard n° 71635 K), [5480], UV [5480], MS [5480].

1-(4-Hydroxy-3,5-dimethylphenyl)-2-(4-methoxyphenyl)ethanone

[74384-32-0] $C_{17}H_{18}O_3$ mol.wt. 270.33



Synthesis

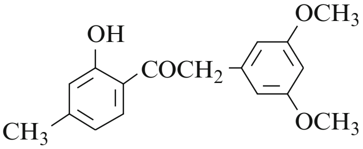
– Preparation by Fries rearrangement of 2,6-dimethyl-phenyl 4-methoxyphenylacetate with aluminium chloride in nitromethane at 20° for 50 h (76%) [5480].

m.p. 177° [5480];

^{1}H NMR (Sadtler: standard n° 44604 M), [5480],
IR (Sadtler: standard n° 71632 K), [5480], UV [5480], MS [5480].

2-(3,5-Dimethoxyphenyl)-1-(2-hydroxy-4-methylphenyl)ethanone

[111191-98-1] $C_{17}H_{18}O_4$ mol.wt. 286.33



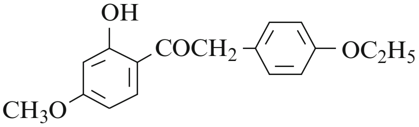
Synthesis

- Obtained (poor yield) by Fries rearrangement of m-cresyl 3,5-dimethoxyphenylacetate (b.p.0.05 95°) in the presence of aluminium chloride, first in carbon disulfide, then at 130–145° for 2 h after solvent elimination (15%) [5482].

brown gum [5482].
N.B.: Methyl ether: m.p. 199–200° [5482], ^{1}H NMR [5482], IR [5482], MS [5482].

2-(4-Ethoxyphenyl)-1-(2-hydroxy-4-methoxyphenyl)ethanone

[89019-87-4] $C_{17}H_{18}O_4$ mol.wt. 286.33



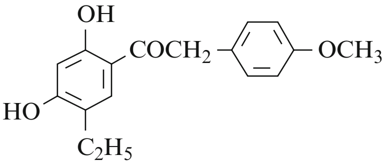
Synthesis

- Obtained (by-product) by reaction of p-hydroxyphenylacetic acid with m-methoxy-phenol in ethylene dichloride in the presence of boron trifluoride at 80° for 2 h under an argon atmosphere (2%) [5439].

m.p. 95–97° [5439]; ^{1}H NMR [5439], IR [5439], UV [5439], MS [5439].

1-(5-Ethyl-2,4-dihydroxyphenyl)-2-(4-methoxyphenyl)ethanone

[96644-03-0] $C_{17}H_{18}O_4$ mol.wt. 286.33



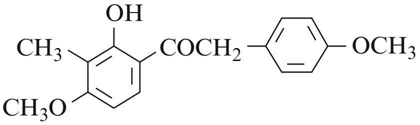
Synthesis

- Preparation by condensation of p-methoxyphenyl-acetonitrile with 4-ethylresorcinol in the presence of boron trifluoride etherate under hydrogen chloride atmosphere (8–10 h) at r.t. overnight (86%) [5367].

m.p. 95–96° [5367]; ^{1}H NMR [5367].

1-(2-Hydroxy-4-methoxy-3-methylphenyl)-2-(4-methoxyphenyl)ethanone

[39604-65-4]



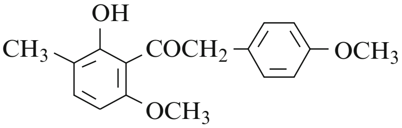
$C_{17}H_{18}O_4$ mol.wt. 286.33

Syntheses

- Preparation by partial methylation of,
  - 2,4-dihydroxy-3-methylphenyl 4-methoxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 6 h [5354], (86%) [5513];
  - 2,4-dihydroxyphenyl 4-methoxybenzyl ketone with methyl iodide in methanolic potash, first in a bath of ice-salt mixture, then at r.t. overnight and at reflux for 7 h (39%) [5354].
- Also refer to: [5381].

m.p. 121–122° [5513], 116–117° [5354].

1-(2-Hydroxy-6-methoxy-3-methylphenyl)-2-(4-methoxyphenyl)ethanone



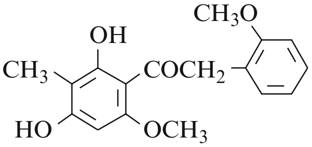
$C_{17}H_{18}O_4$ mol.wt. 286.33

Synthesis

- Obtained by partial methylation of 2,6-dihydroxy-3-methylphenyl 4-methoxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in boiling acetone for 1 h (71%) [5488].

oil [5488].

1-(2,4-Dihydroxy-6-methoxy-3-methylphenyl)-2-(2-methoxyphenyl)ethanone



$C_{17}H_{18}O_5$ mol.wt. 302.33

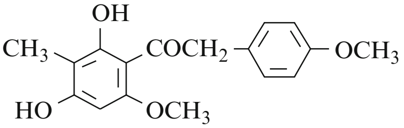
Synthesis

- Obtained by reaction of o-methoxyphenylacetonitrile with 2,6-dihydroxy-4-methoxyyoluene (m.p. 124°) (Hoesch reaction) (36%) [5287], (31%) [5495].

m.p. 195° [5495], 194° [5287].

1-(2,4-Dihydroxy-6-methoxy-3-methylphenyl)-2-(4-methoxyphenyl)ethanone

[56308-09-9]



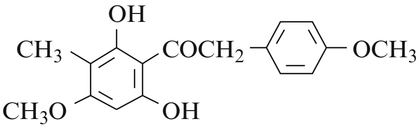
$C_{17}H_{18}O_5$ mol.wt. 302.33

Syntheses

- Obtained by reaction of p-methoxyphenyl-acetonitrile with 2,6-dihydroxy-4-methoxytoluene (m.p. 124°) (Hoesch reaction) [5494], (44%) [5548], (33%) [5495].
- Also refer to: [5281].

m.p. 162–164° [5494], 162° [5495], 125–127° [5548]. One of the reported melting points is obviously wrong.

1-(2,6-Dihydroxy-4-methoxy-3-methylphenyl)-2-(4-methoxyphenyl)ethanone



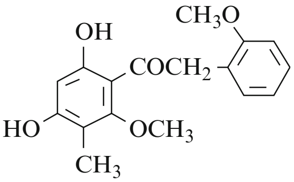
$C_{17}H_{18}O_5$ mol.wt. 302.33

Syntheses

- Obtained by alkaline hydrolysis of 5-hydroxy-7,4′-dimethoxy-8-methylisoflavone (m.p. 164–166°) with potassium hydroxide in refluxing ethanol for 30 min (31%) [5353].
- Also obtained by reaction of p-methoxyphenylacetonitrile with 2,4-dihydroxy-6-methoxytoluene (m.p. 119°) (Hoesch reaction) (41%) [5495].
- Also obtained by alkaline hydrolysis of 5-hydroxy-7,4′-dimethoxy-2,6-dimethylisoflavone (m.p. 198–200°) with 8% alcoholic potassium hydroxide at reflux for 30 min [5353].

m.p. 196–197° [5353], 192° [5495].

1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)-2-(2-methoxyphenyl)ethanone



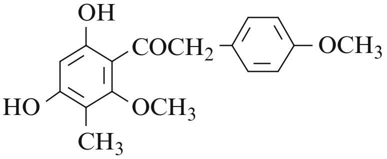
$C_{17}H_{18}O_5$ mol.wt. 302.33

Synthesis

- Obtained by catalytic debenzylation of 4,6-bis(benzyloxy)-2-methoxy-3-methylphenyl 2-methoxybenzyl ketone (m.p. 107°) in acetic acid in the presence of Pd/C for 20 min (quantitative yield) [5549].

m.p. 118° [5549].

1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)-2-(4-methoxyphenyl)ethanone



$C_{17}H_{18}O_5$ mol.wt. 302.33

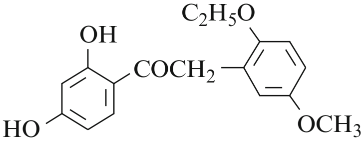
Synthesis

– Obtained by hydrogenolysis of 4,6-bis(benzyloxy)-2-methoxy-3-methylphenyl 4-methoxybenzyl ketone (m.p. 106°) in the presence of Pd/C in acetic acid for 10 min (quantitative yield) [5549].

m.p. 176° [5549].

1-(2,4-Dihydroxyphenyl)-2-(2-ethoxy-5-methoxyphenyl)ethanone

[18086-36-7]



$C_{17}H_{18}O_5$ mol.wt. 302.33

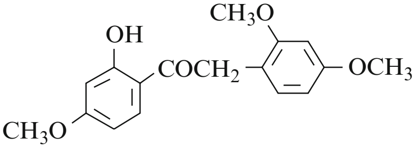
Synthesis

– Obtained by reaction of 2-ethoxy-5-methoxyphenyl-acetonitrile (m.p. 46–48°) with resorcinol (34%) (Hoesch reaction) [5531].

m.p. 114–115° [5531].

2-(2,4-Dimethoxyphenyl)-1-(2-hydroxy-4-methoxyphenyl)ethanone

[70779-11-2]



$C_{17}H_{18}O_5$ mol.wt. 302.33

Syntheses

– Preparation by partial methylation of 2,4-di-hydroxyphenyl 2,4-dimethoxybenzyl ketone [5265],

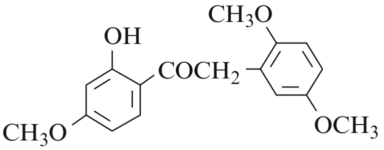
- with methyl iodide in the presence of potassium carbonate in boiling acetone for 70 min (95%) [5527];
- with diazomethane in ethyl ether for 30 min (quantitative yield) [5529].

– Also refer to: [5277,5534].

m.p. 116° [5527], 114–115° [5529]; b.p.$_{0.05}$ 170–180° [5529]; ^{1}H NMR [5341], UV [5550].

2-(2,5-Dimethoxyphenyl)-1-(2-hydroxy-4-methoxyphenyl)ethanone

[18086-26-5]



$C_{17}H_{18}O_5$ mol.wt. 302.33

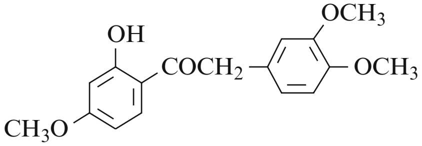
Synthesis

– Preparation by partial methylation of 2,4-dihydroxy-phenyl 2,5-dimethoxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 6 h (92%) [5531].

m.p. 113–114° [5531]; ^{1}H NMR [5341].

2-(3,4-Dimethoxyphenyl)-1-(2-hydroxy-4-methoxyphenyl)ethanone

[53084-05-2] $C_{17}H_{18}O_5$ mol.wt. 302.33



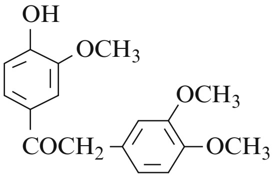
Syntheses

- Preparation by partial methylation of 2,4-di-hydroxyphenyl 3,4-dimethoxybenzyl ketone,
  - with methyl iodide in the presence of potassium carbonate in boiling acetone for 70 min (86–91%) [5527] or for 3 h (81%) [5250];
  - with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 6 h (69%) [5532] or in boiling 10% alcoholic sodium hydroxide for 1 h (31%) [5332].
- Also obtained by reaction of 3,4-dimethoxyphenylacetyl chloride with 1,3-dimethoxybenzene in the presence of aluminium chloride in boiling benzene for 1 h (52%) [5332].
- Also refer to: [4896,5278,5281,5338].

m.p. 119° [5250,5332], 118° [5527], 116–117° [5532].

2-(3,4-Dimethoxyphenyl)-1-(4-hydroxy-3-methoxyphenyl)ethanone

[114847-19-7] $C_{17}H_{18}O_5$ mol.wt. 302.33



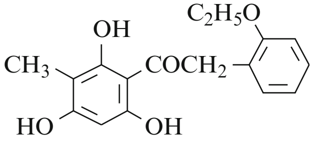
Syntheses

- Preparation by reaction of 3,4-dimethoxyphenylacetic acid with guaiacol in the presence of zinc chloride and phosphorous oxychloride for 24 h at r.t. (40%) [5209].
- Also refer to: [5551].

m.p. 142–144° [5209].

2-(2-Ethoxyphenyl)-1-(2,4,6-trihydroxy-3-methylphenyl)ethanone

$C_{17}H_{18}O_5$ mol.wt. 302.33



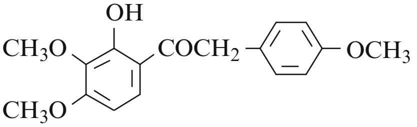
Synthesis

- Obtained by reaction of o-ethoxyphenylacetonitrile with 2-methylphloroglucinol (Hoesch reaction) (11%) [5483].

m.p. 174° [5483].

1-(2-Hydroxy-3,4-dimethoxyphenyl)-2-(4-methoxyphenyl)ethanone

[3606-32-4] $C_{17}H_{18}O_5$ mol.wt. 302.33



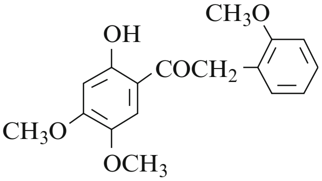
Syntheses

– Obtained by alkaline hydrolysis of *di-O-meth*ylretusin (7,8,4′-trimethoxyisoflavone) (m.p. 151°) (SM) with 10% aqueous potassium hydroxide in refluxing ethanol for 1 h (67%). SM was obtained by partial methylation of *retusin* (m.p. 249°) (7,8-dihydroxy-4′-methoxyisoflavone), itself isolated from *Dalbergia retusa* heartwood (cocobolo) (Leguminosae) [5499].
– Also obtained by Friedel–Crafts reaction of 4-methoxyphenylacetyl chloride with pyrogallol trimethyl ether in the presence of aluminium chloride [5552].
– Also refer to: [5503,5296,5370].

m.p. 122–123° [5499], 121–122° [5552]; ^{13}C NMR [5293].

1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-(2-methoxyphenyl)ethanone

[24195-30-0] $C_{17}H_{18}O_5$ mol.wt. 302.33



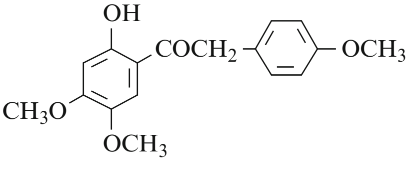
Syntheses

– Obtained by reaction of o-methoxyphenylacetonitrile with 3,4-dimethoxyphenol (Hoesch reaction) [5553], (35%) [5379].
– Also refer to: [5554,5343].

m.p. 108–109° [5379];
IR [5379], UV [5379], MS [5379].

1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-(4-methoxyphenyl)ethanone

[5128-49-4] $C_{17}H_{18}O_5$ mol.wt. 302.33



Syntheses

– Preparation by partial methylation of,

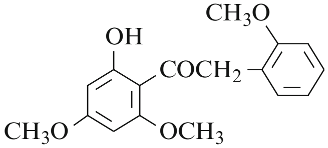
- 2,4-dihydroxy-5-methoxyphenyl 4-methoxybenzyl ketone with methyl iodide in the presence of potassium carbonate in boiling acetone [5501];
- 2,5-dihydroxy-4-methoxyphenyl 4-methoxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 4 h (80%) [5520];
- 2,4,5-trihydroxyphenyl 4-methoxybenzyl ketone with excess diazomethane in ethyl ether at r.t. overnight (45%) [5291] or with methyl iodide in the presence of potassium carbonate in refluxing acetone for 1.5 h [5501] or according to [5273], (86%) [5471].

- Also obtained by Friedel–Crafts acylation of 1,2,4-trimethoxybenzene with p-methoxyphenylacetyl chloride in the presence of aluminium chloride in ethyl ether [5520].
- Also obtained by reaction of p-methoxyphenylacetonitrile with 3,4-dimethoxyphenol (Hoesch reaction) (25%) [5523].
- Also obtained by alkaline degradation of *afromosin 7-methyl ether* (6,7,4′-trimethoxyisoflavone) (SM) (m.p. 178°) [5501], (m.p. 174–175°) [5523] with potassium hydroxide in refluxing ethanol [5501] for 40 min under nitrogen (61%) [5523]. SM was obtained by methylation of *afromosin* (7-hydroxy-6,4′-dimethoxyisoflavone) (m.p. 228–229°), itself isolated from *Afromosia elata* Harms [5523].

m.p. 100–101° [5471], 99–100° [5501,5520,5523], 98–100° [5291];
^{1}H NMR [5471], ^{13}C NMR [5293], IR [5501], UV [5501,5520]; TLC [5471].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-(2-methoxyphenyl)ethanone

[56308-08-8] $C_{17}H_{18}O_5$ mol.wt. 302.33



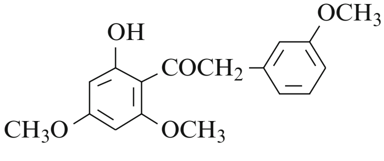
Syntheses

- Preparation by partial methylation of 2,4,6-trihydroxy-phenyl 2-methoxybenzyl ketone,
  - with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 14 h (86%) [5287], for 4 h (70%) [5456] or for 3.5 h (58%) [5495];
  - with methyl iodide and of potassium carbonate in refluxing acetone for 4 h (54%) [5498].
- Also obtained by partial methylation of 2-hydroxy-4,6-dimethoxyphenyl 2-hydroxybenzyl ketone (95%) [5476].
- Also refer to: [5281,5295,5497,5518].

m.p. 122° [5495], 116–118° [5456,5498], 116° [5287].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-(3-methoxyphenyl)ethanone

[109089-92-1] $C_{17}H_{18}O_5$ mol.wt. 302.33



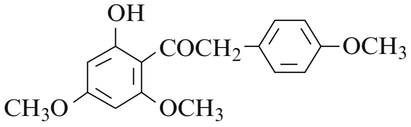
Synthesis

- Preparation by partial methylation of 3-methoxy-benzyl 2,4,6-trihydroxyphenyl ketone with dimethyl sulfate in the presence of potassium carbonate in boiling acetone for 3 h (70%) [5295].

m.p. 66–67° [5295].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-(4-methoxyphenyl)ethanone

[39604-68-7] $C_{17}H_{18}O_5$ mol.wt. 302.33



Syntheses

– Preparation by partial methylation of 2,4,6-tri-hydroxyphenyl 4-methoxybenzyl ketone,
  - with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone [5294,5535,5555], for 3 h (64%) [5250];
  - with diazomethane in ethyl ether at 0° (80%) [5462] or in methanol [5461];
  - with methyl iodide in the presence of potassium carbonate in refluxing acetone for 3 h (42%) [5538].

– Also obtained by reaction of 4-methoxyphenylacetonitrile with phloroglucinol dimethyl ether (Hoesch reaction) [5384], (19%) [5383].

– Also obtained by alkaline degradation of 3-(p-anisoyl)-4,6-dimethoxybenzofuran with potassium hydroxide in refluxing dilute methanol for 1.5 h (78%) [5556].

– Also obtained by alkaline hydrolysis of 5,7,4′-trimethoxyisoflavone (m.p. 162–163°) with 1 N aqueous sodium hydroxide in refluxing ethanol for 2 h [5487].

– Also refer to: [4935,5273,5295,5338,5339,5340,5381,5420,5448,5476,5557,5558].

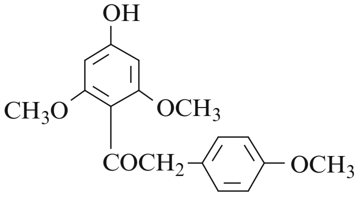
monohydrate [5556];

m.p. 140° [5556], 139–140° [5462]; 89° [5250,5384], 88–89° [5383,5487,5535,5538], 86–87° [5294]. One of the reported melting points is obviously wrong.

^1H NMR [5462], IR [5294], UV [5294,5462]; TLC [5294], HPLC [5462].

1-(4-Hydroxy-2,6-dimethoxyphenyl)-2-(4-methoxyphenyl)ethanone

[109089-93-2] $C_{17}H_{18}O_5$ mol.wt. 302.33



Syntheses

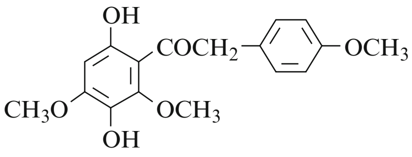
– Obtained by reaction of p-methoxyphenylacetonitrile with phloroglucinol dimethyl ether (Hoesch reaction) [5384].

– Also obtained by saponification of 4-acetoxy-2,6-di-methoxyphenyl 4-methoxybenzyl ketone (m.p. 137°) in ethanol with 3% aqueous sodium hydroxide [5384].

– Also refer to: [5558].

m.p. 73° [5384].

1-(3,6-Dihydroxy-2,4-dimethoxyphenyl)-2-(4-methoxyphenyl)ethanone



$C_{17}H_{18}O_6$ mol.wt. 318.33

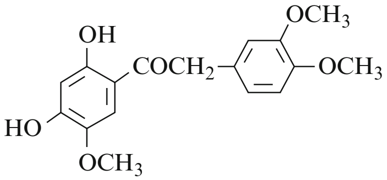
Synthesis

- Obtained (poor yield) by condensation of 2,6-di-methoxyhydroquinone with the complex p-methoxyphenylacetic acid and boron trifluoride in chloroform at r.t. overnight (<2%) [5387].

m.p. 110° [5387].

1-(2,4-Dihydroxy-5-methoxyphenyl)-2-(3,4-dimethoxyphenyl)ethanone

[24126-91-8]



$C_{17}H_{18}O_6$ mol.wt. 318.33

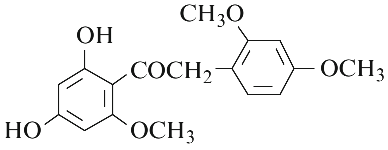
Syntheses

- Obtained by condensation of 3,4-dimethoxyphenyl-acetonitrile with 4-methoxyresorcinol (Hoesch reaction) (14%) [5533].
- Also obtained by heating *Cladrastin* (7-hydroxy-6,3′,4′-trimethoxyisoflavone) (m.p. 206–207°) with 10% aqueous barium hydroxide at reflux for 2 h under nitrogen (62%). *Cladrastin* was isolated from *Cladrastis lutea* (Mich. f.) K. Koch (Leguminosae) [5533].

m.p. 166–167° [5533]; UV [5533], MS [5533].

1-(2,4-Dihydroxy-6-methoxyphenyl)-2-(2,4-dimethoxyphenyl)ethanone

[109091-12-5]



$C_{17}H_{18}O_6$ mol.wt. 318.33

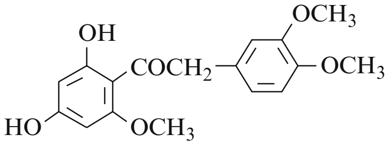
Synthesis

- Preparation by reaction of 2,4-dimethoxyphenyl-acetonitrile with phloroglucinol monomethyl ether (Hoesch reaction) [5295].

m.p. 169–171° [5295].

1-(2,4-Dihydroxy-6-methoxyphenyl)-2-(3,4-dimethoxyphenyl)ethanone

[109092-83-3]



$C_{17}H_{18}O_6$ mol.wt. 318.33

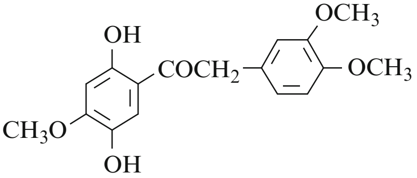
Synthesis

- Obtained by reaction of 3,4-dimethoxyphenyl-acetonitrile with phloroglucinol monomethyl ether (Hoesch reaction) [5295,5559], (20%) [5383].

m.p. 180° [5383], 179–180° [5295], 108–109° [5559]. One of the reported melting points is obviously wrong. IR [5559].

1-(2,5-Dihydroxy-4-methoxyphenyl)-2-(3,4-dimethoxyphenyl)ethanone

[24126-94-1]



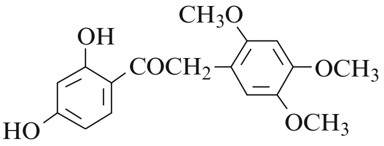
$C_{17}H_{18}O_6$ mol.wt. 318.33

Syntheses

- Obtained by reaction of 3,4-dimethoxyphenyl-acetonitrile with methoxyquinol (2-methoxy-hydroquinone) (Hoesch reaction) (16%) [5533].
- Also refer to: [5351].

m.p. 188–189° [5533];
^{13}C NMR [5293], UV [5533], MS [5533].

1-(2,4-Dihydroxyphenyl)-2-(2,4,5-trimethoxyphenyl)ethanone



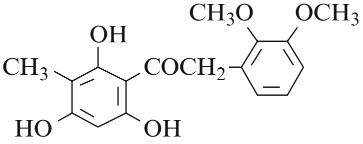
$C_{17}H_{18}O_6$ mol.wt. 318.33

Syntheses

- Obtained by reaction of 2,4,5-trimethoxyphenyl-acetonitrile with resorcinol (Hoesch reaction) (13%) [5560].
- Also obtained by reaction of resorcinol with homoasaronic acid (2,4,5-trimethoxyphenylacetic acid) (m.p. 102–103°),
  - in the presence of zinc chloride at 130–140° for 2 h (31%) (Nencki reaction) [5560];
  - in the presence of phosphorous oxychloride and zinc chloride at 50–60° for 2 h (24%) [5560];
  - in the presence of polyphosphoric acid on a steam bath for 15 min (46%) [5560].

m.p. 201–202° [5560].

2-(2,3-Dimethoxyphenyl)-1-(2,4,6-trihydroxy-3-methylphenyl)ethanone



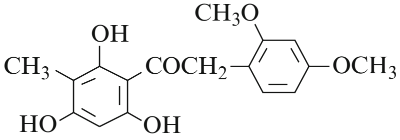
$C_{17}H_{18}O_6$ mol.wt. 318.33

Synthesis

- Obtained by reaction of 2,3-dimethoxyphenyl-acetonitrile with 2-methylphloroglucinol (Hoesch reaction) (47%) [5483].

m.p. 201° [5483].

2-(2,4-Dimethoxyphenyl)-1-(2,4,6-trihydroxy-3-methylphenyl)ethanone



$C_{17}H_{18}O_6$ mol.wt. 318.33

Syntheses

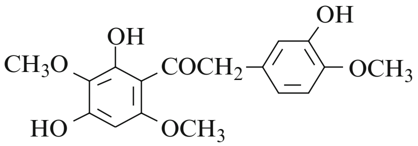
- Obtained by reaction of 2,4-dimethoxyphenyl-acetonitrile with 2-methylphloroglucinol (Hoesch reaction) (56%) [5561].
- Also refer to: [5562].

m.p. 188–189° [5561]; UV [5561].

1-(2,4-Dihydroxy-3,6-dimethoxyphenyl)-2-(3-hydroxy-4-methoxyphenyl) ethanone

[64640-60-4]

$C_{17}H_{18}O_7$ mol.wt. 334.33



Synthesis

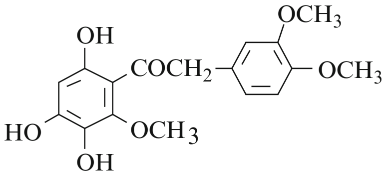
- Preparation by reaction of 3-(benzyloxy)-4-methoxyphenylacetonitrile with 1,3-(dibenzyl-oxy)-2,5-dimethoxybenzene (Hoesch reaction) [5563], (61%) [5508].

m.p. 278–279° [5508], 277–279° [5563];
^{1}H NMR [5508], IR [5563], UV [5508].

2-(3,4-Dimethoxyphenyl)-1-(3,4,6-trihydroxy-2-methoxyphenyl)ethanone

[129207-80-3]

$C_{17}H_{18}O_7$ mol.wt. 334.33



Synthesis

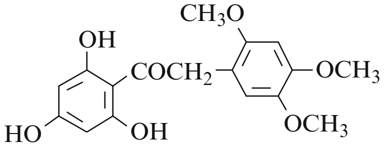
- Obtained by reaction of potassium persulfate with 2,4-dihydroxy-6-methoxyphenyl 3,4-dimethoxy-benzyl ketone in 40% aqueous potassium hydroxide at r.t. overnight (Elbs reaction) [5559].

m.p. 135–137° [5559].

1-(2,4,6-Trihydroxyphenyl)-2-(2,4,5-trimethoxyphenyl)ethanone

[72545-40-5]

$C_{17}H_{18}O_7$ mol.wt. 334.33



Synthesis

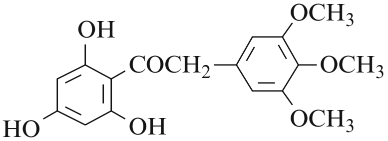
- Obtained by reaction of 2,4,5-trimethoxyphenyl-acetonitrile with phloroglucinol (Hoesch reaction) [5276], (40%) [5564].

N.B.: The phloroglucinol could not be condensed with 2,4,5-trimethoxyphenylacetyl chloride in the presence of aluminium chloride [5249,5567].

m.p. 208–209° [5564].

1-(2,4,6-Trihydroxyphenyl)-2-(3,4,5-trimethoxyphenyl)ethanone

[79744-61-9]



$C_{17}H_{18}O_7$ mol.wt. 334.33

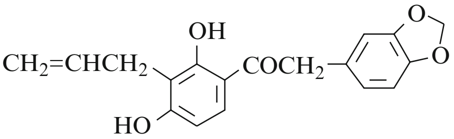
Syntheses

- Preparation by reaction of 3,4,5-trimethoxyphenyl-acetonitrile with phloroglucinol in ethyl ether (63%) (Hoesch reaction) [5496].
- Also refer to: [5351].

m.p. 197–198° [5496]; ^{1}H NMR [5496], ^{13}C NMR [5293], IR [5496], MS [5496].

2-(1,3-Benzodioxol-5-yl)-1-[2,4-dihydroxy-3-(2-propenyl)phenyl]ethanone

[117951-99-2]



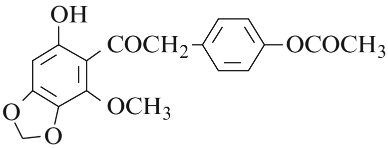
$C_{18}H_{16}O_5$ mol.wt. 312.32

Syntheses

- Obtained by reaction of allyl bromide with 2,4-dihydroxy-3′,4′-methylenedioxydesoxybenzoin in the presence of methanolic potassium hydroxide (16%) [5283].
- Also refer to: [5277].

m.p. 121–122° [5283]; ^{1}H NMR [5283], IR [5283], UV [5283].

2-[4-(Acetyloxy)phenyl]-1-(6-hydroxy-4-methoxy-1,3-benzodioxol-5-yl) ethanone



$C_{18}H_{16}O_7$ mol.wt. 344.32

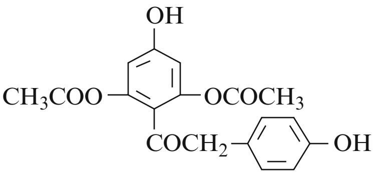
Synthesis

- Obtained by reaction of acetic anhydride with 4′-hydroxybenzyl 2-hydroxy-4,5-methylenedioxy-6-methoxyphenyl ketone in pyridine at r.t. for 1 h [5510].

m.p. 162–163° [5510].

1-[2,6-Bis(acetyloxy)-4-hydroxyphenyl]-2-(4-hydroxyphenyl)ethanone

[147747-31-5]



$C_{18}H_{16}O_7$ mol.wt. 344.32

Syntheses

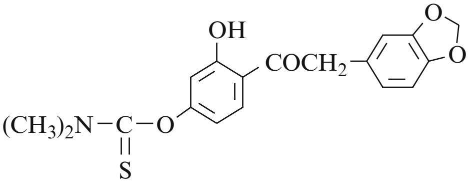
- Obtained (poor yields) by regioselective enzyme-catalyzed deacetylation of 4-acetoxybenzyl 2,4,6-tri-acetoxyphenyl ketone in the dry organic solvents hereafter mentioned containing n-butanol with lipase at 42–45° [5359].

Lipase	Solvent	Time (h)	Yields (%)
PPL	THF/n-BuOH	45	15
CCL	DIPE/n-BuOH	46	15

PPL = porcine pancreas lipase;
CCL = candida cylindracea lipase;
DIPE = diisopropyl ether.

O-[4-(1,3-Benzodioxol-5-ylacetyl)-3-hydroxyphenyl] dimethylcarbamothioate

[142751-39-1]



$C_{18}H_{17}NO_5S$ mol.wt. 359.40

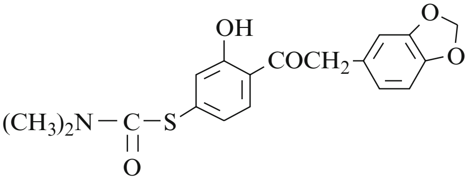
Synthesis

- Obtained by stirring a mixture of 2-(1,3-benzodioxol-5-yl)-1-(2,4-dihydroxy- phenyl)ethanone (1 mol), dimethylthio-carbamoyl chloride (2 mol), 1,4-diaza-bicyclo[2,2,2]octane and N,N-dimethyl-formamide at r.t. for 2 h (92%) [5205].

m.p. 168–169° [5205]; ^{1}H NMR [5205], MS [5205].

S-[4-(1,3-Benzodioxol-5-ylacetyl)-3-hydroxyphenyl] dimethylcarbamothioate

[142751-43-7]



$C_{18}H_{17}NO_5S$ mol.wt. 359.40

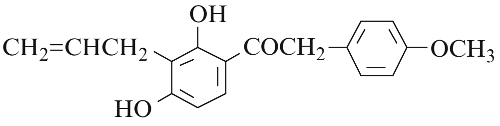
Synthesis

- Obtained by refluxing a solution of O-[4-(1,3-Benzodioxol-5-ylacetyl)-3-hydroxyphenyl] dimethylcarbamothioate [142 751-39-1] in N,N-dimethylaniline for 1 h (91%) (Newman-Kwart rearrangement) [5205].

m.p. 160–161° [5205]; ^{1}H NMR [5205], MS [5205].

1-[2,4-Dihydroxy-3-(2-propenyl)phenyl]-2-(4-methoxyphenyl)ethanone

[117951-88-9]



$C_{18}H_{18}O_4$ mol.wt. 298.34

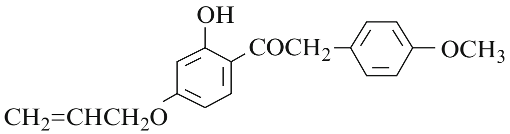
Syntheses

- Obtained by reaction of allyl bromide with 2,4-dihydroxy-4′-methoxydesoxybenzoin in methanolic potassium hydroxide at r.t. overnight (27%) [5283].
- Also refer to: [5277].

m.p. 97–98° [5283]; ^{1}H NMR [5283], IR [5283], UV [5283].

1-[2-Hydroxy-4-(2-propenyloxy)phenyl]-2-(4-methoxyphenyl)ethanone

[73937-48-1]



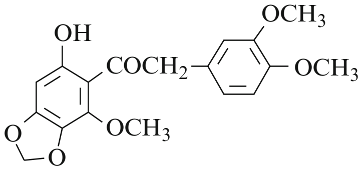
$C_{18}H_{18}O_4$ mol.wt. 298.34

Syntheses

- Refer to: [5565] and [5279] (compound **X**).

2-(3,4-Dimethoxyphenyl)-1-(6-hydroxy-4-methoxy-1,3-benzodioxol-5-yl) ethanone

[2631-85-8]



$C_{18}H_{18}O_7$ mol.wt. 346.34

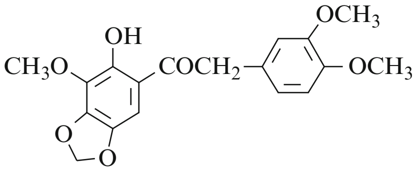
Syntheses

- Obtained by reaction of methylene iodide with 2,4,5-tri-hydroxy-6-methoxyphenyl 3,4-dimethoxybenzyl ketone in the presence of potassium carbonate in refluxing acetone for 45 h [5559].
- Also obtained (poor yield) by reaction of 3,4-di-methoxyphenylacetonitrile with 3-methoxy-4,5-methylenedioxyphenol (Hoesch reaction) (<2%) [5360].

m.p. 125–126° [5559], 121°5–122° [5360]; UV [5360].

2-(3,4-Dimethoxyphenyl)-1-(6-hydroxy-7-methoxy-1,3-benzodioxol-5-yl) ethanone

[61243-79-6]



$C_{18}H_{18}O_7$ mol.wt. 346.34

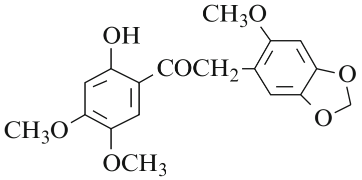
Synthesis

- Obtained by mild base hydrolysis of 8,3′,4′-tri-methoxy-6,7-methylenedioxyisoflavone [5522].

m.p. 162–163° [5522];
^{1}H NMR [5522], IR [5522], UV [5522], MS [5522];
TLC [5522].

1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-(6-methoxy-1,3-benzodioxol-5-yl) ethanone

[24195-24-2]



$C_{18}H_{18}O_7$ mol.wt. 346.34

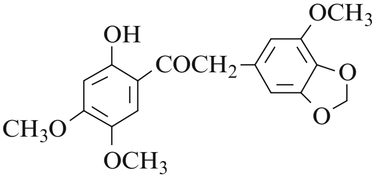
Synthesis

- Obtained by alkaline degradation of 6,7, 3′-tri-methoxy-4′,5′-methylenedioxyisoflavone (m.p. 234°5–235°5) (SM) with sodium hydroxide in refluxing 50% aqueous ethanol (10–45 min) (88%). SM was isolated from the heartwood of *Cordyla africana* (Leguminosae, sub-family: Caesalpinioideae, tribe: Swartzieae) [5379].

m.p. 161–162° [5379]; IR [5379], UV [5379], MS [5379].

1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-(7-methoxy-1,3-benzodioxol-5-yl) ethanone

[24195-23-1] $C_{18}H_{18}O_7$ mol.wt. 346.34



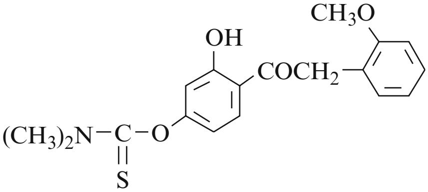
Synthesis

– Obtained by alkaline degradation of 6,7,3′-tri-methoxy-4′,5′-methylenedioxyisoflavone (m.p. 211–212°) (SM) with sodium hydroxide in refluxing 50% aqueous ethanol (10–45 min) (28%). SM was isolated from the heartwood of *Cordyla africana* (Leguminosae, sub-family: Caesalpinioideae, tribe: Swartzieae) [5379].

m.p. 143–144° [5379]; IR [5379], UV [5379], MS [5379].

O-[3-Hydroxy-4-[(2-methoxyphenyl)acetyl]phenyl] dimethylcarbamothioate

[142751-37-9] $C_{18}H_{19}NO_4S$ mol.wt. 345.42



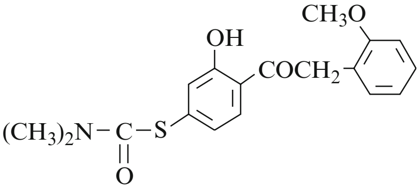
Synthesis

– Obtained by stirring a mixture of 2,4-di-hydroxyphenyl 2-methoxybenzyl ketone (1 mol), dimethylthiocarbamoyl chloride (2 mol), 1,4-diazabicyclo[2,2,2]octane and DMF at r.t. for 2 h (91%) [5205].

m.p. 168–169° [5205];
^{1}H NMR [5205], MS [5205].

S-[3-Hydroxy-4-[(2-methoxyphenyl)acetyl]phenyl] dimethylcarbamothioate

[142751-41-5] $C_{18}H_{19}NO_4S$ mol.wt. 345.42



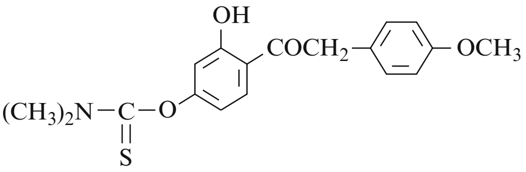
Synthesis

– Obtained by refluxing a solution of O-[3-Hydroxy-4-[(2-methoxyphenyl)acetyl]-phenyl] dimethylcarbamothioate [142751-37-9] in N,N-dimethylaniline for 1 h (89%) (Newman–Kwart rearrangement) [5205].

m.p. 107–108° [5205]; ^{1}H NMR [5205], MS [5205].

O-[3-Hydroxy-4-[(4-methoxyphenyl)acetyl]phenyl] dimethylcarbamothioate

[142751-38-0] $C_{18}H_{19}NO_4S$ mol.wt. 345.42



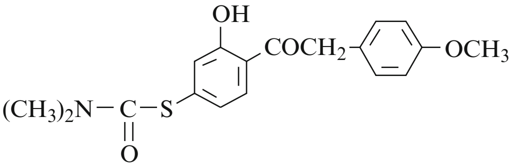
Synthesis

– Obtained by stirring a mixture of 2,4-dihydroxyphenyl 4-methoxybenzyl ketone (1 mol), dimethylthiocarbamoyl chloride (2 mol), 1,4-diazabicyclo[2,2,2] octane (2 mol) and N,N-dimethyl-formamide at r.t. for 2 h (96%) [5205].

m.p. 114–115° [5205]; ^{1}H NMR [5205], MS [5205].

S-[3-Hydroxy-4-[(4-methoxyphenyl)acetyl]phenyl] dimethylcarbamothioate

[142751-42-6] $C_{18}H_{19}NO_4S$ mol.wt. 345.42

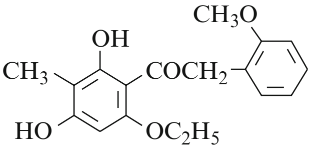


Synthesis

– Obtained by refluxing a solution of O-[3-Hydroxy-4-[(4-methoxyphenyl)-acetyl]phenyl] dimethylcarbamothioate [142751-38-0] in N,N-dimethylaniline for 1 h (92%) (Newman-Kwart rearrangement) [5205].

m.p. 130–131° [5205]; ^{1}H NMR [5205], MS [5205].

1-(6-Ethoxy-2,4-dihydroxy-3-methylphenyl)-2-(2-methoxyphenyl)ethanone



$C_{18}H_{20}O_5$ mol.wt. 316.35

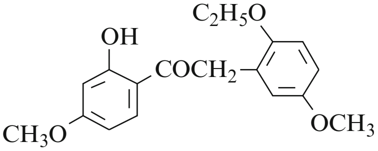
Synthesis

– Obtained by reaction of o-methoxyphenylacetonitrile with 4-ethoxy-2,6-dihydroxytoluene (Hoesch reaction) (32%) [5483].

m.p. 185° [5483].

2-(2-Ethoxy-5-methoxyphenyl)-1-(2-hydroxy-4-methoxyphenyl)ethanone

[18086-37-8]



$C_{18}H_{20}O_5$ mol.wt. 316.35

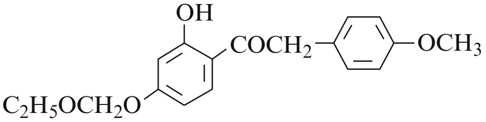
Synthesis

- Obtained by partial methylation of 2,4-dihydroxy-phenyl 2-ethoxy-5-methoxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 6 h (87%) [5531].

m.p. 107–108° [5531].

1-[4-(Ethoxymethoxy)-2-hydroxyphenyl]-2-(4-methoxyphenyl)ethanone

[97714-80-2]

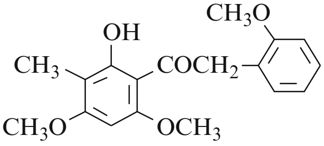


$C_{18}H_{20}O_5$ mol.wt. 316.35

Syntheses

- Obtained by reaction of chloromethyl ethyl ether with 2,4-dihydroxyphenyl 4-methoxybenzyl ketone in acetone in the presence of potassium carbonate at r.t. for 15–45 min [5393].
- Also refer to: [5273].

1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-2-(2-methoxyphenyl)ethanone



$C_{18}H_{20}O_5$ mol.wt. 316.35

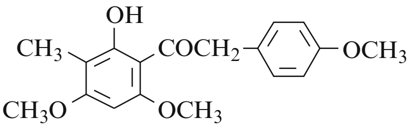
Syntheses

- Preparation by partial methylation of 2,4-dihydroxy-6-methoxy-3-methylphenyl 2-methoxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone (quantitative yield) [5495], for 14 h (89%) [5287].
- Also obtained by partial methylation of 2,4,6-trihydroxy-3-methylphenyl 2-methoxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 4 h (82%) [5497] or for 3 h (66%) [5495].
- Also obtained (by-product) by partial methylation of 2,4,6-trihydroxyphenyl 2-methoxybenzyl ketone with methyl iodide in the presence of potassium carbonate in refluxing acetone for 4 h (11%) [5498].
- Also obtained by reaction of o-methoxyphenylacetonitrile with 2-hydroxy-4,6-dimethoxytoluene (m.p. 67°) (Hoesch reaction) (38%) [5495].
- Also refer to: [5518].

m.p. 150° [5287], 148° [5495], 146–148° [5497,5501].

1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-2-(4-methoxyphenyl)ethanone

[56308-12-4]



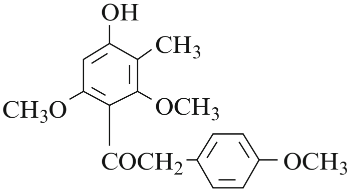
$C_{18}H_{20}O_5$ mol.wt. 316.35

Syntheses

- Obtained by partial methylation of 2,4,6-tri-hydroxy-3-methylphenyl 4-methoxybenzyl ketone,
  - with dimethyl sulfate in the presence of potassium carbonate in boiling acetone for 4 h (73%) [5538] or for 3 h (68%) [5495];
  - with excess methyl iodide in the presence of potassium carbonate in boiling acetone for 4 h [5538].
- Also obtained by reaction of methyl iodide with 2,4,6-trihydroxyphenyl 4-methoxybenzyl ketone in the presence of potassium carbonate in refluxing acetone for 3 h (13%) [5538].
- Also obtained by partial methylation of 2,4-dihydroxy-6-methoxy-3-methylphenyl 4-methoxy-benzyl ketone with methyl iodide in the presence of potassium carbonate in boiling acetone [5495] for 2 h (96%) [5538].
- Also obtained by partial methylation of 2,6-dihydroxy-4-methoxy-3-methylphenyl 4-methoxy-benzyl ketone with dimethyl sulfate with of potassium carbonate in boiling acetone [5495].
- Also obtained by partial methylation of 2,4-dihydroxy-6-methoxy-3-methylphenyl 4-hydroxy-benzyl ketone with dimethyl sulfate with potassium carbonate in boiling acetone [5495].
- Also obtained by reaction of p-methoxyphenylacetonitrile with 2-hydroxy-4,6-dimethoxytoluene (m.p. 67°) (Hoesch reaction) (38%) [5495].
- Also refer to: [5296,5284].

m.p. 116° [5495], 114–115° [5494,5538].

1-(4-Hydroxy-2,6-dimethoxy-3-methylphenyl)-2-(4-methoxyphenyl)ethanone

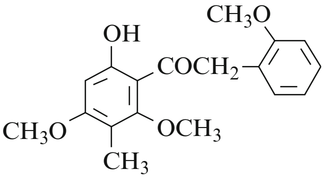


$C_{18}H_{20}O_5$ mol.wt. 316.35

Synthesis

- Obtained (by-product) by reaction of p-methoxyphenyl-acetonitrile with 2-hydroxy-4,6-dimethoxytoluene (Hoesch reaction) (small amounts) [5495].

1-(6-Hydroxy-2,4-dimethoxy-3-methylphenyl)-2-(2-methoxyphenyl)ethanone



$C_{18}H_{20}O_5$ mol.wt. 316.35

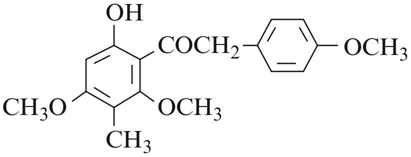
Syntheses

- Obtained by partial methylation of 4,6-dihydroxy-2-methoxy-3-methylphenyl 2-methoxybenzyl ketone with methyl iodide in the presence of potassium carbonate in boiling acetone for 30 min (96%) [5549].
- Preparation by alkaline degradation of 5,7,2′-trimethoxy-6-methylisoflavone (m.p. 220°) with sodium hydroxide in refluxing dilute methanol for 1.5 h (74%) [5483].
- Also refer to: [4935].

m.p. 134° [5483].

1-(6-Hydroxy-2,4-dimethoxy-3-methylphenyl)-2-(4-methoxyphenyl)ethanone

[22081-01-2]



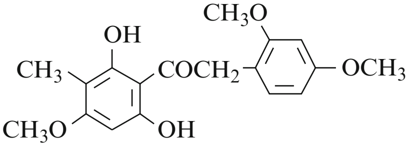
$C_{18}H_{20}O_5$ mol.wt. 316.35

Syntheses

- Obtained by partial methylation of 4,6-di-hydroxy-2-methoxy-3-methylphenyl 4-methoxy-benzyl ketone with excess methyl iodide in the presence of potassium carbonate in boiling acetone for 1.5 h (96%) [5549].
- Also obtained by reaction of p-methoxyphenylacetyl chloride with 4-hydroxy-2,6-dimethoxy-toluene in ethyl ether in the presence of aluminium chloride (18%) [5394].

m.p. 88° [5549], 87–88° [5394];
^{1}H NMR [5341], IR [5394]; TLC [5394].

1-(2,6-Dihydroxy-4-methoxy-3-methylphenyl)-2-(2,4-dimethoxyphenyl)ethanone



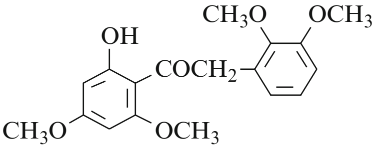
$C_{18}H_{20}O_6$ mol.wt. 332.35

Synthesis

- Obtained by alkaline degradation of dehydro-ougenin dimethyl ether acetate (5-acetoxy-6-methyl-7,2′,4′-trimethoxyisoflavanone) (m.p. 216–217°) with refluxing alcoholic potash for 2 h (83%) [5561].

m.p. 174–175° [5561]; UV [5561].

2-(2,3-Dimethoxyphenyl)-1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone



$C_{18}H_{20}O_6$ mol.wt. 332.35

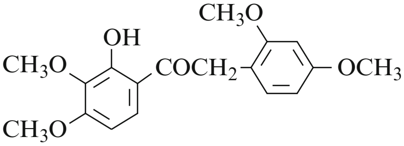
Synthesis

- Preparation by partial methylation of 2,4,6-tri-hydroxy-2′,3′-dimethoxydeoxybenzoin with dimethyl sulfate in the presence of potassium carbonate in boiling acetone (92%) [5483].

m.p. 132° [5483].

2-(2,4-Dimethoxyphenyl)-1-(2-hydroxy-3,4-dimethoxyphenyl)ethanone

[6502-87-0]



$C_{18}H_{20}O_6$ mol.wt. 332.35

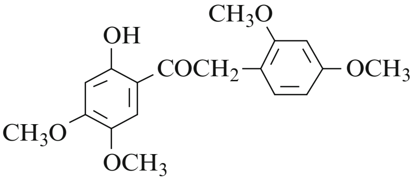
Syntheses

- Preparation by partial methylation of 2,3,4-tri-hydroxyphenyl 2,4-dimethoxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 6 h (82%) [5540].
- Also obtained by Friedel–Crafts acylation of pyrogallol trimethyl ether with 2,4-dimethoxy-phenylacetyl chloride [5566], (41%) [5540].
- Also refer to: [5567].

m.p. 134–135° [5566,5543]; ^{1}H NMR [5341].

2-(2,4-Dimethoxyphenyl)-1-(2-hydroxy-4,5-dimethoxyphenyl)ethanone

[15402-24-1]



$C_{18}H_{20}O_6$ mol.wt. 332.35

Syntheses

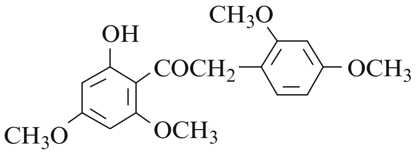
- Preparation by Hoesch condensations of 1,2,4-tri-hydroxybenzene with 2,4-dimethoxyphenyl-acetonitrile or with 2,4-dihydroxyphenylacetonitrile, followed by partial methylations of the ketones so obtained [5265].
- Also obtained by Friedel–Crafts acylation of 1,2,4-trimethoxybenzene with 2,4-dimethoxyphenylacetyl chloride in the presence of aluminium chloride [5567] in ethyl ether [5553], (31%) [5568].
- Also refer to: [5277].

m.p. 122–123° [5568,5570].

2-(2,4-Dimethoxyphenyl)-1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone *(Albizoin)*

[39604-69-8]

$C_{18}H_{20}O_6$ mol.wt. 332.35



Syntheses

- Preparation by partial methylation of 2,4-di-methoxybenzyl 2,4,6-trihydroxyphenyl ketone with dimethyl sulfate in the presence of potassium carbonate in boiling acetone [5295], (92%) [5569].
- Preparation by partial demethylation of 2,4-dimethoxybenzyl 2,4,6-trimethoxyphenyl ketone with aluminium chloride in refluxing ethyl ether for 10 h (65%) [5570].
- Also obtained by alkaline degradation of 5,7,2′,4′-tetramethoxyisoflavone (m.p. 203–204°) with potassium hydroxide in refluxing dilute ethanol for 1 h (93%) [5570].
- Also obtained by alkaline degradation of *ferreirin* trimethyl ether (m.p. 163°) [5570], so called *dihydrodalbergioidin tetramethyl ether* (m.p. 165–166°) [5561], (5,7,2′,4′-tetramethoxy-isoflavanone) with potassium hydroxide in refluxing dilute ethanol for 1 h (21%) [5570] or for 6 h (26%) [5561].
- Also obtained by Friedel–Crafts acylation of 1,3,5-trimethoxybenzene with 2,4-dimethoxyphenyl-acetyl chloride in the presence of aluminium chloride in ethyl ether at 0° for 16 h [5571].
- Also refer to: [4923,5277,5381,5483,5534].

Isolation from natural sources

- From the marine mollusc *Nerita albicilla* (Class Gastropoda, family Neritidae) [5571].

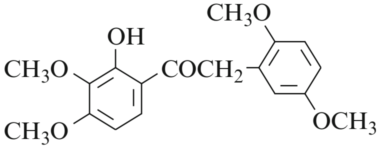
m.p. 140–142° [5571], 139° [5570], 138–139° [5561], 137–138° [5569], 136–137° [5295];

^{1}H NMR [5341,5571], IR [5561,5571], UV [5561,5571], MS [5571];
HPLC [5571].

2-(2,5-Dimethoxyphenyl)-1-(2-hydroxy-3,4-dimethoxyphenyl)ethanone

[20569-19-1]

$C_{18}H_{20}O_6$ mol.wt. 332.35



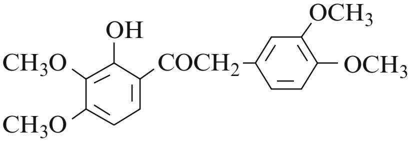
Synthesis

- Obtained by Friedel–Crafts acylation of pyrogallol trimethyl ether with 2,5-dimethoxyphenylacetyl chloride in the presence of aluminium chloride in ethyl ether at 0° overnight (39%) [5568].

m.p. 153–154° [5568].

2-(3,4-Dimethoxyphenyl)-1-(2-hydroxy-3,4-dimethoxyphenyl)ethanone

[61243-86-5] $C_{18}H_{20}O_6$ mol.wt. 332.35



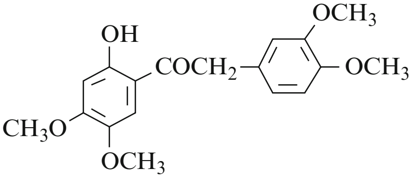
Syntheses

- Obtained by partial methylation of 2,3,4-tri-hydroxyphenyl 3,4-dimethoxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 6 h (82%) [5470].
- Also obtained by Friedel–Crafts acylation of pyrogallol trimethyl ether with 3,4-dimethoxyphenyl-acetyl chloride (homoveratroyl chloride) in ethyl ether in the presence of aluminium chloride, first at 0°, then at r.t. overnight (33–34%) [5214,5473] or in refluxing methylene chloride for 2.5 h (70%) [5572,5576].
- Obtained by partial demethylation of 2,3,4-trimethoxyphenyl 3′,4′-dimethoxybenzyl ketone (oil) with aluminium chloride in refluxing ethyl ether for 1 h (67%) [5522].
- Also refer to: [5574].

m.p. 139–140° [5573], 135–137° [5522], 134° [5470], 133–134° [5214], ^{1}H NMR [5573], IR [5573].

2-(3,4-Dimethoxyphenyl)-1-(2-hydroxy-4,5-dimethoxyphenyl)ethanone

[24195-22-0] $C_{18}H_{20}O_6$ mol.wt. 332.35



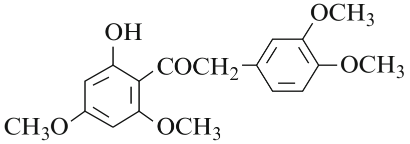
Syntheses

- Obtained by alkaline degradation of 6,7,3′,4′-tetra-methoxyisoflavone (m.p. 187–188°) (SM) with sodium hydroxide in refluxing 50% aqueous ethanol (45%) [5379]. SM was isolated from the heartwood of *Cordyla africana* (Leguminosae).
- Also obtained by partial demethylation of 3,4-di-methoxybenzyl 2,4,5-trimethoxyphenyl ketone (m.p. 120–121°) with aluminium chloride in refluxing acetonitrile for 45 min (10%) [5379].
- Also obtained by partial methylation of 3,4-dimethoxybenzyl 2,4,5-trihydroxyphenyl ketone (85%) [5471] according to [5273].

m.p. 138–139° [5471], 137–138° [5379];
^{1}H NMR [5341], ^{13}C NMR [5293], IR [5379], UV [5379], MS [5379]; TLC [5471].

2-(3,4-Dimethoxyphenyl)-1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone

[109250-71-7]



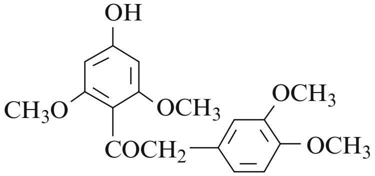
$C_{18}H_{20}O_6$ mol.wt. 332.35

Syntheses

- Preparation by partial methylation of 2,4-di-hydroxy-6,3′,4′-trimethoxydeoxybenzoin with methyl iodide in the presence of potassium carbonate in boiling acetone for 1 h (80%) [5383].
- Also obtained by partial methylation of 2,4,6-trihydroxy-3′,4′-dimethoxydeoxybenzoin with dimethyl sulfate in the presence of potassium carbonate in boiling acetone [5294], (80%) [5295], for 3 h [5250] or for 10 h [5546].
- Also obtained by hydrolysis of *O-trimethylsantal* (5,7,3′,4′-tetramethoxyisoflavone) (m.p. 155–156°) with potassium hydroxide in boiling dilute ethanol for 1 h (82%) [5383].
- Also obtained by reaction of 3,4-dimethoxyphenylacetonitrile with phloroglucinol dimethyl ether (Hoesch reaction) (12%) [5383].
- Also refer to: [4935,5476].

m.p. 120–121° [5546], 117°5 [5294], 117° [5250,5383], 101–103° [5295];
One of the reported melting points is obviously wrong.
IR [5294], UV [5294]; TLC [5294].

2-(3,4-Dimethoxyphenyl)-1-(4-hydroxy-2,6-dimethoxyphenyl)ethanone



$C_{18}H_{20}O_6$ mol.wt. 332.35

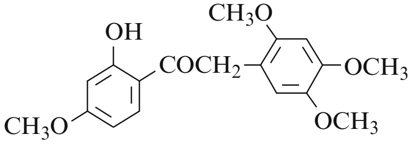
Synthesis

- Obtained (by-product) by reaction of 3,4-dimethoxy-phenylacetonitrile with phloroglucinol dimethyl ether (Hoesch reaction) (<3%) [5383].

m.p. 140° [5383].

1-(2-Hydroxy-4-methoxyphenyl)-2-(2,4,5-trimethoxyphenyl)ethanone

[85288-48-8]



$C_{18}H_{20}O_6$ mol.wt. 332.35

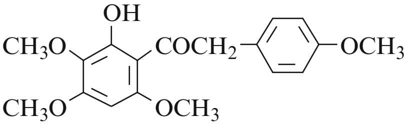
Syntheses

- Obtained by partial methylation of 2,4-di-hydroxyphenyl2,4,5-trimethoxybenzyl ketone in acetone,
  - with an etheral solution of diazomethane (96%) [5560];
  - with methyl iodide in the presence of potassium carbonate in refluxing acetone for 70 min (96%) [5560].

m.p. 135–136° [5560]; ^{1}H NMR [5341].

1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-2-(4-methoxyphenyl)ethanone

[13539-22-5]



^{1}H NMR [5341].

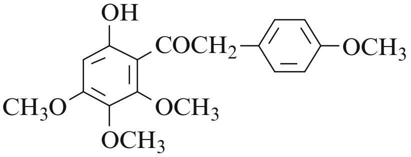
$C_{18}H_{20}O_6$ mol.wt. 332.35

Synthesis

- Refer to: [5341] (compound **XII**).

1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-2-(4-methoxyphenyl)ethanone

[22110-04-9]



$C_{18}H_{20}O_6$ mol.wt. 332.35

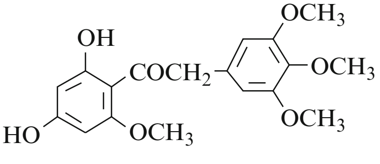
Syntheses

- Preparation by acylation of antiarol,
  - with p-methoxyphenylacetyl chloride in ethyl ether in the presence of aluminium chloride at r.t. for 12 h (28%) [5396] or for 24 h (55%) [5251];
  - with p-methoxyphenylacetic acid in chloroform in the presence of boron trifluoride at r.t. overnight (37%) [5387].
- Also obtained by alkaline hydrolysis of *munigin* dimethyl ether (m.p. 176°) (5,6,7,4′-tetramethoxy-isoflavone) with potassium hydroxide in refluxing ethanol for 30 min (84%) [5575].

m.p. 91–92° [5396], 73° [5575], 69° [5251,5390].
One of the reported melting points is obviously wrong.
b.p.$_1$ 190–200° [5575], b.p.$_4$ 207–222° [5251], b.p.$_2$ 210–220° [5387].

1-(2,4-Dihydroxy-6-methoxyphenyl)-2-(3,4,5-trimethoxyphenyl)ethanone

[129207-78-9]



m.p. 130–131° [5559]; IR [5559].

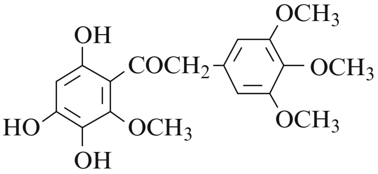
$C_{18}H_{20}O_7$ mol.wt. 348.35

Synthesis

- Obtained by reaction of 3,4,5-tri methoxyphenyl-acetonitrile with phloroglucinol monomethyl ether (Hoech reaction) [5559].

1-(3,4,6-Trihydroxy-2-methoxyphenyl)-2-(3,4,5-trimethoxyphenyl)ethanone

[129207-79-0]



$C_{18}H_{20}O_8$ mol.wt. 364.35

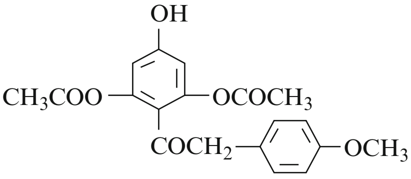
Synthesis

- Obtained by reaction of potassium persulfate with 2,4-dihydroxy-6-methoxyphenyl 3,4,5-trimethoxybenzyl ketone in 40% aqueous potassium hydroxide at r.t. overnight (Elbs reaction) (21%) [5559].

m.p. 164–165° [5559].

1-[2,6-Bis(acetyloxy)-4-hydroxyphenyl]-2-(4-methoxyphenyl)ethanone

[204068-63-3]



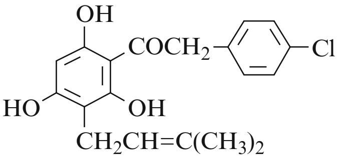
$C_{19}H_{18}O_7$ mol.wt. 358.35

Synthesis

- Obtained (small amount) by selective deacetylation of 1-acetoxy-1-(2,4,6-triacetoxy-phenyl)-2-(4-methoxyphenyl)ethene catalyzed by porcine pancreas lipase in THF at 42–45° for 72 h [5265].

2-(4-Chlorophenyl)-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl] ethanone

[85602-22-8]



$C_{19}H_{19}ClO_4$ mol.wt. 346.81

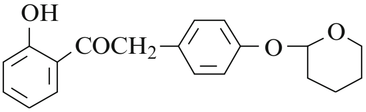
Synthesis

- Preparation by reaction of prenyl chloride with 2-(4-chlorophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone in ethyl ether in the presence of a saturated aqueous sodium carbonate solution and a catalytic amount of cuprous chloride for 3 h at r.t. (45%) [5576].

m.p. 182–184° [5576]; ^{13}C NMR [5576], IR [5576], MS [5576].

1-(2-Hydroxyphenyl)-2-[4-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone

[130064-19-6]



$C_{19}H_{20}O_4$ mol.wt. 312.37

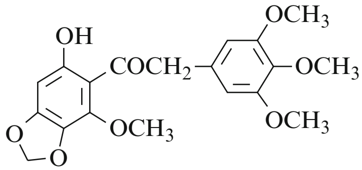
Synthesis

- Preparation by reaction of 3,4-dihydro-2*H*-pyran with 2,4′-dihydroxydeoxybenzoin in dioxane in the presence of PTSA (p-toluenesulfonic acid) at r.t. for 4 h (80%) [5280].

m.p. 95° [5280]; ^1H NMR [5280], IR [5280], MS [5280].

1-(6-Hydroxy-4-methoxy-1,3-benzodioxol-5-yl)-2-(3,4,5-trimethoxyphenyl) ethanone

[50901-33-2]



$C_{19}H_{20}O_8$ mol.wt. 376.38

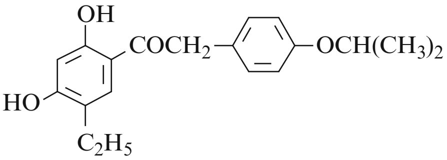
Synthesis

- Obtained by reaction of methylene iodide with 2,4,5-trihydroxy-6-methoxyphenyl 3,4,5-trimethoxy-benzyl ketone in the presence of potassium carbonate in refluxing acetone for 50 h [5559].

m.p. 119–120° [5559].

1-(5-Ethyl-2,4-dihydroxyphenyl)-2-[4-(1-methylethoxy)phenyl]ethanone

[96644-04-1]



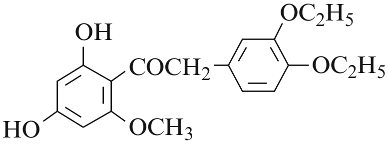
$C_{19}H_{22}O_4$ mol.wt. 314.38

Synthesis

- Preparation by condensation of p-isopropyl-oxyphenylacetonitrile with 4-ethylresorcinol in the presence of boron trifluoride etherate under hydrogen chloride atmosphere (8–10 h) at r.t. overnight (62%) [5367].

m.p. 95–96° [5367]; ^{1}H NMR [5367].

2-(3,4-Diethoxyphenyl)-1-(2,4-dihydroxy-6-methoxyphenyl)ethanone



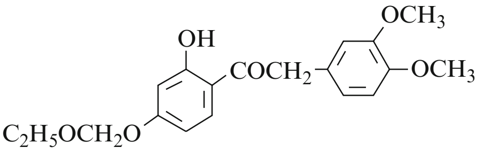
$C_{19}H_{22}O_6$ mol.wt. 346.38

Synthesis

- Obtained by reaction of 3,4-diethoxyphenyl-acetonitrile with phloroglucinol monomethyl ether (Hoesch reaction) (24%) [5383].

m.p. 129–130° [5383].

2-(3,4-Dimethoxyphenyl)-1-(4-ethoxymethoxy-2-hydroxyphenyl)ethanone



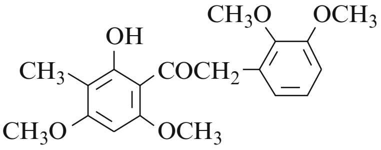
$C_{19}H_{22}O_6$ mol.wt. 346.38

Synthesis

- Obtained by reaction of ethoxymethyl chloride with 2,4-dihydroxyphenyl 3,4-dimethoxybenzyl ketone in the presence of potassium carbonate in acetone for 45 min [5534].

TLC [5534].

2-(2,3-Dimethoxyphenyl)-1-(2-hydroxy-4,6-dimethoxy-3-methylphenyl) ethanone



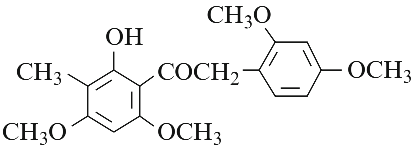
$C_{19}H_{22}O_6$ mol.wt. 346.38

Synthesis

- Preparation by partial methylation of 2,4,6-tri-hydroxy-3-methylphenyl 2, 3-dimethoxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in boiling acetone (69%) [5483].

m.p. 160° [5483].

2-(2,4-Dimethoxyphenyl)-1-(2-hydroxy-4,6-dimethoxy-3-methylphenyl) ethanone



$C_{19}H_{22}O_6$ mol.wt. 346.38

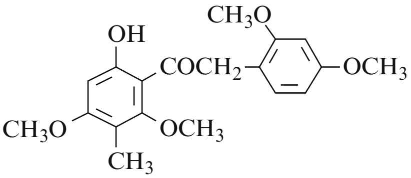
Syntheses

- Preparation by partial methylation of 2,4,6-tri-hydroxy-3-methylphenyl 2,4-dimethoxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 40 h (55%) [5561].
- Also refer to: [5562].

m.p. 142–143° [5561]; UV [5561].

2-(2,4-Dimethoxyphenyl)-1-(6-hydroxy-2,4-dimethoxy-3-methylphenyl) ethanone

[22081-04-5]



$C_{19}H_{22}O_6$ mol.wt. 346.38

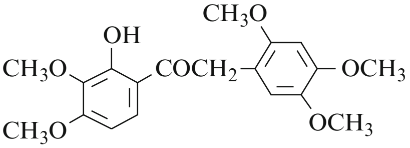
Synthesis

- Obtained by reaction of 2,4-dimethoxyphenyl-acetyl chloride with 4-hydroxy-2,6-dimethoxy-toluene in ethyl ether in the presence of aluminium chloride [5394].

m.p. 115–116° [5394]; IR [5394], UV [5394].

1-(2-Hydroxy-3,4-dimethoxyphenyl)-2-(2,4,5-trimethoxyphenyl)ethanone

[20390-13-0]



$C_{19}H_{22}O_7$ mol.wt. 362.38

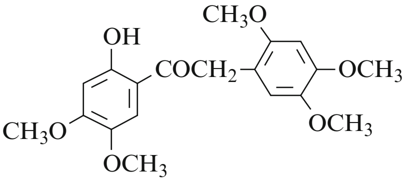
Synthesis

- Preparation by Friedel–Crafts acylation of pyrogallol trimethyl ether with 2,4,5-trimethoxy-phenylacetyl chloride in the presence of aluminium chloride in ethyl ether at 0° overnight (68%) [5568].

m.p. 170–171° [5568].

1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-(2,4,5-trimethoxyphenyl)ethanone

[24195-21-9]



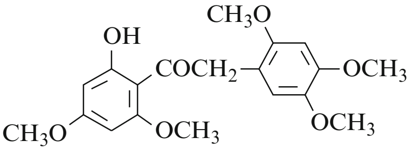
$C_{19}H_{22}O_7$ mol.wt. 362.38

Synthesis

- Obtained by alkaline degradation of 6,7,2′,4′,5′-pentamethoxyisoflavone (m.p. 171–172°) (SM) with sodium hydroxide in refluxing 50% aqueous ethanol (10–45 min) (84%). SM was isolated from the heartwood of Cordyla africana (Leguminosae; sub-family: Caesal-pinioideae, tribe: Swartzieae) [5379].

m.p. 137–138° and 127°5–128°5 [5379]; IR [5379], UV [5379], MS [5379].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-(2,4,5-trimethoxyphenyl)ethanone



$C_{19}H_{22}O_7$ mol.wt. 362.38

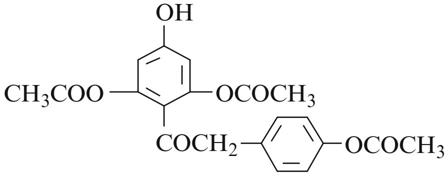
Synthesis

- Obtained by partial methylation of 2,4,6-tri-hydroxyphenyl 2,4,5-trimethoxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 14 h (74%) [5564].

m.p. 144–145° [5564].

2-[4-(Acetyloxy)phenyl]-1-[2,6-bis(acetyloxy)-4-hydroxyphenyl]ethanone

[145747-30-4]



$C_{20}H_{18}O_8$ mol.wt. 386.36

Syntheses

- Obtained by regioselective enzyme-catalyzed deacetylation of 4-acetoxybenzyl 2,4,6-triacetoxyphenyl ketone in the dry organic solvents hereafter mentioned containing n-butanol with lipase at 42–45° [5359].

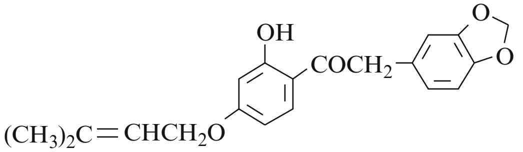
Lipase	Solvent	Time (h)	Yields (%)
PPL	Acetone/n-BuOH	48	18
PPL	CH_3CN/n-BuOH	48	18
PPL	THF/n-BuOH	45	55
CCL	DIPE/n-BuOH	46	52

PPL = porcine pancreas lipase; CCL = candida cylindracea lipase; DIPE = diisopropyl ether.

TLC [5359].

2-(1,3-Benzodioxol-5-yl)-1-[2-hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]ethanone

[94683-36-0]



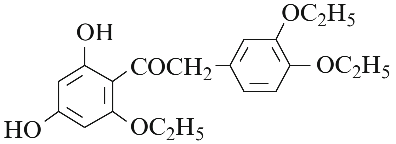
$C_{20}H_{20}O_5$ mol.wt. 340.38

Syntheses

- Obtained by alkaline degradation of *Tephrosia maxima* Pers. (7-γ,γ-di-methylallyloxy)-3′,4′-methylenedioxy-isoflavone so called *7-O-γ,γ-dimethyl-allylpseudobaptigenin* (m.p. 126–128°)$C_{21}H_{18}O_5$, with sodium hydroxide (12%) in refluxing dilute ethanol for 15 min [5469].
- Also obtained by partial allylation of *Ψ-baptigenetin* (2,4-dihydroxyphenyl 3,4-methylenedioxy-benzyl ketone) with γ,γ-dimethylallyl bromide in the presence of potassium carbonate in refluxing acetone for 8 h (64%) [5470].

m.p. 81–82° [5470], 73–74° [5469].

2-(3,4-Diethoxyphenyl)-1-(2-ethoxy-4,6-dihydroxyphenyl)ethanone



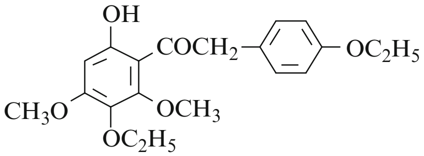
$C_{20}H_{24}O_6$ mol.wt. 360.41

Synthesis

- Obtained by condensation of 3,4-diethoxyphenyl-acetonitrile with phloroglucinol monoethyl ether (Hoesch reaction) (16%) [5383].

rhombic prisms [5383];
4-methyl ether m.p. 99° [5383]

1-(3-Ethoxy-6-hydroxy-2,4-dimethoxyphenyl)-2-(4-ethoxyphenyl)ethanone



$C_{20}H_{24}O_6$ mol.wt. 360.41

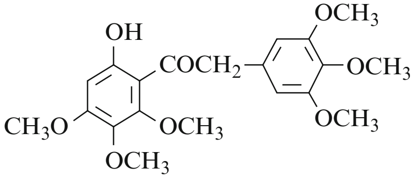
Syntheses

- Obtained by acylation of 4-ethoxy-3,5-dimethoxy-phenol,
  - with p-ethoxyphenylacetic acid in chloroform in the presence of boron trifluoride at r.t. overnight (31%) [5387];
  - with p-ethoxyphenylacetyl chloride in ethyl ether in the presence of aluminium chloride at 0° for 2 h, then at r.t. overnight (9%) [5577].

m.p. 104° [5387,5580]; b.p.$_{0.5}$ 220–230° [5387].

1-(6-hydroxy-2,3,4-trimethoxyphenyl)-2-(3,4,5-trimethoxyphenyl)ethanone

[64554-42-3]



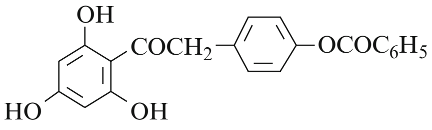
$C_{20}H_{24}O_8$ mol.wt. 392.41

Synthesis

- Obtained by alkaline degradation of *irigenin trimethyl ether* (5,6,7,3′,4′,5′-hexamethoxy-isoflavone) (m.p. 163°) with potassium hydro xide in refluxing dilute ethanol for 3 h (82%). *Irigenin* (5,7,3′-trihydroxy-6,4′,5′-tri-methoxyisoflavone) (m.p. 185°) was prepared by acidic hydrolysis of *iridin* (7-glucopyranosyloxy-5,3′-dihydroxy-6,4′,5′-trimethoxyisoflavone) (m.p. 216–217°), itself isolated from *iris kumaonensis* Wall. [5578].

m.p. 92° [5578]; IR [5578], UV [5578]; TLC [5578].

2-[4-(Benzoyloxy)phenyl]-1-(2,4,6-trihydroxyphenyl)ethanone



$C_{21}H_{16}O_6$ mol.wt. 364.35

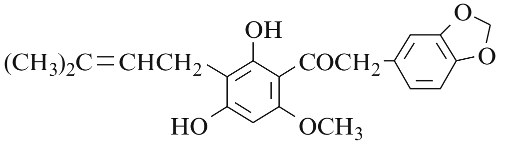
Synthesis

- Preparation by reaction of 4-benzoyloxy-acetonitrile with phloroglucinol (Hoesch reaction) (44%) [5330].

m.p. 224° [5330].

2-(1,3-Benzodioxol-5-yl)-1-[2,4-dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[55607-37-9] $C_{21}H_{22}O_6$ mol.wt. 370.95



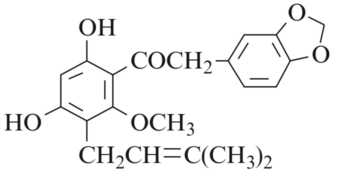
Synthesis

- Obtained (poor yield) by prenylation of 2,4-dihydroxy-6-methoxyphenyl 3,4-methylenedioxybenzyl ketone with 2-hydroxy-2-methyl-3-butene in dioxane in the presence of boron tri-fluoride etherate for 1 h at r.t. (4%) [5352].

m.p. 155–156° [5352]; ^{1}H NMR [5352]; TLC [5352].

2-(1,3-Benzodioxol-5-yl)-1-[4,6-dihydroxy-2-methoxy-3-(3-methyl-2-butenyl) phenyl]ethanone

[55607-38-0] $C_{21}H_{22}O_6$ mol.wt. 370.95



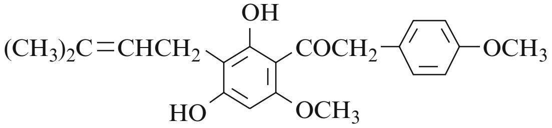
Synthesis

- Obtained (poor yield) by prenylation of 2,4-dihydroxy-6-methoxyphenyl 3,4-methylenedioxybenzyl ketone with 2-hydroxy-2-methyl-3-butene in dioxane in the presence of boron trifluoride etherate for 1 h at r.t. (3%) [5352].

m.p. 105–106° [5352]; ^{1}H NMR [5352]; TLC [5352].

1-[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-(4-methoxyphenyl)ethanone

[35817-95-9] $C_{21}H_{24}O_5$ mol.wt. 356.42



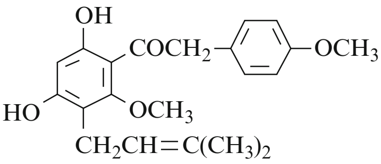
Syntheses

- Obtained by nuclear prenylation of 2,4-dihydroxy-6-methoxyphenyl 4-methoxybenzyl ketone,
  - using 2-methyl-2-hydroxy-3-methylbutene in dioxane in the presence of boron trifluoride etherate, first at 0°, then for 1 h at r.t. (9%) [5524];
  - with prenyl bromide in the presence methanolic potassium hydroxide, first with cooling, then keeping the reaction mixture for 20 h at r.t. (12%) [5524].

m.p. 153–154° [5524]; ^{1}H NMR [5524]; TLC [5524].

1-[4,6-Dihydroxy-2-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-(4-methoxyphenyl)ethanone

[35817-96-0] $C_{21}H_{24}O_5$ mol.wt. 356.42



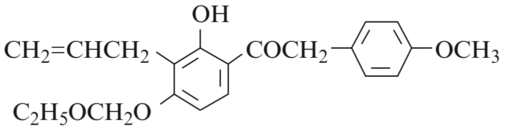
Synthesis

– Obtained by nuclear prenylation of 2,4-dihydroxy-6-methoxyphenyl4-methoxybenzyl ketone using 2-methyl-2-hydroxy-3-methylbutene in dioxane in the presence of boron trifluoride etherate, first at 0°, then for 1 h at r.t. (7%) [5524].

m.p. 91–92° [5524]; ^{1}H NMR [5524]; TLC [5524].

1-[4-(Ethoxymethoxy)-2-hydroxy-3-(2-propenyl)phenyl]-2-(4-methoxyphenyl)ethanone

[117951-89-0] $C_{21}H_{24}O_5$ mol.wt. 356.42



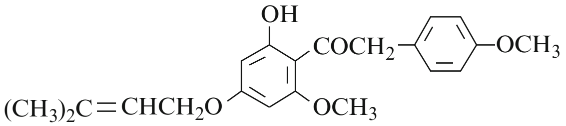
Synthesis

– Obtained by reaction of ethoxymethyl chloride with 3-allyl-2,4-dihydroxy-4′-methoxydesoxybenzoin in the presence of potassium carbonate in acetone for 10 min at r.t. [5283].

TLC [5283].

1-[2-Hydroxy-6-methoxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-2-(4-methoxyphenyl)ethanone

[35817-38-0] $C_{21}H_{24}O_5$ mol.wt. 356.42

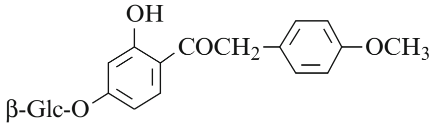


Synthesis

– Obtained by reaction of prenyl bromide with 2,4-dihydroxy-6,4′-dimethoxydesoxybenzoin in the presence of potassium carbonate in refluxing acetone for 3 h (81%) or in the presence of methanolic potassium hydroxide, first with cooling, then keeping the reaction mixture for 20 h at r.t. (2%) [5524].

m.p. 76–77° [5524]; ^{1}H NMR [5524]; TLC [5524].

1-[4-(β-D-Glucopyranosyloxy)-2-hydroxyphenyl]-2-(4-methoxyphenyl)ethanone *(Onospin)*



$C_{21}H_{24}O_9$ mol.wt. 420.42

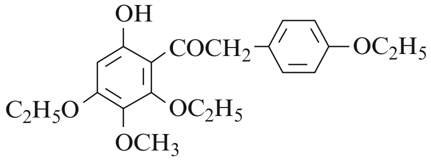
Syntheses

- Obtained by reaction of acetobromoglucose with 1-(2,4-dihydroxyphenyl)-2-(4-methoxy-phenyl) ethanone in acetone in the presence of 10% aqueous sodium hydroxide at r.t. for 12 h (25%) [5486].
- Also obtained by alkaline degradation of *Ononin* — 7-(β-D-glucopyranosyloxy)-4′-methoxy isoflavone (SM) — [5251] with boiling aqueous barium hydroxide [5486]. SM was isolated from the roots of thorny restharrow (*Ononis spinosa*) (Leguminosae, sub-family Fabaceae) [5251,5489].

m.p. 179°5 [5486];

$(\alpha)_D$ = 65°9–67°2 (methanol) [5486].

1-(2,4-Diethoxy-6-hydroxy-3-methoxyphenyl)-2-(4-ethoxyphenyl)ethanone



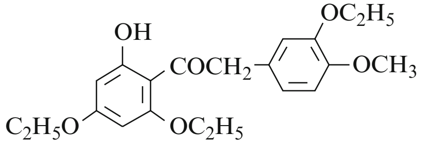
$C_{21}H_{26}O_6$ mol.wt. 374.43

Synthesis

- Obtained by Friedel–Crafts acylation of 3,5-di-ethoxy-4-methoxy-phenol with p-ethoxy-phenylacetyl chloride in ethyl ether in the presence of aluminium chloride at r.t. for 12 h [5396].

oil [5396].

1-(2,4-Diethoxy-6-hydroxyphenyl)-2-(3-ethoxy-4-methoxyphenyl)ethanone



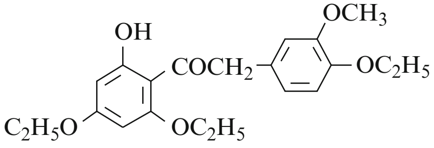
$C_{21}H_{26}O_6$ mol.wt. 374.43

Synthesis

- Obtained by condensation of 3-ethoxy-4-methoxyphenylacetonitrile with phloroglucinol diethyl ether (Hoesch reaction) (16%) [5383].

m.p. 117° [5383].

1-(2,4-Diethoxy-6-hydroxyphenyl)-2-(4-ethoxy-3-methoxyphenyl)ethanone



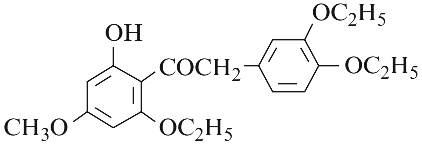
$C_{21}H_{26}O_6$ mol.wt. 374.43

Synthesis

- Obtained by condensation of 4-ethoxy-3-methoxyphenylacetonitrile with phloro-glucinol diethyl ether (Hoesch reaction) (16%) [5383].

m.p. 138° [5383].

2-(3,4-Diethoxyphenyl)-1-(2-ethoxy-6-hydroxy-4-methoxyphenyl)ethanone



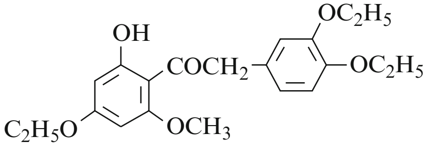
$C_{21}H_{26}O_6$ mol.wt. 374.43

Syntheses

- Obtained by alkaline degradation of O-triethyl-santal (7-methoxy-5, 3′,4′-triethoxyisoflavone) (m.p. 111–112°) with potassium hydroxide in boiling dilute ethanol for 1.5 h (67%) [5383].
- Also obtained by partial methylation of 2,4-di-hydroxy-6,3′,4′-triethoxydeoxybenzoin [5383].

m.p. 99° [5383].

2-(3,4-Diethoxyphenyl)-1-(4-ethoxy-2-hydroxy-6-methoxyphenyl)ethanone



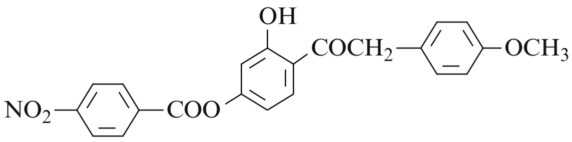
$C_{21}H_{26}O_6$ mol.wt. 374.43

Synthesis

- Obtained by partial ethylation of 2,4-di-hydroxy-6-methoxyphenyl 3,4-diethoxybenzyl ketone with ethyl iodide in the presence of potassium carbonate in boiling acetone for 2 h (77%) [5383].

m.p. 111–112° [5383].

1-[2-Hydroxy-4-(4-nitrobenzoyloxy)phenyl]-2-(4-methoxyphenyl)ethanone



$C_{22}H_{17}NO_7$ mol.wt. 407.38

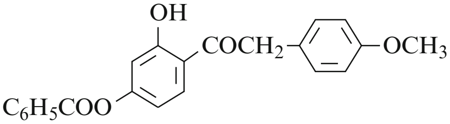
Synthesis

- Obtained by partial esterification of 2, 4-dihydroxyphenyl 4-methoxybenzylketone with p-nitrobenzoyl chloride in the presence of pyridine [5217].

m.p. 166–167° [5217].

1-[4-(Benzoyloxy)-2-hydroxyphenyl]-2-(4-methoxyphenyl)ethanone

[102706-12-7]



$C_{22}H_{18}O_5$ mol.wt. 362.38

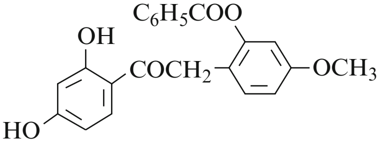
Syntheses

- Obtained by partial esterification of 2,4-di-hydroxyphenyl 4-methoxybenzyl ketone with benzoyl chloride (Schotten-Baumann method) [5217].
- Also refer to: [4846].

m.p. 120–121° [5217].

2-[2-(Benzoyloxy)-4-methoxyphenyl]-1-(2,4-dihydroxyphenyl)ethanone

[52250-27-8]



$C_{22}H_{18}O_6$ mol.wt. 378.38

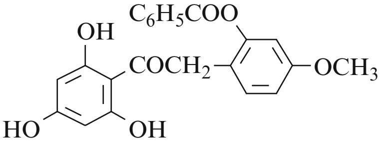
Synthesis

- Preparation by reaction of 2-benzoyloxy-4-methoxy-phenylacetonitrile with resorcinol (Hoesch reaction) [5579].

oil [5579].

2-[2-(Benzoyloxy)-4-methoxyphenyl]-1-(2,4,6-trihydroxyphenyl)ethanone

[32884-28-9]



$C_{22}H_{18}O_7$ mol.wt. 394.38

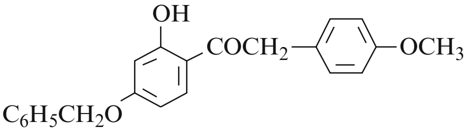
Synthesis

- Obtained by reaction of 2-benzoyloxy-4-methoxy-phenylacetonitrile with phloroglucinol [5562].

m.p. 207–208° [5562]; IR [5562].

1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-2-(4-methoxyphenyl)ethanone

[95307-71-4]



$C_{22}H_{20}O_4$ mol.wt. 348.40

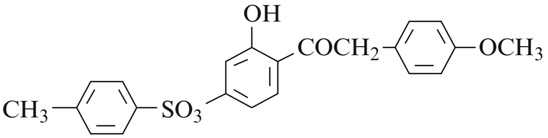
Syntheses

- Preparation by partial alkylation of 2,4-di-hydroxyphenyl 4-methoxybenzyl ketone with benzyl chloride in the presence of potassium carbonate in refluxing acetone for 6 h (80%) [5420] or 8 h [5580], (37%) [5581].
- Also refer to: [5277,5385].

m.p. 103° [5581], 93–95° [5420]; ^1H NMR [5420].

1-[2-Hydroxy-4-[[(4-methylphenyl)sulfonyl]oxy]phenyl]-2-(4-methoxyphenyl)ethanone

[102599-68-8] $C_{22}H_{20}O_6S$ mol.wt. 412.46



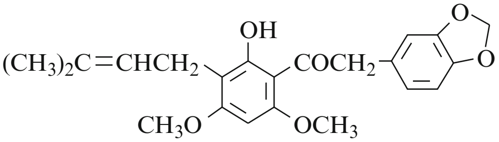
Synthesis

– Obtained by partial esterification of 2,4-dihydroxyphenyl 4-methoxybenzyl ketone with p-toluenesulfonyl chloride in acetone in the presence of potassium carbonate [5217].

m.p. 91° [5217].

2-(1,3-Benzodioxol-5-yl)-1-[2-hydroxy-4,6-dimethoxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[55607-39-1] $C_{22}H_{24}O_6$ mol.wt. 384.43



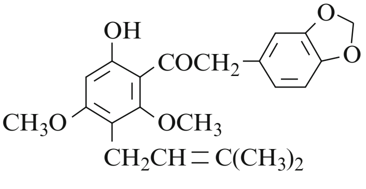
Synthesis

– Obtained by partial methylation of 2,4-dihydroxy-6-methoxy-3-prenyl-phenyl 3,4-methylenedioxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 3.5 h (96%) [5352].

m.p. 118–119° [5352]; ^{1}H NMR [5352]; TLC [5352].

2-(1,3-Benzodioxol-5-yl)-1-[6-hydroxy-2,4-dimethoxy-3-(3-methyl-2-butenyl)phenyl]ethanone

[55607-41-5] $C_{22}H_{24}O_6$ mol.wt. 384.43



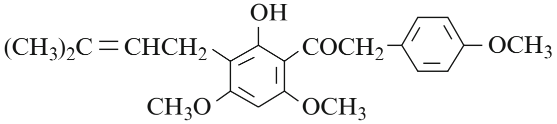
Synthesis

– Obtained by partial methylation of 2,4-dihydroxy-6-methoxy-5-prenylphenyl 3,4-methylenedioxybenzyl ketone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 4 h (96%) [5352].

m.p. 83–84° [5352]; ^{1}H NMR [5352]; TLC [5352].

1-[2-Hydroxy-4,6-dimethoxy-3-(3-methyl-2-butenyl)phenyl]-2-(4-methoxyphenyl)ethanone

[51323-85-4] $C_{22}H_{26}O_5$ mol.wt. 370.45



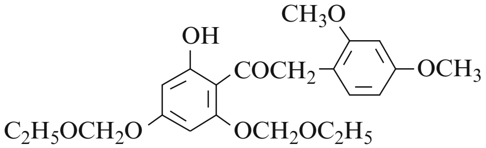
Synthesis

– Obtained by partial methylation of 3-prenyl-2,4-dihydroxy-6,4′-di-methoxydesoxybenzoinwith dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 3.5 h [5524].

m.p. 94–95° [5524].

2-(2,4-Dimethoxyphenyl)-1-[4,6-bis(ethoxymethoxy)-2-hydroxyphenyl] ethanone

$C_{22}H_{28}O_8$ mol.wt. 420.46

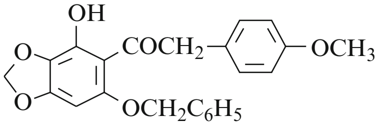


Synthesis

– Refer to: [5534] (compound **1d**).

1-[4-Hydroxy-6-(phenylmethoxy)-1,3-benzodioxol-5-yl]-2-(4-methoxyphenyl) ethanone

$C_{23}H_{20}O_6$ mol.wt. 392.41



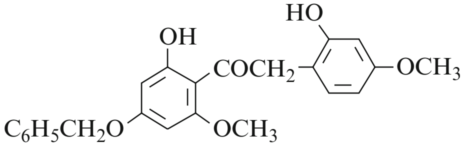
Synthesis

– Obtained by alkaline degradation of 5-benzyloxy-4′-methoxy-7,8-methylenedioxy-2-methylisoflavone with 10% aqueous potassium hydroxide in refluxing ethanol for 2 h under a stream of nitrogen gas (74%) [5507].

m.p. 148–149° [5507]; IR [5507], UV [5507].

1-[2-Hydroxy-6-methoxy-4-(phenylmethoxy)phenyl]-2-(2-hydroxy-4-methoxyphenyl)ethanone

[32884-33-6]



$C_{23}H_{22}O_6$ mol.wt. 394.42

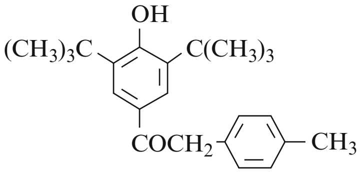
Synthesis

- Obtained by alkaline degradation of 2′-benzoyloxy-7-benzyloxy-4′,5-di-methoxy-2-methoxycarbonylisoflavone (m.p. 183–184°) with potassium hydroxide in refluxing dilute ethanol for 2 h (98%) [5562].

m.p. 120–122° [5562]; ^{1}H NMR [5562], IR [5562].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-2-(4-methylphenyl)ethanone

[81116-01-0]



$C_{23}H_{30}O_2$ mol.wt. 338.49

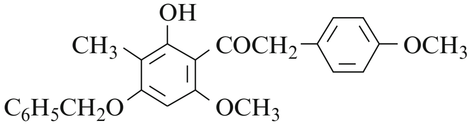
Syntheses

- Obtained by acylation of 2,6-di-tert-butylphenol with p-methylphenylacetyl chloride according to [5402], (35%) [5403].
- Also refer to: [5582].

m.p. 114–115° [5403]; IR [5403].

1-[2-Hydroxy-6-methoxy-3-methyl-4-(phenylmethoxy)phenyl]-2-(4-methoxyphenyl)ethanone

[102749-29-1]



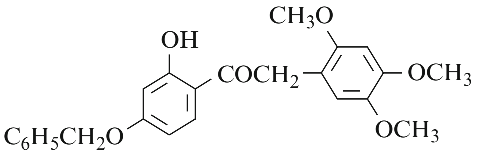
$C_{24}H_{24}O_5$ mol.wt. 392.45

Synthesis

- Obtained by reaction of benzyl bromide with 2,4-dihydroxy-6-methoxy-3-methylphenyl 4-methoxybenzyl ketone in the presence of potassium carbonate in boiling acetone for 3 h (77%) [5488].

m.p. 118° [5488].

1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-2-(2,4,5-trimethoxyphenyl)ethanone



$C_{24}H_{24}O_6$ mol.wt. 408.45

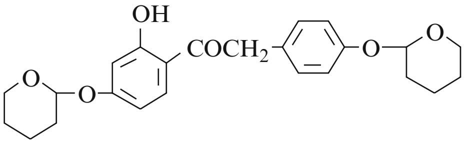
Synthesis

- Obtained by reaction of 2,4-dihydroxyphenyl 2,4,5-trimethoxybenzyl ketone with benzyl chloride in the presence of potassium carbonate in refluxing acetone for 8 h (94%) [5560].

m.p. 149–150° [5560].

1-[2-Hydroxy-4-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]-2-[4-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone

[130064-21-0]



$C_{24}H_{28}O_6$ mol.wt. 412.48

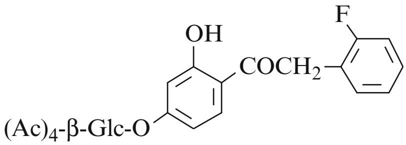
Synthesis

- Preparation by reaction of 3,4-dihydro-2*H*-pyran with 1-(2,4-dihydroxyphenyl)-2-(4-hydroxyphenyl)ethanone in concentrated hydrochloric acid and stirring in an ice bath for 4 h (87%) [5280].

m.p. 118° [5280]; ^{1}H NMR [5280], IR [5280], MS [5280].

2-(2-Fluorophenyl)-1-[2-hydroxy-4-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy]-phenyl]ethanone

[121060-06-8]



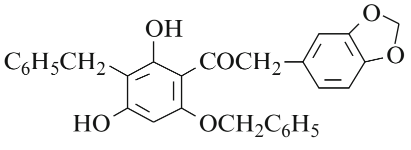
$C_{28}H_{29}FO_{12}$ mol.wt. 576.53

Synthesis

- Obtained by glycosidation of 1-(2,4-dihydroxy-phenyl)-2-(2-fluorophenyl)ethanone with acetobromo-α-D-glucose in aqueous acetone containing potassium hydroxide [5418].

2-(1,3-Benzodioxol-5-yl)-1-[2,4-dihydroxy-6-(phenylmethoxy)-3-(phenylmethyl)phenyl]ethanone

[39549-01-4]



$C_{29}H_{24}O_6$ mol.wt. 468.51

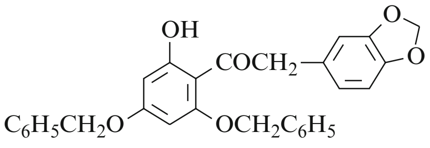
Synthesis

- Obtained by benzylation of 2,4,6-trihydroxy-phenyl 3,4-methylenedioxybenzyl ketone with benzyl chloride in the presence of potassium carbonate in refluxing acetone for 7 h (10%) [5408].

m.p. 145–146° [5408]; ^{1}H NMR [5408], UV [5408].

2-(1,3-Benzodioxol-5-yl)-1-[2-hydroxy-4,6-bis(phenylmethoxy)phenyl] ethanone

[39549-00-3]



$C_{29}H_{24}O_6$ mol.wt. 468.51

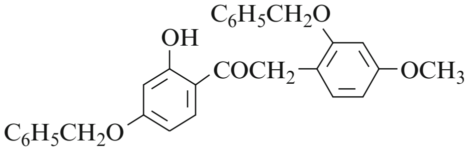
Synthesis

- Obtained by benzylation of 2,4,6-tri-hydroxyphenyl 3,4-methylenedioxy-benzyl ketone with benzyl chloride in the presence of potassium carbonate in refluxing acetone for 7 h (17%) [5408].

m.p. 78–79° [5408]; ^{1}H NMR [5408], UV [5408].

1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-2-[4-methoxy-2-(phenylmethoxy) phenyl]ethanone

[67685-29-4]



$C_{29}H_{26}O_5$ mol.wt. 454.52

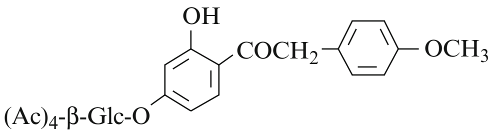
Syntheses

- Obtained by decarbonylation of the acetal—2-(2-benzyloxy-4-methoxyphenyl)-1-(2,4-dibenzyloxyphenyl)-3,3-dimethoxy-propan-1-one—(colourless oil) in refluxing methanol (200 ml) containing 60% perchloric acid (30 ml) for 1.5 h (12%) [5521].
- Also obtained by selective debenzylation of 2,2′,4-tribenzyloxy-4′-methoxydeoxybenzoin (m.p. 111°) in acetonitrile in the presence of boron trifluoride etherate and sodium iodide at r.t. (88%) [5521].

m.p. 101° [5521]; ^{1}H NMR [5521], MS [5521]; TLC [5521].

1-[2-Hydroxy-4-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy]phenyl]-2-(4-methoxyphenyl)ethanone

[42868-73-5]

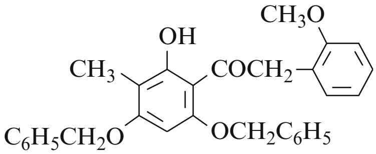


$C_{29}H_{32}O_{13}$ mol.wt. 588.57

Synthesis

- Obtained by glycosidation of 1-(2,4-dihydroxyphenyl)-2-(4-methoxyphenyl)ethanone with acetobromo-α-D-glucose in aqueous acetone containing potassium hydroxide [5418].

1-[2-Hydroxy-3-methyl-4,6-bis(phenylmethoxy)phenyl]-2-(2-methoxyphenyl) ethanone



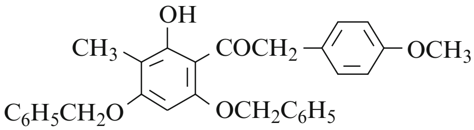
$C_{30}H_{28}O_5$ mol.wt. 468.55

Synthesis

- Obtained by reaction of benzyl bromide (2 mol) with 2,4,6-trihydroxy-3-methylphenyl 2-methoxybenzyl ketone in the presence of potassium carbonate in boiling acetone for 3 h (31%) [5549].

m.p. 146° [5549].

1-[2-Hydroxy-3-methyl-4,6-bis(phenylmethoxy)phenyl]-2-(4-methoxyphenyl)ethanone



$C_{30}H_{28}O_5$ mol.wt. 468.55

Synthesis

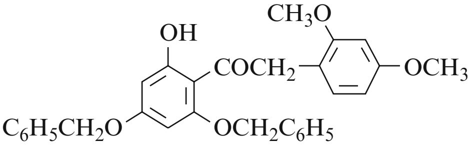
- Obtained by reaction of benzyl bromide (2 mol) with 2,4,6-trihydroxy-3-methylphenyl 4-methoxybenzyl ketone in the presence of potassium carbonate in boiling acetone for 3 h (37%) [5549].

m.p. 129° [5549].

2-(2,4-Dimethoxyphenyl)-1-[2-hydroxy-4,6-bis(phenylmethoxy)phenyl] ethanone

[39604-84-7]

$C_{30}H_{28}O_6$ mol.wt. 484.55



Synthesis

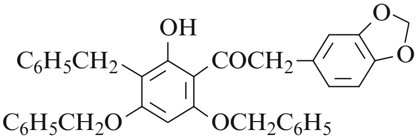
– Obtained by reaction of benzyl chloride with 2,4,6-trihydroxyphenyl 2,4-dimethoxybenzyl ketone in the presence of potassium carbonate in refluxing acetone for 5 h (38%) [5381].

m.p. 136–137° [5381].

2-(1,3-Benzodioxol-5-yl)-1-[2-hydroxy-4,6-bis(phenylmethoxy)-3-(phenylmethyl)phenyl]ethanone

[39548-99-7]

$C_{36}H_{30}O_6$ mol.wt. 558.63



Synthesis

– Obtained (poor yield) by benzylation of 2,4,6-trihydroxyphenyl 3′, 4′-methylenedioxy-benzyl ketone with benzyl chloride in the presence of potassium carbonate in refluxing acetone for 7 h (10%) [5408].

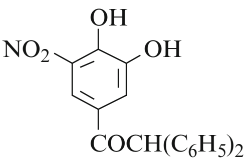
m.p. 135–136° [5408]; ^{1}H NMR [5408], UV [5408].

18.3 Compounds Derived from Di- and Triphenylacetic Acids

1-(3,4-Dihydroxy-5-nitrophenyl)-2,2-diphenylethanone

[400871-22-9]

$C_{20}H_{15}NO_5$ mol.wt. 349.34



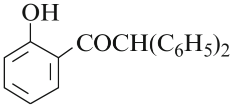
Synthesis

– Preparation by treatment of 2,2-diphenyl-1-(4-hydroxy-3-methoxy-5-nitrophenyl)ethanone with aluminium chloride in refluxing ethyl acetate/pyridine mixture for 2 h (90–96%) [5193].

m.p. 204–205° [5193]; ^{1}H NMR [5193], ^{13}C NMR [5193], IR [5193].

1-(2-Hydroxyphenyl)-2,2-diphenylethanone

[4970-24-5] $C_{20}H_{16}O_2$ mol.wt. 288.34



Syntheses

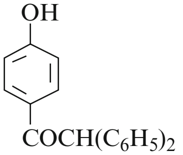
- Obtained (by-product) by Fries rearrangement of phenyl diphenylacetate with aluminium chloride in nitrobenzene for 4 h at 60° (3%) [5201].
- Also obtained (poor yield) by refluxing phenyl diphenyl-acetate (pyrolysis, 300°) [5204].

N.B.: By using "*Kupferbronze*" or various silicates ("*Bleicherde*", for example) as catalysts, the yield increases with appreciable change from 140°.

m.p. 99–100° [5201].

1-(4-Hydroxyphenyl)-2,2-diphenylethanone

[4873-38-5] $C_{20}H_{16}O_2$ mol.wt. 288.34



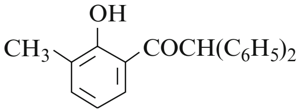
Syntheses

- Preparation by Fries rearrangement of phenyl diphenyl-acetate with aluminium chloride,
  - in nitrobenzene at 60° for 4 h (70%) [5201];
  - in nitroethane at r.t. for 24 h (86%) or at 60° for 4 h (79%) [5202].

m.p. 178–180° [5202], 174–177° [5201].

1-(2-Hydroxy-3-methylphenyl)-2,2-diphenylethanone

[133859-03-7] $C_{21}H_{18}O_2$ mol.wt. 302.37



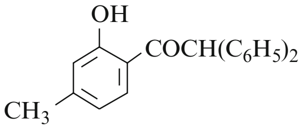
Synthesis

- Obtained by Fries rearrangement of o-tolyl diphenylacetate with aluminium chloride in nitrobenzene at 60° for 4 h (20%) [5201].

IR [5201].

1-(2-Hydroxy-4-methylphenyl)-2,2-diphenylethanone

[133859-04-8] $C_{21}H_{18}O_2$ mol.wt. 302.37



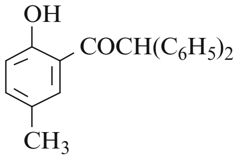
Synthesis

- Obtained by Fries rearrangement of m-tolyl diphenylacetate with aluminium chloride in nitrobenzene at 60° for 4 h (7%) [5201].

m.p. 179–180° [5201]; IR [5201].

1-(2-Hydroxy-5-methylphenyl)-2,2-diphenylethanone

[133859-05-9] $C_{21}H_{18}O_2$ mol.wt. 302.37



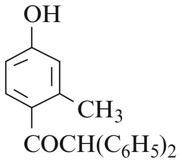
Syntheses

- Obtained by Fries rearrangement of p-tolyl diphenylacetate (m.p. 76°) [4548] with aluminium chloride,
  - without solvent in boiling water bath for 1 h [4548];
  - in nitrobenzene at 60° for 4 h (22%) [5201].

IR [5201].

1-(4-Hydroxy-2-methylphenyl)-2,2-diphenylethanone

[133859-06-0] $C_{21}H_{18}O_2$ mol.wt. 302.37



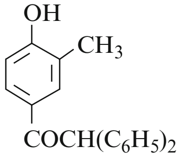
Syntheses

- Obtained (poor yields) by Fries rearrangement of m-tolyl diphenylacetate with aluminium chloride,
  - in nitromethane or in nitroethane at r.t. for 12 h (19–21%) [5202];
  - in nitrobenzene at r.t. for 10 h (10%) or at 60° for 4 h (10%) [5202], (6%) [5201].

m.p. 150–151° [5202], 125–127° [5201].
One of the reported melting points is obviously wrong.

1-(4-Hydroxy-3-methylphenyl)-2,2-diphenylethanone

[122918-54-1] $C_{21}H_{18}O_2$ mol.wt. 302.37



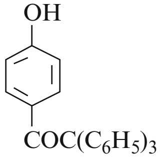
Synthesis

- Preparation by Fries rearrangement of o-cresyl diphenyl-acetate with aluminium chloride in nitrobenzene at 60° for 4 h (49%) [5201] or in nitroethane at r.t. for 12 h (73%) [5202].

m.p. 211–212° [5201].

1-(4-Hydroxyphenyl)-2,2,2-triphenylethanone

[133859-07-1] $C_{26}H_{20}O_2$ mol.wt. 364.44



Synthesis

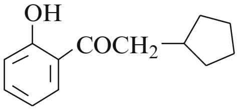
- Obtained (poor yield) by Fries rearrangement of phenyl triphenylacetate with aluminium chloride in nitrobenzene for 4 h at 60° (5%) [5201].

IR [5201].

18.4 Compounds Derived from Cycloalkylacetic Acids

2-Cyclopentyl-1-(2-hydroxyphenyl)ethanone

[56234-70-9] $C_{13}H_{16}O_2$ mol.wt. 204.27



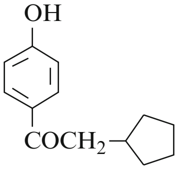
Synthesis

– Obtained by reaction of cyclopentylacetyl chloride with phenol in the presence of aluminium chloride at 140° for 15 min (46%) [5583].

b.p.,$_1$ 112–114° [5583].

2-Cyclopentyl-1-(4-hydroxyphenyl)ethanone

[56184-10-2] $C_{13}H_{16}O_2$ mol.wt. 204.27



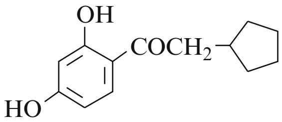
Synthesis

– Obtained by reaction of cyclopentylacetyl chloride with phenol in the presence of aluminium chloride at 140° for 15 min (36%) [5583].

b.p.,$_1$ 175–185° [5583].

2-Cyclopentyl-1-(2,4-dihydroxyphenyl)ethanone

[59108-69-9] $C_{13}H_{16}O_3$ mol.wt. 220.27

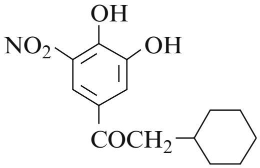


Synthesis

– Obtained by reaction of cyclopentylacetonitrile with resorcinol (Hoesch reaction) [4852].

2-Cyclohexyl-1-(3,4-dihydroxy-5-nitrophenyl)ethanone

[400871-12-7] $C_{14}H_{17}NO_5$ mol.wt. 279.29



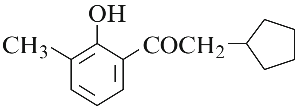
Synthesis

– Preparation by treatment of 2-cyclohexyl-1-(4-hydroxy-3-methoxy-5-nitrophenyl)ethanone with aluminium chloride in refluxing ethyl acetate/pyridine mixture for 2 h (90–96%) [5193].

m.p. 113–114° [5193];
^{1}H NMR [5193], ^{13}C NMR [5193], IR [5193].

2-Cyclopentyl-1-(2-hydroxy-3-methylphenyl)ethanone

[56184-11-3] $C_{14}H_{18}O_2$ mol.wt. 218.30



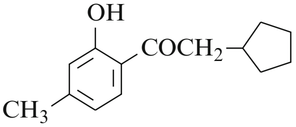
Synthesis

– Preparation by reaction of cyclopentylacetyl chloride with o-cresol in the presence of aluminium chloride at 180° for 15 min (40–45%) [5583].

b.p.$_1$ 121–123° [5583].

2-Cyclopentyl-1-(2-hydroxy-4-methylphenyl)ethanone

[56184-13-5] $C_{14}H_{18}O_2$ mol.wt. 218.30



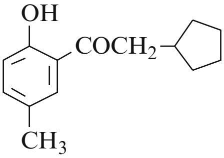
Synthesis

– Preparation by reaction of cyclopentylacetyl chloride with m-cresol in the presence of aluminium chloride at 140° for 15 min (81%) [5583].

b.p.$_1$ 126–128° [5583].

2-Cyclopentyl-1-(2-hydroxy-5-methylphenyl)ethanone

[56184-14-6] $C_{14}H_{18}O_2$ mol.wt. 218.30



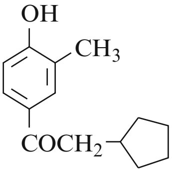
Synthesis

– Preparation by reaction of cyclopentylacetyl chloride with p-cresol in the presence of aluminium chloride at 140° for 15 min (80%) [5583].

b.p.$_1$ 124–126° [5583].

2-Cyclopentyl-1-(4-hydroxy-3-methylphenyl)ethanone

[56184-12-4] $C_{14}H_{18}O_2$ mol.wt. 218.30



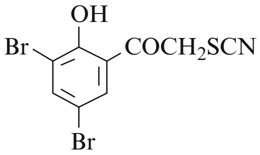
Synthesis

– Preparation by reaction of cyclopentylacetyl chloride with o-cresol in the presence of aluminium chloride at 140° for 15 min (56%) [5583].

b.p.$_1$ 180–190° [5583].

Chapter 19
Compounds Derived from S-Substituted Mercaptoacetic Acids

2-(3,5-Dibromo-2-hydroxyphenyl)-2-oxoethyl thiocyanate



$C_9H_5Br_2NO_2S$ mol.wt. 351.02

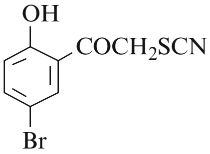
Synthesis

- Obtained by reaction of ammonium thiocyanate with 3,5,α-tribromo-2-hydroxyacetophenone in aqueous acetone at r.t. for 6 h (69%) [4383].

m.p. 142–143° [4383]; IR [4383].

2-(5-Bromo-2-hydroxyphenyl)-2-oxoethyl thiocyanate

[260430-29-3] $C_9H_6BrNO_2S$ mol.wt. 272.12



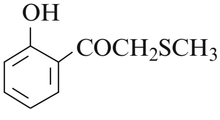
Synthesis

- Obtained by reaction of ammonium thiocyanate with 5,α-di-bromo-2-hydroxyacetophenone in aqueous acetone at r.t. for 6 h (88%) [4383].

m.p. 131–132° [4383]; IR [4383].

1-(2-Hydroxyphenyl)-2-(methylthio)ethanone

[56986-82-4] $C_9H_{10}O_2S$ mol.wt. 182.96



Synthesis

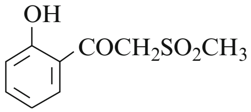
- Preparation by adding a solution of 15% sodium methylmercaptide to a solution of 2-hydroxy-α-bromo-acetophenone in methanol in an ice bath. The reaction mixture was stirred for 30 min at r.t. (98%) [4395].

yellow liquid [4395]; MS [4395].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_19,

1-(2-Hydroxyphenyl)-2-(methylsulfonyl)ethanone

[39068-36-5] $C_9H_{10}O_4S$ mol.wt. 214.24



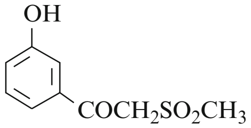
Synthesis

- Obtained by condensation of methyl 2-hydroxybenzoate with dimethylsulfone carbanion in DMSO (65%) [5584].

m.p. 139–140° [5584].

1-(3-Hydroxyphenyl)-2-(methylsulfonyl)ethanone

[52945-17-2] $C_9H_{10}O_4S$ mol.wt. 214.24



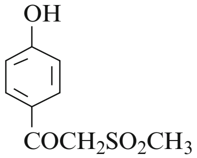
Synthesis

- Obtained by condensation of methyl 3-hydroxybenzoate with dimethylsulfone carbanion in DMSO (60%) [5584].

m.p. 168–169° [5584].

1-(4-Hydroxyphenyl)-2-(methylsulfonyl)ethanone

[52945-18-3] $C_9H_{10}O_4S$ mol.wt. 214.24



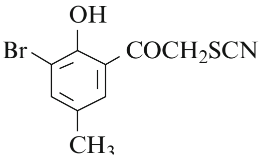
Synthesis

- Obtained by condensation of methyl 4-hydroxybenzoate with dimethylsulfone carbanion in DMSO (58%) [5584].

m.p. 173–174° [5584].

2-(3-Bromo-2-hydroxy-5-methylphenyl)-2-oxoethyl thiocyanate

$C_{10}H_8BrNO_2S$ mol.wt. 286.15



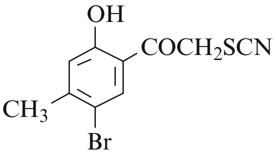
Synthesis

- Obtained by reaction of ammonium thiocyanate with 3,α-dibromo-2-hydroxy-5-methylacetophenone in aqueous acetone at r.t. for 6 h (66%) [4383].

m.p. 148–149° [4383]; IR [4383].

2-(5-Bromo-2-hydroxy-4-methylphenyl)-2-oxoethyl thiocyanate

$C_{10}H_8BrNO_2S$ mol.wt. 286.15



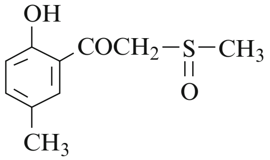
Synthesis

- Obtained by reaction of ammonium thiocyanate with 5,α-di-bromo-2-hydroxy-4-methylacetophenone in aqueous acetone at r.t. for 6 h (73%) [4383].

m.p. 122–123° [4383]; IR [4383].

1-(2-Hydroxy-5-methylphenyl)-2-(methylsulfinyl)ethanone

[52159-50-9]



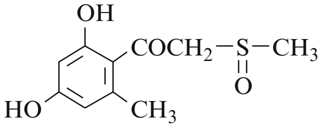
$C_{10}H_{12}O_3S$ mol.wt. 212.27

Synthesis

- Refer to: [5585].

1-(2,4-Dihydroxy-6-methylphenyl)-2-(methylsulfinyl)ethanone

[478795-87-8]



$C_{10}H_{12}O_4S$ mol.wt. 228.27

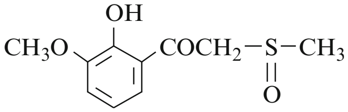
Synthesis

- Obtained by reaction of ethyl 2,4-dihydroxy-6-methyl-benzoate with sodium methylsulfinylmethide [5586] according to [5587].

oil [5586].

1-(2-Hydroxy-3-methoxyphenyl)-2-(methylsulfinyl)ethanone

[65220-47-5]



$C_{10}H_{12}O_4S$ mol.wt. 228.27

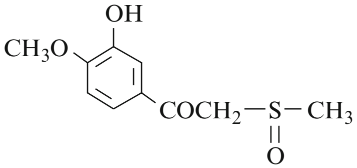
Synthesis

- Preparation by reaction of methyl 3-methoxysalicylate (methyl 2-hydroxy-3-methoxybenzoate) with methylsulfinyle carbanion, itself obtained from DMSO and sodium hydride (83%) [5588].

m.p. 140–142° [5588]; TLC [5588].

1-(3-Hydroxy-4-methoxyphenyl)-2-(methylsulfinyl)ethanone

[66100-55-8]



$C_{10}H_{12}O_4S$ mol.wt. 228.27

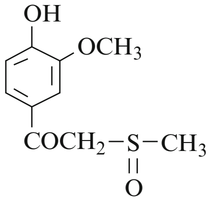
Synthesis

- Preparation by reaction of methyl isovanillinate (methyl 3-hydroxy-4-methoxybenzoate) with methylsulfinyle carbanion, itself obtained from DMSO and sodium hydride (89%) [5589].

m.p. 158–161° [5589];
^1H NMR [5589], IR [5589].

1-(4-Hydroxy-3-methoxyphenyl)-2-(methylsulfinyl)ethanone

[66100-54-7]



m.p. 151–152° [5589];
^{1}H NMR [5589], IR [5589].

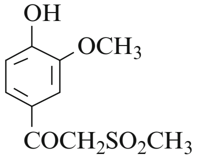
$C_{10}H_{12}O_4S$ mol.wt. 228.27

Synthesis

- Preparation by reaction of methyl vanillinate (methyl 4-hydroxy-3-methoxybenzoate) with methylsulfinyle, carbanion itself obtained from DMSO and sodium hydride (90%) [5589].

1-(4-Hydroxy-3-methoxyphenyl)-2-(methylsulfonyl)ethanone

[52945-22-9]



m.p. 157–158° [5584].

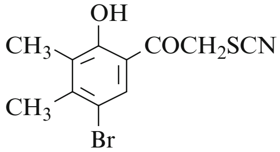
$C_{10}H_{12}O_5S$ mol.wt. 244.27

Synthesis

- Obtained by condensation of methyl 4-hydroxy-3-methoxy-benzoate with dimethylsulfone carbanion in DMSO (76%) [5584].

2-(5-Bromo-2-hydroxy-3,4-dimethylphenyl)-2-oxoethyl thiocyanate

[260430-31-7]



m.p. 149–150° [4383]; IR [4383].

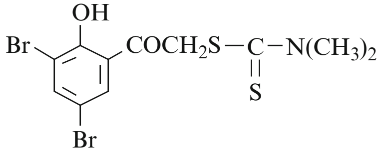
$C_{11}H_{10}BrNO_2S$ mol.wt. 300.18

Synthesis

- Obtained by reaction of ammonium thiocyanate with 5,α-di-bromo-2-hydroxy-3,4-dimethylacetophenone in aqueous acetone at r.t. for 6 h (52%) [4383].

2-(3,5-Dibromo-2-hydroxyphenyl)-2-oxoethyl dimethylcarbamodithioate

[214959-26-9]



m.p. 164° [4382];
^{1}H NMR [4382], IR [4382], UV [4382].

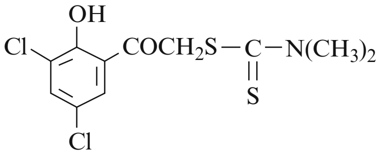
$C_{11}H_{11}Br_2NO_2S_2$ mol.wt. 413.15

Synthesis

- Obtained by reaction of 3,5-dibromo-2-hydroxy-α-bromoacetophenone with sodium or piperidinium N,N-dimethyldithiocarbamate in methanol at r.t. for 12 h (72%) [4382].

2-(3,5-Dichloro-2-hydroxyphenyl)-2-oxoethyl dimethylcarbamodithioate

[87669-75-8]



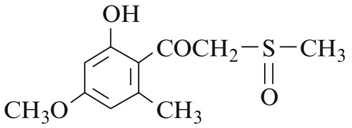
$C_{11}H_{11}Cl_2NO_2S_2$ mol.wt. 324.2

Syntheses

- Refer to: [5590,5591].

1-(2-Hydroxy-4-methoxy-6-methylphenyl)-2-(methylsulfinyl)ethanone

[104783-89-3]



$C_{11}H_{14}O_4S$ mol.wt. 242.30

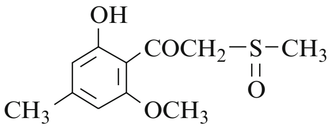
Syntheses

- Preparation by reaction of ethyl 2-hydroxy-4-methoxy-6-methylbenzoate (m.p. 73–74°) with sodium methyl-sulfinylmethide, itself obtained from DMSO and sodium hydride (85%) [5587].
- Also refer to: [5586].

m.p. 146–148° [5587]; ^{1}H NMR [5587], IR [5587].

1-(2-Hydroxy-6-methoxy-4-methylphenyl)-2-(methylsulfinyl)ethanone

[205880-83-7]



$C_{11}H_{14}O_4S$ mol.wt. 242.30

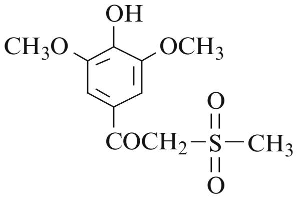
Syntheses

- Preparation by treatment of methyl 2-hydroxy-6-methoxy-4-methylbenzoate (methyl mono-O-methyl-p-orsellinate) (m.p. 94–96°) with sodium methylsulfinyl-methide (SM) formed in situ. SM was obtained by action of sodium hydride (3 equiv) with DMSO (7 equiv) in benzene at 50° for 1 h. (78%) [5592].
- Also refer to: [5593].

m.p. 62–64° [5592].

1-(4-Hydroxy-3,5-dimethoxyphenyl)-2-(methylsulfonyl)ethanone

[52945-23-0]



$C_{11}H_{14}O_6S$ mol.wt. 274.29

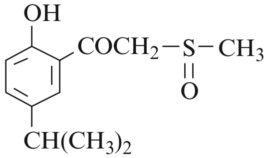
Synthesis

- Obtained by condensation of methyl 4-hydroxy-3,5-di-methoxybenzoate with dimethylsulfonyl carbanion in DMSO (74%) [5584].

m.p. 154–155° [5584].

1-[2-Hydroxy-5-(1-methylethyl)phenyl]-2-(methylsulfinyl)ethanone

[63220-58-6]



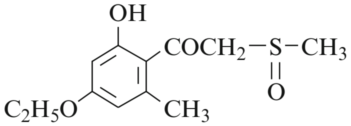
$C_{12}H_{16}O_3S$ mol.wt. 240.32

Syntheses

- Refer to: [5585,5594].

1-(4-Ethoxy-2-hydroxy-6-methylphenyl)-2-(methylsulfinyl)ethanone

[478795-93-6]



$C_{12}H_{16}O_4S$ mol.wt. 256.32

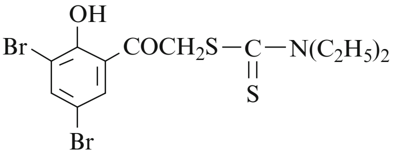
Synthesis

- Obtained by reaction of ethyl 4-ethoxy-2-hydroxy-6-methylbenzoate with sodium methylsulfinylmethide [5586] according to [5587].

oil [5586].

2-(3,5-Dibromo-2-hydroxyphenyl)-2-oxoethyl diethylcarbamodithioate

[214959-27-0]



$C_{13}H_{15}Br_2NO_2S_2$ mol.wt. 441.21

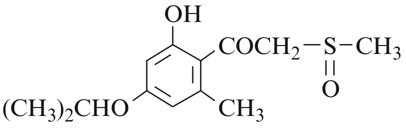
Synthesis

- Obtained by action of 3,5-dibromo-2-hydroxy-α-bromoacetophenone with sodium or piperidinium N,N-diethyldithiocarbamate in methanol at r.t. for 12 h (58%) [4382].

m.p. 132° [4382]; ^{1}H NMR [4382], IR [4382], UV [4382].

1-[2-Hydroxy-6-methyl-4-(1-methylethoxy)phenyl]-2-(methylsulfinyl)ethanone

[478795-95-8]



$C_{13}H_{18}O_4S$ mol.wt. 270.35

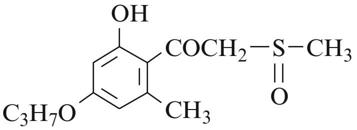
Synthesis

- Obtained by reaction of ethyl 2-hydroxy-4-iso-propoxy-6-methylbenzoate with sodium methyl-sulfinylmethide [5586] according to [5587].

oil [5586].

1-(2-Hydroxy-6-methyl-4-propoxyphenyl)-2-(methylsulfinyl)ethanone

[478795-94-7]



$C_{13}H_{18}O_4S$ mol.wt. 270.35

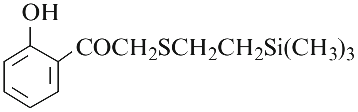
Synthesis

- Obtained by reaction of ethyl 2-hydroxy-6-methyl-4-propoxybenzoate with sodium methylsulfinylmethide [5586] according to [5587].

oil [5586].

1-(2-Hydroxyphenyl)-2-[2-(trimethylsilyl)ethylthio]ethanone

[193075-79-5]



$C_{13}H_{20}O_2SSi$ mol.wt. 268.45

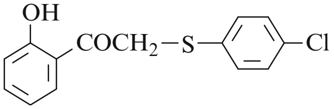
Synthesis

- Preparation by adding an ethanolic solution of sodium 2-(trimethylsilyl)ethanethiolate in a solution of 2-chloro-1-(2-hydroxyphenyl)ethanone in dioxane at r.t. for 2 h under nitrogen (99%) [4570].

pale yellow oil [4570]; ^{1}H NMR [4570], IR [4570].

2-[(4-Chlorophenyl)thio]-1-(2-hydroxyphenyl)ethanone

[113272-14-3]



$C_{14}H_{11}ClO_2S$ mol.wt. 278.76

Syntheses

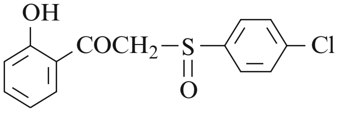
- Preparation by adding dropwise at r.t. an ethanolic solution of sodium 4-chlorothiophenoxide to a solution of 2-hydroxy-α-bromoacetophenone in dioxane and stirring the mixture for a further hour (84%) [5595].
- Also refer to: [5596].

m.p. 83–84° [5595]; ^{1}H NMR [5595], IR [5595], MS [5595].

2-[(4-Chlorophenyl)sulfinyl]-1-(2-hydroxyphenyl)ethanone

[113272-15-4]

[131137-71-8] (±)



$C_{14}H_{11}ClO_3S$ mol.wt. 294.76

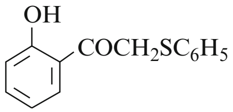
Syntheses

- Preparation by oxidation of α-(4-chlorophenylthio)-2-hydroxyacetophenone in methylene chloride with 3-chloroperoxybenzoic acid at 0° for 5 h (93%) [5595].
- Also refer to: [5596].

m.p. 131–132° [5595];
^{1}H NMR [5595], IR [5595], MS [5595].

1-(2-Hydroxyphenyl)-2-(phenylthio)ethanone

[56307-98-3] $C_{14}H_{12}O_2S$ mol.wt. 244.31



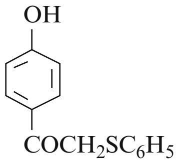
Syntheses

- Preparation by adding an ethanolic solution of sodium benzenethiolate to a solution of 2-hydroxy-α-bromoacetophenone in dioxane at r.t. and stirring for 1 h at the same temperature (77%) [5596,5597].
- Preparation by condensation of 2-hydroxy-α-bromoacetophenone (or 2-hydroxy-α-chloro-acetophenone) with thiophenol in the presence of sodium ethoxide in an ethanol/dioxane mixture (77%) [5598].
- Also refer to: [4395].

m.p. 55° [5597,5598], 52–54° [5596];
^{1}H NMR [5596,5597], IR [5596,5597], UV [5597,5598].

1-(4-Hydroxyphenyl)-2-(phenylthio)ethanone

[137524-65-3] $C_{14}H_{12}O_2S$ mol.wt. 244.31



Syntheses

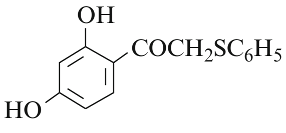
- Preparation by adding thiophenol (0.1 ml) and rhodium (II) acetate dimer (2 mg) to a suspension of resin **6** (52 mg) in benzene and the mixture agitated at 50° for 2 h. Resin was filtered, successively washed with methylene chloride, THF and ethyl ether and dried in vacuo. A 50% (v/v) solution of TFA in methylene chloride was added to the above resin and the mixture was agitated at r.t. for 30 min. After, the resin was filtered and washed with methylene chloride, the combined filtrates were concentrated and purified by preparative TLC (ethyl acetate/toluene) to give the titled compound (64%) [4814].

  N.B.: Resin **6** (resin-bound α-TMS diazoketon 6) (preparation given).
- Also refer to: [4815].

^{1}H NMR [4814], ^{13}C NMR [4814], IR [4814], MS [4814].

1-(2,4-Dihydroxyphenyl)-2-(phenylthio)ethanone

[56307-99-4] $C_{14}H_{12}O_3S$ mol.wt. 260.31



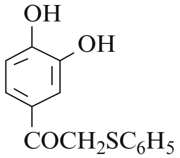
Syntheses

- Preparation by condensation of 2,4-dihydroxy-α-chloro-acetophenone with thiophenol in the presence of sodium ethoxide in a mixture of ethanol/dioxane (93%) [5598].
- Also obtained by condensation of phenylthioacetonitrile with resorcinol (Hoesch reaction) (54%) [5598].

m.p. 152–153° [5598]; UV [5598].

1-(3,4-Dihydroxyphenyl)-2-(phenylthio)ethanone

[131985-77-8] $C_{14}H_{12}O_3S$ mol.wt. 260.31

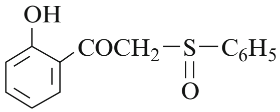


Synthesis

– Refer to: [5599] (Japanese patent).

1-(2-Hydroxyphenyl)-2-(phenylsulfinyl)ethanone

[131137-70-7] $C_{14}H_{12}O_3S$ mol.wt. 260.31



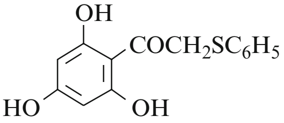
Synthesis

– Preparation by oxidation of 1-(2-hydroxyphenyl)-2-(phenyl-thio)ethanone with *m*-CPBA in methylene chloride at 0° for 5 h and water then added (90%) [5596].

m.p. 117–118° [5596];
^{1}H NMR [5596], IR [5596], MS [5596].

2-(Phenylthio)-1-(2,4,6-trihydroxyphenyl)ethanone

[56308-00-0] $C_{14}H_{12}O_4S$ mol.wt. 276.31



Syntheses

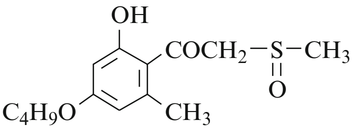
– Preparation by condensation of phenylthioacetonitrile with phloroglucinol (Hoesch reaction) (85%) [5598].

– Also obtained by condensation of 2,4,6-trihydroxy-α-chloro-acetophenone with thiophenol in the presence of sodium ethoxide in an ethanol/dioxane mixture (20%) [5598].

m.p. 173–174° [5598]; UV [5598].

1-(4-Butoxy-2-hydroxy-6-methylphenyl)-2-(methylsulfinyl)ethanone

[478795-98-1] $C_{14}H_{20}O_4S$ mol.wt. 284.38



Synthesis

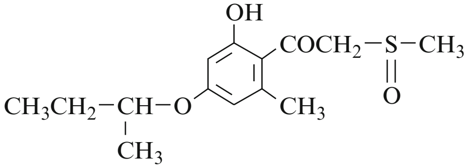
– Obtained by reaction of ethyl 4-butoxy-2-hydroxy-6-methylbenzoate with sodium methylsulfinylmethide [5586] according to [5587].

oil [5586].

1-[2-Hydroxy-6-methyl-4-(1-methylpropoxy)phenyl]-2-(methylsulfinyl)ethanone

[478795-97-0]

$C_{14}H_{20}O_4S$ mol.wt. 284.38



Synthesis

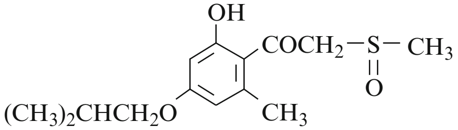
- Obtained by reaction of ethyl 4-sec-butoxy-2-hydroxy-6-methylbenzoate with sodium methylsulfinylmethide [5586] according to [5587].

oil [5586].

1-[2-Hydroxy-6-methyl-4-(2-methylpropoxy)phenyl]-2-(methylsulfinyl)ethanone

[478795-96-9]

$C_{14}H_{20}O_4S$ mol.wt. 284.38



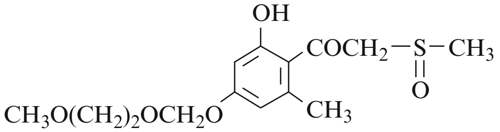
Synthesis

- Obtained by reaction of ethyl 4-isobutoxy-2-hydroxy-6-methylbenzoate with sodium methylsulfinylmethide [5586] according to [5587].

oil [5586].

1-[2-Hydroxy-4-[(2-methoxyethoxy)methoxy]-6-methylphenyl]-2-(methylsulfinyl)-ethanone

$C_{14}H_{20}O_6S$ mol.wt. 316.38



Synthesis

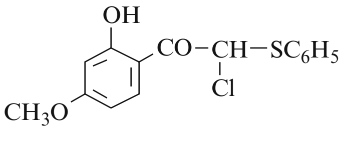
- Preparation by reaction of ethyl 2-hydroxy-4-(2-meth oxyethoxy)-methoxy-6-methylbenzoate with methylsulfinyl carbanion, itself obtained from DMSO and sodium hydride (86%) [5587].

m.p. 106–107° [5587]; ^{1}H NMR [5587], IR [5587].

2-Chloro-1-(2-hydroxy-4-methoxyphenyl)-2-(phenylthio)ethanone

[153432-53-2]

$C_{15}H_{13}ClO_3S$ mol.wt. 308.79



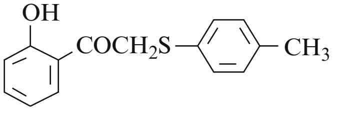
Synthesis

- Preparation by reaction of N-chlorosuccinimide with 2′-hydroxy-4′-methoxy-2-(phenylthio)acetophenone in carbon tetrachloride under argon at r.t. for 2 h (83%) [5600].

m.p. 49–50°5 [5600]; ^{1}H NMR [5600], ^{13}C NMR [5600], IR [5600], MS [5600].

1-(2-Hydroxyphenyl)-2-[(4-methylphenyl)thio]ethanone

[108378-94-5] $C_{15}H_{14}O_2S$ mol.wt. 258.34



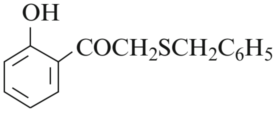
Synthesis

- Preparation by adding an ethanolic solution of sodium 4-methylbenzenethiolate to a solution of 2-hydroxy-α-bromoacetophenone in dioxane at r.t. and stirring for 1 h at the same temperature (83%) [5601,5596].

m.p. 66–67° [5596]; ^{1}H NMR [5596], IR [5596].

1-(2-Hydroxyphenyl)-2-[(phenylmethyl)thio]ethanone

[111809-47-3] $C_{15}H_{14}O_2S$ mol.wt. 258.34



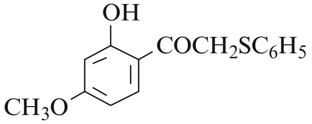
Synthesis

- Preparation by adding a solution of sodium phenylmethane-thiolate in ethanol to a solution of o-hydroxy-α-chloro-acetophenone in dioxane at 20° during 40 min and then stirring at the same temperature for 30 min more (84%) [4571].

m.p. 64° [4571]; ^{1}H NMR [4571], IR [4571].

1-(2-Hydroxy-4-methoxyphenyl)-2-(phenylthio)ethanone

[56308-01-1] $C_{15}H_{14}O_3S$ mol.wt. 274.34



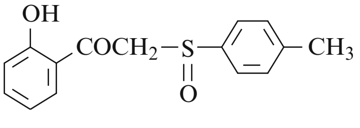
Syntheses

- Preparation by reaction of sodium thiophenolate with 2-hydroxy-4-methoxy-α-chloroacetophenone in tetrahydrofuran under argon, at r.t. for 10 min (73%) [5600].
- Also obtained by partial methylation of 2,4-dihydroxy-α-phenylthioacetophenone with dimethyl sulfate in the presence of potassium carbonate in acetone for 3 h (64%) [5598].
- Also obtained by alkaline degradation of 3-(phenylthio)-7-methoxychromone (m.p. 101–102°) with N-sodium hydroxide in dilute methanol for 3 h (96%) [5598].

m.p. 42–44° [5600], 42–43° [5598]; ^{1}H NMR [5600], ^{13}C NMR [5600], UV [5598].

1-(2-Hydroxyphenyl)-2-[(4-methylphenyl)sulfinyl]ethanone

[108378-95-6] (±)
[108448-95-9] (R) $C_{15}H_{14}O_3S$ mol.wt. 274.34



Syntheses

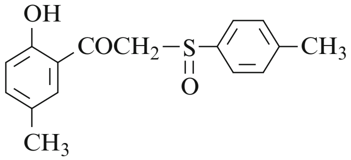
- Preparation by oxidation of 1-(2-hydroxyphenyl)-2-[(4-methylphenyl)thio]ethanone with *m*-CPBA in methylene chloride at 0° for 5 h and water then added (95%) [5596].

– Also refer to: [5601].

m.p. 118–119° [5596]; ^{1}H NMR [5596], IR [5596].

1-(2-Hydroxy-5-methylphenyl)-2-[(R)-(4-methylphenyl)sulfinyl]ethanone

[371258-74-1] $C_{16}H_{16}O_3S$ mol.wt. 288.37



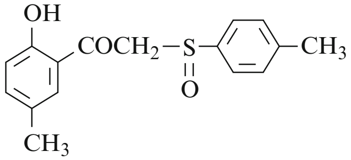
Syntheses

– Preparation at −78° by reaction between methyl 2-hydroxy-5-methylbenzoate and (R)-(+)-methyl p-tolyl sulfoxide in the presence of lithium diisopropylamide (LDA) in THF (91%) [5602].

m.p. 128° [5602]; $(\alpha)_D^{22}$= +134° to +148° (c = 1 in chloroform) [5602]; ^{1}H NMR [5602], IR [5602], MS [5602].

1-(2-Hydroxy-5-methylphenyl)-2-[(S)-(4-methylphenyl)sulfinyl]ethanone

[371258-72-9] $C_{16}H_{16}O_3S$ mol.wt. 288.37

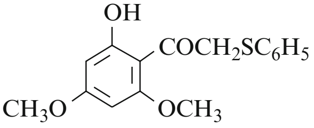


Synthesis

– Refer to: [5602].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-(phenylthio)ethanone

[56308-02-2] $C_{16}H_{16}O_4S$ mol.wt. 304.37



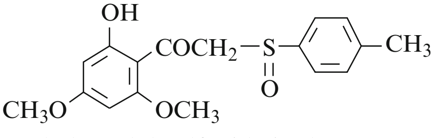
Synthesis

– Preparation by partial methylation of 2-(phenylthio)-2′,4′,6′-trihydroxy-acetophenone with dimethyl sulfate in the presence of potassium carbonate in acetone for 2 h (88%) [5598].

m.p. 75° [5598]; UV [5598].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-[(R)-(4-methylphenyl)sulfinyl]ethanone

[371258-80-9] $C_{17}H_{18}O_5S$ mol.wt. 334.39



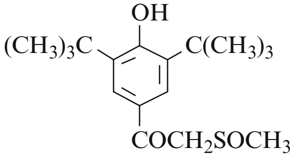
Synthesis

– Preparation at −78° by reaction between methyl 2-hydroxy-4,6-dimethoxybenzoate and (R)-(+)-methyl p-tolyl sulfoxide in the presence of lithium diisopropylamide (LDA) in THF (58%) [5602].

m.p. 107–109° [5602]; $(\alpha)_D^{20}$= −28° to −32° (c = 1 in chloroform) [5602]; ^{1}H NMR [5602], IR [5602], MS [5602]; TLC [5602].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-2-(methylsulfinyl)ethanone

[115207-18-6] $C_{17}H_{26}O_3S$ mol.wt. 310.46



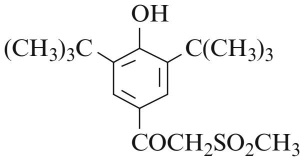
Syntheses

- Obtained by reaction of 3,5-di-tert-butyl-4-hydroxybenzoyl chloride with excess DMSO at r.t. (16–18%) [5603].
- Obtained by acylation of 2,6-di-tert-butylphenol with 2-(methylthio)acetic acid and the intermediate oxidized with m-CPBA [5604].

m.p. 143°6–144°9 [5604], 61–62° [5603].
One of the reported melting points is obviously wrong.
^{1}H NMR [5603], IR [5603].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-2-(methylsulfonyl)ethanone

[191157-34-3] $C_{17}H_{26}O_4S$ mol.wt. 326.46

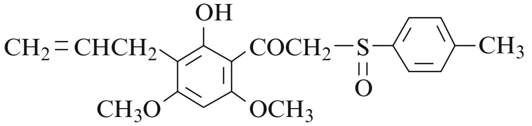


Synthesis

- Refer to: [5604].

1-[2-Hydroxy-4,6-dimethoxy-3-(2-propenyl)phenyl]-2-[(S)-(4-methylphenyl)sulfinyl]ethanone

[371258-84-3] $C_{20}H_{22}O_5S$ mol.wt. 374.46



Synthesis

- Obtained at −78° in THF using lithium diisopropylamide (LDA), either by reaction between methyl 2-hydroxy-4,6-dimethoxy-3-(2-propenyl)-benzoate and (R)-(+)-methyl p-tolyl sulfoxide in the presence of 1,3-dimethyl-3,4,5,6-tetrahydro-2[1*H*]-pyrimidinone (DMPU) (this method yields only a trace of the desired compound), or by reaction between 2-hydroxy-4,6-dimethoxy-3-(2-propenyl)benzaldehyde and (R)-(+)-methyl p-tolyl sulfoxide via the subsequent oxidation ^{a}t r.t. by MnO_2 of the intermediate 1-[2-hydroxy-4,6-dimethoxy-3-(2-propenyl)phenyl]-2-(4-methylsulfinyl)ethanol (57%) [5602].

m.p. 150–151° [5602]; $(\alpha)_D^{30}$=+52° to +56° (c = 1.5 in chloroform) [5602];
^{1}H NMR [5602], ^{13}C NMR [5602], IR [5602], MS [5602];
TLC [5602].

Part VIII
Di- and Polyketones

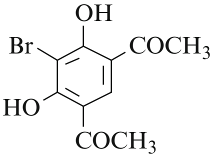
Chapter 20
Aromatic Ketones Containing Only Acetyl Groups

20.1 Acetyl Groups Located on One Ring

20.1.1 Unsubstituted Acetyl Groups and Homologues

1,1′-(5-Bromo-4,6-dihydroxy-1,3-phenylene)bis-ethanone

[117156-78-2] $C_{10}H_9BrO_4$ mol.wt. 273.08



Syntheses

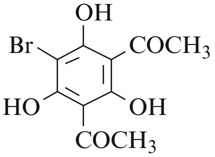
– Preparation by bromination of resodiacetophenone,
  - with NBS in refluxing dioxane for 10 h (97%) [5605];
  - with bromine, for 6 h at r.t. [5606], in cooled acetic acid [4538].

– Also refer to: [5607].

m.p. 205° [4538], 202–203° [5606]; ^1H NMR [5605], IR [5605], UV [5605].

1,1′-(5-Bromo-2,4,6-trihydroxy-1,3-phenylene)bis-ethanone

[98149-38-3] $C_{10}H_9BrO_5$ mol.wt. 289.08



Synthesis

– Preparation by Friedel–Crafts acylation of 2-bromophloroglucinol with acetyl chloride or acetic anhydride in the presence of boron trifluoride (72–78%) [5608].

m.p. 150–152° [5608].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
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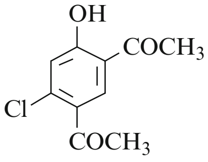
1,1′-(4-Chloro-6-hydroxy-1,3-phenylene)bis-ethanone

[30335-99-0] $C_{10}H_9ClO_3$ mol.wt. 212.63



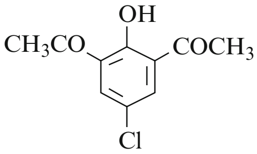
Syntheses

- Obtained by Fries rearrangement,
    - of 4-acetyl-3-chlorophenyl acetate with aluminium chloride at 120° for 20 min (35%) [5609];
    - of 3-chlorophenyl acetate with aluminium chloride at 175–180° for 3 h (by-product) [5610].
- Also obtained from 5-acetyl-6-chloro-2,3-dimethyl-benzofuran by oxidation with chromium trioxide in dilute acetic acid at 50° for 30 min, followed by hydrolysis of the resulting keto ester (18%) [5609].

m.p. 84° [5610], 75° [5609]
b.p.$_9$ 167° [5609]; ^{1}H NMR [5610], MS [5610].

1,1′-(5-Chloro-2-hydroxy-1,3-phenylene)bis-ethanone

[71643-62-4] $C_{10}H_9ClO_3$ mol.wt. 212.63



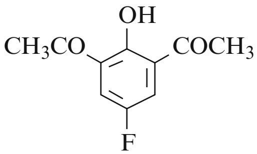
Syntheses

- Preparation by Fries rearrangement of 2-(acetyloxy)-5-chloroacetophenone with aluminium chloride for 1 h at 130°, then 1 h at 140° (80%) [5611].
- Also refer to: [5612, 5613].

b.p.$_6$ 120° [5611].

1,1′-(5-Fluoro-2-hydroxy-1,3-phenylene)bis-ethanone

[106823-62-5] $C_{10}H_9FO_3$ mol.wt. 196.18



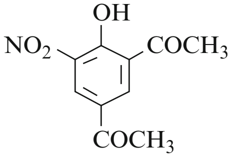
Synthesis

- Preparation by Fries rearrangement of 2-acetyl-4-fluoro-phenyl acetate (b.p. 124–126°) with aluminium chloride at 130–140° for 3 h (71%) [5614].

b.p. 130–135° [5614]; IR [5614].

1,1′-(4-Hydroxy-5-nitro-1,3-phenylene)bis-ethanone

[100245-07-6] $C_{10}H_9NO_5$ mol.wt. 223.19



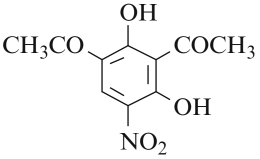
Synthesis

- Preparation by nitration of 5-acetyl-2-hydroxyacetophenone at −20° using standard reagents (51%) [4699].

m.p. 104–105° [4699].

1,1′-(2,4-Dihydroxy-5-nitro-1,3-phenylene)bis-ethanone

[103264-32-0] $C_{10}H_9NO_6$ mol.wt. 239.18



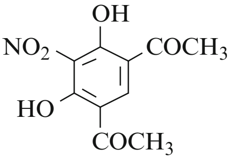
Syntheses

- Preparation by reaction of 1,3-dinitroquinolizin-4-one with sodio-2,4,6-heptanetrione in DMF for 1.5 h between −15° and −10° (57%) [5615].
- Also obtained by nitration of 2,4-diacetylresorcinol with a nitric acid (d = 1.42)/sulfuric acid (d = 1.84) mixture in acetic acid at 0° for 1 h (41%) [5616].
- Also obtained by Fries rearrangement of 4-nitroresorcinol diacetate in nitrobenzene with aluminium chloride at 95–100° for 2 h or at r.t. for 72 h (38%) [5616].

m.p. 142–143° [5615], 139–140° [5616]; ^{1}H NMR [5615], IR [5615].

1,1′-(4,6-Dihydroxy-5-nitro-1,3-phenylene)bis-ethanone

[103262-48-2] $C_{10}H_9NO_6$ mol.wt. 239.18



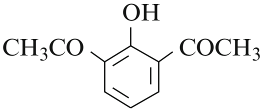
Syntheses

- Preparation by Fries rearrangement of 2-nitroresorcinol diacetate with aluminium chloride,
  - without solvent, at 140° [5617], at 100–110° (30%) [5618];
  - in nitrobenzene, at 140° [5617], at 100–110° for 3 h (60%) [5618] and at 25–28° for 70 h (73%) [5618].
- Preparation by Friedel–Crafts acetylation of 2-nitroresorcinol with acetic anhydride in the presence of aluminium chloride in nitrobenzene at 120–130° for 3 h (76%) [5618].
- Preparation by nitration of 4,6-diacetylresorcinol,
  - with nitric acid (d = 1.42) at 80° (84%) [5605], or first at 80°, then at r.t. for 2 h [5619];
  - with nitric acid in sulfuric acid at 0° [5617];
  - with concentrated nitric acid in a concentrated sulfuric acid/acetic acid mixture at 0° for 1 h [5618];
  - with cooled fuming nitric acid, then at r.t. for 30 min [4538];
  - with fuming nitric acid in acetic acid, first at 0°, then at r.t. for a few minutes [4538].

m.p. 235° [5605], 235°(d) [5619], 234° [5617,5618], 231° [4538];
^{1}H NMR [5605], IR [5605], UV [5605].

1,1′-(2-Hydroxy-1,3-phenylene)bis-ethanone

[103867-89-6] $C_{10}H_{10}O_3$ mol.wt. 178.19



Syntheses

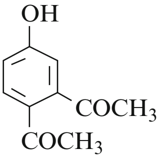
– Obtained by hydrolysis of 2-(3-acetyl-2-hydroxyphenyl)-2-methyl-1,3-dioxolane with a 5% aqueous hydrochloric acid/ethanol mixture at r.t. for 5 min (almost quantitative yield) [5620].

– Also obtained by UV light irradiation of 2-(2-acetoxy-phenyl)-2-methyl-1,3-dioxolane in hexane (7%) [5621] or in hexane in the presence of potassium carbonate for 6 h (7%) [5620].

m.p. 71–73° [5621]; ^{1}H NMR [5621], IR [5621], UV [5621].

1,1′-(4-Hydroxy-1,2-phenylene)bis-ethanone

[90464-79-2] $C_{10}H_{10}O_3$ mol.wt. 178.19

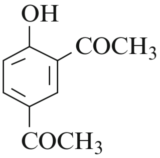


Synthesis

– Refer to: [5622] (Japanese patent).

1,1′-(4-Hydroxy-1,3-phenylene)bis-ethanone

[30186-16-4] $C_{10}H_{10}O_3$ mol.wt. 178.19



Syntheses

– Preparation by reaction of acetyl chloride (2 mol) with o-methoxyacetophenone (1 mol) in the presence of aluminium chloride (2 mol) in boiling carbon disulfide for 12 h (87%) [5623].

– Preparation by Fries rearrangement of various substituted phenyl esters (1 mol) in the presence of aluminium chloride,

- of p-acetylphenyl acetate,
  - at 150° for 3 h: ($AlCl_3$ 3.5 mol) (80%) [5624,5625] or ($AlCl_3$ 4 mol) (60%) [5626];
  - at 140–150° for 2 h ($AlCl_3$ 3.4 mol) (78%) [5627] or for 1 h ($AlCl_3$ 3.3 mol) (40%) [5628];
  - first at 130–140°, then at 160° for 10 min ($AlCl_3$ 2.7 mol) (poor yields) [5623,5629];
- of o-acetylphenyl acetate,
  - first at 50°, then at 80° for 15 min (AlCl3 2.7 mol) (46%) [5623,5629];
  - in nitrobenzene at r.t. overnight (AlCl3 3.3 mol) (43%) [5628];

  - of o-bromophenyl acetate at 180° for 5 h ($AlCl_3$ 3.2 mol) (by-product) (18%) [5630].

- Also obtained by photo-Fries rearrangement of two different esters in hexane for 6 h,
  - of 2-(4-acetoxyphenyl)-2-methyl-1,3-dioxolane (12%) [5620,5621];
  - of 2-(2-acetoxyphenyl)-2-methyl-1,3-dioxolane (4%) [5620,5621].
- Also obtained by Friedel–Crafts acylation of p-hydroxyacetophenone with acetyl chloride in tetrachloroethane in the presence of aluminium chloride (4 mol) at 130° for 4 h (49%) [5626].
- Also obtained by treatment of two different substituted acetophenones with 5% aqueous hydrochloric acid/ethanol (30v/1v) at r.t. for 5 min,
  - of 2-(5-acetyl-2-hydroxyphenyl)-2-methyl-1,3-dioxolane (almost quantitative yield) [5620];
  - of 2-(3-acetyl-4-hydroxyphenyl)-2-methyl-1,3-dioxolane (almost quantitative yield) [5620].
- Also obtained by decarboxylation of 3,5,3′,3′-tetraacetylxanthyrone in boiling water for 4 h (18%) [5631].
- Also refer to: [5632,5633,5634] and also [5635] (Fries rearrangement).

Isolation from natural sources

- From the aerial parts of *Ophryosporus floribundus* (Compositae, tribe Eupatorieae) [5636].
- From the *Artemisia campestris* L. subsp. *glutinosa* (Gay ex Besser) (Compositae) [5637].

m.p. 95° [5623], 93° [5630], 92–93° [5628], 92 [5624], 91–92° [5626], 90–92° [5621], 90–91° [5629], 72° [5631], 64–65° [5637].

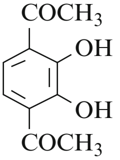
One of the reported melting points is obviously wrong.

^{1}H NMR [5621,5626,5627,5630,5631,5637],

IR [5036,5626,5627,5631,5637,5638], UV [5631,5637], MS [5631,5637].

1,1′-(2,3-Dihydroxy-1,4-phenylene)bis-ethanone

[39126-03-9] $C_{10}H_{10}O_4$ mol.wt. 194.19



Synthesis

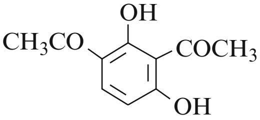
- Refer to: [5639].

Hueckel MO calculations (compound **XI**) [5640];

^{1}H NMR [5639], ^{13}C NMR [5639].

1,1′-(2,4-Dihydroxy-1,3-phenylene)bis-ethanone

[2163-12-4] $C_{10}H_{10}O_4$ mol.wt. 194.19



Syntheses

- Obtained from resorcinol by a typical Friedel–Crafts reaction (40%) [5641],
  - with acetic acid in the presence of boron trifluoride,
    - at 140° for 3 h in a sealed tube (30%) [5257];
    - at 125° for 6 h (20%) [5642];
  - with acetic anhydride,
    - in the presence of concentrated sulfuric acid at 130° for 15 min (15%) [5643];
    - in the presence of zinc chloride at 145–150° (7%) [5644] or at 150–160° for 20 min (6%) [5645];
  - with acetyl chloride,
    - by heating in the presence of concentrated sulfuric acid (10%) [5643];
    - in ethyl ether in the presence of aluminium chloride at r.t. for 3 days (7%) [5251].
- Also obtained by acetylation of resacetophenone with acetic anhydride,
  - in the presence of boron trifluoride in acetic acid at 80° for 1.5 h (31%) [5646];
  - in the presence of boron trifluoride at 70° for 2 h in a sealed tube (30%) [5257];
  - in the presence of aluminium chloride in nitrobenzene at 105–110° for 2 h (15%) [5647].
- Also obtained by acetylation of 2-acetylresorcinol with acetic acid in the presence of zinc chloride at reflux for 5 min [5648].
- Also obtained by Fries rearrangement of resorcinol diacetate,
  - on heating with concentrated sulfuric acid (45%) [5643];
  - with aluminium chloride,
    - at 180–185° for 1.5 h ($AlCl_3$ 3 mol) (60%) [5649];
    - at 160–170° for 2 h ($AlCl_3$ 3 mol) [5650];
    - at 130–135° for 4.5 h ($AlCl_3$ > 2 mol) (crude, 90%) [5651];
    - in nitrobenzene at 100° for 3 h ($AlCl_3$ 3.3 mol) [5616].
- Also obtained by treatment of 4-acetoxy-2-hydroxyacetophenone with aluminium chloride in nitrobenzene at 115° [5652,5653], (26%) [5619] (Fries rearrangement).
- Also obtained by heating 2,4-diacetoxyacetophenone with aluminium chloride for 3 h (26%) [5654] (Fries rearrangement).
- Also obtained by decarboxylation of 3,5-diacetyl-2,4-dihydroxybenzoic acid,
  - with refluxing very dilute hydrochloric acid in water for 12–18 h [5655];
  - with very dilute hydrochloric acid in acetic acid at 160–170° in a sealed tube for 7–8 h [5656].

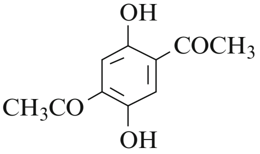
- Also obtained by degradation of 7,7′-diacetoxy-4,4′-dimethyl-3,4-dihydro-4,6′-bicoumarin with aluminium chloride between 135° and 170° for 2 h (19%) [5654].
- Also obtained by Claisen rearrangement of 3-acetyl-2,4-bis(3-methyl-2-butenyloxy)acetophenone, resulting from deprenylation,
  - in trifluoroacetic acid at 0° for 3 h (95%) [5657];
  - in the presence of palladium chloride-bis(acetonitrile) in refluxing dioxane for 45 min (31%) [5657];
  - by heating neat at 185° for 2 h (22%) [5657].
- Also refer to: [5658,5659,5660,5661,5662,5663,5664].

N.B.: Mono Na salt [5665]; di Na salt [5660].

m.p. 96–97° [5641], 95–96° [5656], 92° [5643,5652], 91–92° [5654], 90° [5644,5645], 89° [5648], 88–89° [5651], 88° [5257], 86–87° [5655], 86° [5251], 85–87° [5619], 85–86° [5647], 85° [5642,5646].
b.p.$_{26}$ 170–172° [5651];
^{1}H NMR [5639,5641,5645], ^{13}C NMR [5639,5641], IR [5645], UV [5320].

1,1′-(2,5-Dihydroxy-1,4-phenylene)bis-ethanone

[20129-52-6] $C_{10}H_{10}O_4$ mol.wt. 194.19



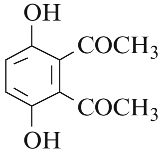
Syntheses

- Obtained by reaction of acetyl chloride (7.6 mol) with hydroquinone dimethyl ether (1.8 mol) in nitrobenzene in the presence of aluminium chloride (5 mol), first at r.t. for 67 h, then at 95° for 40 h (10%) [5666].
- Also obtained by photo-Fries rearrangement of hydroquinone diacetate in methanol under nitrogen for 12 h (10%) [5667].
- Also refer to: [5668].

m.p. 192° [5666], 155° [5667]. One of the reported melting points is obviously wrong.
^{1}H NMR [5639,5667], ^{13}C NMR [5639], IR [5667], MS [5668];
Crystals data [5669,5670,5671]; Hueckel MO calculations (compound **VIII**) [5640].

1,1′-(3,6-Dihydroxy-1,2-phenylene)bis-ethanone

[39125-99-0] $C_{10}H_{10}O_4$ mol.wt. 194.19



Synthesis

- Preparation by oxidative cyclization of 1,3-bis(trimethylsilyloxy)-1-methyl-1,3-butadiene: to a acetonitrile solution of sodium bicarbonate (12 equiv.) and CAN (6 equiv.) was slowly added an acetonitrile solution of 1,3-bis(trimethylsilyloxy)-1-methyl-1,3-butadiene (2 equiv.) at −45°.

The temperature of the reaction mixture was allowed to rise to 20° during 2 h.

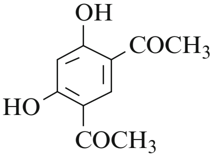
After stirring for 1 h at 20°, a saturated aqueous solution of brine was added, the organic layer was separated and the aqueous layer was extracted with ethyl ether.

The combined organic extracts were dried, filtered and the solvent was removed in vacuo. The residue was purified by column chromatography (compound **4** h) (28%) [5672].

Hueckel MO calculations (compound **VII**) [5640].

1,1′-(4,6-Dihydroxy-1,3-phenylene)bis-ethanone

[2161-85-5] $C_{10}H_{10}O_4$ mol.wt. 194.19



Syntheses

- Preparation by Friedel–Crafts acylation of resorcinol,
  - with acetic anhydride,
    - in the presence of zinc chloride [5673,5674,5675], (80%) [5676], at 142–150° for 15 min (96%) [5677], (90%) [5644] or at 150–160° for 20 min (68%) [5645];
    - in the presence of ferric chloride [5678].
    - in the presence of concentrated sulfuric acid at 130° for 15 min (15%) [5643];
    - in the presence of 70% perchloric acid at 125–135° for 15–20 min (42%) [5679].
  - with acetyl chloride,
    - in the presence of zinc chloride [5680,5681], at 120° [5682];
    - in the presence of ferric chloride [4598,5678], at 150° for 15 min (60%) [5683] or at reflux for 30 min [5673];
    - in the presence of concentrated sulfuric acid (18%) [5643].
  - by a typical Friedel–Crafts reaction (24%) [5641].
- Also obtained by acylation of resorcinol with acetic acid,
  - in the presence of polyphosphoric acid for 15 min in a boiling water bath (9%) [5684];
  - in the presence of boron trifluoride at 125° for 6 h (20%) [5642].
- Also obtained by Friedel–Crafts acylation of paeonol with acetic anhydride in nitrobenzene in the presence of aluminium chloride [5685].
- Also obtained by Fries rearrangement of resorcinol diacetate,
  - with hot concentrated sulfuric acid (31%) [5643];
  - with polyphosphoric acid at 70° for 2 h (19%) [5686];
  - with aluminium chloride,
    - in nitrobenzene in a boiling water bath (70%) [4471];
    - without solvent (15%) [5687], at 205–210° for 1.5 h (14%) [5649].
  - with fused zinc chloride [5606,5673,5680,5688,5689], at 120° [5682] or at 130° (40–50%) [5690];
  - with ferric chloride at 180° for 3 h [5691], (32%) [5692], under nitrogen (16%) [5693,5694] or under carbon dioxide [5673], (15%) [5695].

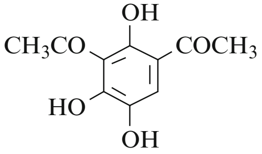
- Also obtained by photo-Fries rearrangement of resorcinol diacetate in methanol at 25° under nitrogen [5696].
- Also obtained by acylation of resacetophenone,
  - with acetic acid,
    - in the presence of zinc chloride (Nencki reaction) [5618];
    - in the presence of zinc chloride and phosphorous oxychloride at 140–150° for 30 min [5606,5673,5697].
    - in the presence of polyphosphoric acid (14%) [5698], in a boiling water bath for 10 min (21%) [5684].
  - with acetic anhydride,
    - in the presence of boron trifluoride in acetic acid at 80° for 1.5 h (35%) [5646].
    - in the presence of aluminium chloride in nitrobenzene at 105–110° for 2 h (15%) [5647].
- Also obtained by Friedel–Crafts acylation of resorcinol dimethyl ether with acetyl chloride in carbon disulfide at 10° for 1 h (9%) [5646].
- Also obtained by Fries rearrangement of 2-acetoxy-4-hydroxyacetophenone with ferric chloride at 180° for 3 h (12%) [5695].
- Also obtained by Fries rearrangement of 2,4-diacetoxyacetophenone with aluminium chloride (9%) [4660].
- Also obtained (poor yield) by treatment of 7,7′-diacetoxy-4,4′-dimethyl-3,4-dihydro-4,6′-bi-coumarin with aluminium chloride between 135° and 170° for 2 h (< 3%) [4660].
- Also obtained by total dealkylation,
  - of resodiacetophenone diallyl ether with trifluoroacetic acid at 60° for 1 h (85%) [5605];
  - of resodiacetophenone dimethyl ether with 48% aqueous hydrobromic acid in refluxing acetic acid for 2 h (34%) [5699,5700].
  - of resodiacetophenone diprenyl ether,
    - with trifluoroacetic acid at 0° for 24 h (95%) [5657];
    - with boron trifluoride etherate in refluxing carbon tetrachloride (98%) [5657].
- Also refer to: [5607,5619,5651,5653,5658,5661,5701,5702,5703,5704,5705, 5706,5707,5708,5709].

N.B.: Mono Na salt [52597-47-4] [4857], di Na salt [52814-43-4] [4836].

m.p. 185° [5647,5685], 183° [5682], 182–184° [5699], 182–183° [5618], 182° [5619,5642,5643,5646,5679,5684,5690,5692,5695], 181–184° [5700], 181–182° [5686], 180–180°5 [4660], 180° [5697,5698], 179°5 [5689], 179–181° [5694], 178–180° [5677], 178–179° [5644,5645,5683], 178° [5710], 177–178° [4471], 176–177° [5641];
^{1}H NMR [5639, 5641,5645,5693, 5694], ^{13}C NMR [5639,5641],
IR [5645,5683], UV [5653,5711], MS [5693,5694];
Crystal data [5674]; Conductimetry [5711]; Polarography [5711].

1,1′-(2,4,5-Trihydroxy-1,3-phenylene)bis-ethanone

[2999-24-8] $C_{10}H_{10}O_5$ mol.wt. 210.19



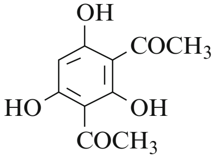
Syntheses

- Preparation by Fries rearrangement of 1,2,4-triacetoxy-benzene with aluminium chloride,
  - at 140° for 35 min [5712], 64% [5713], 30% [5714];
  - at 160–170° for 2 h (60%) [5650].
- Also obtained by oxidation of 2,4-diacetylresorcinol with potassium persulfate (Elbs reaction) [5712].

m.p. 186–187° [5712], 186° [5714], 185° [5650], 183° [5713].

1,1′-(2,4,6-Trihydroxy-1,3-phenylene)bis-ethanone

[2161-86-6] $C_{10}H_{10}O_5$ mol.wt. 210.19



Syntheses

- Preparation by Friedel–Crafts acylation of phloroglucinol,
  - with acetic acid,
    - by using boron trifluoride-acetic acid complex at 28–30° for 18 h (85%) [5642];
    - without cooling (71%) [5715];
    - or by heating on a steam bath for 2 h (60%) [5716];
  - with acetic anhydride,
    - in the presence of boron trifluoride-ethyl ether complex at 20° for 1 h (80%) [5646];
    - in the presence of zinc chloride at 145–150° for 15 min (25%) [5644];
    - in the presence of concentrated sulfuric acid (24%) [5643];
  - with acetyl chloride,
    - (4 equiv.) in the presence of ferric chloride [5673,5717], (3%) [5678];
    - in ethyl ether in the presence of aluminium chloride at r.t. for 5 days (8%) [5251].
- Also obtained by Fries rearrangement of phloroglucinol triacetate with aluminium chloride at 160–170° for 2 h (10%) [5650].
- Also obtained by monodecarbonylation of 2,4,6-triacetylphloroglucinol in 73% sulfuric acid at r.t. for 72 h [5650].
- Also obtained by hydrolysis of 5-acetoxy-2,4-diacetyl-1,3-dihydroxybenzene [5667], in the presence of 70% sulfuric acid [5678].
- Also refer to: [5718,5719,5720].

Isolation from natural sources

- This compound is one of the antifungal metabolites produced by *Pseudomonas fluorescens* [5721,5722,5723,5724,5725,5726,5727,5728,5729,5730,5731,5732, 5733,5734,5735,5736,5737,5738,5739].
- Production in the rhizosphere by strains of fluorescent *Pseudomonas* spp. [5740].
- Also produced by a bacterial symbiot of the white-backed planthopper (insect), *Sogatella furcifera* [5741,5742].
- Also produced by a fungal ectosymbiot of an ambrosia beetle (insect), *Scolytoplatypus mikado* [5742].

N.B.: An hemihydrate was obtained by crystallisation of ketone in aqueous ethanol [5725] and a monohydrate in 20% aqueous acetic acid [5717]. The melting points are determined after solvents elimination.

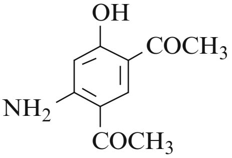
m.p. 173° [5644], 172–173° [5667], 171° [5251], 170° [5717], 168–170° [5716,5725], 168° [5642,5646,5650,5715,5743], 153° [5643].

One of the reported melting points is obviously wrong.

^{1}H NMR [5716], IR [5716,5725], UV [5715,5725]; TLC [5725].

1,1′-(4-Amino-6-hydroxy-1,3-phenylene)bis-ethanone

[79324-45-1] $C_{10}H_{11}NO_3$ mol.wt. 193.20



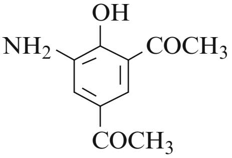
Syntheses

- Preparation by hydrolysis of 3-acetamido-4,6-diacetyl-phenol (m.p. 201–202°) with concentrated hydrochloric acid in refluxing ethanol for 5 h (91%) [5744] or for 3.5 h (60%) [5745].
- Preparation by hydrolysis of 2-amino-5-(1-iminoethyl)-4-hydroxyacetophenone (SM) on heating with aqueous hydrochloric acid. SM was obtained by heating a mixture of resodiacetophenone, aqueous ammonia and concentrated hydrochloric acid as a catalyst in an autoclave during 8–72 h [5746].

m.p. 227–230° [5744].

1,1′-(5-Amino-4-hydroxy-1,3-phenylene)bis-ethanone

[100245-11-2] $C_{10}H_{11}NO_3$ mol.wt. 193.20

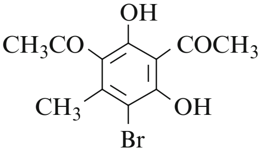


Synthesis

- Preparation by hydrogenation of 5-acetyl-2-hydroxy-3-nitroacetophenone using 5% Pd/C as a catalyst in ethanol (49%) [4699].

m.p. 156–160° (d) [4699].

1,1′-(5-Bromo-2,4-dihydroxy-6-methyl-1,3-phenylene)bis-ethanone



$C_{11}H_{11}BrO_4$ mol.wt. 287.11

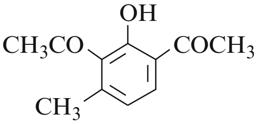
Synthesis

- Obtained by reaction of bromine with 2,4-diacetyl-3,5-di-hydroxytoluene (diacetylorcinol) in acetic acid [5747].

m.p. 79° [5747].

1,1′-(2-Hydroxy-4-methyl-1,3-phenylene)bis-ethanone

[131941-97-4]



$C_{11}H_{12}O_3$ mol.wt. 192.21

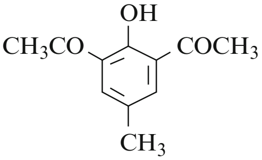
Synthesis

- Obtained by Fries rearrangement of m-cresyl acetate with aluminium chloride [5748,5749].

^{13}C NMR [5748].

1,1′-(2-Hydroxy-5-methyl-1,3-phenylene)bis-ethanone

[55108-28-6]



$C_{11}H_{12}O_3$ mol.wt. 192.21

Syntheses

- Preparation by Friedel–Crafts acylation of p-cresol with excess acetyl chloride in nitrobenzene in the presence of aluminium chloride [5750,5751], at 60° for 6 h (42%) [5752], (20%) [5753].
- Also obtained by Friedel–Crafts acylation of p-cresol methyl ether with excess acetyl chloride in the presence of aluminium chloride [4625].

Also obtained by Fries rearrangement of p-cresyl acetate with aluminium chloride [5748,5754].

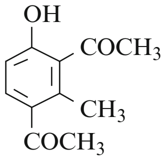
- Preparation by Fries rearrangement of 2-(acetyloxy)-5-methylacetophenone with aluminium chloride,
  - at 100–120° for 10 min (76%) [5755];
  - at 130° for 1 h, then at 140° for 1 h (70%) [5611].
- Also refer to: [5756,5757,5758,5759,5760,5761,5762,5763,5764,5765,5766].

N.B.: Metal complexes of binucleating ligands: Cu (II) [5750,5759,5760,5764], Ni (II) [5759] and UO_2 (VI) [5759]; Li salt (compound **2**) [5767].
Dioxime [5768].

m.p. 83° [5755], 82–83° [4625], 82° [5752,5753];
b.p.$_5$ 85–87° [5611], b.p.$_{18}$ 194° [5755];
^1H NMR [5748,5752,5769], ^{13}C NMR [5748], IR [5752],
UV [4856,5752,5762]; emission spectra [5765].

1,1′-(4-Hydroxy-2-methyl-1,3-phenylene)bis-ethanone

[170802-46-7] $C_{11}H_{12}O_3$ mol.wt. 192.21



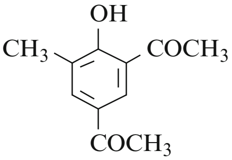
Synthesis

– Obtained by Fries rearrangement of m-cresyl acetate with aluminium chloride [5748].

^{1}H NMR [5748], ^{13}C NMR [5748].

1,1′-(4-Hydroxy-5-methyl-1,3-phenylene)bis-ethanone

[23133-81-5] $C_{11}H_{12}O_3$ mol.wt. 192.21



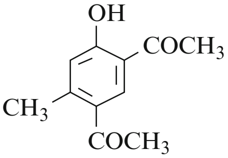
Syntheses

– Obtained by Fries rearrangement of o-cresyl acetate with aluminium chloride [5748].
– Also refer to: [5770].

^{13}C NMR [5748].

1,1′-(4-Hydroxy-6-methyl-1,3-phenylene)bis-ethanone

[16475-85-7] $C_{11}H_{12}O_3$ mol.wt. 192.21



Syntheses

– Preparation by cyclization of 1,1,3,3-tetraacetylpropene (formerly so called methenylbisacetylacetone) (SM) (m.p. 117–118°) [5771],

- by adding a solution of SM (1 mol) in benzene to a sodium methoxide (4 mol) or magnesium methoxide (4 mol) solution in methanol and set aside for 24 h (quantitative yields) [5772];
- on heating of its potassium salt in alcoholic solution for 6–8 h at reflux. SM was prepared by treatment of ethoxymethyleneacetylacetone (m.p. 140–142°) with the potassium salt of acetylacetone in ethanol [5771].

– Also obtained directly by heating together sodioacetylacetone and ethoxymethyleneacetylacetone at 100° for 30 min [5773].
– Also obtained by Fries rearrangement of m-cresyl acetate with aluminium chloride [5635,5748,5749].
– Also obtained by heating a mixture of 3,5-diacetyl-2,4-heptanedione (m.p. 33–35°) and triethyl-ammonium formate (TEAF) at 145–150° for 5 h with stirring in a constant stream of air (28%) [5774].
– Also obtained by heating a mixture of 4,6-diacetyl-3-methyl-2-cyclohexen-1-one and TEAF at 145–150° for 4 h with stirring in a stream of oxygen (33%) [5774].
– Also obtained from 1,1,3,3-tetraacetylpropane (formerly so called methylenebisacetylacetone) (SM1) (m.p. 87–88°),

- by reaction with concentrated sulfuric acid under oxygen of the air during short-lived—via the formation of 4,6-diacetyl-3-methyl-2-cyclohexen-1-one—(m.p. 75°) [5775];
- in chloroformic solution with hydrogen chloride under oxygen of the air [5775];
- SM1 (1 vol) in solution of 20% hydrochloric acid (3–4 vol) during 5–8 days at r.t. (44%).

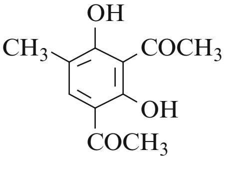
  SM1 was obtained by condensation of formaldehyde with acetylacetone [5775].

– Also refer to: [5755,5776,5777,5778].

N.B.: Ba [5771] and K salts [5771,5775].

m.p. 112° [5771], 108° [5772], 106° [5775], 105° [5773], 104–105° [5774];
b.p. 310° (without decomposition) [5771]; TLC [5772];
^{1}H NMR [5748,5772,5773,5774], ^{13}C NMR [5748],
IR [5772,5773,5774], UV [5772,5773], MS [5772].

1,1′-(2,4-Dihydroxy-5-methyl-1,3-phenylene)bis-ethanone



$C_{11}H_{12}O_4$ mol.wt. 208.21

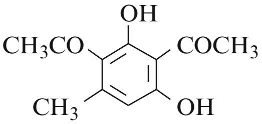
Synthesis

– Obtained by Fries rearrangement of 4,6-dimethylresorcinol diacetate (m.p. 44°) with aluminium chloride by heating, first at 120° and then raising the temperature to 180° over a period of an hour (19%) [5779]. **N.B.**: One of the methyl groups was displaced during the reaction.

m.p. 83–84° [5779].

1,1′-(2,4-Dihydroxy-6-methyl-1,3-phenylene)bis-ethanone

[13444-19-4]



$C_{11}H_{12}O_4$ mol.wt. 208.21

Syntheses

– Preparation by Fries rearrangement of orcinol diacetate (m.p. 25°) [5780] with aluminium chloride, at 140–150° for 1.5 h (80%) [5649], at 150° for 2 h (42%) [5781].

– Also obtained by acylation of γ-orcacetophenone or β-orcacetophenone with acetic anhydride in nitrobenzene in the presence of aluminium chloride in a water bath for 6 h (15–20%) [5782].

– Also obtained by reaction of acetyl chloride with an anhydrous disodium salt (SM) in chloroform (major product). SM was prepared by action of sodium ethoxide with diacetylacetone or dimethylpyrone in ethanol [5747].

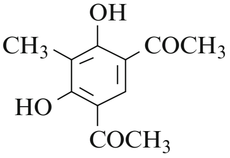
– Also obtained (small amount) during an attempt to acylate 2-acetylfuran with a three-fold excess acetyl chloride in the presence of aluminium chloride, first between 20° and 45°, then at 115° for 3 h. This diketone was formed by self-condensation of acetyl chloride in these conditions [5783].

- Also obtained by decarboxylation of 3,5-diacetyl-o-orsellinic acid [5781].
- Also obtained in two steps: first, reaction of acetyl chloride with diacetyl acetone disodium salt in chloroform at 20° for 1 h, then, after elimination of solvent, treatment of the residue in refluxing 3 N sodium hydroxide for 30 min [5783].
- Also obtained by reaction of acetic anhydride with orcinol in concentrated sulfuric acid at 130° for 15 min [5783], according to the method [5643].

m.p. 96° [5781], 95° [5747,5782,5784], 94–95° [5783];
^{1}H NMR [5783,5784], IR [5784], MS [5783,5784].

1,1′-(4,6-Dihydroxy-5-methyl-1,3-phenylene)bis-ethanone

[22304-66-1] $C_{11}H_{12}O_4$ mol.wt. 208.21



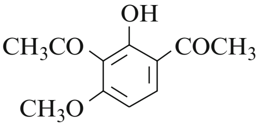
Syntheses

- Preparation by Fries rearrangement of 2,6-diacetoxy-toluene with aluminium chloride in nitrobenzene,
  - at 75° for 3 h, under nitrogen (63%) [5693,5694];
  - at 67° for 4 h (58%) [5687].
- Preparation by reaction of acetic anhydride with 2-methyl-resorcinol,
  - in the presence of sodium acetate at reflux for 8 h (83%) [5687];
  - in the presence of zinc chloride at 142° for 15 min (60%) [5785].

m.p. 146–147° [5785], 139–142° [5687], 137–139° [5693,5694];
^{1}H NMR [5693,5694,5785], IR [5785], MS [5693,5694].

1,1′-(2-Hydroxy-4-methoxy-1,3-phenylene)bis-ethanone

[64857-81-4] $C_{11}H_{12}O_4$ mol.wt. 208.21



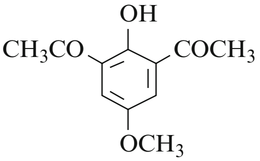
Syntheses

- Preparation by Friedel–Crafts acylation of paeonol with acetic anhydride,
  - in acetic acid in the presence of boron trifluoride at 50° for 1 h [5646];
  - in nitrobenzene in the presence of aluminium chloride at r.t. for 72 h [5685].
- Preparation by methylation of 2,4-diacetylresorcinol [5682], with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 2 h (51%) [5646] or for 8 h (50%) [5652].
- Also obtained by hydrolysis of 2-(3-acetyl-2-hydroxy-4-methoxyphenyl)-2-methyl-1,3-dioxolane with a mixture of 5% aqueous hydrochloric acid and ethanol at r.t. for 5 min (almost quantitative yield) [5652].

m.p. 104° [5646,5652], 102° [5685,5786], 101–102° [5720]; IR [5720].

1,1′-(2-Hydroxy-5-methoxy-1,3-phenylene)bis-ethanone

[103867-90-9] $C_{11}H_{12}O_4$ mol.wt. 208.21



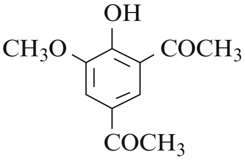
Synthesis

- Obtained by hydrolysis of 2-(3-acetyl-2-hydroxy-5-methoxyphenyl)-2-methyl-1,3-dioxolane with a mixture of 5% aqueous hydrochloric acid and ethanol at r.t. for 5 min (almost quantitative yield) [5620].

m.p. 116–117° [5620]; ^{1}H NMR [5620], IR [5620].

1,1′-(4-Hydroxy-5-methoxy-1,3-phenylene)bis-ethanone

[294888-77-0] $C_{11}H_{12}O_4$ mol.wt. 208.21

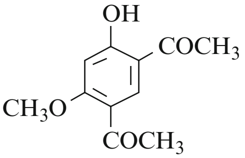


Synthesis

- Refer to: [5787].

1,1′-(4-Hydroxy-6-methoxy-1,3-phenylene)bis-ethanone

[99865-77-7] $C_{11}H_{12}O_4$ mol.wt. 208.21



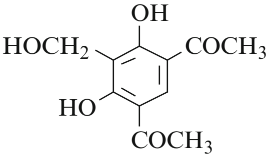
Syntheses

- Preparation by action of methyl iodide with the potassium salt of resodiacetophenone in ethanol [5606].
- Preparation by reaction of acetyl chloride (1 mol) with resorcinol dimethyl ether (0.15 mol) in the presence of aluminium chloride in carbon disulfide for 1 h (27%) [5788].
- Preparation by reaction of acetic acid with resacetophenone monomethyl ether (m.p. 51°) in the presence of polyphosphoric acid for 10 min in a boiling water bath (36%) [5698].

m.p. 121–122° [5788], 121°5 [5682], 121° [5698], 120° [5606];
UV [5711]; Conductimetry [5711]; Polarography [5711].

1,1′-[4,6-Dihydroxy-5-(hydroxymethyl)-1,3-phenylene]bis-ethanone

[58805-54-2] $C_{11}H_{12}O_5$ mol.wt. 224.21



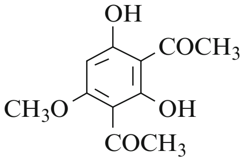
Synthesis

- Obtained by action of a 40% formaldehyde solution with resodiacetophenone in 1% aqueous sodium hydroxide at r.t. for 5 min (78%) [5789].

m.p. 150–151° [5789].

1,1′-(2,4-Dihydroxy-6-methoxy-1,3-phenylene)bis-ethanone

[3098-38-2] $C_{11}H_{12}O_5$ mol.wt. 224.21



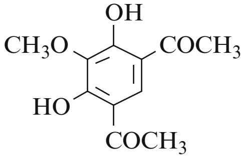
Syntheses

- Preparation by reaction of phloroglucinol monomethyl ether,
    - with boron trifluoride-acetic acid complex at 100° for 4 h (80%) [5642];
    - with acetic anhydride and acetic acid in the presence of boron trifluoride at 20° for 1 h (81%) [5646].
- Preparation by Fries rearrangement of phloroglucinol monomethyl ether diacetate in acetic acid in the presence of boron trifluoride at 75° for 15 min (66%) [5790].
- Also obtained by partial demethylation,
    - of 2,4-diacetylphloroglucinol trimethyl ether with boron trichloride, first at –70°, then at r.t. for 20 min (72%) [5791];
    - of 2,4-diacetyl-3,5-dimethoxyphenol with boron trifluoride in ethyl ether containing a small amount of acetic acid at r.t. for 24 h (10%) [5646].
- Also obtained by monomethylation of 2,4-diacetylphloroglucinol [5789,5792],
    - with diazomethane in benzene [5646];
    - with methyl iodide in the presence of potassium carbonate in boiling acetone for 3 h [5646].
- Also obtained by hydrogenolysis of 3-(benzyloxy)-2,6-diacetyl-5-methoxyphenol [5646].
- Also obtained by treatment of 2,6-dihydroxy-4-methoxy-3-trichloroacetylacetophenone with zinc dust in acetic acid on a steam bath for 3 min (quantitative yield) [4680].
- Also refer to: [5793].

m.p. 106° [5642,5646], 105–106° [5790], 105° [4680];
^{1}H NMR [5794], ^{2}H NMR [5794], ^{3}H NMR [5794].

1,1′-(4,6-Dihydroxy-5-methoxy-1,3-phenylene)bis-ethanone

[144632-80-4] $C_{11}H_{12}O_5$ mol.wt. 224.21



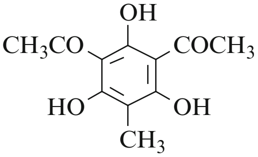
Synthesis

- Obtained by reaction of acetonitrile with pyrogallol 2-methyl ether in the presence of triflic acid, first at r.t. for 8 days, and at reflux for 30 min (20%) [5795].

m.p. 130–132° [5795];
^{1}H NMR [5795], IR [5795], MS [5795].

1,1′-(2,4,6-Trihydroxy-5-methyl-1,3-phenylene)bis-ethanone

[2999-42-0] $C_{11}H_{12}O_5$ mol.wt. 224.21



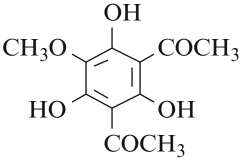
Syntheses

- Preparation by Friedel–Crafts acylation of 2-methylphloroglucinol with acetic anhydride/acetic acid in the presence of excess boron trifluoride,
  - at r.t. for 20 h (56%) [5646];
  - first at r.t., then heating on a steam bath for 2 h [5796], (21%) [5716].
- Also obtained by UV irradiation of a d-usnic acid solution in THF for 12 h at −20° under oxygen (13%) [5797].
- Also obtained by UV irradiation of a decarbousnic acid solution in THF for 8 h at −20° under oxygen (6%) [5797].
- Also obtained by hydrolysis of 2,4-diacetyl-3,5-dihydroxy-6-methylphenyl acetate with concentrated sulfuric acid for 10 min in cold (78%) [5797].
- Also obtained by saponification of its diacetate (SM)—1,1′-[4,6-di(acetyloxy)-2-hydroxy-5-methyl-1,3-phenylene]bis-ethanone—with refluxing 2 N sodium carbonate for 10 min (69%). SM was prepared from the ozonid of diacetyldecarbousnic acid (C21H22O11, m.p. 146°) by treatment with boiling 3% methanolic hydrogen chloride for 5 min (73%, m.p. 116°) [5798].
- Also refer to: [5720,5799].

m.p. 172° [5797], 169–170° [5716], 168° [5798], 160° [5646]; TLC [5797]; ^{1}H NMR [5716,5797], IR [5716], UV [5797], MS [5797].

1,1′-(2,4,6-Trihydroxy-5-methoxy-1,3-phenylene)bis-ethanone

[17678-03-4] $C_{11}H_{12}O_6$ mol.wt. 240.21



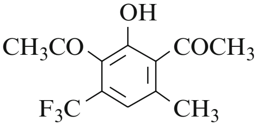
Synthesis

- Obtained by reaction of iretol with boron trifluoride-acetic acid complex at r.t. for 20 h (52%) [4919].

m.p. 140° [4919].

1,1′-[2-Hydroxy-4-methyl-6-(trifluoromethyl)-1,3-phenylene]bis-ethanone

[76716-15-9] $C_{12}H_{11}F_3O_3$ mol.wt. 260.21



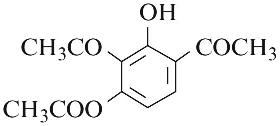
Synthesis

- Obtained (poor yield) by condensation of 2,4,6-heptanetrione (1 mol) and 1,1,1-trifluoro-2,4-pentanedione (1 mol) in the presence of sodium hydroxide in 50% aqueous methanol at 25° for 25 h (13%) [5800].

m.p. 83–85° [5800]; ^{1}H NMR [5800], ^{19}F NMR [5800].

1,1′-[4-(Acetyloxy)-2-hydroxy-1,3-phenylene]bis-ethanone

[116470-16-7] $C_{12}H_{12}O_5$ mol.wt. 236.22



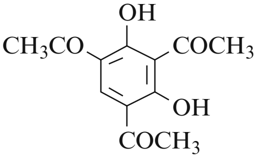
Synthesis

– Obtained by heating at reflux (180°) for 1 h a mixture of 2,4-diacetylresorcinol, sodium acetate and acetic anhydride (29%) [5658].

m.p. 147–148° [5658]; ^{1}H NMR [5658], IR [5658], UV [5658].

1,1′,1″-(2,4-Dihydroxy-1,3,5-benzenetriyl)tris-ethanone

[64857-82-5] $C_{12}H_{12}O_5$ mol.wt. 236.22



Syntheses

– Obtained by heating 2,4-diacetoxyacetophenone with aluminium chloride (29%) (Fries rearrangement) [5654].

– Also obtained (by-product) by Friedel–Crafts acylation of resorcinol dimethyl ether with acetyl chloride in the presence of aluminium chloride in carbon disulfide at 10° for 1 h (5%) [5646].

– Also obtained by Friedel–Crafts acylation of paeonol with acetic anhydride in the presence of aluminium chloride in nitrobenzene at 100° for 2 h [5685].

– Also obtained by reaction of acetic anhydride (2 mol) with resacetophenone (1 mol) in the presence of aluminium chloride (3 mol) in nitrobenzene on a steam bath for 4 h (51%) [5664].

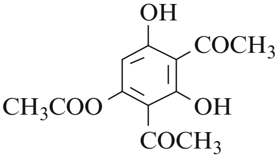
– Also obtained by reaction of acetyl chloride with 4,6-diacetylresorcinol in the presence of aluminium chloride, first at 110° for 15 min, then at 130° for 1 h (73%) [5665].

– Also obtained by degradation of 7,7′-diacetoxy-4,4′-dimethyl-3,4-dihydro-4,6′-bicoumarin with aluminium chloride in dilute hydrochloric acid between 135° and 170° for 2 h (5%) [5654].

m.p. 137–138° [5664], 137° [5646], 136° [5665,5685], 135–136°5 [5654]; ^{1}H NMR [5654], MS [5654].

1,1′-[4-(Acetyloxy)-2,6-dihydroxy-1,3-phenylene]bis-ethanone

[104654-31-1] $C_{12}H_{12}O_6$ mol.wt. 252.22



Synthesis

– Obtained by UV light irradiation of 1,3,5-triacetoxybenzene in methanol at r.t. for 12 h under nitrogen (25%) [5667].

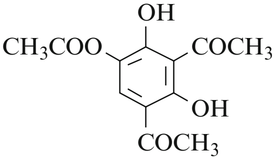
Isolation from natural sources

– From *Hypericum japonicum* Thunb. and *Agromonia pilosa* Ledb. [5720].

m.p. 150° [5667].

1,1′-[5-(Acetyloxy)-2,4-dihydroxy-1,3-phenylene]bis-ethanone

[55168-30-4] $C_{12}H_{12}O_6$ mol.wt. 252.22



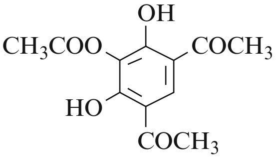
Synthesis

- Obtained by treatment of 1,2,4-triacetoxybenzene with acetic acid and zinc chloride at 130° for 1 h (24%) [5713] or at 140° for 30 min (9%) [5801].

m.p. 142–143° [5801], 142° [5713];
^{1}H NMR [5713,5801], IR [5713,5801], MS [5713].

1,1′-[5-(Acetyloxy)-4,6-dihydroxy-1,3-phenylene]bis-ethanone

[104654-32-2] $C_{12}H_{12}O_6$ mol.wt. 252.22



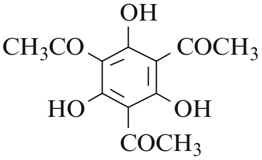
Syntheses

- Obtained by photolysis of 1,2,3-triacetoxybenzene in methanol at r.t. for 12 h under nitrogen (20%) [5667].
- Also obtained by reaction of acetic acid with gallacetophenone in the presence of zinc chloride and phosphorous oxychloride at 140–150° for 30 min [5697], according to [5802].

m.p. 209–210° [5667], 207–209° [5697].

1,1′,1″-(2,4,6-Trihydroxy-1,3,5-benzenetriyl)tris-ethanone

[2161-87-7] $C_{12}H_{12}O_6$ mol.wt. 252.22



Syntheses

- Obtained by Friedel–Crafts acylation of phloroglucinol,
  - with acetic anhydride in the presence of boron trifluoride in acetic acid at r.t. (60%) [5646];
  - with acetyl chloride in the presence of aluminium chloride in ethyl ether at r.t. for 6 days (17%) [5251];
  - with acetyl chloride and acetic acid in the presence of ferric chloride in ethyl acetate [5717].
- Also obtained by Fries rearrangement of phloroglucinol triacetate,
  - in the presence of aluminium chloride,
    - without solvent [5803], at 160–170° (40%) [5650];
    - in nitrobenzene at r.t. for 4 h [5650];
  - in the presence of zinc chloride at 130°(40–50%) [5690], for 3 h (60%) [5804].

- Also refer to: [5720,5743,5793,5805,5806].

N.B.: Tri-Na salt [5807,5808].

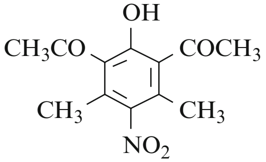
Isolation from natural sources

- From *Pseudomonas fluorescens* [5725].

sublimation at 140°/15 mm [5251]; TLC [52515725];
m.p. 158–159° [5650], 156° [5646,5717,5801], 152–153° [5251];
^{1}H NMR [5251,5639,5794], ^{2}H NMR [5794], ^{3}H NMR [5794], ^{13}C NMR [5639], MS [5725].

1,1′-(2-Hydroxy-4,6-dimethyl-5-nitro-1,3-phenylene)bis-ethanone

[85450-67-5] $C_{12}H_{13}NO_5$ mol.wt. 251.24



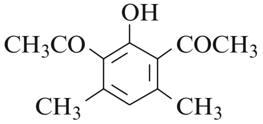
Synthesis

- Obtained by reaction of nitromethane with 3,5-diacetyl-2,6-dimethyl-4-pyrone in the presence of potassium tert-butoxide in tert-butanol at 30–40° for 75 min (74%) [5809].

m.p. 117–119° [5809].

1,1′-(2-Hydroxy-4,6-dimethyl-1,3-phenylene)bis-ethanone

[66634-65-9] $C_{12}H_{14}O_3$ mol.wt. 206.24



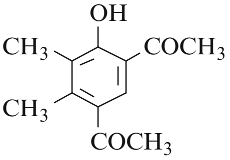
Syntheses

- Obtained by Friedel–Crafts acylation of 3,5-dimethylphenol with acetyl chloride in the presence of aluminium chloride in boiling carbon disulfide for some hours [5810], (40%) [5811], (36%) [5812].
- Also obtained by acylation of 3,5-dimethylanisole with acetyl chloride (6 mol) in the presence of a large excess of aluminium chloride in boiling carbon disulfide for 2 h, then, after solvent elimination, heating in a water bath for 4 h (33%) [5811].
- Also obtained by Fries rearrangement of 3,5-dimethylphenyl acetate with aluminium chloride (2 mol) in a water bath for 2 h [5810].
- Also obtained by condensation of 2,4,6-heptanetrione (1 mol) and 2,4-pentanedione (1 mol) in the presence of sodium hydroxide in 50% aqueous methanol at 25° for 25 h (50%) [5800].

m.p. 109–110° [5810,5811,5812], 102–105° [5800]; ^{1}H NMR [5800].

1,1′-(4-Hydroxy-5,6-dimethyl-1,3-phenylene)bis-ethanone

[51233-76-2] $C_{12}H_{14}O_3$ mol.wt. 206.24



Syntheses

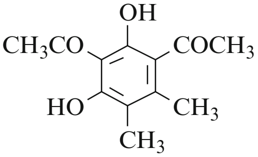
- Obtained (by-product) by Fries rearrangement of 2,3-di-methylphenyl acetate with aluminium chloride at 135° for 30 min (<10%) [5813].
- A sample of pure 4-hydroxy-2,3-dimethylacetophenone (m.p. 144°) [5814], stored in a stoppered bottle, was analyzed 10 years later. The melting point (144°) was lowered to 138°. By treatment of the mixture in boiling heptane, the pure insoluble 4-hydroxy-2,3-dimethylacetophenone was recovered by filtration and thoroughly washed with boiling heptane (50%). The solution was then concentrated and the residue chromatographied on silica gel with benzene-ethyl acetate-acetic acid mixture (90/5/5) as eluent. The 2,3-dimethylphenol (25%) and the pure entitled diketone (25%) were isolated [5815].

m.p. 101° [5815]; TLC [5815];
IR [5815] 1685 cm^{-1} (**C**=O para), 1640 cm^{-1} (**C**=O ortho), UV [5815].

N.B.: This transformation was unexpectedly obtained by a simple storage in a dry dull place.

1,1′-(2,4-Dihydroxy-5,6-dimethyl-1,3-phenylene)bis-ethanone

[82817-51-4] $C_{12}H_{14}O_4$ mol.wt. 222.24



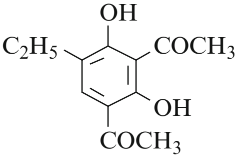
Synthesis

- Obtained by Fries rearrangement of 1,3-diacetoxy-4,5-di-methylbenzene with aluminium chloride at 115–120° for 30 min (54%) [5816].

m.p. 78–80° [5816]; IR [5816], UV [5816].

1,1′-(5-Ethyl-2,4-dihydroxy-1,3-phenylene)bis-ethanone

[63411-83-6] $C_{12}H_{14}O_4$ mol.wt. 222.24

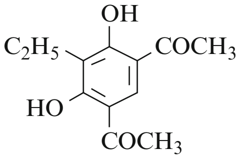


Synthesis

- Obtained by Fries rearrangement of 4,6-diethylresorcinol diacetate (1 mol) with aluminium chloride (2.2 mol) at 155° for 1 h (57%) [5817].

m.p. 71–73° [5817]; TLC [5817].

1,1′-(5-Ethyl-4,6-dihydroxy-1,3-phenylene)bis-ethanone



$C_{12}H_{14}O_4$ mol.wt. 222.24

Synthesis

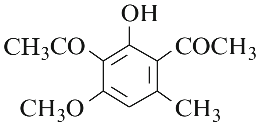
- Obtained by Fries rearrangement of 2-ethylresorcinol diacetate (m.p. 70–71°) with aluminium chloride at 150° for 30 min (84%) [5665].

m.p. 110° [5665].

1,1′-(2-Hydroxy-4-methoxy-6-methyl-1,3-phenylene)bis-ethanone

[78274-03-0]

$C_{12}H_{14}O_4$ mol.wt. 222.24



Synthesis

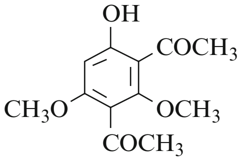
- Obtained by Friedel–Crafts acylation of 2-hydroxy-6-methoxy-4-methylacetophenone (yield 25%) or 2-hydroxy-4-methoxy-6-methylacetophenone with acetyl chloride in the presence of aluminium chloride [5818].

m.p. 98° [5818]; ^{1}H NMR [5818], IR [5818], MS [5818].

1,1′-(4-Hydroxy-2,6-dimethoxy-1,3-phenylene)bis-ethanone

[72221-04-6]

$C_{12}H_{14}O_5$ mol.wt. 238.24



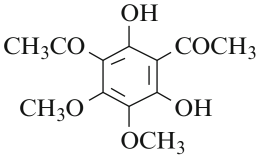
Syntheses

- Obtained by debenzylation of 3-acetyl-6-(benzyloxy)-2,4-di-methoxyacetophenone (83%) [5646].
- Also obtained (by-product) by Friedel–Crafts acylation of phloroglucinol trimethyl ether with acetyl chloride in the presence of aluminium chloride in boiling carbon disulfide [5819]. **N.B.**: No direct proof of the constitution of the diketone was described, but it would appear most probable that it is 3-acetyl-6-hydroxy-2,4-dimethoxyacetophenone [5819].
- Also obtained by saponification of its veratric ester (m.p. 198°) in pyridine with powdered potassium hydroxide (pre-heated at 100°) at 50° for 1 h [5820].

m.p. 192° [5820], 127–128° [5819], 106° [5646]. One note a very large dispersion of the various melting points.

1,1′-(2,4-Dihydroxy-5,6-dimethoxy-1,3-phenylene)bis-ethanone

[91498-04-3] $C_{12}H_{14}O_6$ mol.wt. 254.24



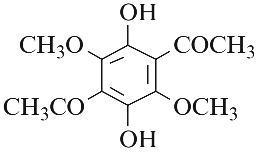
Synthesis

- Obtained from antiarol by reaction,
  - with acetic anhydride in the presence of boron trifluoride in acetic acid at 30° (max), followed by standing overnight (quantitative yield) [5821];
  - with acetyl chloride in the presence of aluminium chloride in nitrobenzene, during a short time on a steam bath (poor yield) [4946].

m.p. 93°5–94°5 [5821], 91–93° [4946].

1,1′-(2,5-Dihydroxy-3,6-dimethoxy-1,4-phenylene)bis-ethanone

[34554-37-5] $C_{12}H_{14}O_6$ mol.wt. 254.24



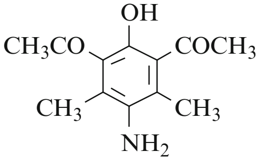
Synthesis

- Obtained (by-product) by metallation of 2,5-dimethoxy-hydroquinonebis[tetrahydropyranyl (2) ether], followed by treatment of the intermediate aryllithium compound with acetic anhydride in THF at r.t. (3–5%) [5822].

m.p. 159° [5822].

1,1′-(5-Amino-2-hydroxy-4,6-dimethyl-1,3-phenylene)bis-ethanone

[85450-76-6] $C_{12}H_{15}NO_3$ mol.wt. 221.26



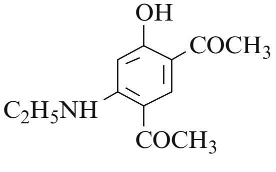
Synthesis

- Preparation by catalytic hydrogenation of 3-acetyl-2-hydroxy-4,6-dimethyl-5-nitroacetophenone in ethanol in the presence of 10% Pd/C at 40° for 3 days (61%) [5809].

m.p. 111° [5809].

1,1′-[4-(Ethylamino)-6-hydroxy-1,3-phenylene]bis-ethanone

[79324-49-5] $C_{12}H_{15}NO_3$ mol.wt. 221.26



Syntheses

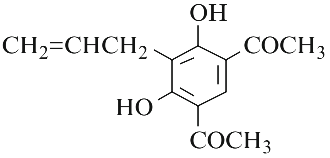
- Obtained by hydrolysis of 2-(ethylamino)-5-[1-(ethylimino)-ethyl]-4-hydroxyacetophenone on heating with aqueous hydrochloric acid [5746].
- Also obtained first, by treatment of 3-acetamido-4,6-diacetyl-phenol with sodium hydride in N-methylpyrrolidone at <5°.

After 15 min, the mixture was treated with ethyl iodide at <5° for 2 h, then acidified with concentrated hydrochloric acid/ethanol (1:1) and heated to reflux for 2.5 h (63%) [5744].

m.p. 103–104° [5744], 99° [5746].

1,1′-[4,6-Dihydroxy-5-(2-propenyl)-1,3-phenylene]bis-ethanone

[75631-42-4] $C_{13}H_{14}O_4$ mol.wt. 234.25



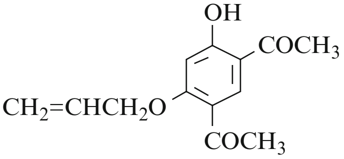
Syntheses

- Obtained (poor yield) by Claisen rearrangement of 4,6-di-acetylresorcinol diallyl ether (m.p. 92°) in refluxing N,N-diethylaniline for 6 h (6%) [5823].
- Also refer to: [5746].

m.p. 93–94° [5823]; MS [5823].

1,1′-[4-Hydroxy-6-(2-propenyloxy)-1,3-phenylene]bis-ethanone

[117156-74-8] $C_{13}H_{14}O_4$ mol.wt. 234.25



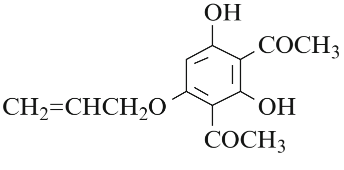
Synthesis

- Obtained by partial deallylation of 3-acetyl-4,6-di-(allyloxy)acetophenone (m.p. 92°) in trifluoroacetic acid, with stirring at 0°. Stirring was continued at r.t. for a further 24 h (70%) [5605].

m.p. 95° [5605]; TLC [5605]; ^{1}H NMR [5605], IR [5605], UV [5605].

1,1′-[2,4-Dihydroxy-6-(2-propenyloxy)-1,3-phenylene]bis-ethanone

[35075-32-2] $C_{13}H_{14}O_5$ mol.wt. 250.24



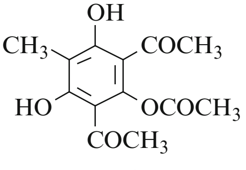
Synthesis

- Obtained by reaction of allyl bromide with 2,4-diacetyl-phloroglucinol in the presence of potassium carbonate in refluxing acetone for 48 h (27%) [5789,5792].

m.p. 111–112° [5789,5792].

1,1′-[2-(Acetyloxy)-4,6-dihydroxy-5-methyl-1,3-phenylene]bis-ethanone

$C_{13}H_{14}O_6$ mol.wt. 266.25



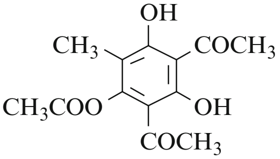
Synthesis

- Obtained by treatment of usnetol with ozone, in 20 parts of chloroform or acetic acid for 1 h [5798], (15–20%) [5824].

m.p. 172° [5824].

1,1′-[4-(Acetyloxy)-2,6-dihydroxy-5-methyl-1,3-phenylene]bis-ethanone

[69150-72-7] $C_{13}H_{14}O_6$ mol.wt. 266.25



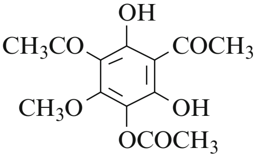
Synthesis

– Obtained by ozonolysis of diacetyldecarbousnic acid in carbon tetrachloride for 4 h at 15° [5797].

m.p. 120–121° [5797]; TLC [5797];
^{1}H NMR [5797], IR [5797], MS [5797].

1,1′-[5-(Acetyloxy)-2,4-dihydroxy-6-methoxy-1,3-phenylene]bis-ethanone

$C_{13}H_{14}O_7$ mol.wt. 282.25



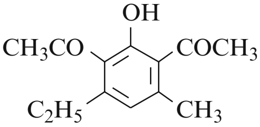
Synthesis

– Obtained by reaction of acetic anhydride with 2,6-di-methoxyhydroquinone diacetate in the presence of boron trifluoride in acetic acid at 30° (max), followed by standing overnight (60%) [5821].

m.p. 98°5–100°2 [5821].

1,1′-(4-Ethyl-2-hydroxy-6-methyl-1,3-phenylene)bis-ethanone

[76716-12-6] $C_{13}H_{16}O_3$ mol.wt. 220.27



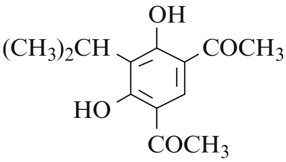
Synthesis

– Obtained by condensation of 2,4,6-heptanetrione (1 mol) and 2,4-hexanedione (1 mol) in the presence of sodium hydroxide in 50% aqueous methanol at 25° for 25 h (41%) [5800].

m.p. 86–89° [5800]; ^{1}H NMR [5800].

1,1′-[4,6-Dihydroxy-5-(1-methylethyl)-1,3-phenylene]bis-ethanone

[75643-06-0] $C_{13}H_{16}O_4$ mol.wt. 236.27

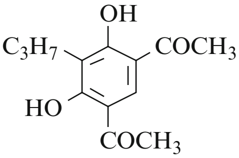


Synthesis

– Refer to: [5825] (Japanese patent).

1,1′-(4,6-Dihydroxy-5-propyl-1,3-phenylene)bis-ethanone

[58805-52-0] $C_{13}H_{16}O_4$ mol.wt. 236.27

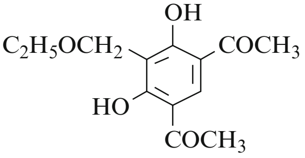


Syntheses

– Obtained by Fries rearrangement of 2-propylresorcinol diacetate with aluminium chloride for 1 h at 130–150° [5789].
– Also refer to: [5746,5825] (Japanese patent).

1,1′-[5-(Ethoxymethyl)-4,6-dihydroxy-1,3-phenylene]bis-ethanone

[58805-51-9] $C_{13}H_{16}O_5$ mol.wt. 252.27



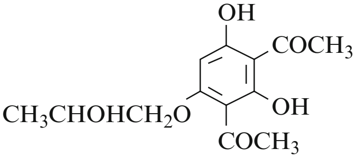
Synthesis

– Obtained by refluxing an ethanolic solution of 4,6-diacetyl-2-(hydroxymethyl)resorcinol in the presence of a small amount of concentrated sulfuric acid for 2 h (84%) [5789].

m.p. 163–165° [5789].

1,1′-[2,4-Dihydroxy-6-(2-hydroxypropoxy)-1,3-phenylene]bis-ethanone

[23937-51-1] $C_{13}H_{16}O_6$ mol.wt. 268.27



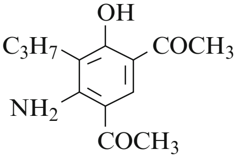
Synthesis

– Preparation by reaction of propylene oxide with 2,4-diacetylphloroglucinol in the presence of benzyl trimethyl ammonium hydroxide at 100° for 48 h (63%) [5792].

m.p. 152–154° [5792].

1,1′-(4-Amino-6-hydroxy-5-propyl-1,3-phenylene)bis-ethanone

[79324-47-3] $C_{13}H_{17}NO_3$ mol.wt. 235.28



Syntheses

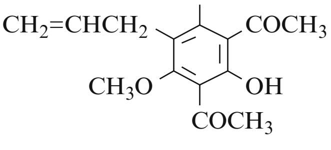
– Preparation by hydrogenation of 2-allyl-3-amino-4,6-di-acetylphenol (SM) in ethanol in the presence of 5% Pd/C at atmospheric pressure and at r.t. (82%). SM was obtained by Claisen rearrangement of 3-(allyloxy)-4,6-diacetylaniline (m.p. 131–134°) in N-methylpyrrolidone under nitrogen at 200° for 3 h [5744].

– Also refer to: [5801,5826,5827].

m.p. 138–139° [5744].

1,1′-[2,4-Dihydroxy-6-methoxy-5-(2-propenyl)-1,3-phenylene]bis-ethanone

[37126-09-3] $C_{14}H_{16}O_5$ mol.wt. 264.28



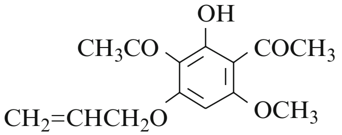
Synthesis

– Obtained by Claisen rearrangement of 2,6-diacetyl-3-(allyloxy)-5-methoxyphenol in refluxing tetralin for 3.5 h (25%) [5789].

m.p. 84°5–85° [5789].

1,1′-[2-Hydroxy-4-methoxy-6-(2-propenyloxy)-1,3-phenylene]bis-ethanone

[58805-53-1] $C_{14}H_{16}O_5$ mol.wt. 264.28



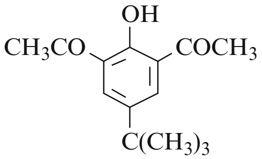
Synthesis

- Obtained by reaction of methyl iodide with 2,4-diacetyl-5-(allyloxy)resorcinol in the presence of potassium carbonate in refluxing acetone for 16 h (quantitative yield) [5789].

red oil (crude product) [5789]; b.p.$_{0.3}$ 148–162° [5789].

1,1′-[5-(1,1-Dimethylethyl)-2-hydroxy-1,3-phenylene]bis-ethanone

[203004-96-0] $C_{14}H_{18}O_3$ mol.wt. 234.20



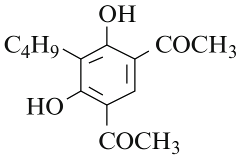
Synthesis

- Obtained by Friedel–Crafts acylation of p-tert-butylphenol with acetyl chloride (3 mol) in nitrobenzene in the presence of aluminium chloride at 65–70° overnight (45%) [5766].

m.p. 53–54° [5766]; ^{1}H NMR [5766], MS [5766].

1,1′-(5-Butyl-4,6-dihydroxy-1,3-phenylene)bis-ethanone

[40449-66-9] $C_{14}H_{18}O_4$ mol.wt. 250.29



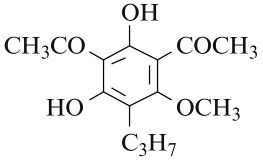
Syntheses

- Obtained by Fries rearrangement of 2-butylresorcinol diacetate with aluminium chloride for 1 h at 130–150° [5789].
- Also refer to: [5793,5825] (Japanese patent).

m.p. 61–64° [5789].

1,1′-(2,4-Dihydroxy-6-methoxy-5-propyl-1,3-phenylene)bis-ethanone

[37126-10-6] $C_{14}H_{18}O_5$ mol.wt. 266.29



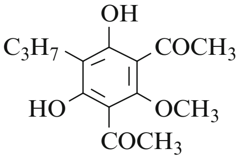
Synthesis

- Obtained by hydrogenation of 2,4-diacetyl-6-allyl-5-methoxyresorcinol in ethanol over 5% Pd/C at three atmospheres for 2 h (61%) [5789].

m.p. 48–49° [5789].

1,1′-(4,6-Dihydroxy-2-methoxy-5-propyl-1,3-phenylene)bis-ethanone

[37126-08-2] $C_{14}H_{18}O_5$ mol.wt. 266.29



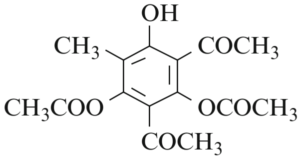
Syntheses

- Obtained by hydrogenation of 2,4-diacetyl-6-allyl-5-(benzyl-oxy)-3-methoxyphenol in ethanol containing hydrochloric acid at three atmospheres in the presence of 5% Pd/C for 1 h (61%) [5789].
- Also refer to: [5793,5828].

b.p.$_{0.6}$ 150–170° [5789]; m.p. 80° [5789].

1,1′-[2,4-(Diacetyloxy)-6-hydroxy-5-methyl-1,3-phenylene]bis-ethanone

$C_{15}H_{16}O_7$ mol.wt. 308.29



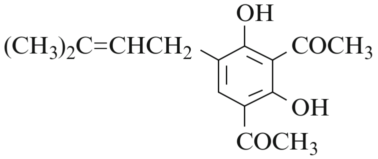
Synthesis

- Obtained from the ozonid of diacetyldecarbousnic acid ($C_{21}H_{22}O_{11}$, m.p. 146°) by treatment with boiling 3% methanolic hydrogen chloride for 5 min (73%) [5798].

m.p. 116° [5798]; sublimation without decomposition at 110°/12 mm [5798].

1,1′-[2,4-Dihydroxy-5-(3-methyl-2-butenyl)-1,3-phenylene]bis-ethanone

[117374-55-7] $C_{15}H_{18}O_4$ mol.wt. 262.31



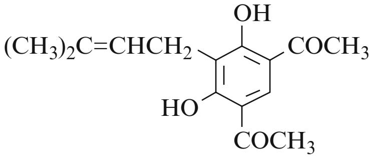
Syntheses

- Obtained by thermal rearrangement,
  - of 3-acetyl-2,4-bis(3-methyl-2-butenyloxy)-acetophenone (m.p. 62°) in refluxing N,N-dimethylaniline for 1.5 h (11%) [5829];
  - of 5-acetyl-2,4-bis(3,3-dimethylallyloxy)-acetophenone (m.p. 103–104°), in refluxing N,N-dimethylaniline for 3 h (6%) [5830], in refluxing n-decane for 18 h (6%) [5830], in refluxing o-xylene for 90 h (10%) [5830] or by heating in a sealed tube at 150° for 18 h (6%) or at 185° for 8 h (2%) [5830].

m.p. 65–66° [5830]; TLC [5830]; ^{1}H NMR [5830], IR [5830], UV [5830], MS [5830].

1,1′-[4,6-Dihydroxy-5-(3-methyl-2-butenyl)-1,3-phenylene]bis-ethanone

[117374-56-8] $C_{15}H_{18}O_4$ mol.wt. 262.31



Syntheses

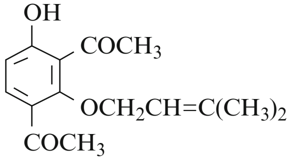
- Obtained (poor yields) by thermal rearangement of 5-acetyl-2,4-bis(3, 3-dimethylallyloxy)acetophenone (m.p. 103–104°),

- in refluxing n-decane for 18 h (8%) [5830];
- in refluxing o-xylene for 90 h (11%) [5830];
- by heating in a sealed tube at 150° for 8 h (10%) [5830].

m.p. 89–90° [5830]; TLC [5830]; ^{1}H NMR [5830], IR [5830], UV [5830], MS [5830].

1,1′-[4-Hydroxy-2-[(3-methyl-2-butenyl)oxy]-1,3-phenylene]bis-ethanone

[136811-82-0] $C_{15}H_{18}O_4$ mol.wt. 262.31



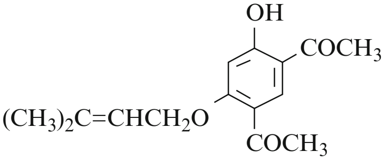
Syntheses

- Obtained by Claisen rearrangement of 3-acetyl-2,4-bis (3-methyl-2-butenyloxy)acetophenone with palladium chloride-bis(acetonitrile) in refluxing dioxane for 45 min (29%) [5657].
- Also refer to: [5831].

m.p. 51° [5657]; ^{1}H NMR [5657], IR [5657], UV [5657], MS [5657].

1,1′-[4-Hydroxy-6-[(3-methyl-2-butenyl)oxy]-1,3-phenylene]bis-ethanone

[136811-83-1] $C_{15}H_{18}O_4$ mol.wt. 262.31



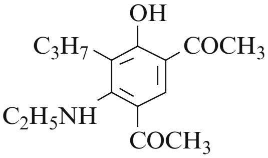
Syntheses

- Obtained by Claisen rearrangement of 5-acetyl-2,4-bis(3-methyl-2-butenyloxy) acetophenone,
  - with palladium chloride-bis(acetonitrile) in refluxing dioxane for 4 h (95%) [5657];
  - with boron trifluoride etherate at r.t. for 7 days (48%) [5657].
- Also refer to: [5831].

m.p. 83–87° [5657]; ^{1}H NMR [5657], UV [5657], MS [5657].

1,1′-[4-(Ethylamino)-6-hydroxy-5-propyl-1,3-phenylene]bis-ethanone

[79324-51-9] $C_{15}H_{21}NO_3$ mol.wt. 263.34



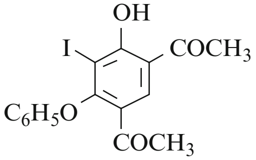
Syntheses

- Preparation by hydrogenation of 2-allyl-3-hydroxy-4,6-di-acetyl-N-ethylaniline (SM) in ethanol in the presence of 5% Pd/C at 15–20 psi for 2.5 h (63%). SM was obtained by Claisen rearrangement of 3-(allyloxy)-4,6-diacetyl-N-ethyl-aniline (m.p. 82–83°) in refluxing N-methylpyrrolidone under nitrogen for 1 h [5744].
- Also refer to: [5826].

m.p. 114–115° [5744].

1,1′-(4-Hydroxy-5-iodo-6-phenoxy-1,3-phenylene)bis-ethanone

[145489-92-5] $C_{16}H_{13}IO_4$ mol.wt. 396.18



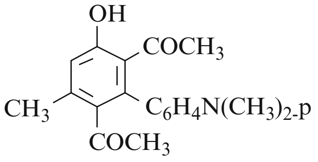
Synthesis

– Preparation by thermal rearrangement of 4,6-diacetyl-3-hydroxy-2-phenyliodoniophenolate (SM) in refluxing acetonitrile for 30 min (50%). SM was obtained by reaction of iodosobenzene diacetate with resodiacetophenone in methanol in the presence of potassium hydroxide, at 0° for 30 min (40%, m.p. 120–130°) [5709].

m.p. 132–136° [5709]; ^{1}H NMR [5709], IR [5709], MS [5709].

1,1′-[4′-(Dimethylamino)-3-hydroxy-5-methyl[1,1′-biphenyl]-2,6-diyl] bis-ethanone

[108909-50-8] $C_{19}H_{21}NO_3$ mol.wt. 311.38



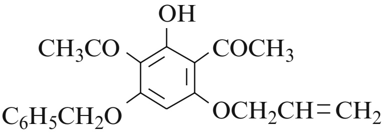
Synthesis

– Obtained by aromatization of 4,6-diacetyl-5-[4-(dimethyl-amino)phenyl]-3-methyl-2-cyclohexen-1-one (m.p. 117°) with bromine in chloroform (45%) or by heating at 170° for 3 h [5832].

m.p. 153° [5832]; IR [5832].

1,1′-[2-Hydroxy-4-(phenylmethoxy)-6-(2-propenyloxy)-1,3-phenylene] bis-ethanone

[37126-05-9] $C_{20}H_{20}O_5$ mol.wt. 340.38



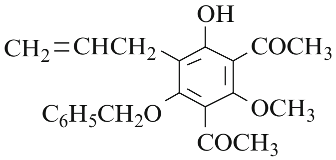
Synthesis

– Obtained by reaction of benzyl chloride with 2,4-di-acetyl-5-(allyloxy)resorcinol in the presence of potassium carbonate and potassium iodide in refluxing acetone for 43 h (33%) [5789].

m.p. 92° [5789].

1,1′-[4-Hydroxy-2-methoxy-6-(phenylmethoxy)-5-(2-propenyl)-1,3-phenylene]bis-ethanone

$C_{21}H_{22}O_5$ mol.wt. 354.40



Syntheses

– Obtained by Claisen rearrangement of 3-acetyl-4-allyloxy-6-benzyloxy-2-methoxyacetophenone in refluxing tetralin under nitrogen for 4 h (36%) [5789].

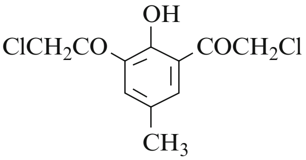
– Also refer to: [5793,5828].

oil [5789].

20.1.2 Diversely Substituted Acetyl Groups

1,1′-(2-Hydroxy-5-methyl-1,3-phenylene)bis[2-chloroethanone]

[99984-12-0] $C_{11}H_{10}Cl_2O_3$ mol.wt. 261.10



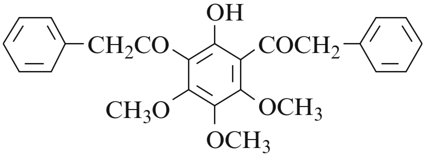
Syntheses

- Obtained (by-product) by Friedel–Crafts acylation of p-cresol methyl ether with chloroacetyl chloride in the presence of aluminium chloride in refluxing carbon disulfide for 4–5 h [4625].
- Also obtained by Friedel–Crafts acylation of p-cresol with chloroacetyl chloride in the presence of aluminium chloride at 140° for 4 h [4623].

m.p. 168° [4623], 167–168° [4625].

1,1′-(2-Hydroxy-4,5,6-trimethoxy-1,3-phenylene)bis[2-phenylethanone]
3,4,5-Trimethoxy-2,6-bis(phenylacetyl)phenol

[22228-86-0] $C_{25}H_{24}O_6$ mol.wt. 420.46



Syntheses

- Obtained by Friedel–Crafts acylation of 6-hydroxy-2,3,4-trimethoxyphenyl benzyl ketone with phenylacetyl chloride in the presence of aluminium chloride [5251].
- Also obtained (by-product) by Friedel–Crafts acylation of antiarol with phenylacetyl chloride in the presence of aluminium chloride (<3%) [5251].

m.p. 106° [5251]; ^{1}H NMR [5251], IR [5251].

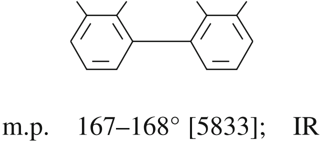
20.2 Acetyl Groups Located on Different Rings

20.2.1 Diphenyl Derivatives

Symmetrical ketones

1,1′-(2,2′-Dihydroxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[60312-44-9] $C_{16}H_{14}O_4$ mol.wt. 270.28



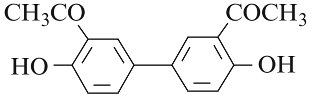
Synthesis

- Obtained by alkaline degradation of 8,8′-bichromonyl (m.p. 326°) with refluxing 10% sodium hydroxide for 20 min [5833].

m.p. 167–168° [5833]; IR [5833].

1,1′-(4,4′-Dihydroxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[13938-28-8] $C_{16}H_{14}O_4$ mol.wt. 270.28



Syntheses

– Preparation by Fries rearrangement of 4,4′-diacetoxy-biphenyl,

- with a mixture of aluminium chloride and sodium chloride (5:1, w/w), first at 140°, then at 200° for 2 min (melting) (70%) [5834];
- with a mixture of aluminium chloride and zinc chloride (5:1, w/w), first at 140°, then at 200° for 2 min (melting) (82%) [5834];
- with aluminium chloride at 120° [5835], (75%) [5836,5837];
- with aluminium chloride in refluxing chlorobenzene for 24 h (19%) [5838].

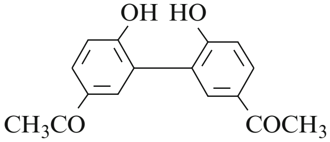
– Also obtained by alkaline degradation of 6,6′-bichromonyl (m.p. 298–299°) with refluxing 10% sodium hydroxide for 20 min [5833].

– Also refer to: [5839,5840].

m.p. 219–220° [5836], 219–219°5 [5837], 215–216° [5838], 209–210° [5833]; IR [5838].

1,1′-(6,6′-Dihydroxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[23080-48-0] $C_{16}H_{14}O_4$ mol.wt. 270.28



Syntheses

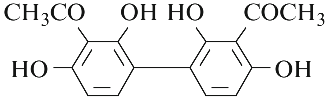
– Preparation by Fries rearrangement of 2,2′-diacetoxy-biphenyl (1 mol) with aluminium chloride (1 mol) at 110–120° for 4 h (52%) [5835].

– Also obtained by Friedel–Crafts acylation of 2,2′-di-hydroxybiphenyl (1 mol) with acetyl chloride (4 mol) in the presence of aluminium chloride (4 mol) at 110–120° [5835].

m.p. 275° [5835].

1,1′-(2,2′,4,4′-Tetrahydroxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[2551-44-2] $C_{16}H_{14}O_6$ mol.wt. 302.28



Syntheses

– Obtained by refluxing a solution of 9,9′-di-O-methylergoflavinone—m.p. 330° (d)—in 50% aqueous potassium hydroxide for 30 min (10%) [5841].

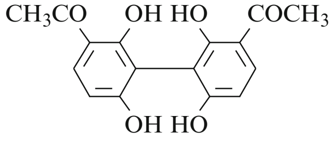
– Also obtained (poor yield) by Fries rearrangement of 6,6′-bi-(7-acetoxy-4-methylcoumarin) (m.p. 327°) with aluminium chloride at 260° for 75 min, followed by heating the resulting 6,6′-bi-(8-acetyl-7-hydroxy-4-methylcoumarin) with 20% (w/v) aqueous sodium hydroxide on a steam bath for 5 h under nitrogen (<3%) [5842].

- Also obtained by Fries rearrangement of 2,2′,4,4′-tetraacetoxybiphenyl (1 mol) with aluminium chloride (4 mol),
  - without solvent at 130–140° for 4 h (27%) [5835];
  - in nitrobenzene at r.t. for 24 h [5835].
- Also refer to: [5843].

m.p. 249–250° [5841], 248–249° [5842], 245° [5835]; IR [5841].

1,1′-(2,2′,6,6′-Tetrahydroxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[93107-98-3]



$C_{16}H_{14}O_6$ mol.wt. 302.28

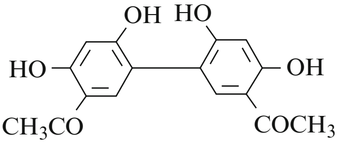
Synthesis

- Obtained by oxidative coupling of resacetophenone using silica-bound ferric chloride, first in methylene chloride, then, after solvent elimination, the residue left at r.t. for a week (13%) [5844].

m.p. 286–287° [5844]; TLC [5844]; IR [5844].

1,1′-(4,4′,6,6′-Tetrahydroxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[23080-53-7]



$C_{16}H_{14}O_6$ mol.wt. 302.28

Syntheses

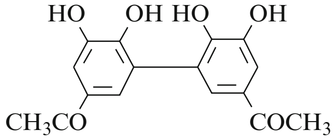
- Obtained by Fries rearrangement of 2,2′,4,4′-tetra-acetoxybiphenyl (1 mol) with aluminium chloride (4 mol),
  - without solvent at 130–140° for 4 h (30%) [5835];
  - in nitrobenzene at r.t. for 24 h [5835].
- Also obtained by oxidative coupling of resacetophenone using silica-bound ferric chloride, first in methylene chloride, then, after solvent elimination, the residue left at r.t. for a week (10%) [5844].

m.p. 236° [5835], 197–198° [5844]. One of the reported melting points is obviously wrong.

^{1}H NMR [5844], IR [5844]; TLC [5844].

1,1′-(5,5′,6,6′-Tetrahydroxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[224030-70-0]



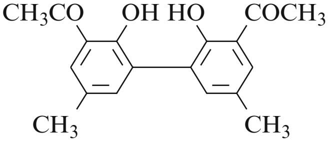
$C_{16}H_{14}O_6$ mol.wt. 302.28

Synthesis

- Refer to: [5845].

1,1′-(2,2′-Dihydroxy-5,5′-dimethyl[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[13938-30-2] $C_{18}H_{18}O_4$ mol.wt. 298.34



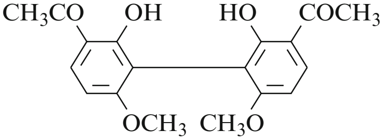
Syntheses

- Obtained by hydrolysis of [m,m′-bitolyl]-6,6′-diol-5,5′-bis(2-methyl-1,3-dioxolan-2-yl), its diketal—[24046-06-8], $C_{22}H_{26}O_6$, m.p. 169°5–170°—with hydrogen chloride in methanol (almost quantitative yield) [5846].
- Also obtained (poor yield) by Fries rearrangement of 2,2′-diacetoxy-5,5′-dimethylbiphenyl with aluminium chloride in nitrobenzene at 120° for 2 h (11%) [5846].
- Also refer to: [5847].

m.p. 189°5–190° [5846]; TLC [5846];
^{1}H NMR [5846,5847], IR [5846,5847].

1,1′-(2,2′-Dihydroxy-6,6′-dimethoxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[93107-89-2] $C_{18}H_{18}O_6$ mol.wt. 330.34



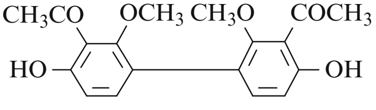
Synthesis

- Obtained by oxidative coupling of resacetophenone 4-methyl ether using silica-bound ferric chloride, first in methylene chloride, then, after solvent elimination, the residue left at r.t. for a week (10%) [5844].

m.p. 322° [5844]; TLC [5844]; IR [5844].

1,1′-(4,4′-Dihydroxy-2,2′-dimethoxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[2551-38-4] $C_{18}H_{18}O_6$ mol.wt. 330.34



Syntheses

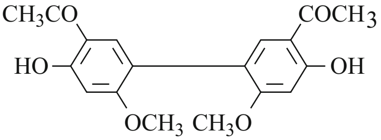
- Obtained by degradation of 1,1′,9,9′-tetra-O-methylergoflavinone in 10% sodium hydroxide solution at reflux for 2 h under nitrogen [5848], (40%) [5841].
- Also obtained by degradation of 9,9′-di-O-ethyl-1,1′-di-O-methylergoflavinone (m.p. 271°) in 1% sodium hydroxide solution on a steam bath for 2 h (31%) [5841].
- Also obtained by degradation of 6,6′-bis(5-methoxy-2-methylchromone) in 80% (w/v) aqueous sodium hydroxide solution on a steam bath for 2.5 h under nitrogen (11%) [5848].
- Also obtained by degradation of 1,1′,9-tri-O-methylchrysinone A in 10% aqueous sodium hydroxide solution on a steam bath for 1.5 h under nitrogen (9%) [5849].

- Also obtained by degradation of 1,1′,9,9′-tetra-O-methylergoflavin (m.p. 282° (d)) with barium hydroxide octahydrate in boiling water for 5 h (5%) [5850].
- Also obtained by degradation of 1,1′-di-O-methyl-9,9′-di-O-ethylergoflavin (m.p. 280° (d)) in a 50% (w/v) barium hydroxide octahydrate solution in boiling water for 5 h (<2%) [5841].

m.p. 168–169° [5848], 168° [5841,5850];
^{1}H NMR [5848], IR [5841,5848,5849], UV [5841].

1,1′-(4,4′-Dihydroxy-6,6′-dimethoxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[93107-86-9] $C_{18}H_{18}O_6$ mol.wt. 330.34



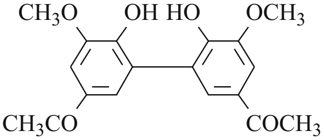
Synthesis

- Obtained by oxidative coupling of resacetophenone 4-methyl ether using silica-bound ferric chloride, first in methylene chloride, then, after solvent elimination, the residue left at r.t. for a week (10%) [5844].

m.p. 125° [5844]; TLC [5844]; ^{1}H NMR [5844], IR [5844].

1,1′-(6,6′-Dihydroxy-5,5′-dimethoxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[29799-22-2] $C_{18}H_{18}O_6$ mol.wt. 330.34



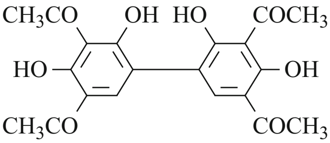
Syntheses

- Obtained by alkaline CuO oxidation of lignin (compound **Vn-Vn**) named dehydrodiacetovanillone [5851].
- Also refer to: [5852].

GC [5851], GC-MS [5851].
N.B.: Utilisation in the long-lasting perfume compositions [5852].

1,1-(2,2′,4,4′-Tetrahydroxy[1,1′-biphenyl]-3,3′,5,5′-tetrayl)tetrakis-ethanone

[23080-58-2] $C_{20}H_{18}O_8$ mol.wt. 386.36



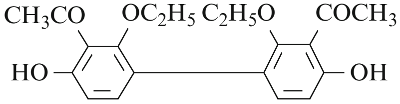
Syntheses

- Obtained by Fries rearrangement of 2,2′, 4,4′-tetra-acetoxybiphenyl (1 mol) with aluminium chloride (4 mol),
  - without solvent at 130–140° for 4 h (20%) [5835];
  - in nitrobenzene at r.t. for 24 h [5835].
- Also obtained by Friedel–Crafts acylation of 2,2′,4,4′-tetrahydroxybiphenyl (1 mol) with acetyl chloride (4 mol) in the presence of aluminium chloride (7 mol) at 120° for 2 h [5835].

m.p. 302° [5835].

1,1′-(2,2′-Diethoxy-4,4′-dihydroxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[35292-40-1]



$C_{20}H_{22}O_6$ mol.wt. 358.39

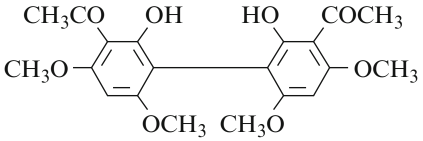
Synthesis

- Obtained by degradation of 1,1′,9,9′-tetra-O-ethyl-ergoflavinone (m.p. 303–306°) (SM) in 10% aqueous sodium hydroxide solution on asteam bath for 2 h under nitrogen (22%). SM was obtained by oxidation of 1,1′,9,9′-tetra-O-ethylergoflavin with Jones reagent [5848].

m.p. 99° [5848]; ^{1}H NMR [5848].

1,1′-(2,2′-Dihydroxy-4,4′,6,6′-tetramethoxy[1,1′-biphenyl]-3,3′-diyl) bis-ethanone

[35134-71-5]



$C_{20}H_{22}O_8$ mol.wt. 390.39

Syntheses

- Obtained by oxidative coupling of phloraceto-phenone 4,6-dimethyl ether *(Xanthoxylin)* using silica-bound ferric chloride, either at 43–45° for 6 days (81%) [5853], or first in methylene chloride, then, after solvent elimination, the residue left at r.t. for a week (40%) [5844].
- Obtained by Friedel–Crafts acylation of 2,2′,4,4′,6,6′-hexamethoxybiphenyl,
  - with acetic anhydride in the presence of aluminium chloride in nitrobenzene (20%) [5854];
  - with acetyl chloride in the presence of aluminium chloride in ethyl ether (15%) [5854].

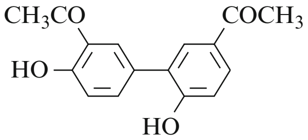
m.p. 262–264° [5844], 254–257° [5854], 211–212° [5853]. One of the reported melting points is obviously wrong.

^{1}H NMR [5844,5853,5854], ^{13}C NMR [5853], IR [5844,5853], MS [5853].

Asymmetrical ketones

1,1′-(4,6′-Dihydroxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[131844-78-5]



$C_{16}H_{14}O_4$ mol.wt. 270.28

Synthesis

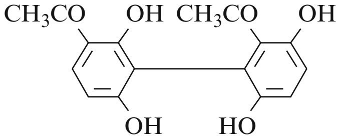
- Refer to: [4664] (European patent).

1,1′-(2′,3,6,6′-Tetrahydroxy[1,1′-biphenyl]-2,3′-diyl)bis-ethanone *(Cynandione A)*

1,1′-(2,2′,3′,6,-Tetrahydroxy[1,1′-biphenyl]-3,4′-diyl)bis-ethanone

(Present name attributed by CAS Registry Handbook Number Section-1995 Supplement)

[168706-29-4] $C_{16}H_{14}O_6$ mol.wt. 302.28



Isolation from natural sources

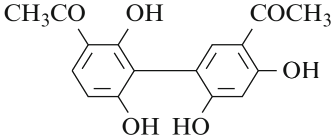
- From the *Cynanchum taiwanianum* (Asclepiadaceae) [5855,5856,5857, 5858,5859].
- From the *Cynanchum wilfordii* Hemsley (Asclepiadaceae) [5860,5861,5862,5863].

N.B.: The structure of *Cynandione A*, previously designated as 3′,4-diacetyl-2,2′,3,6′-tetrahydroxy-biphenyl [5855], has been revised as 2,3′-diacetyl-2′,3,6,6′-tetrahydroxybiphenyl [5859] in 1997.

m.p. 203–206° [5855]; ^{1}H NMR [5855,5857,5859], ^{1}H NMR-NOE [5859], ^{13}C NMR [5855], IR [5855,5859], UV [5855], MS [5855,5857].

1,1′-(2,4′,6,6′-Tetrahydroxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[93108-00-0] $C_{16}H_{14}O_6$ mol.wt. 302.28



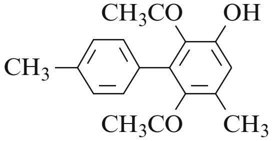
Synthesis

- Obtained by oxidative coupling of resacetophenone using silica-bound ferric chloride, first in methylene chloride, then, after solvent elimination, the residue left at r.t. for a week (15%) [5844].

m.p. 130° [5844]; TLC [5844]; IR [5844].

1,1′-(3-Hydroxy-4′,5-dimethyl[1,1′-biphenyl]-2,6-diyl)bis-ethanone

[108909-49-5] $C_{18}H_{18}O_3$ mol.wt. 282.34



Synthesis

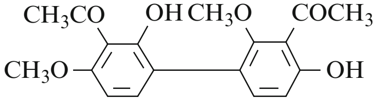
- Obtained by aromatization of 4,6-diacetyl-3-methyl-5-(4-methylphenyl)-2-cyclohexen-1-one (m.p. 140°) with bromine in chloroform (40%) or by heating at 170° for 3 h [5832].

m.p. 165° [5832].

1,1′-(2,4′-Dihydroxy-2′,4-dimethoxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[35287-64-0]

$C_{18}H_{18}O_6$ mol.wt. 330.34



Synthesis

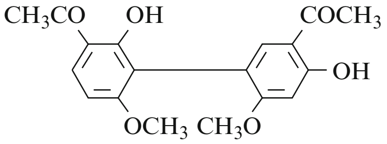
- Obtained by degradation of 6,8′-bis (5-methoxy-2-methylchromone) (m.p. 250–252°) in 80% (w/v) aqueous sodium hydroxide solution on a steam bath for 2 h (13%) [5848].

m.p. 130–132° [5848]; ^{1}H NMR [5848].

1,1′-(2,4′-Dihydroxy-6,6′-dimethoxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[93107-87-0]

$C_{18}H_{18}O_6$ mol.wt. 330.34



Synthesis

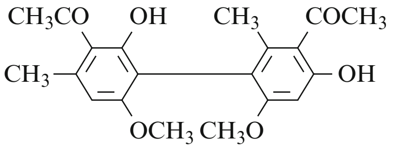
- Obtained by oxidative coupling of resaceto-phenone 4-methyl ether using silica-bound ferric chloride, first in methylene chloride, then, after solvent elimination, the residue left at r.t. for a week (20%) [5844].

m.p. 194–195° [5844]; TLC [5844]; ^{1}H NMR [5844], IR [5844].

1,1′-(2,4′-Dihydroxy-6,6′-dimethoxy-2′,4-dimethyl[1,1′-biphenyl]-3,3′-diyl) bis-ethanone

[110325-66-1]

$C_{20}H_{22}O_6$ mol.wt. 358.40



Synthesis

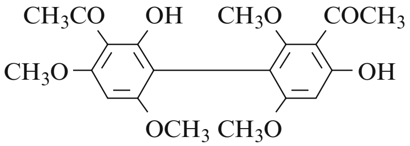
- Obtained by alkaline hydrolysis of *desertorin* C (m.p. 235–237°) (SM) in a mixture of 10% aqueous potassium hydroxide and dioxane (1:1) at reflux for 2 h (54%). SM was isolated from *Emericella desertorum* Samson & Mouchacca strain CBS 653.73. [5864].

m.p. 149–150° [5864];
^{1}H NMR [5864], ^{13}C NMR [5864], IR [5864], UV [5864], MS [5864].

1,1′-(2,4′-Dihydroxy-2′,4,6,6′-tetramethoxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[37879-22-4]

$C_{20}H_{22}O_8$ mol.wt. 390.39



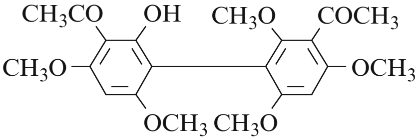
Synthesis

- Obtained by Friedel–Crafts acylation of 2,2′,4,4′,6,6′-hexamethoxybiphenyl with acetyl chloride in the presence of aluminium chloride in ethyl ether (23%) [5854].

m.p. 185–186° [5854]; ^{1}H NMR [5854].

1,1′-(2-Hydroxy-2′,4,4′,6,6′-pentamethoxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone

[37879-23-5]



$C_{21}H_{24}O_8$ mol.wt. 404.42

Synthesis

- Obtained by Friedel–Crafts acylation of 2,2′,4,4′,6,6′-hexamethoxybiphenyl with acetyl chloride in the presence of aluminium chloride in ethyl ether (16%) [5854].

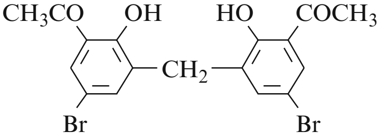
m.p. 213–215° [5854]; ^{1}H NMR [5854].

20.2.2 Diphenylmethane Derivatives

20.2.2.1 Unsubstituted Acetyl Groups

1,1-[Methylenebis(5-bromo-2-hydroxy-3,1-phenylene)]bis-ethanone

[83143-04-8]



$C_{17}H_{14}Br_2O_4$ mol.wt. 442.10

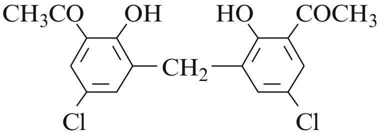
Synthesis

- Preparation by Fries rearrangement of 2,2′-diacetoxy-5,5′-dibromodiphenylmethane with aluminium chloride at 160–180° for 20 min (60%) [5865].

m.p. 232–235° [5865].

1,1′-[Methylenebis(5-chloro-2-hydroxy-3,1-phenylene)]bis-ethanone

[60011-06-5]



$C_{17}H_{14}Cl_2O_4$ mol.wt. 353.20

Syntheses

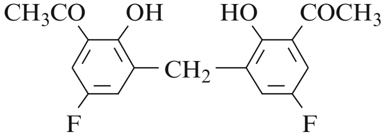
- Preparation by Fries rearrangement of 2,2′-diacetoxy-5,5′-dichlorodiphenylmethane with aluminium chloride at 150–155° for 20 min (80–85%) [5866], at 160–180° for 20 min (70%) [5865] or at 170–180° for 30 min (90%) [5867], (40%) [5868].
- Also obtained by adding 38% formaldehyde to a cooled solution of 2-acetyl-4-chlorophenol (SM) in concentrated sulfuric acid/methanol solution (2:1 v/v) and stirring for 1.5 h at 20°, then for 4 h at 60–70° (quantitative yield) [5867] or first at −10° under stirring for 2 h, then at r.t. overnight (60%) [5868]. SM was prepared by Fries rearrangement of p-chlorophenyl acetate with aluminium chloride at 160° for 20 min (98%, m.p. 54°) [5867].

N.B.: Mono- and binuclear complexes of Cu (II), Ni (II), Co (II), Fe (III) and V (V) [5868].

m.p. 202–203° [5865,5866,5867,5868]; sublimation 155–160°/0.01 mm [5867]; X-ray data [5866,5869,5870].

1,1′-[Methylenebis(5-fluoro-2-hydroxy-3,1-phenylene)]bis-ethanone

[78563-09-4]



$C_{17}H_{14}F_2O_4$ mol.wt. 310.78

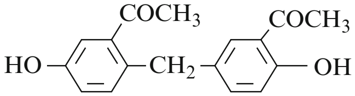
Synthesis

- Preparation by Fries rearrangement of 2,2′-diacetoxy-5,5′-difluorodiphenylmethane with aluminium chloride at 160–180° for 20 min (64%) [5865].

m.p. 155–156° [5865]; ^{13}C NMR [5865].

1-[2-[(3-Acetyl-4-hydroxyphenyl)methyl]-5-hydroxyphenyl]ethanone

[52977-39-6]



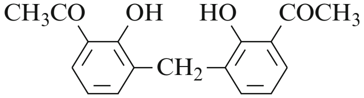
$C_{17}H_{16}O_4$ mol.wt. 284.31

Synthesis

- Refer to: [5871] (Russian patent).

1,1′-[Methylenebis(2-hydroxy-3,1-phenylene)]bis-ethanone

[60312-53-0]



$C_{17}H_{16}O_4$ mol.wt. 284.31

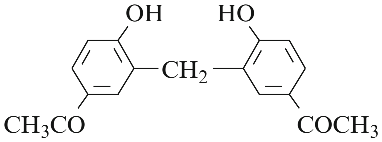
Syntheses

- Obtained by Fries rearrangement of 2,2′-diacetoxy-diphenylmethane with aluminium chloride, first at 140° for 5 min, then at 160–180° for 20 min (70%) [5865].
- Also obtained by alkaline degradation of 8,8′-bichromonyl methane (m.p. 222–223°) with refluxing aqueous 10% sodium hydroxide for 20 min [5833].

m.p. 183–184° [5865], 108–109° [5833]. One of the reported melting points is obviously wrong. Sublimation at 220°/0.03 mm [5865];
IR [5833], MS [5865].

1,1′-[Methylenebis(4-hydroxy-3,1-phenylene)]bis-ethanone

[38782-68-2]



$C_{17}H_{16}O_4$ mol.wt. 284.31

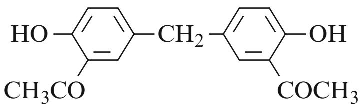
Syntheses

- Obtained by Fries rearrangement of 2,2′-diacetoxy-diphenylmethane with aluminium chloride,
  - in nitrobenzene at 45° for 3 h (30%) [5872];
  - without solvent (by-product), first at 140° for 5 min, then at 160–180° for 20 min (20%) [5865].

m.p. 272–274° [5872], 271–274° [5865];
sublimation at 180–190°/0.035 mm [5865]; MS [5865].

1,1′-[Methylenebis(6-hydroxy-3,1-phenylene)]bis-ethanone

[28467-22-3] $C_{17}H_{16}O_4$ mol.wt. 284.31



Syntheses

– Obtained by Fries rearrangement of 4,4′-diacetoxy-diphenylmethane,

- with aluminium chloride at 130–140° for 1 h (50%) [5873];
- with aluminium chloride and sodium chloride mixture at 140–150° for 4 h (28%) [5874,5875].

– Also obtained by reaction of 1,3,5-trioxane with o-hydroxyacetophenone in acetic acid in the presence of 98% sulfuric acid under nitrogen at 95–100° for 24 h [5840].

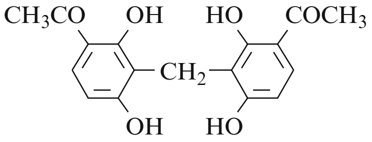
– Also obtained by alkaline degradation of 6,6′-bichromonyl methane (m.p. 193–194°) with refluxing aqueous 10% sodium hydroxide for 20 min [5833].

– Also refer to: [5876].

m.p. 156–157° [5874,5875], 155–156° [5833], 155° [5873], 130° [5840].
One of the reported melting points is obviously wrong.
IR [5840], UV [5840], MS [5840].

1,1′-[Methylenebis(2,4-dihydroxy-3,1-phenylene)]bis-ethanone

[10508-84-6] $C_{17}H_{16}O_6$ mol.wt. 316.31



Syntheses

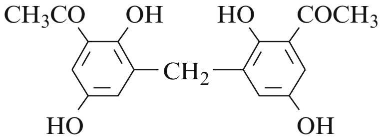
– Obtained by treatment of resacetophenone with methylene iodide in the presence of ethanolic sodium ethoxide for 18 h at r.t., then for 1 h at 60–70° [5877] (19%) [5878].

– Also refer to: [5876].

m.p. 204–205° [5878]; UV [5878].

1,1′-[Methylenebis(2,5-dihydroxy-3,1-phenylene)]bis-ethanone

[78563-10-7] $C_{17}H_{16}O_6$ mol.wt. 316.31

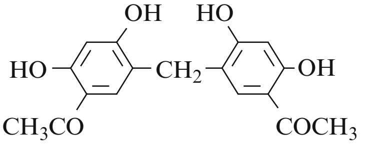


Synthesis

– Preparation by Fries rearrangement of 2,2′,5,5′-tetra-acetoxydiphenylmethane with aluminium chloride at 150–155° for 20 min (50%) [5866].

m.p. 227–228° [5866]; IR [5866], MS [5866].

1,1′-[Methylenebis(4,6-dihydroxy-3,1-phenylene)]bis-ethanone



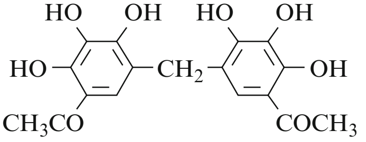
$C_{17}H_{16}O_6$ mol.wt. 316.31

Synthesis

- Obtained by heating at reflux a mixture of resaceto-phenone, 40% formaldehyde and concentrated hydrochloric acid for 2 h [5879].

m.p. >250° [5879].

1,1′-[Methylenebis(4,5,6-trihydroxy-3,1-phenylene)]bis-ethanone



$C_{17}H_{16}O_8$ mol.wt. 348.31

Synthesis

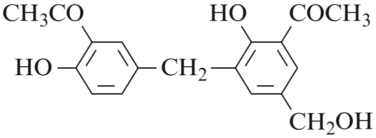
- Obtained by reaction of formaldehyde with gallaceto-phenone in the presence of hydrogen chloride [5182,5879].

m.p. 265° [5879].

1-[3-[(3-Acetyl-4-hydroxyphenyl)methyl]-2-hydroxy-5-(hydroxymethyl) phenyl]ethanone

2′,6′′′-Dihydroxy-5′-(hydroxymethyl)-3′,3′′′-methylenediacetophenone

[30787-44-1]



$C_{18}H_{18}O_5$ mol.wt. 314.34

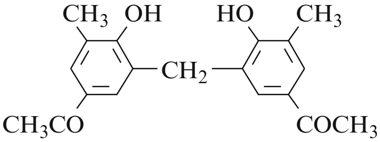
Synthesis

- Obtained by hydrolyzing 2-hydroxy-5-(chloro-methyl)acetophenone [5880,5881].

1,1′-[Methylenebis(4-hydroxy-5-methyl-3,1-phenylene)]bis-ethanone

[38782-67-1]

$C_{19}H_{20}O_4$ mol.wt. 312.37



Synthesis

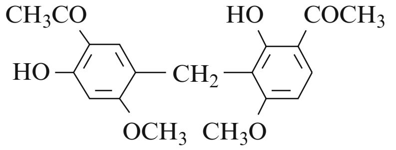
- Preparation by Fries rearrangement of 2,2′-di-acetoxy-3,3′-dimethyldiphenyl-methane in nitrobenzene with aluminium chloride at 60° for 3 h [5882], (59%) [5872].

m.p. 257° [5872]; ^{1}H NMR [5882], IR [5882].

1-[3-[(5-Acetyl-4-hydroxy-2-methoxyphenyl)methyl]-2-hydroxy-4-methoxyphenyl]ethanone

[71204-08-5]

$C_{19}H_{20}O_6$ mol.wt. 344.36



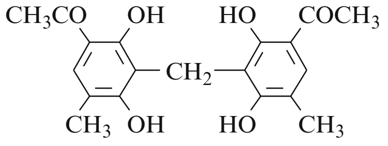
Synthesis

- Obtained by acid-catalyzed condensation of formaldehyde with 2-hydroxy-4-methoxy-acetophenone in the presence of 35% aqueous sulfuric acid [4897].

m.p. 161–162° [4897]; ^{1}H NMR [4897].

1,1′-[Methylenebis(2,4-dihydroxy-5-methyl-3,1-phenylene)]bis-ethanone

$C_{19}H_{20}O_6$ mol.wt. 344.36



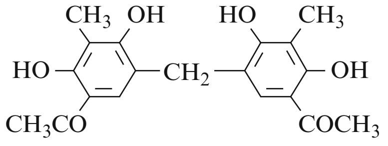
Synthesis

- Preparation by action of 40% aqueous formaldehyde with 2,4-dihydroxy-5-methylacetophenone in ethanol in the presence of concentrated sulfuric acid at 10° (77%) [5883].

m.p. 258° (d) [5883].

1,1′-[Methylenebis(4,6-dihydroxy-5-methyl-3,1-phenylene)]bis-ethanone

$C_{19}H_{20}O_6$ mol.wt. 344.36



Synthesis

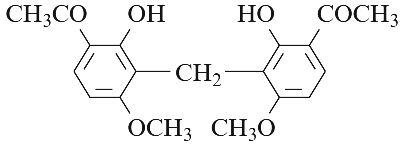
- Preparation by action of 40% aqueous formaldehyde with 2,4-dihydroxy-3-methylacetophenone in ethanol in the presence of concentrated sulfuric acid at r.t. for 3 days (68%) [5883].

m.p. 263–264° [5883].

1,1′-[Methylenebis(2-hydroxy-4-methoxy-3,1-phenylene)]bis-ethanone

[28466-42-4]

$C_{19}H_{20}O_6$ mol.wt. 344.36



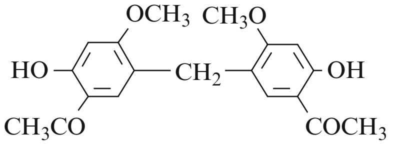
Synthesis

- Obtained by acid-catalyzed condensation of formaldehyde with 2-hydroxy-4-methoxy-acetophenone in the presence of 35% aqueous sulfuric acid [4897].

m.p. 255–256° [4897];
diacetate derivative: m.p. 161–162° [4897]; ^{1}H NMR [4897].

1,1′-[Methylenebis(6-hydroxy-4-methoxy-3,1-phenylene)]bis-ethanone

[71204-07-4]



$C_{19}H_{20}O_6$ mol.wt. 344.36

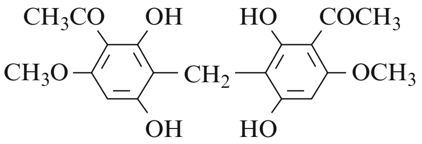
Synthesis

- Obtained by acid-catalyzed condensation of formaldehyde with 2-hydroxy-4-methoxy-acetophenone in the presence of 35% aqueous sulfuric acid [4897].

m.p. 204–205° [4897]; ^{1}H NMR [4897].

1,1′-[Methylenebis(2,4-dihydroxy-6-methoxy-3,1-phenylene)] bis-ethanone *(Didemethylpseudoaspidin)*

[142382-28-3]



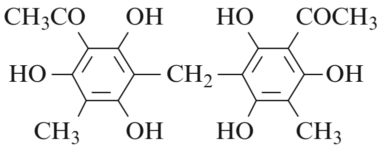
$C_{19}H_{20}O_8$ mol.wt. 376.36

Isolation from natural sources

- From the roots of *Euphorbia ebracteolata Hayata* (Euphorbiaceae) [5884].
- From the roots of *Euphorbia kansui* (Euphorbiaceae) [5885].

m.p. 232–233° [5885];
^{1}H NMR [5885], ^{13}C NMR [5885], IR [5885], EIMS [5885].

1,1′-[Methylenebis(2,4,6-trihydroxy-5-methyl-3,1-phenylene)]bis-ethanone



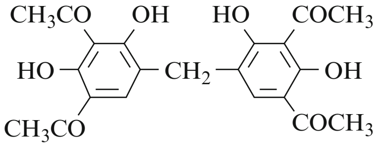
$C_{19}H_{20}O_8$ mol.wt. 376.36

Synthesis

- Obtained by hydrolysis of 8-isobutyryl-5-methoxy-methyleneoxy-2,2-dimethylchroman-7-ol (SM) in the presence of 2,4,6-trihydroxy-3-methyl-acetophenone. The hydrolysis of SM proceeds with the liberation of formaldehyde which condenses with phenol under these acidic conditions [5716].

1,1′,1″,1‴-[Methylenebis(2,4-dihydroxy-5,1,3-benzenetriyl)]tetrakis-ethanone

[84422-46-8]



$C_{21}H_{20}O_8$ mol.wt. 400.39

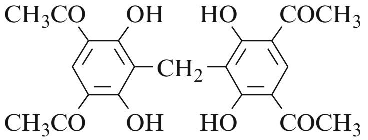
Synthesis

- Obtained by reaction of formaldehyde with 3-acetylresacetophenone in the presence of dilute sulfuric acid in refluxing ethanol for 16 h (42%) [5661].

m.p. 184° [5661]; ^{1}H NMR [5661].

1,1′,1″,1‴-[Methylenebis(4,6-dihydroxy-5,1,3-benzenetriyl)]tetrakis-ethanone

[84422-38-8] $C_{21}H_{20}O_8$ mol.wt. 400.39



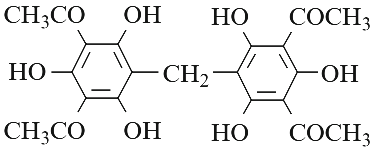
Synthesis

- Obtained by acid-catalysed condensation of formaldehyde with 5-acetylresacetophenone in the presence of dilute sulfuric acid in refluxing ethanol for 30 min [5661].

m.p. 345–346° [5661]; TLC [5661].

1,1′,1″,1‴-[Methylenebis(2,4,6-trihydroxy-5,1,3-benzenetriyl)]tetrakis-ethanone

[58316-48-6] $C_{21}H_{20}O_{10}$ mol.wt. 432.39



Synthesis

- Obtained by condensation of formaldehyde with 2,4-diacetylphloroglucinol (35%) [5608].

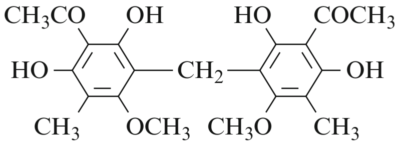
Isolation from natural sources

- From the culture fluid of *Pseudomonas aurantiaca* [5886].
- Also refer to: [5720] (compound **28**).

m.p. 284–286° [5608].

1,1′-[Methylenebis(2,6-dihydroxy-4-methoxy-5-methyl-3,1-phenylene)] bis-ethanone *(Mallotophenone)*

[98569-63-2] $C_{21}H_{24}O_8$ mol.wt. 404.42



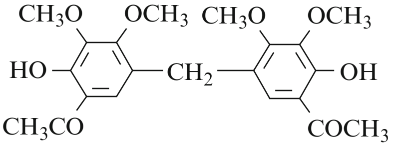
Isolation from natural sources

- From the pericarps of *Mallotus japonicus* Muell. Arg. (Euphorbiaceae) [5887,5888,5889,5890,5891,5892, 5893,5894,5895].

m.p. 223–225° [5893];
^{1}H NMR [5889,5893], ^{13}C NMR [5889,5893,5894], IR [5893],
UV [5893], MS [5893]; Cytotoxicity [5894].

1,1′-[Methylenebis(6-hydroxy-4,5-dimethoxy-3,1-phenylene)]bis-ethanone

[71204-14-3]



$C_{21}H_{24}O_8$ mol.wt. 404.42

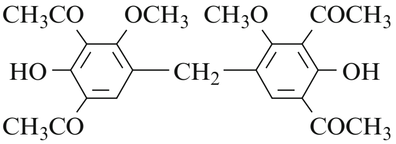
Synthesis

- Obtained from 2-hydroxy-3,4-dimethoxy-acetophenone with formaldehyde and 35% aqueous sulfuric acid [4897].

m.p. 141–142° [4897].

1,1′,1″,1‴-[Methylenebis(2-hydroxy-4-methoxy-5,1,3-benzenetriyl)]tetrakis-ethanone

[84422-49-1]



$C_{23}H_{24}O_8$ mol.wt. 428.44

Synthesis

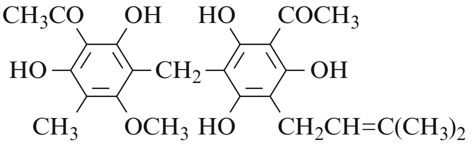
- Obtained by partial methylation of 3,3′,5,5′-tetra-acetyl-2,2′,4,4′-tetrahydroxydiphenylmethane with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 12 h (29%) [5661].

m.p. 132° [5661]; ^{1}H NMR [5661];
TLC [5661], column chromatography [5661].

1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-2,4,6-trihydroxy-5-(3-methyl-2-butenyl)phenyl]ethanone

*Proposed name mallotojaponin**

[86828-07-1]



$C_{24}H_{28}O_8$ mol.wt. 444.48

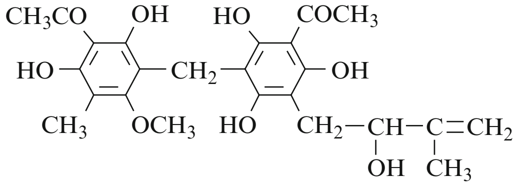
Isolation from natural sources

- From the pericarps of *Mallotus japonicus* Muell. Arg. (Euphorbiaceae) [5896]* [5887, 5889,5890,5891,5892,5893,5894, 5897,5898].

m.p. 190–191° [5893], 188–189° [5898];
^{1}H NMR [5893,5898], ^{13}C NMR [5897,5898], IR [5893,5898],
UV [5893,5898], MS [5893,5898]; Cytotoxicity [5894].

1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-2,4,6-trihydroxy-5-(2-hydroxy-3-methyl-3-butenyl)phenyl]ethanone *(proposed name mallotolerin)**

[86828-08-2]



$C_{24}H_{28}O_9$ mol.wt. 460.48

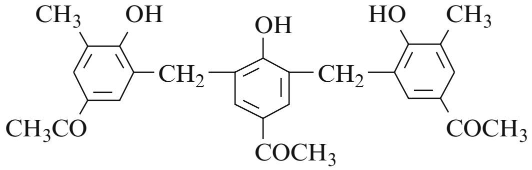
Isolation from natural sources

- From the pericarps of *Mallotus japonicus* Muell. Arg. (Euphorbiaceae) [5896]* [5889,5890,5891, 5892,5894], [5897,5898].

m.p. 197–199° [5898];
^{1}H NMR [5892,5898], ^{13}C NMR [5898], IR [5892,5898], UV [5892,5898] MS [5892,5898]; Cytotoxicity [5894].

1-[3,5-Bis[(5-acetyl-2-hydroxy-3-methylphenyl)methyl]-4-hydroxyphenyl]ethanone

[38782-69-3]



$C_{28}H_{28}O_6$ mol.wt. 460.53

Synthesis

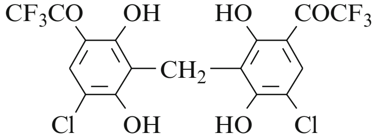
- Obtained by Fries rearrangement of 2,6-bis (2-acetoxy-3-methylbenzyl)-acetoxybenzene with aluminium chloride in nitrobenzene at 50° for 5 h (41%) [5872].

m.p. 242° [5872].

20.2.2.2 Halogenated Acetyl Groups

1,1′-[Methylenebis(5-chloro-2,4-dihydroxy-3,1-phenylene)]bis[2,2,2-trifluoroethanone

[65240-40-6]



$C_{17}H_8Cl_2F_6O_6$ mol.wt. 493.14

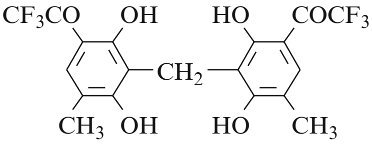
Synthesis

- Preparation by reaction of paraformaldehyde with 5-chloro-2,4-dihydroxy-α,α,α-trifluoro-acetophenone in methanol in the presence of concentrated sulfuric acid at 0° for 5 h (87%) [4689].

m.p. 205° [4689].

1,1′-[Methylenebis(2,4-dihydroxy-5-methyl-3,1-phenylene)]bis[2,2,2-trifluoroethanone

[65240-30-4]



m.p. 234° [4689].

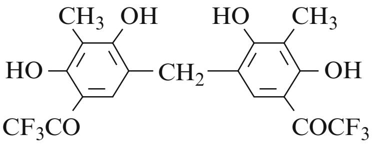
$C_{19}H_{14}F_6O_6$ mol.wt. 452.31

Synthesis

- Preparation by reaction of paraformaldehyde with 2,4-dihydroxy-5-methyl-α,α,α-trifluoro-acetophenone at 140° (92%) [4689].

1,1′-[Methylenebis(4,6-dihydroxy-5-methyl-3,1-phenylene)]bis[2,2,2-trifluoroethanone

[65240-39-3]



m.p. 195° [4689].

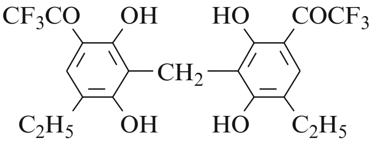
$C_{19}H_{14}F_6O_6$ mol.wt. 452.31

Synthesis

- Preparation by reaction of paraformaldehyde with 2,4-dihydroxy-3-methyl-α,α,α-trifluoro-acetophenone in methanol in the presence of concentrated sulfuric acid at 0° for 5 h (87%) [4689].

1,1′-[Methylenebis(5-ethyl-2,4-dihydroxy-3,1-phenylene)]bis[2,2,2-trifluoroethanone

[65240-29-1]



m.p. 170° [4689].

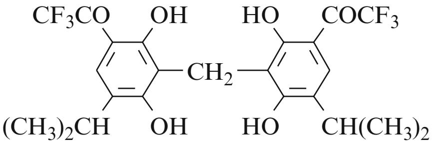
$C_{21}H_{18}F_6O_6$ mol.wt. 480.36

Synthesis

- Preparation by reaction of paraformaldehyde with 2,4-dihydroxy-5-ethyl-α,α,α-trifluoroacetophenone at 140° for 1 h (92%) [4689].

1,1′-[Methylenebis[2,4-dihydroxy-5-(1-methylethyl)-3,1-phenylene]]bis[2,2,2-trifluoroethanone

[65240-35-9]



m.p. 140° [4689].

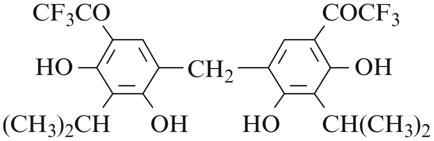
$C_{23}H_{22}F_6O_6$ mol.wt. 508.41

Synthesis

- Preparation by reaction of paraformaldehyde with 2,4-dihydroxy-5-isopropyl-α,α,α-tri-fluoroacetophenone at 140° (89%) [4689].

1,1′-[Methylenebis[4,6-dihydroxy-5-(1-methylethyl)-3,1-phenylene]] bis[2,2,2-trifluoroethanone

[65240-38-2]



m.p. 123° [4689].

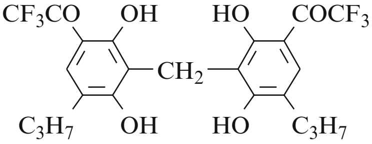
$C_{23}H_{22}F_6O_6$ mol.wt. 508.41

Synthesis

- Preparation by reaction of paraformaldehyde with 2,4-dihydroxy-3-isopropyl-α,α,α-tri-fluoroacetophenone at 140° (90%) [4689].

1,1′-[Methylenebis(2,4-dihydroxy-5-propyl-3,1-phenylene)]bis [2,2,2-trifluoroethanone

[65240-31-5]



m.p. 153° [4689].

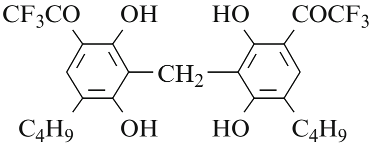
$C_{23}H_{22}F_6O_6$ mol.wt. 508.41

Synthesis

- Preparation by reaction of paraformaldehyde with 2,4-dihydroxy-5-propyl-α,α,α-trifluoro-acetophenone at 140° (90%) [4689].

1,1′-[Methylenebis(5-butyl-2,4-dihydroxy-3,1-phenylene)]bis [2,2,2-trifluoroethanone

[65290-78-0]



m.p. 145° [4689].

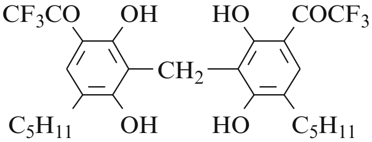
$C_{25}H_{26}F_6O_6$ mol.wt. 536.47

Synthesis

- Preparation by reaction of paraformaldehyde with 5-butyl-2,4-dihydroxy-α,α,α-trifluoroacetophenone at 140° (91%) [4689].

1,1′-[Methylenebis(2,4-dihydroxy-5-pentyl-3,1-phenylene)]bis [2,2,2-trifluoroethanone

[65240-32-6]



m.p. 131° [4689].

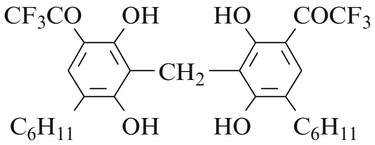
$C_{27}H_{30}F_6O_6$ mol.wt. 564.52

Synthesis

- Preparation by reaction of paraformaldehyde with 2,4-dihydroxy-5-pentyl-α,α,α-trifluoro-acetophenone at 140° (90%) [4689].

1,1′-[Methylenebis(5-cyclohexyl-2,4-dihydroxy-3,1-phenylene)] bis[2,2,2-trifluoroethanone

[65240-37-1]



m.p. 206° [4689].

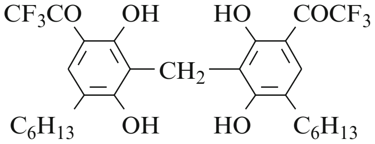
$C_{29}H_{30}F_6O_6$ mol.wt. 588.54

Synthesis

- Preparation by reaction of paraformaldehyde with 5-cyclohexyl-2,4-dihydroxy-α,α,α-trifluoro-acetophenone at 140° (85%) [4689].

1,1′-[Methylenebis(5-hexyl-2,4-dihydroxy-3,1-phenylene)] bis[2,2,2-trifluoroethanone

[65240-33-7]



m.p. 120° [4689].

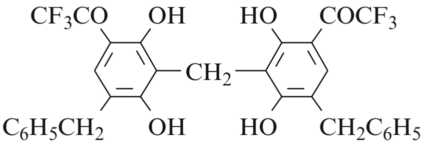
$C_{29}H_{34}F_6O_6$ mol.wt. 592.58

Synthesis

- Preparation by reaction of paraformaldehyde with 2,4-dihydroxy-5-hexyl-α,α,α-trifluoroacetophenone at 140° (93%) [4689].

1,1′-[Methylenebis[2,4-dihydroxy-5-(phenylmethyl)-3,1-phenylene]] bis[2,2,2-trifluoroethanone

[65240-36-0]



m.p. 183° [4689].

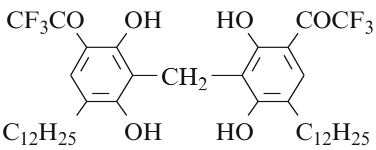
$C_{31}H_{22}F_6O_6$ mol.wt. 604.50

Synthesis

- Preparation by reaction of paraformaldehyde with 5-benzyl-2,4-dihydroxy-α,α,α-trifluoro-acetophenone at 140° (90%) [4689].

1,1′-[Methylenebis(5-dodecyl-2,4-dihydroxy-3,1-phenylene)] bis[2,2,2-trifluoroethanone

[65240-34-8]



m.p. 110° [4689].

$C_{41}H_{58}F_6O_6$ mol.wt. 760.90

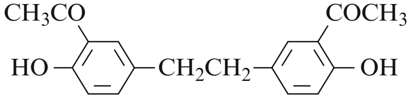
Synthesis

- Preparation by reaction of paraformaldehyde with 2,4-dihydroxy-5-dodecyl-α,α,α-trifluoro-acetophenone at 140° (85%) [4689].

20.2.3 Diphenylalkanes Derivatives and Homologues

1,1′-[1,2-Ethanediylbis(6-hydroxy-3,1-phenylene)]bis-ethanone

[34036-53-8] $C_{18}H_{18}O_4$ mol.wt. 298.34



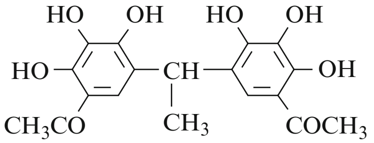
Synthesis

- Preparation by Friedel–Crafts acylation of 1,2-bis(p-methoxyphenyl) ethane (m.p. 127–129°) with acetyl chloride in the presence of aluminium chloride in ethylene dichloride at 65° for 2.5 h (86%) [5899].

m.p. 194–195°5 [5899].

1,1′-[Ethylidenebis(4,5,6-trihydroxy-3,1-phenylene)]bis-ethanone

[128197-51-3] $C_{18}H_{18}O_8$ mol.wt. 362.34

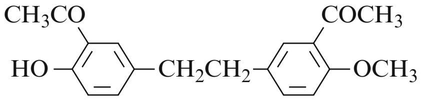


Syntheses

- Refer to: [5900,5901,5902,5903] (Japanese patents).

1-[5-[2-(3-Acetyl-4-hydroxyphenyl)ethyl]-2-methoxyphenyl]ethanone

[27171-77-3] $C_{19}H_{20}O_4$ mol.wt. 312.37



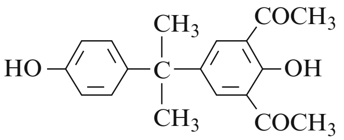
Syntheses

- Obtained by partial methylation of 1,2-bis-(3′-acetyl-4′-hydroxyphenyl)ethane with dimethyl sulfate in ethyl ether in the presence of 2 N aqueous potassium hydroxide at 100° for 8 h (29%) [5899] or with methyl halide [5904].
- Also obtained by acetylation of 1,2-bis(4′-methoxyphenyl)ethane with acetyl chloride in the presence of aluminium chloride [5904].

m.p. 62–63°5 [5899].

1,1′-[2-Hydroxy-5-[1-(4-hydroxyphenyl)-1-methylethyl]-1,3-phenylene] bis-ethanone

[104676-23-5] $C_{19}H_{20}O_4$ mol.wt. 312.37



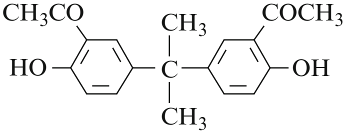
Syntheses

- Obtained by action of acetyl chloride with 2,2-bis (4-acetoxyphenyl)propane in ethylene dichloride in the presence of aluminium chloride at 50° for 5 h (38%) [5905].
- Also refer to: [5906].

m.p. 151–152° [5905].

1,1′-[(1-Methylethylidene)bis(6-hydroxy-3,1-phenylene)]bis-ethanone

[3511-69-1] $C_{19}H_{20}O_4$ mol.wt. 312.37



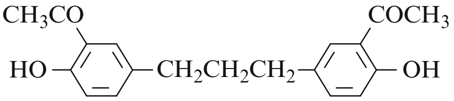
Syntheses

- Preparation by reaction of acetyl chloride,
  - with 2,2-bis(4-ethoxyphenyl)propane (bisphenol A diethyl ether) in the presence of aluminium chloride in methylene chloride at 30° for 30 min (61%) [5905] or in ethylene dichloride at 50° for 3 h (63–65%) [5907];
  - with 2,2-bis(4-methoxyphenyl)propane (bisphenol A dimethyl ether) in the presence of aluminium chloride in ethylene dichloride at 70° for 2 h (45%) [5908].
- Also obtained by Fries rearrangement of bisphenol A diacetate in nitrobenzene,
  - in the presence of aluminium chloride, first at r.t., then at 120–130° for 3 h (28%) [5909];
  - in the presence of titanium tetrachloride, first at r.t. for 24 h, then at 55° for 6 h (11%) [5910].

m.p. 142–143° [5910], 141–142° [5905,5907,5908], 107–109° [5909].
One of the reported melting points is obviously wrong.
^{1}H NMR [5910], ^{13}C NMR [5910], IR [5910].

1,1′-[1,3-Propanediylbis(6-hydroxy-3,1-phenylene)]bis-ethanone

[29668-20-0] $C_{19}H_{20}O_4$ mol.wt. 312.37



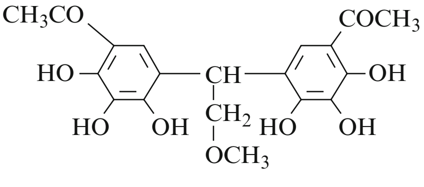
Syntheses

- Obtained by Friedel–Crafts acylation of 1,3-bis(4-methoxyphenyl)propane with acetyl chloride in tetrachloroethane in the presence of aluminium chloride, first at 0–5°, then below 15° overnight [5875].
- Also refer to: [5911,5912,5913].

m.p. 112° [5875].

1,1′-[(2-Methoxyethylidene)bis(4,5,6-trihydroxy-3,1-phenylene)]bis-ethanone

[143868-77-3] $C_{19}H_{20}O_9$ mol.wt. 392.36

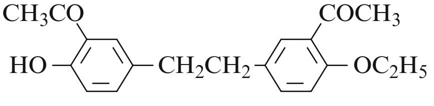


Synthesis

- Refer to: [5902] (Japanese patent).

1-[5-[2-(3-Acetyl-4-hydroxyphenyl)ethyl]-2-ethoxyphenyl]ethanone

[27171-79-5] $C_{20}H_{22}O_4$ mol.wt. 326.39



Syntheses

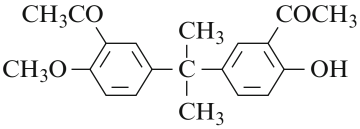
– Obtained by partial ethylation of 1,2-bis-(3′-acetyl-4′-hydroxyphenyl)ethane with ethyl iodide in the presence of potassium carbonate in refluxing acetone for 12 h (17%) [5899] or with ethyl halide [5904].
– Also obtained by acetylation of 1,2-bis(4-ethoxyphenyl)ethane with acetyl chloride in the presence of aluminium chloride [5904].

m.p. 75°5–76° [5899].

1-[5-[1-(3-Acetyl-4-hydroxyphenyl)-1-methylethyl]-2-methoxyphenyl]ethanone

6′-Hydroxy-6‴-methoxy-3′,3‴-isopropylidenediacetophenone

[27171-78-4] $C_{20}H_{22}O_4$ mol.wt. 326.39



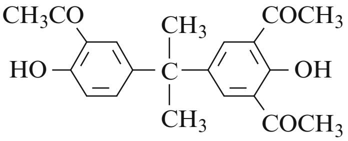
Synthesis

– Preparation by acetylation of bisphenol A dimethyl ether with acetyl chloride in the presence of aluminium chloride, and subsequent partial methylation with methyl bromide of the obtained 2,2-bis (3-acetyl-4-hydroxyphenyl)propane [5904].

^{1}H NMR [5904], IR [5904].

1,1′-[5-[1-(3-Acetyl-4-hydroxyphenyl)-1-methylethyl]-2-hydroxy-1,3-phenylene]bis-ethanone

[104676-24-6] $C_{21}H_{22}O_5$ mol.wt. 354.40



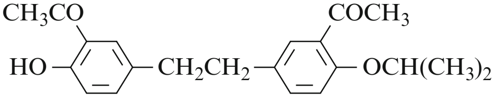
Synthesis

– Obtained by action of acetyl chloride with 2,2-bis (4-acetoxyphenyl)propane in ethylene dichloride in the presence of aluminium chloride at 50° for 30 h (15%) [5905].

m.p. 111°5–112° [5905].

1-[5-[2-(3-Acetyl-4-hydroxyphenyl)ethyl]-2-isopropoxyphenyl]ethanone

[34036-60-7] $C_{21}H_{24}O_4$ mol.wt. 340.42



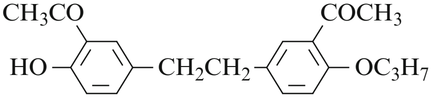
Synthesis

– Obtained by partial alkylation of 1,2-bis-(3-acetyl-4-hydroxyphenyl)ethane with isopropyl iodide in the presence of potassium hydroxide in ethanol at 80° for 14 h (25%) [5899].

m.p. 70–71° [5899].

1-[5-[2-(3-Acetyl-4-hydroxyphenyl)ethyl]-2-propoxyphenyl]ethanone

[27171-80-8] $C_{21}H_{24}O_4$ mol.wt. 340.42



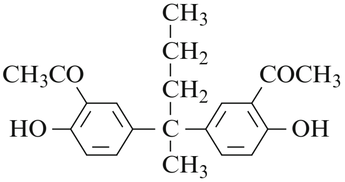
Synthesis

- Preparation by acetylation of 1,2-bis-(4-hydroxyphenyl)ethane diisopropyl ether with acetyl chloride in the presence of aluminium chloride and subsequent partial alkylation of the obtained 1,2-bis(3-acetyl-4-hydroxy-phenyl) ethane with isopropyl bromide (or chloride) [5904].

^{1}H NMR [5904].

1,1′-[(1-Ethylpropylidene)bis(6-hydroxy-3,1-phenylene)]bis-ethanone

[20636-45-7] $C_{21}H_{24}O_4$ mol.wt. 340.42

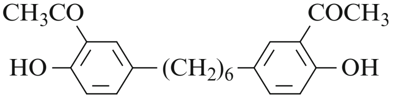


Syntheses

- Refer to: [5907,5914].

1,1′-[1,6-Hexanediylbis(6-hydroxy-3,1-phenylene)]bis-ethanone

[29668-19-7] $C_{22}H_{26}O_4$ mol.wt. 354.45



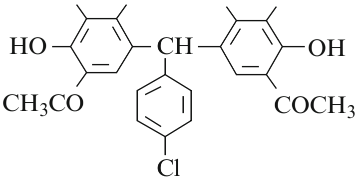
Synthesis

- Obtained by Friedel–Crafts acylation of 1,6-bis-(4-methoxyphenyl)hexane with acetyl chloride in the presence of aluminium chloride in tetrachloro-ethane, first at 0–5°, then below 15° overnight (65%) [5875].

m.p. 97–98° [5875].

1,1′-[[(4-Chlorophenyl)methylene]bis(4,5,6-trihydroxy-3,1-phenylene)] bis-ethanone

$C_{23}H_{19}ClO_8$ mol.wt. 458.85



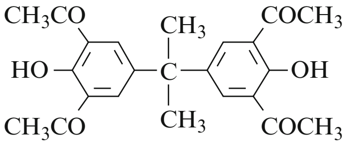
Synthesis

- Obtained by condensation of 1 mol of p-chloro-benzaldehyde (m.p. 47–50°) with two mol of gallacetophenone [5182].

m.p. 230–231° [5182].

1,1′,1″,1‴-[(1-Methylethylidene)bis(2-hydroxy-5,1,3-benzenetriyl)] tetrakis-ethanone

[104676-25-7] $C_{23}H_{24}O_6$ mol.wt. 396.44



Syntheses

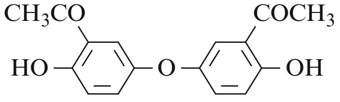
- Preparation by Friedel–Crafts acylation of 2,2-bis (4-ethoxyphenyl)propane (bisphenol A diethyl ether) with acetyl chloride in ethylene dichloride in the presence of aluminium chloride at 60° for 7 h (52%) [5905].
- Also obtained by Fries rearrangement of 2,2-bis(4-acetoxyphenyl)propane (bisphenol A diacetate) with aluminium chloride in an acetyl chloride/ethylene dichloride mixture at 50° for 30 h (10%) [5905].

m.p. 204°5–205° [5905].

20.2.4 Diphenyl Ethers and Related Compounds

1,1′-[Oxybis(6-hydroxy-3,1-phenylene)]bis-ethanone

[28467-08-5] $C_{16}H_{14}O_5$ mol.wt. 286.28



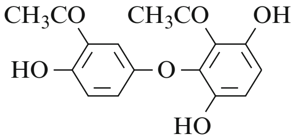
Syntheses

- Obtained by Fries rearrangement of 4,4′-diacetoxy-diphenyl ether with aluminium chloride and sodium chloride at 140–150° for 4 h [5875] or at 140° for 3 h (40%) [5915].
- Also refer to: [5874,5876].

m.p. 185° [5915], 181–183° [5875].

1-[5-(2-Acetyl-3,6-dihydroxyphenoxy)-2-hydroxyphenyl]ethanone

[72926-21-7] $C_{16}H_{14}O_6$ mol.wt. 302.28



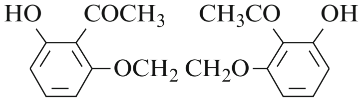
Synthesis

- Preparation by adding an aqueous solution of sodium hydrosulfite to an ethereal solution of 2-acetyl-3-(3-acetyl-4-hydroxyphenoxy)-1, 4-benzoquinone (m.p. 120–121°) and stirring the mixture at r.t. for 30 min (80%) [5916].

^{1}H NMR [5916].

1,1′-[1,2-Ethanediylbis[oxy(6-hydroxy-2,1-phenylene)]]bis-ethanone

[16139-62-1] $C_{18}H_{18}O_6$ mol.wt. 330.34



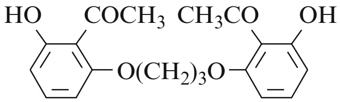
Syntheses

- Obtained by reaction of 1,2-dibromoethane with 2,6-di-hydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 48 h [5875] or for 72 h [5917].
- Also refer to: [5918].

m.p. 188–189° [5875,5917].

1,1′-[1,3-Propanediylbis[oxy(6-hydroxy-2,1-phenylene)]]bis-ethanone

[16150-42-8] $C_{19}H_{20}O_6$ mol.wt. 344.36



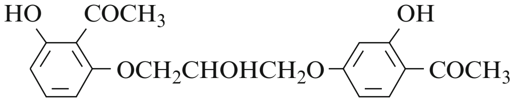
Synthesis

- Obtained by reaction of 1,3-dibromopropane with 2,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 48 h [5875].

m.p. 184–185° [5875].

1-[4-[3-(2-Acetyl-3-hydroxyphenoxy)-2-hydroxypropoxy]-2-hydroxyphenyl]ethanone

[16130-16-8] $C_{19}H_{20}O_7$ mol.wt. 360.36



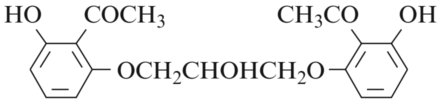
Synthesis

- Obtained by reaction of 2-(3-chloro-2-hydroxypropoxy)-6-hydroxyacetophenone (SM) with resaceto-phenone in the presence of potassium carbonate in refluxing acetone for 48 h (31%). SM was prepared by reaction of epichlorohydrin with 2,6-dihydroxyacetophenone in the presence of benzyltrimethylammonium hydroxide in dioxane at 100° for 72 h (37%, oil) [5875].

m.p. 182–185° [5875].

1,1′-[(2-Hydroxy-1,3-propanediyl)bis[oxy(6-hydroxy-2,1-phenylene)]] bis-ethanone

[16150-44-0] $C_{19}H_{20}O_7$ mol.wt. 360.36



Syntheses

- Preparation by reaction of 2,6-hydroxy-acetophenone,
  - with 1,3-dichloro-2-propanol [5919], in the presence of potassium carbonate in refluxing acetone for 72 h [5917];

- with 1,3-dibromo-2-propanol in the presence of potassium carbonate in refluxing acetone for 48 h (21%) [5875].
- with epichlorohydrin in the presence of potassium hydroxide in refluxing isopropanol for 48 h (59%) [5875].

m.p. 165–166° [5875,5917].

1,1′-[(2-Hydroxy-1,3-propanediyl)bis[oxy(6-hydroxy-3,1-phenylene)]] bis-ethanone

[16139-50-7] $C_{19}H_{20}O_7$ mol.wt. 360.36

Syntheses

– Obtained by reaction of 1,3-dibromo-2-hydroxypropane with 2,5-dihydroxyacetophenone (quinacetophenone) in the presence of potassium carbonate in refluxing acetone for 48 h [5875].

– Also obtained by reaction of epichlorohydrin with quinacetophenone in the presence of potassium hydroxide in boiling isopropanol for 48 h [5875].

m.p. 127–129° [5875].

1,1′-[(2-Hydroxy-1,3-propanediyl)bis[oxy(2-hydroxy-4,1-phenylene)]] bis-ethanone

[16139-45-0] $C_{19}H_{20}O_7$ mol.wt. 360.36

Syntheses

– Obtained by reaction of 1,3-di-bromo-2-hydroxypropane with resacetophenone in the presence of potassium carbonate in refluxing acetone for 48 h [5875].

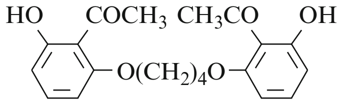
– Also obtained by reaction of epichlorohydrin with resacetophenone in the presence of potassium hydroxide, in boiling acetone for 48 h [5875] or in boiling water for 3 h (20%) (by-product) [5920].

– Also obtained by heating a mixture of epichlorohydrin, sodium, ethanol and resacetophenone under reflux for 4 h [5920].

m.p. 178–180° [5875], 161° [5920]. One of the reported melting points is obviously wrong.

1,1′-[1,4-Butanediylbis[oxy-(6-hydroxy-2,1-phenylene)]]bis-ethanone

[16129-95-6] $C_{20}H_{22}O_6$ mol.wt. 358.39



Synthesis

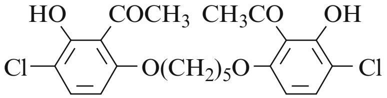
– Preparation by reaction of 1,4-dibromobutane with 2,6-di-hydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 72 h [5917] or for 48 h [5875].

m.p. 219–221° [5875,5917].

1,1′-[1,5-Pentanediylbis[oxy(5-chloro-6-hydroxy-2,1-phenylene)]]bis-ethanone

2′,2‴-(Pentamethylenedioxy)bis[5′-chloro-6′-hydroxyacetophenone

[16130-26-0]



$C_{21}H_{22}Cl_2O_6$ mol.wt. 441.31

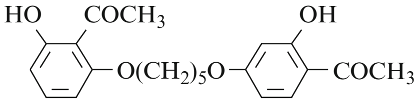
Synthesis

– Preparation by reaction of 1,5-dibromopentane with 3-chloro-2,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 72 h [5917].

m.p. 96° [5917].

1-[4-[[5-(2-Acetyl-3-hydroxyphenoxy)pentyl]oxy]-2-hydroxyphenyl]ethanone

[16130-20-4]



$C_{21}H_{24}O_6$ mol.wt. 372.42

Syntheses

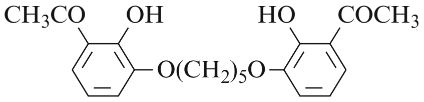
– Obtained by reaction of 2-(5-bromopentyloxy)-6-hydroxyacetophenone (SM) with resaceto-phenone in the presence of potassium carbonate in refluxing acetone for 18 h (60%). SM was formed by reaction of 1,5-dibromopentane with 2,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 20 h (oil, 50%) [5875].

– Also refer to: [5917].

m.p. 91–91°5 [5875,5917].

1,1′-[1,5-Pentanediylbis[oxy(2-hydroxy-3,1-phenylene)]]bis-ethanone

[16139-26-7]



$C_{21}H_{24}O_6$ mol.wt. 372.42

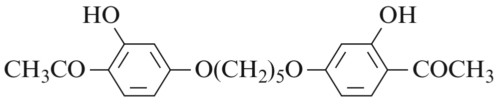
Synthesis

– Obtained by reaction of 1,5-dibromopentane with 2,3-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 48 h [5875,5917].

m.p. 103°5–104°5 [5875,5917].

1,1′-[1,5-Pentanediylbis[oxy(2-hydroxy-4,1-phenylene)]]bis-ethanone

[37086-37-6]



$C_{21}H_{24}O_6$ mol.wt. 372.42

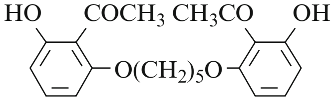
Synthesis

– Obtained by reaction of 1,5-dibromo-pentane with resacetophenone in the presence of potassium carbonate in refluxing acetone for 48 h [5875].

m.p. 119–121° [5875].

1,1′-[1,5-Pentanediylbis[oxy(6-hydroxy-2,1-phenylene)]]bis-ethanone

[16130-01-1] $C_{21}H_{24}O_6$ mol.wt. 372.42



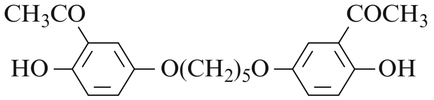
Synthesis

– Preparation by reaction of 1,5-dibromopentane with 2,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 72 h [5917] or for 48 h [5875].

m.p. 131–133° [5875,5917].

1,1′-[1,5-Pentanediylbis[oxy(6-hydroxy-3,1-phenylene)]]bis-ethanone

[16139-42-7] $C_{21}H_{24}O_6$ mol.wt. 372.42



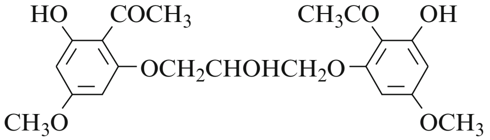
Synthesis

– Obtained by reaction of 1,5-dibromopentane with quinacetophenone in the presence of potassium carbonate in refluxing acetone for 48 h [5875,5917].

m.p. 107–109° [5875,5917].

1,1′-[(2-Hydroxy-1,3-propanediyl)bis[oxy(6-hydroxy-4-methoxy-2,1-phenylene)]]bis-ethanone

[23937-88-4] $C_{21}H_{24}O_9$ mol.wt. 420.42



Syntheses

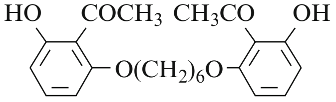
– Obtained by reaction of 1,3-dibromo-2-hydroxypropane with 2,6-dihydroxy-4-methoxyacetophenone in the presence of potassium carbonate in refluxing acetone for 48 h [5875].

– Also obtained by reaction of epichlorohydrin with 2,6-dihydroxy-4-methoxyacetophenone in the presence of potassium hydroxide in refluxing isopropanol for 48 h [5875].

m.p. 108–182° [5875]. A typing error probably occurred in the published data.

1,1′-[1,6-Hexanediylbis[oxy-(6-hydroxy-2,1-phenylene)]]bis-ethanone

[16130-02-2] $C_{22}H_{26}O_6$ mol.wt. 386.44

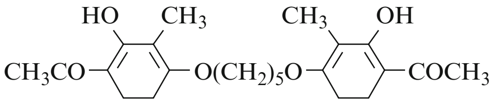


Synthesis

– Preparation by reaction of 1,6-dibromohexane with 2,6-di-hydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 72 h [5917] or for 48 h [5875].

m.p. 147°5–148°5 [5875,5917].

1,1′-[1,5-Pentanediylbis[oxy(2-hydroxy-3-methyl-4,1-phenylene)]] bis-ethanone



$C_{23}H_{28}O_6$ mol.wt. 400.47

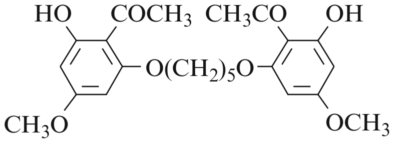
Synthesis

- Preparation by reaction of 1,5-dibromo-pentane with 2,4-dihydroxy-3-methyl-acetophenone in the presence of potassium carbonate in refluxing acetone for 72 h [5917].

m.p. 116–117° [5917].

1,1′-[1,5-Pentanediylbis[oxy(6-hydroxy-4-methoxy-2,1-phenylene)]]bis-ethanone

[23937-90-8]



$C_{23}H_{28}O_8$ mol.wt. 432.47

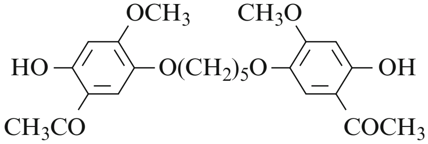
Synthesis

- Obtained by reaction of 1,5-dibromopentane with 2,6-dihydroxy-4-methoxyacetophenone in the presence of potassium carbonate in refluxing acetone for 48 h [5875].

m.p 146–147° [5875].

1,1′-[1,5-Pentanediylbis[oxy(6-hydroxy-4-methoxy-3,1-phenylene)]]bis-ethanone

[23937-59-9]



$C_{23}H_{28}O_8$ mol.wt. 432.47

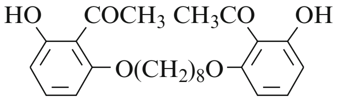
Synthesis

- Obtained by reaction of 1,5-dibromopentane with 2,5-dihydroxy-4-methoxyacetophenone in the presence of potassium carbonate in refluxing acetone for 48 h [5875].

m.p. 145–148° [5875].

1,1′-[1,8-Octanediylbis[oxy-(6-hydroxy-2,1-phenylene)]]bis-ethanone

[16139-58-5]



$C_{24}H_{30}O_6$ mol.wt. 414.50

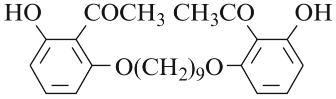
Synthesis

- Preparation by reaction of 1,8-dibromooctane with 2,6-di-hydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 72 h [5917] or for 48 h [5875].

m.p. 108–109° [5875], 107–109° [5917].

1,1′-[1,9-Nonanediylbis[oxy-(6-hydroxy-2,1-phenylene)]]bis-ethanone

[16139-60-9] $C_{25}H_{32}O_6$ mol.wt. 428.53



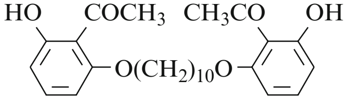
Synthesis

- Preparation by reaction of 1,9-dibromononane with 2,6-di-hydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 72 h [5917] or for 48 h [5875].

m.p. 55–59° [5875,5917].

1,1′-[1,10-Decanediylbis[oxy-(6-hydroxy-2,1-phenylene)]]bis-ethanone

[16258-59-6] $C_{26}H_{34}O_6$ mol.wt. 442.55



Synthesis

- Preparation by reaction of 1,10-dibromodecane with 2,6-dihydroxyacetophenone in the presence of potassium carbonate in refluxing acetone for 72 h [5917] or for 48 h [5875].

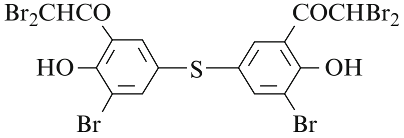
m.p. 102°5–104° [5875,5917].

20.2.5 Diphenyl Sulfide Derivatives and Related Compounds

20.2.5.1 Diphenyl Sulfide Derivatives

1,1v-[Thiobis(5-bromo-6-hydroxy-3,1-phenylene)]bis[2,2-dibromoethanone

$C_{16}H_8Br_6O_4S$ mol.wt. 775.73



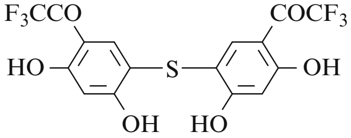
Synthesis

- Obtained by reaction of excess bromine with 3,3′-diacetyl-4,4′-dihydroxydiphenyl sulfide in acetic acid in a boiling water bath for 3 h [4530].

m.p. 168–170° [4530].

1,1′-[Thiobis(4,6-dihydroxy-3,1-phenylene)]bis[2,2,2-trifluoroethanone

[65239-96-5] $C_{16}H_8F_6O_6S$ mol.wt. 442.29

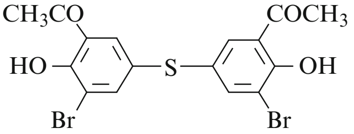


Synthesis

- Obtained by Friedel–Crafts acylation of 2,2′,4,4′-tetrahydroxydiphenylsulfide with trifluoroacetic anhydride in the presence of aluminium chloride in ethylene dichloride at r.t. (10%) [4689].

m.p. 172° [4689].

1,1′-[Thiobis(5-Bromo-6-hydroxy-3,1-phenylene)]bis-ethanone



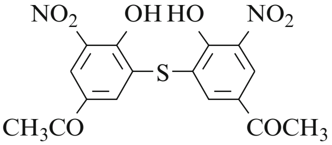
$C_{16}H_{12}Br_2O_4S$ mol.wt. 460.14

Synthesis

- Obtained by reaction of bromine with 3,3′-di-acetyl-4,4′-dihydroxydiphenyl sulfide in acetic acid, first at 90°, then at r.t. for 6 h [4530].

m.p. 218–219° [4530].

1,1′-[Thiobis(4-hydroxy-5-nitro-3,1-phenylene)]bis-ethanone



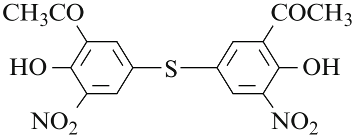
$C_{16}H_{12}N_2O_8S$ mol.wt. 392.35

Synthesis

- Obtained by reaction of thionyl chloride with 4-hydroxy-3-nitroacetophenone in the presence of copper, first at r.t. overnight, then at reflux for 30 min [5921].

m.p. >300° [5921].

1,1′-[Thiobis(6-hydroxy-5-nitro-3,1-phenylene)]bis-ethanone



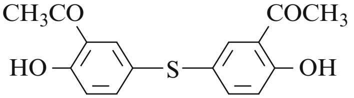
$C_{16}H_{12}N_2O_8S$ mol.wt. 392.35

Synthesis

- Obtained by reaction of 3,3′-diacetyl-4,4′-di-hydroxydiphenyl sulfide with dilute nitric acid at reflux for 2 h [4530].

m.p. 206–208° [4530].

1,1′-[Thiobis(6-hydroxy-3,1-phenylene)]bis-ethanone



$C_{16}H_{14}O_4S$ mol.wt. 302.35

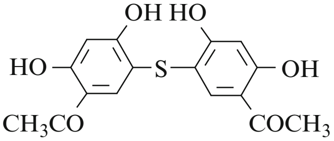
Syntheses

- Obtained by reaction of thionyl chloride or sulfur dichloride with o-hydroxyacetophenone in the presence of copper powder, first at r.t. overnight, then in a boiling water bath for 10 min [4530].
- Also refer to: [5922].

m.p. 196–197° [4530].

1,1′-[Thiobis(4,6-dihydroxy-3,1-phenylene)]bis-ethanone

[56923-41-2] $C_{16}H_{14}O_6S$ mol.wt. 334.35



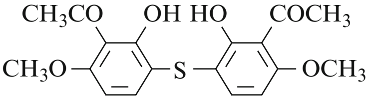
Syntheses

- Obtained by reaction of thionyl chloride with resacetophenone in chloroform in the presence of copper powder, first at 0°, then at r.t. overnight and, the next day, at reflux (60°) for 5 min (11%) [5923].
- Also refer to: [5922].

m.p. 209–210° [5923].

1,1′-[Thiobis(2-hydroxy-6-methoxy-3,1-phenylene)]bis-ethanone

[103154-01-4] $C_{18}H_{18}O_6S$ mol.wt. 362.40



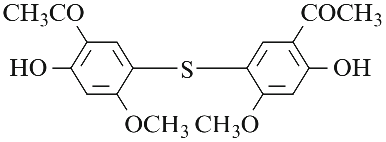
Syntheses

- Obtained by reaction of thionyl chloride with 2-hydroxy-6-methoxyacetophenone in chloroform in the presence of copper powder at r.t. overnight (23%) [4457].
- Also obtained by reaction of sulfur monochloride or sulfur dichloride with 2-hydroxy-6-methoxy-acetophenone in ethyl ether, first at 0° for 1 h and at r.t. overnight (<15%) [4457].

m.p. 184–185° [4457].

1,1′-[Thiobis(6-hydroxy-4-methoxy-3,1-phenylene)]bis-ethanone

[56923-42-3] $C_{18}H_{18}O_6S$ mol.wt. 362.40



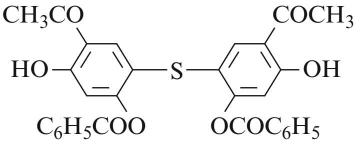
Syntheses

- Obtained by reaction of thionyl chloride with paeonol in chloroform in the presence of copper powder, first at r.t. overnight, then at 60° for 10 min (19%) [4467].
- Also refer to: [5922].

m.p. 223–224° [4467].

1,1′-[Thiobis[4-(benzoyloxy)-6-hydroxy-3,1-phenylene]]bis-ethanone

[56923-50-3] $C_{30}H_{22}O_8S$ mol.wt. 542.57



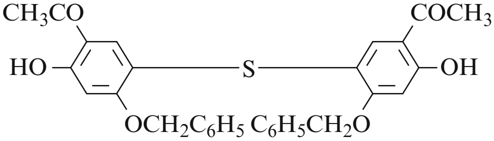
Syntheses

- Obtained by reaction of thionyl chloride or sulfur dichloride with 4-(benzoyloxy)-2-hydroxy-acetophenone in the presence of copper powder in chloroform at 60° for 10 min [5924].
- Also refer to: [5922].

m.p. 228–229° [5924].

1,1′-[Thiobis[6-hydroxy-4-(phenylmethoxy)-3,1-phenylene]]bis-ethanone

[56923-49-0] $C_{30}H_{26}O_6S$ mol.wt. 514.60



Syntheses

– Obtained by reaction of thionyl chloride or sulfur chloride with 4-(benzyloxy)-2-hydroxy-acetophenone in chloroform in the presence of copper powder, first at 0°, then at r.t. overnight and at 60° for 10 min [5925].

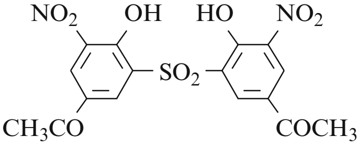
– Also refer to: [5922].

m.p. 202–203° [5925].

20.2.5.2 *Diphenyl Sulfone Derivatives*

1,1′-[Sulfonylbis(4-hydroxy-5-nitro-3,1-phenylene)]bis-ethanone

$C_{16}H_{12}N_2O_{10}S$ mol.wt. 424.34



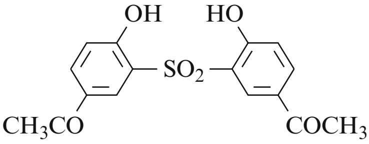
Synthesis

– Obtained by oxidation of 5,5′-diacetyl-2,2′-dihydroxy-3,3′-dinitrodiphenyl sulfide with hydrogen peroxide in acetone at r.t. overnight (74%) [5921].

m.p. 135° [5921].

1,1′-[Sulfonylbis(4-hydroxy-3,1-phenylene)]bis-ethanone

$C_{16}H_{14}O_6S$ mol.wt. 334.35



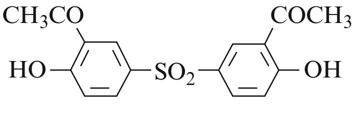
Synthesis

– Obtained by oxidation of 5,5′-diacetyl-2,2′-dihydroxy-diphenyl sulfide with hydrogen peroxide in acetone at r.t. overnight (73%) [5921].

m.p. >300° [5921].

1,1′-[Sulfonylbis(6-hydroxy-3,1-phenylene)]bis-ethanone

[56923-31-0] $C_{16}H_{14}O_6S$ mol.wt. 334.35



Syntheses

– Obtained by Fries rearrangement of bisphenol S diacetate with aluminium chloride (10 equiv.) at 160° (23%) [5926].

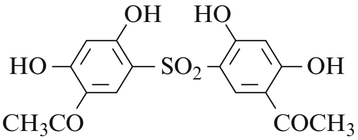
N.B.: The UV irradiation of a diester solution in 0.02 M acetonitrile does not lead to the above mentioned diketone.

- Also obtained by Fries rearrangement of 4,4′-diacetoxydiphenyl sulfone with aluminium chloride (5 equiv.) at 150–160° for 5 h (30%) [5915].
- Also obtained by oxidation of 3,3′-diacetyl-4,4′-dihydroxydiphenyl sulfide with hydrogen peroxide (73%) [5915] according to [5921] or with 30% hydrogen peroxide in acetic acid at r.t. for 48 h (63%) [5922].
- Also obtained by alkaline degradation of 6,6′-bichromonyl sulfone (m.p. 266–268°) with refluxing aqueous 10% sodium hydroxide for 20 min [5833].
- Also refer to: [5927].

m.p. 189–190° [5915], 189° [5921,5922], 187–188° [5833], 186°6 [5926]; ^{1}H NMR [5926], IR [5922], UV [5922].

1,1′-[Sulfonylbis(4,6-dihydroxy-3,1-phenylene)]bis-ethanone

[56923-32-1]



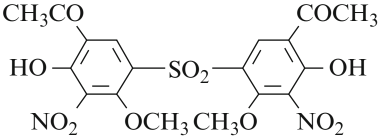
$C_{16}H_{14}O_8S$ mol.wt. 366.35

Synthesis

- Obtained by oxidation of 5,5′-diacetyl-2,2′-4,4′-tetra-hydroxydiphenyl sulfide with 30% hydrogen peroxide in acetic acid at r.t. for 48 h (75%) [5922] or in acetone at r.t. overnight (73%) [5921].

m.p. 195° [5921,5922]; IR [5922], UV [5922].

1,1′-[Sulfonylbis(6-hydroxy-4-methoxy-5-nitro-3,1-phenylene)]bis-ethanone



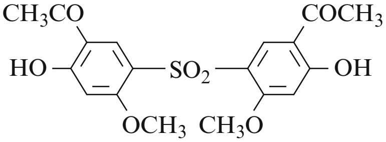
$C_{18}H_{16}N_2O_{12}S$ mol.wt. 484.40

Synthesis

- Obtained by nitration of 3,3′-diacetyl-4,4′-dihydroxy-6,6′-dimethoxy-diphenyl sulfone in concentrated sulfuric acid with concentrated nitric acid at 60° for 15 min [5922].

1,1′-[Sulfonylbis(6-hydroxy-4-methoxy-3,1-phenylene)]bis-ethanone

[56923-33-2]



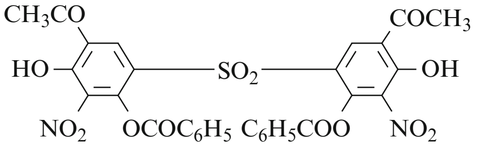
$C_{18}H_{18}O_8S$ mol.wt. 394.40

Synthesis

- Obtained by oxidation of 3,3′-diacetyl-4,4′-di-hydroxy-6,6′-dimethoxydiphenyl sulfide with 30% hydrogen peroxide in acetic acid at r.t. for 48 h (68%) [5922].

m.p. 281–282° [5922]; IR [5922], UV [5922].

1,1′-[Sulfonylbis[4-(benzoyloxy)-6-hydroxy-5-nitro-3,1-phenylene]] bis-ethanone



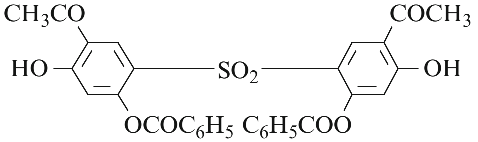
$C_{30}H_{20}N_2O_{14}S$ mol.wt. 664.56

Synthesis

- Obtained by nitration of 5,5′-diacetyl-2,2′-bis(benzoyloxy)-4,4′-dihydroxy-diphenyl sulfone in concentrated sulfuric acid with concentrated nitric acid at 50° for 15 min [5922].

1,1′-[Sulfonylbis[4-(benzoyloxy)-6-hydroxy-3,1-phenylene]]bis-ethanone

[56923-35-4]



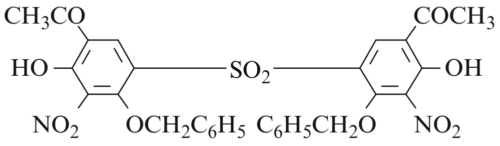
$C_{30}H_{22}O_{10}S$ mol.wt. 574.56

Synthesis

- Obtained by oxidation of 5,5′-diacetyl-2,2′-bis(benzoyloxy)-4,4′-dihydroxy-diphenyl sulfide with 30% hydrogen peroxide in acetic acid at r.t. for 48 h (71%) [5922].

m.p. 245–246° [5922]; IR [5922], UV [5922].

1,1′-[Sulfonybis[6-hydroxy-5-nitro-4-(phenylmethoxy)-3,1-phenylene]] bis-ethanone



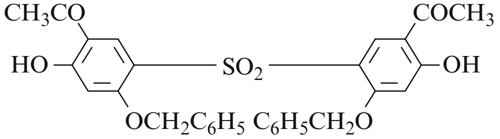
$C_{30}H_{24}N_2O_{12}S$ mol.wt. 636.59

Synthesis

- Obtained by nitration of 5,5′-diacetyl-2,2′-bis(benzyloxy)-4,4′-dihydroxy-diphenyl sulfone in concentrated sulfuric acid with concentrated nitric acid at 60° for 15 min [5922].

1,1′-[Sulfonylbis[6-hydroxy-4-(phenylmethoxy)-3,1-phenylene]]bis-ethanone

[56923-34-3]



$C_{30}H_{26}O_8S$ mol.wt. 546.60

Synthesis

- Obtained by oxidation of 5, 5′-diacetyl-2,2′-bis(benzyloxy)-4,4′-dihydroxy-diphenyl sulfide with 30% hydrogen peroxide in acetic acid at r.t. for 48 h (75%) [5922].

m.p. 222–223° [5922]; IR [5922], UV [5922].

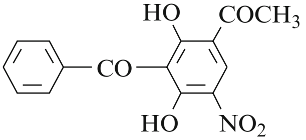
Chapter 21
Aromatic Ketones Containing At Least One Acetyl Group and One Other Acyl Group

21.1 Acyl Groups Located on One Ring

21.1.1 Diphenyl Ketone Derivatives

1-(3-Benzoyl-2,4-dihydroxy-5-nitrophenyl)ethanone

[54917-81-6] $C_{15}H_{11}NO_6$ mol.wt. 301.26



Synthesis

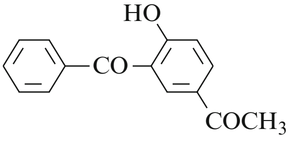
– Obtained by heating 3-benzoyl-2,4-dihydroxy-acetophenone-4-β-D-glucopyranoside with dilute nitric acid for 3 min (44%) [5928].

N.B: A sesquihydrate was obtained by crystallisation of the ketone in water [5928]. The melting point is determined after water elimination (100°/0.04 mm/1 h).

m.p. 114–118° [5928].

1-(3-Benzoyl-4-hydroxyphenyl)ethanone

[13043-37-3] $C_{15}H_{12}O_3$ mol.wt. 240.26

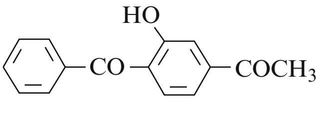


Synthesis

– Refer to: [5929,5930].

1-(4-Benzoyl-3-hydroxyphenyl)ethanone

[39954-75-1] $C_{15}H_{12}O_3$ mol.wt. 240.26



Synthesis

– Obtained by alkaline hydrolysis of 3-(benzoyloxy)-4-benzoylacetophenone (SM) (m.p. 88°)

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
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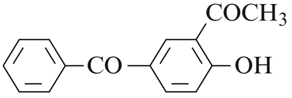
with sodium hydroxide in boiling ethanol for 15 min. SM was prepared by oxidation of 6-acetyl-2,3-diphenyl-benzofuran (m.p. 119°) with chromium trioxide in acetic acid at 70–75° for 2 h [5931].

m.p. 103° [5931]; IR [5931].

1-(5-Benzoyl-2-hydroxyphenyl)ethanone

[2589-80-2]



$C_{15}H_{12}O_3$ mol.wt. 240.26

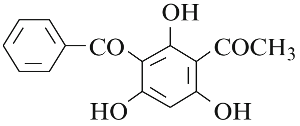
Syntheses

- Obtained by Fries rearrangement of 4-(acetyloxy)-benzophenone with aluminium chloride (3.3 mol) at 150–160° for 1 h [5628].
- Also obtained by hydrolysis of 3-acetyl-4-(acetyloxy)-benzophenone [5932].
- Also refer to: [5929].

m.p. 102–103° [5628], 95–96° [5932]; ^{1}H NMR [5932], IR [5932], MS [5932].

1-(3-Benzoyl-2,4,6-trihydroxyphenyl)ethanone

[31188-65-5]



$C_{15}H_{12}O_5$ mol.wt. 272.26

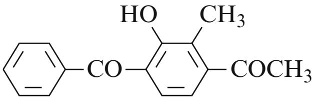
Syntheses

- Preparation by C-acetylation of 2-benzoylphloroglucinol with boron trifluoride-acetic acid complex (76%) [5933].
- Also refer to: [5929,5934].

m.p. 145–146° [5933].

1-(4-Benzoyl-3-hydroxy-2-methylphenyl)ethanone

[51846-39-0]



$C_{16}H_{14}O_3$ mol.wt. 254.29

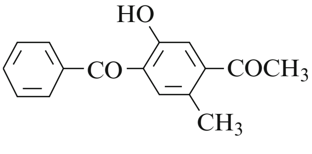
Synthesis

- Obtained by alkaline hydrolysis of 4-benzoyl-3-benzoyl-oxy-2-methylacetophenone (m.p. 102°) [5935].

m.p. 39° [5935]; IR [5935].

1-(4-Benzoyl-5-hydroxy-2-methylphenyl)ethanone

[39954-81-9]



$C_{16}H_{14}O_3$ mol.wt. 254.29

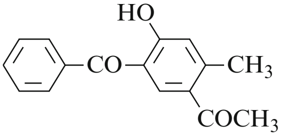
Synthesis

- Obtained by alkaline hydrolysis of 3-(benzoyloxy)-4-benzoyl-6-methylacetophenone (m.p. 135°) (SM) with sodium hydroxide in boiling ethanol for 15 min. SM was obtained by oxidation of 6-acetyl-5-methyl-2,3-diphenyl-benzofuran (m.p. 133°) with chromium trioxide in acetic acid at 70–75° for 2 h [5931].

m.p. 92° [5931]; IR [5931].

1-(5-Benzoyl-4-hydroxy-2-methylphenyl)ethanone

[51846-51-6] $C_{16}H_{14}O_3$ mol.wt. 254.29



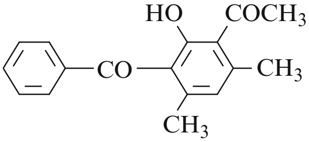
Synthesis

- Obtained by alkaline hydrolysis of 5-benzoyl-4-benzoyl-oxy-2-methylacetophenone [5935].

m.p. 108–112° [5935]; IR [5935].

1-(3-Benzoyl-2-hydroxy-4,6-dimethylphenyl)ethanone

[84312-32-3] $C_{17}H_{16}O_3$ mol.wt. 268.31



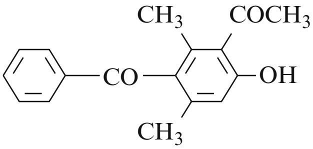
Synthesis

- Obtained by UV light irradiation of 2-acetyl-3,5-di-methylphenyl benzoate in benzene for 10 h (13%) [5936].

m.p. 138–139° [5936]; ^{1}H NMR [5936], IR [5936], UV [5936], MS [5936].

1-(3-Benzoyl-6-hydroxy-2,4-dimethylphenyl)ethanone

[84312-33-4] $C_{17}H_{16}O_3$ mol.wt. 268.31



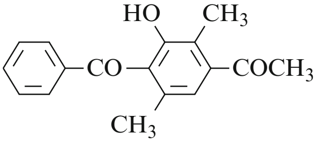
Synthesis

- Obtained by UV light irradiation of 2-acetyl-3,5-dimethyl-phenyl benzoate in benzene for 10 h (6%) [5936].

m.p. 61–65° [5936]; ^{1}H NMR [5936], IR [5936], UV [5936], MS [5936].

1-(4-Benzoyl-3-hydroxy-2,5-dimethylphenyl)ethanone

[51846-44-7] $C_{17}H_{16}O_3$ mol.wt. 268.31



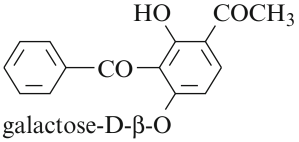
Synthesis

- Obtained by alkaline hydrolysis of 4-benzoyl-3-benzoyl-oxy-2,5-dimethylacetophenone (m.p. 145–146°) [5935].

m.p. 95–102° [5935].

1-[3-Benzoyl-4-(β-D-galactopyranosyloxy)-2-hydroxyphenyl]ethanone

[54917-83-8]



$C_{21}H_{22}O_9$ mol.wt. 418.40

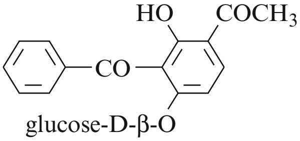
Synthesis

- Obtained by action of 4-formyl-1,2-phenylene dibenzoate with 2-hydroxy-4-(β-D-galactopyranosyloxy)-acetophenone in acetone in the presence of 2 N aqueous sodium hydroxide at 20° (16%) [5928].

m.p. 199–201° [5928]; $(\alpha)_D^{18} = +78°$ (c = 0.4 in pyridine) [5928].

1-[3-Benzoyl-4-(β-D-glucopyranosyloxy)-2-hydroxyphenyl]ethanone

[54918-25-1]



$C_{21}H_{22}O_9$ mol.wt. 418.40

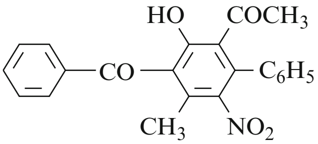
Synthesis

- Obtained by reaction of 4-formyl-1,2-phenylene dibenzoate with 2-hydroxy-4-(β-D-glucopyranosyloxy)-acetophenone in acetone in the presence of 2 N aqueous sodium hydroxide at 20° for 17 h (29%) [5928].

m.p. 192–194° [5928]; $(\alpha)_D^{30} = -89°$ (c = 0.55 in acetone/water 1:1) [5928].

1-(4-Benzoyl-3-hydroxy-5-methyl-6-nitro[1,1′-biphenyl]-2-yl)ethanone

[85450-70-0]



$C_{22}H_{17}NO_5$ mol.wt. 375.38

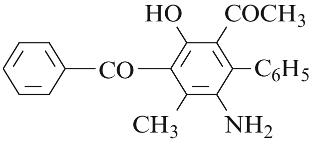
Synthesis

- Obtained (by-product) by reaction of 1,3-dibenzoyl-4,6-dimethylpyrone with nitromethane in tert-butanol in the presence of potassium tert-butoxide at 60° for 4 h (13%) [5809].

m.p. 141–142° [5809].

1-(6-Amino-4-benzoyl-3-hydroxy-5-methyl[1,1′-biphenyl]-2-yl)ethanone

[85450-81-3]



$C_{22}H_{19}NO_3$ mol.wt. 345.40

Synthesis

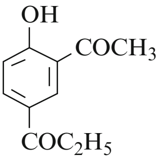
- Obtained (poor yield) by catalytic hydrogenation of 3-acetyl-2-hydroxy-6-methyl-5-nitro-4-phenyl-benzophenone in ethanol in the presence of 10% Pd/C at 40° for 3 days (9%) [5809].

m.p. 120–123° [5809].

21.1.2 Miscellaneous

1-(3-Acetyl-4-hydroxyphenyl)-1-propanone

[79010-36-9] $C_{11}H_{12}O_3$ mol.wt. 192.21



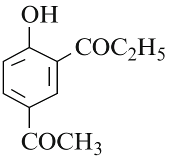
Synthesis

- Preparation by Fries rearrangement of 4-propionylphenyl acetate with aluminium chloride at 150° for 3 h (80%) [5937].

m.p. 69° [5937]; ^{1}H NMR [5937], IR [5937].

1-(5-Acetyl-2-hydroxyphenyl)-1-propanone

[36039-26-6] $C_{11}H_{12}O_3$ mol.wt. 192.20



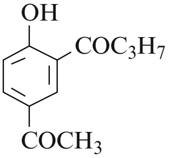
Syntheses

- Preparation by Fries rearrangement of p-(propionyloxy)-acetophenone with aluminium chloride (4 mol) without solvent at 150° for 3 h (62%) [5626].
- Also obtained by Friedel–Crafts acylation of p-hydroxyacetophenone with propionyl chloride in the presence of aluminium chloride (4 mol) in tetrachloroethane at 130° for 4 h (47%) [5626].
- Also obtained by deacylation of 2-(LD-2′-acetoxypropionyloxy)-5-acetylpropiophenone (24%) [5633].

m.p. 67–69° [5626]; ^{1}H NMR [5626], IR [5626].

1-(5-Acetyl-2-hydroxyphenyl)-1-butanone

[92757-66-9] $C_{12}H_{14}O_3$ mol.wt. 206.24



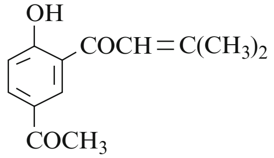
Syntheses

- Preparation by Fries rearrangement of p-(butyryloxy)-acetophenone with aluminium chloride (4 mol) without solvent at 150° for 3 h (58%) [5626].
- Also obtained by Friedel–Crafts acylation of p-hydroxyacetophenone with butyryl chloride in the presence of aluminium chloride (4 mol) in tetrachloroethane at 130° for 4 h (47%) [5626].

m.p. 54–55° [5626]; ^{1}H NMR [5626], IR [5626].

1-(5-Acetyl-2-hydroxyphenyl)-3-methyl-2-buten-1-one

[65580-31-6]



$C_{13}H_{14}O_3$ mol.wt. 218.25

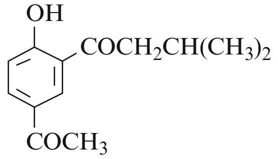
Isolation from natural sources

- From the aerial parts of *Ophryosporus floribundus* (Compositae, tribe Eupatorieae) [5636].
- From the leaves of ageratina *altissima* (L) K. et R. (Compositae) [5938].
- From the aerial parts of *senecio behnii* Ric. et Martic [5939].
- From the aerial parts of *Ophryosporus charua* (Griseb.) Hieron (Compositae) [5940].
- From *Ophryosporus chilca* [5940].

m.p. 75°5 [5938]; TLC [5938];
^{1}H NMR [5938], IR [5938], UV [5938], MS [5938].

1-(5-Acetyl-2-hydroxyphenyl)-3-methyl-1-butanone

[62458-64-4]



$C_{13}H_{16}O_3$ mol.wt. 220.27

Syntheses

- Preparation by Fries rearrangement of p-acetylphenyl isovalerate without solvent in the presence of aluminium chloride at 140–160° for 2.5 h (51%) [5941] or at 150° for 3 h (32%) [5626].
- Also obtained by Friedel–Crafts acylation of p-hydroxy-acetophenone with isovaleryl chloride (4 mol) in tetrachloroethane at 130° for 4 h (37%) [5626].

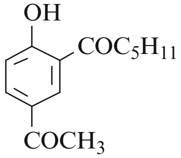
Isolation from natural sources

- From the genus *Flourensia cernua* DC (Compositae) [5942].
- From the aerial parts of *Ophryosporus floribundus* (Compositae, tribe Eupatorieae) [5636].
- From sliced yacon tubers after inoculation with the bacterium *Pseudomonas cichorii* and incubation at 20° for 3 days, then extraction with acetone. Yacon (*Polymnia sonchifolia*) (Compositae) is cultivated in South America and has recently been introduced in Japan [5943].

Colourless oil [5942];
m.p. 94°5–96° [5943]; 64–66° [5626], 61°5 [5941].
One of the reported melting points is obviously wrong.
^{1}H NMR [5626,5942,5943], ^{13}C NMR [5943], IR [5626,5942,5943],
UV [5943], MS [5942,5943].

1-(5-Acetyl-2-hydroxyphenyl)-1-hexanone

[92757-67-0] $C_{14}H_{18}O_3$ mol.wt. 234.30



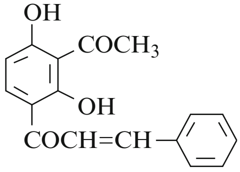
Syntheses

- Obtained by Fries rearrangement of p-(caproyloxy)-acetophenone with aluminium chloride (4 mol) without solvent at 150° for 3 h (37%) [5626].
- Also obtained by Friedel–Crafts acylation of p-hydroxy-acetophenone with caproyl chloride in the presence of aluminium chloride in tetrachloroethane at 130° for 4 h (39%) [5626].

m.p. 52° [5626]; ^{1}H NMR [5626], IR [5626].

1-(3-Acetyl-2,4-dihydroxyphenyl)-3-phenyl-2-propen-1-one

[116470-07-6] $C_{17}H_{14}O_4$ mol.wt. 282.30
[84422-44-6] (*E*)



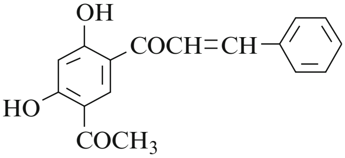
Synthesis

- Obtained by reaction of benzaldehyde with 2,4-diacetyl-resorcinol in ethanol in the presence of concentrated aqueous potassium hydroxide at r.t. (Claisen–Schmidt condensation), for 48 h (34%) [5661] or for 24 h (9%) [5658].

m.p. 149° [5658], 134° [5661]; TLC [5658,5661].
One of the reported melting points is obviously wrong.
^{1}H NMR [5658,5661], IR [5658], UV [5658], MS [5658].

1-(5-Acetyl-2,4-dihydroxyphenyl)-3-phenyl-2-propen-1-one (*E*)

[104236-84-2] $C_{17}H_{14}O_4$ mol.wt. 282.30

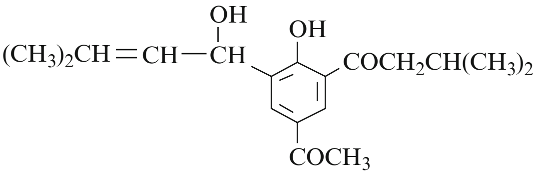


Synthesis

- No details of synthesis are given [5944].

1-[5-Acetyl-2-hydroxy-3-(1-hydroxy-3-methyl-2-butenyl)phenyl]-3-methyl-1-butanone

[94413-27-1] $C_{17}H_{24}O_4$ mol.wt. 292.38

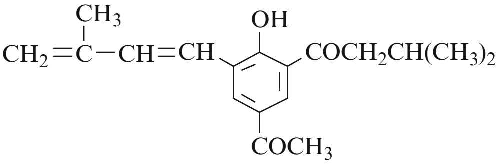


Isolation from natural sources

- From the aerial parts of *Ophryosporus peruvianus* (Gmel.) K. et R. (Compositae) [5945].

1-[5-Acetyl-2-hydroxy-3-(3-methyl-1,3-butadienyl)phenyl]-3-methyl-1-butanone *(E)*

[148707-32-8] $C_{18}H_{22}O_3$ mol.wt. 286.37



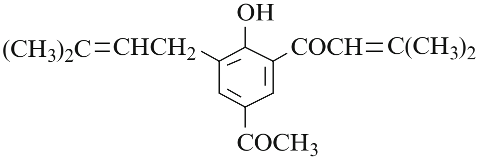
Synthesis

N.B.: After several days in a $CDCl_3$ solution used for the ^1H NMR measurements, 3-(3-hydroxy-3-methyl-1-butenyl)-5-isovaleryl-p-hydroxy-acetophenone (*E*) was converted into the titled substance [5946].

^1H NMR [5946].

1-[5-Acetyl-2-hydroxy-3-(3-methyl-2-butenyl)phenyl]-3-methyl-2-buten-1-one *(Piloselloidon)*

[94413-26-0] $C_{18}H_{22}O_3$ mol.wt. 286.37



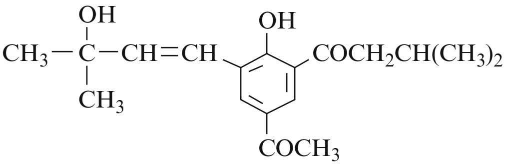
Isolation from natural sources

- From the roots of *Gerbera piloselloides* Cass. (Compositae, tribe Arctotideae) [5947].
- From the aerial parts of *Ophryosporus chilca* (Compositae, tribe Eupatorieae) [5945].
- From the aerial parts of *Ophryosporus peruvianus* (Compositae) [5945].

Colourless oil [5947]; TLC [5945]; ^1H NMR [5947], IR [5947], MS [5947].

1-[5-Acetyl-2-hydroxy-3-(3-hydroxy-3-methyl-1-butenyl)phenyl]-3-methyl-1-butanone (*E*)

[54963-60-9] $C_{18}H_{24}O_4$ mol.wt. 304.39



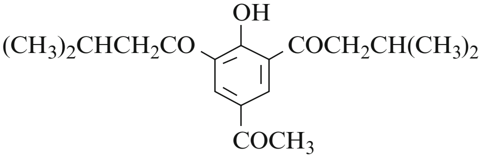
Isolation from natural sources

- From the aerial parts of *Ophryosporus charua* (Griseb.) Hieron (Compo-sitae) [5940].
- From the aerial parts of *Ophryosporus macrodon* Griseb. (Compositae, tribe Eupatorieae) [5946].

m.p. 103–105° [5946]; ^1H NMR [5946], MS [5946].

1,1′-(5-Acetyl-2-hydroxy-1,3-phenylene)bis[3-methylbutanone

[94413-28-2]



$C_{18}H_{24}O_4$ mol.wt. 304.39

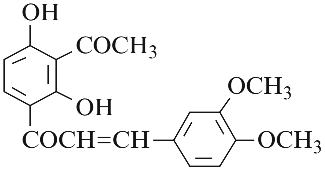
Isolation from natural sources

- From the aerial parts of *Ophryosporus peruvianus* (Gmel.) K. et R. (Compositae) [5945].

^{1}H NMR [5945], IR [5945], MS [5945]; TLC [5945].

1-(3-Acetyl-2,4-dihydroxyphenyl)-3-(3,4-dimethoxyphenyl)-2-propen-1-one

[116470-11-2]



$C_{19}H_{18}O_6$ mol.wt. 342.35

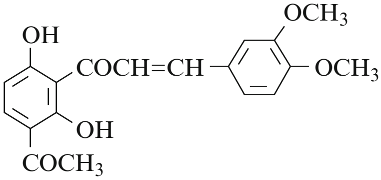
Synthesis

- Obtained by reaction of veratraldehyde with 2,4-diacetylresorcinol in ethanol in the presence of aqueous potassium hydroxide at r.t. for 24 h (18%) [5658].

m.p. 189–190° [5658]; TLC [5658];
^{1}H NMR [5658], IR [5658], UV [5658], MS [5658].

1-(3-Acetyl-2,6-dihydroxyphenyl)-3-(3,4-dimethoxyphenyl)-2-propen-1-one

[116470-10-1]



$C_{19}H_{18}O_6$ mol.wt. 342.35

Synthesis

- Obtained (by-product) by reaction of veratraldehyde with 2,4-diacetylresorcinol in ethanol in the presence of aqueous potassium hydroxide at r.t. for 24 h (6%) [5658].

m.p. 164° [5658]; TLC [5658]; ^{1}H NMR [5658], IR [5658], UV [5658], MS [5658].

21.2 Acyl Groups Located on Different Rings

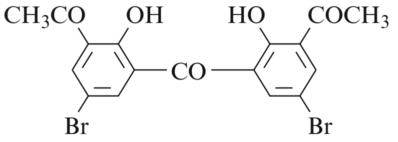
21.2.1 Diphenyl Ketone Derivatives

Symmetrical ketones

1,1′-[Carbonylbis(5-bromo-2-hydroxy-3,1-phenylene)]bis-ethanone

[83143-07-1]

$C_{17}H_{12}Br_2O_5$ mol.wt. 456.09



Synthesis

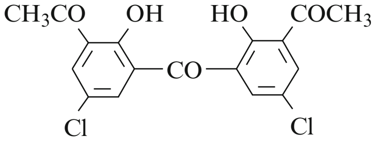
- Preparation by Fries rearrangement of 2,2′-di-acetoxy-5,5′-dibromobenzophenone with aluminium chloride at 160–180° for 20 min (50%) [5865].

m.p. 230–231° [5865].

1,1′-[Carbonylbis(5-chloro-2-hydroxy-3,1-phenylene)]bis-ethanone

[83143-06-0]

$C_{17}H_{12}Cl_2O_5$ mol.wt. 367.18



Synthesis

- Preparation by Fries rearrangement of 2,2′-di-acetoxy-5,5′-dichlorobenzophenone with aluminium chloride,

- at 160–180° for 20 min (80%) [5865];
- at 170–180° for 30 min (80%) [5867].

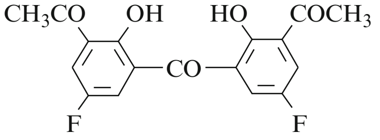
m.p. 222–224° [5865,5867].

N.B.: Na salt, m.p. >360° [5867].

1,1′-[Carbonylbis(5-fluoro-2-hydroxy-3,1-phenylene)]bis-ethanone

[83143-05-9]

$C_{17}H_{12}F_2O_5$ mol.wt. 334.28



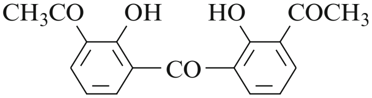
Synthesis

- Preparation by Fries rearrangement of 2,2′-di-acetoxy-5,5′-difluorobenzophenone with aluminium chloride at 160–180° for 20 min (72%) [5865].

m.p. 149–150° [5865].

1,1′-[Carbonylbis(2-hydroxy-3,1-phenylene)]bis-ethanone

[83143-08-2]



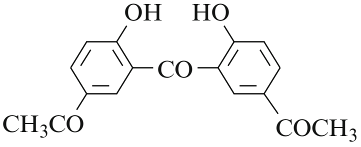
$C_{17}H_{14}O_5$ mol.wt. 298.30

Synthesis

- Preparation by Fries rearrangement of 2,2′-di-acetoxybenzophenone with aluminium chloride at 160–180° for 20 min (63%) [5865].

m.p. 170–171° [5865].

1,1′-[Carbonylbis(4-hydroxy-3,1-phenylene)]bis-ethanone



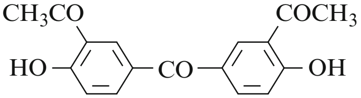
$C_{17}H_{14}O_5$ mol.wt. 298.30

Synthesis

- Obtained (by-product) by Fries rearrangement of 2,2′-diacetoxybenzophenone with aluminium chloride at 160–180° for 20 min (10%) [5865].

1,1′-[Carbonylbis(6-hydroxy-3,1-phenylene)]bis-ethanone

[20795-69-1]



$C_{17}H_{14}O_5$ mol.wt. 298.30

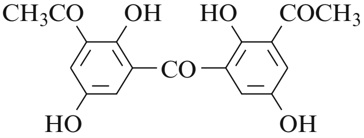
Syntheses

- Obtained by Fries rearrangement of 4,4′-diacetoxy-benzophenone,
  - with aluminium chloride at 140° for 4 h (40%) [5948];
  - with aluminium chloride and sodium chloride at 140–150° for 6 h [5875].
- Also obtained by alkaline degradation of 6,6′-bichromonyl ketone (m.p. 249–250°) with refluxing 10% aqueous sodium hydroxide for 20 min [5833].

m.p. 182–183° [5948], 180–181° [5833], 174–176° [5875].

1,1′-[Carbonylbis(2,5-dihydroxy-3,1-phenylene)]bis-ethanone

[78563-21-0]



$C_{17}H_{14}O_7$ mol.wt. 330.29

Synthesis

- Obtained by Fries rearrangement of 2,2′,5,5′-tetra-acetoxybenzophenone with aluminium chloride at 175–178° for 20 min (30%) [5866].

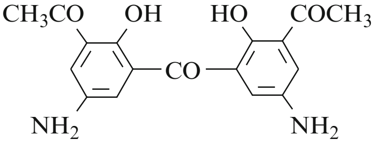
m.p. >300° [5866].

N.B.: This ketone (**5d**) was characterized by its corresponding tetraacetate (**5d**$_1$)

m.p. 254–256° [5866]; IR [5866], MS [5866].

1,1′-[Carbonylbis(5-amino-2-hydroxy-3,1-phenylene)]bis-ethanone

[78563-23-2]



$C_{17}H_{16}N_2O_5$ mol.wt. 328.31

Synthesis

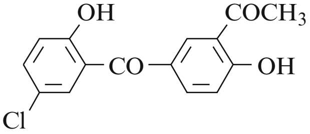
- Preparation by Fries rearrangement of 5,5′-di-acetamido-2,2′-diacetoxy-benzophenone with aluminium chloride at 175–178° for 20 min (80%) [5866].

m.p. 254–255° [5866]; IR [5866], MS [5866].

Asymmetrical ketones

1-[5-(5-Chloro-2-hydroxybenzoyl)-2-hydroxyphenyl]ethanone

[220042-68-2]



$C_{15}H_{11}ClO_4$ mol.wt. 290.70

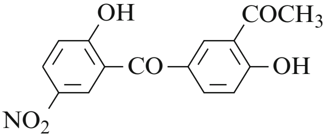
Synthesis

- Obtained (20% yield) by adding a solution of 6-chloro-4-oxo-4*H*-1-benzopyran-3-carboxaldehyde in acetic acid to a preheated (70–80°) mixture of acetylacetone in acetic acid containing a catalytic amount of concentrated hydrochloric acid [5949].

m.p. 144° [5949]; ^{1}H NMR [5949], IR [5949].

1-[2-Hydroxy-5-(2-hydroxy-5-nitrobenzoyl)phenyl]ethanone

[220042-69-3]



$C_{15}H_{11}NO_6$ mol.wt. 301.26

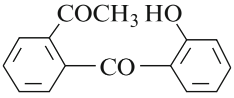
Synthesis

- Obtained (30% yield) by adding a solution of 6-nitro-4-oxo-4*H*-1-benzopyran-3-carboxaldehyde in acetic acid to a preheated (70–80°) mixture of acetylacetone in acetic acid containing a catalytic amount of concentrated hydrochloric acid [5949].

m.p. 174° [5949]; ^{1}H NMR [5949], IR [5949].

1-[2-(2-Hydroxybenzoyl)phenyl]ethanone

[17526-21-5]



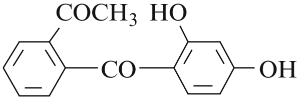
$C_{15}H_{12}O_3$ mol.wt. 240.26

Synthesis

- Refer to: [5929].

1-[2-(2,4-Dihydroxybenzoyl)phenyl]ethanone

[36414-93-4]



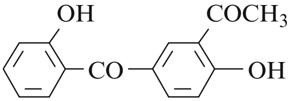
$C_{15}H_{12}O_4$ mol.wt. 256.26

Syntheses

- Refer to: [5929,5950].

1-[2-Hydroxy-5-(2-hydroxybenzoyl)phenyl]ethanone

[124208-69-1]



$C_{15}H_{12}O_4$ mol.wt. 256.26

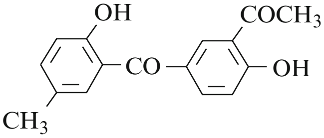
Syntheses

- Obtained (17% yield) by adding a solution of 4-oxo-4*H*-1-benzopyran-3-carboxaldehyde in acetic acid to a preheated (70–80°) mixture of acetylacetone in acetic acid containing a catalytic amount of concentrated hydrochloric acid [5949].
- Also refer to: [5929].

m.p. 128° [5949]; ^{1}H NMR [5949], IR [5949].

1-[2-Hydroxy-5-(2-hydroxy-5-methylbenzoyl)phenyl]ethanone

[220042-67-1]



$C_{16}H_{14}O_4$ mol.wt. 270.28

Synthesis

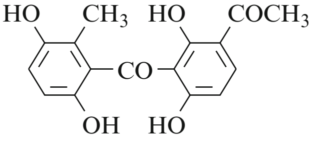
- Obtained (15% yield) by adding a solution of 6-methyl-4-oxo-4H-1-benzopyran-3-carboxaldehyde in acetic acid to a preheated (70–80°) mixture of acetylacetone in acetic acid containing a catalytic amount of concentrated hydrochloric acid [5949].

m.p. 141° [5949]; ^{1}H NMR [5949], IR [5949].

1-[3-(3,6-Dihydroxy-2-methylbenzoyl)-2,4-dihydroxyphenyl]ethanone

(Baishouwubenzophenone)

[115834-34-9]



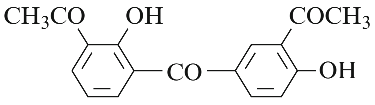
$C_{16}H_{14}O_6$ mol.wt. 302.28

Isolation from natural sources

- From *Baishouwu*, the botanical source of which being chiefly the tuber of *Cynanchum auriculatum* Royle ex Wight (Asclepiadaceae) [5951].
- Also refer to: [5929].

^{1}H NMR [5951], ^{13}C NMR [5951], IR [5951], UV [5951], MS [5951].

1-[3-(3-Acetyl-4-hydroxybenzoyl)-2-hydroxyphenyl]ethanone



$C_{17}H_{14}O_5$ mol.wt. 298.30

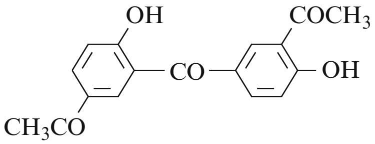
Synthesis

- Obtained (by-product) by Fries rearrangement of 2,4′-diacetoxybenzophenone with aluminium chloride at 158–160° for 2 h (<2%) [5952].

m.p. 152° [5952]; IR [5952].

1-[3-(3-Acetyl-4-hydroxybenzoyl)-4-hydroxyphenyl]ethanone

[124208-68-0]



$C_{17}H_{14}O_5$ mol.wt. 298.30

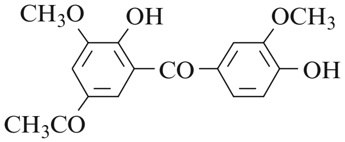
Synthesis

- Preparation by Fries rearrangement of 2,4′-di-acetoxybenzophenone with aluminium chloride at 158–160° for 2 h (60%) [5952].

m.p. 184–185° [5952]; IR [5952].

1-[4-Hydroxy-3-(4-hydroxy-3-methoxybenzoyl)-5-methoxyphenyl]ethanone

[147904-65-2]



$C_{17}H_{16}O_6$ mol.wt. 316.31

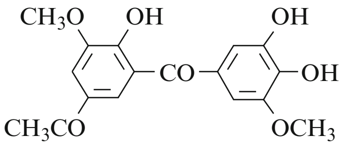
Synthesis

- Obtained by alkaline CuO oxidation of lignin (compound **Vo5Vn**) named 5-vanilloacetovanillone [5851].

GC [5851], GC-MS [5851].

1-[3-(3,4-Dihydroxy-5-methoxybenzoyl)-4-hydroxy-5-methoxyphenyl]ethanone

[147904-69-6]



$C_{17}H_{16}O_7$ mol.wt. 332.31

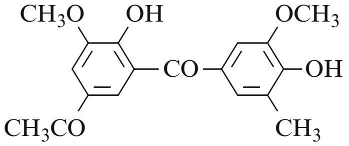
Synthesis

- Obtained by alkaline CuO oxidation of lignin [5851].

GC [5851], GC-MS [5851].

1-[4-Hydroxy-3-(4-hydroxy-3-methoxy-5-methylbenzoyl)-5-methoxyphenyl] ethanone

[147904-68-5]



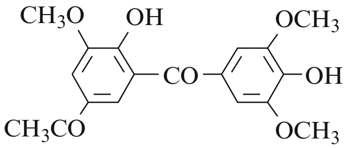
$C_{18}H_{18}O_6$ mol.wt. 330.34

Synthesis

- Obtained by alkaline CuO oxidation of lignin [5851].

GC [5851], GC-MS [5851].

1-[4-Hydroxy-3-(4-hydroxy-3,5-dimethoxybenzoyl)-5-methoxyphenyl]ethanone



$C_{18}H_{18}O_7$ mol.wt. 346.34

Synthesis

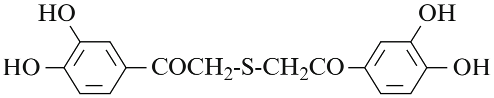
- Obtained by alkaline CuO oxidation of lignin (compound **S.5Vn**) named 5-syringoacetovanillon [5851].

GC [5851], GC-MS [5851].

21.2.2 Miscellaneous

2,2′-Thiobis-1-(3,4-dihydroxyphenyl)ethanone

[215431-54-2]



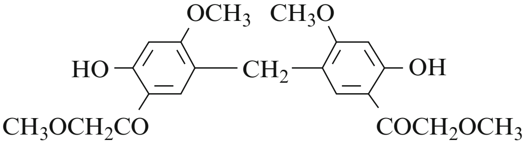
$C_{16}H_{14}O_6S$ mol.wt. 334.35

Synthesis

- Refer to: [5953] (Japanese patent).

1,1′-[Methylenebis(6-hydroxy-4-methoxy-3,1-phenylene)]bis[2-methoxyethanone]

[71204-18-7]



$C_{21}H_{24}O_8$ mol.wt. 404.42

Synthesis

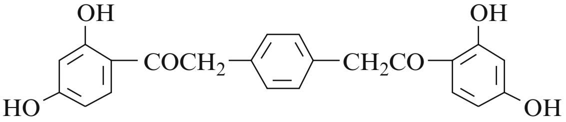
- Obtained from 2-hydroxy-4,α-di-methoxyacetophenone with formaldehyde and 35% aqueous sulfuric acid [4897].

m.p. 152° [4897]; (dibenzoate: m.p. 95–96°) [4897].

1-(2,4-Dihydroxyphenyl)-2-[4-[2-(2,4-dihydroxyphenyl)-2-oxoethyl]phenyl] ethanone

αα,α-bis-(2,4-dihydroxybenzoyl)-p-xylene

[97829-54-4]



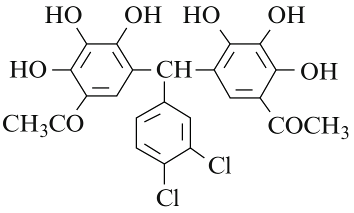
$C_{22}H_{18}O_6$ mol.wt. 378.38

Syntheses

- Preparation by reaction of 1,4-di-cyanobenzene (terephthalonitrile) with resorcinol (Hoesch reaction) (52%) [5954].
- Also refer to: [5955].

m.p. 282° (d) [5954,5955].

1,1′-[[(3,4-Dichlorophenyl)methylene]bis(4,5,6-trihydroxy-3,1-phenylene)] bis-ethanone



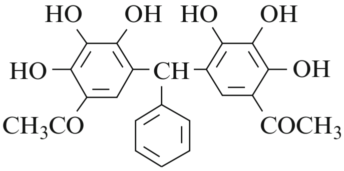
$C_{23}H_{18}Cl_2O_8$ mol.wt. 493.30

Synthesis

- Obtained by condensation of 1 mol of 3,4-dichloro-benzaldehyde (m.p. 41–44°) with 2 mol of gallacetophenone [5182].

m.p. 259–260° [5182].

1,1′-[(Phenylmethylene)bis(4,5,6-trihydroxy-3,1-phenylene)]bis-ethanone



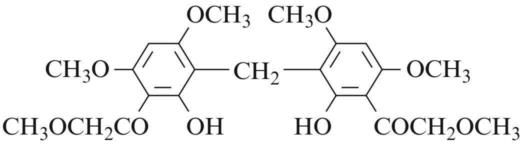
$C_{23}H_{20}O_8$ mol.wt. 424.41

Synthesis

- Obtained by condensation of benzaldehyde (1 mol) with gallacetophenone (2 mol) in saturated ethanol with hydrogen chloride [5182,5956].
- dihydrate [5956]; m.p. 226° [5956].

1,1′-[Methylenebis(2-hydroxy-4,6-dimethoxy-3,1-phenylene)]bis[2-methoxyethanone

[71204-19-8]



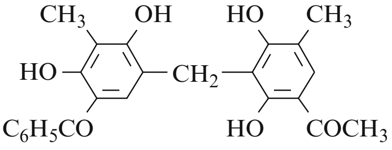
$C_{23}H_{28}O_{10}$ mol.w.464.47

Synthesis

- Obtained from 2-hydroxy-4,6,a-tri-methoxyacetophenone on refluxing with formaldehyde and 35% aqueous sulfuric acid [4897].

m.p. 232–234° [4897]; (monobenzoate, m.p. 176–178°) [4897].

1-[3-[(5-Benzoyl-2,4-dihydroxy-3-methylphenyl)methyl]-2,4-dihydroxy-5-methylphenyl]ethanone



$C_{24}H_{22}O_6$ mol.wt. 406.44

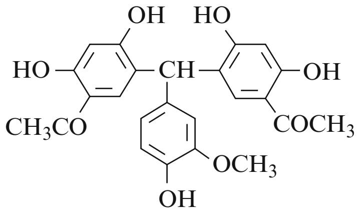
Synthesis

- Obtained by action of 40% aqueous formaldehyde with a mixture of 5-methyl-resacetophenone and 3-methylresbenzophenone (1:1) in ethanol in the presence of concentrated sulfuric acid at r.t. for 3 days (27%) [5883].

m.p. 239–240° [5883].

1,1′-[[(4-Hydroxy-3-methoxyphenyl)methylene]bis(4,6-dihydroxy-3,1-phenylene)] bis-ethanone

[146533-78-0]



$C_{24}H_{22}O_8$ mol.wt. 438.44

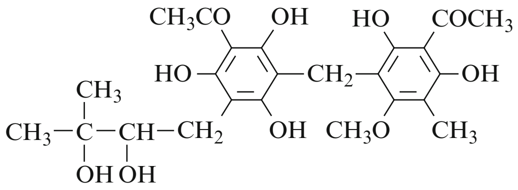
Synthesis

- Refer to: [5957].

1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-5-(2,3-dihydroxy-3-methylbutyl)-2,4,6-trihydroxyphenyl]ethanone

(Mallotojaponol)

[131836-01-6] (racemic)



$C_{24}H_{30}O_{10}$ mol.wt. 478.18

Isolation from natural sources

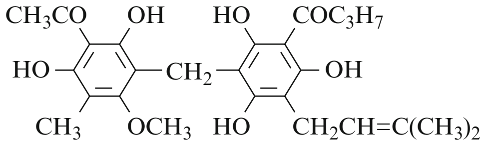
- From the pericarps of *Mallotus japonicus* Muell. Arg. (Euphorbiaceae) [5896].

m.p. 150–151° [5896];
n_{D}^{23} = 0° (c = 0.1 in chloroform) [5896];
^{1}H NMR [5896], IR [5896], UV [5896], EIMS [5896].

1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-2,4,6-trihydroxy-5-(3-methyl-2-butenyl)phenyl]-1-butanone

(Butyrylmallotojaponin)

[96853-73-5]



$C_{26}H_{32}O_8$ mol.wt. 72.54

Isolation from natural sources

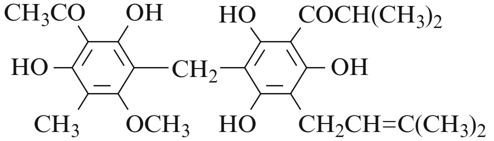
- From the pericarps of *Mallotus japonicus* Muell. Arg. (Euphorbiaceae) [5887, 5893, 5892–5894, 5899, 5897].

^{1}H NMR [5897], ^{13}C NMR [5897], IR [5897], MS [5897]; Cytotoxicity [5894].

1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-2,4,6-trihydroxy- 5-(3-methyl-2-butenyl)phenyl]-2-methyl-1-propanone

(Isobutyrylmallotojaponin)

[96853-74-6] $C_{26}H_{32}O_8$ mol.wt. 72.54



Isolation from natural sources

- From the pericarps of *Mallotus japonicus* Muell. Arg. (Euphorbiaceae) [5887, 5890, 5892–5894, 5896, 5897].

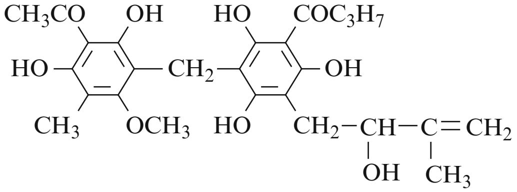
^{1}H NMR [5897], ^{13}C NMR [5897], IR [5897], MS [5897]; Cytotoxicity [5894].

1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-2,4,6-trihydroxy-5-(2-hydroxy-3-methyl-3-butenyl)phenyl]-1-butanone

Proposed name butyrylmallotolerin (old name mallotolerin)***

[102904-17-6] (racemic) $C_{26}H_{32}O_9$ mol.wt. 88.54

[130778-21-1] (optical isomer not indicated)



Isolation from natural sources

- From the pericarps of *Mallotus japonicus* Muell. Arg. (Euphorbiaceae) [5896],* [5887,5888,5889,5890, 5892],** [5894].

m.p. 197–198° [5892];

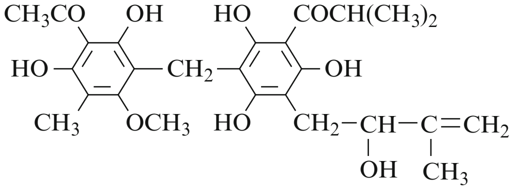
^{1}H NMR [5889,5892], ^{13}C NMR [5888,5889,5892], IR [5892], UV [5892], MS [5892];

Cytotoxicity [5894].

1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-2,4,6-trihydroxy-5-(2-hydroxy-3-methyl-3-butenyl)phenyl]-2-methyl-1-propanone

Proposed name isobutyrylmallotolerin (old name Isomallotolerin)*

[126026-30-0] $C_{26}H_{32}O_9$ mol.wt. 488.54



Isolation from natural sources

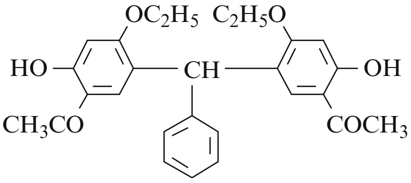
- From the pericarps of Mallotus japonicus Muell. Arg. (Euphorbiaceae) [5896],* [5887,5888, 5889,5890].

m.p. 216–217° [5888];

$(\alpha)_D^{23}$ = 0° (c = 0.63 in chloroform) [5888];

^{1}H NMR [5888,5889], ^{13}C NMR [5888,5889], IR [5888], UV [5888], MS [5888].

1,1′-[(Phenylmethylene)bis(4-ethoxy-6-hydroxy-3,1-phenylene)]bis-ethanone



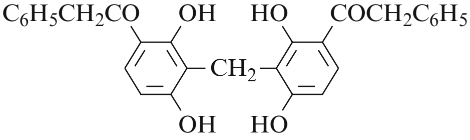
$C_{27}H_{28}O_6$ mol.wt. 448.52

Synthesis

- Obtained by condensation of resacetophenone 4-ethyl ether (2 mol) with benzaldehyde (1 mol) in the presence of hydrogen chloride in ethanol [5956].

m.p. 211° [5956].

1,1′-[Methylenebis(2,4-dihydroxy-3,1-phenylene)]bis[2-phenylethanone



$C_{29}H_{24}O_6$ mol.wt. 468.50

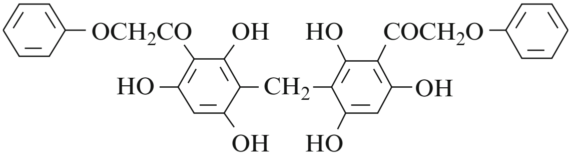
Synthesis

- Obtained by treatment of 2,4-dihydroxy-deoxybenzoin with methylene iodide in the presence of sodium ethoxide in ethanol (29%) [595].

m.p. 191–192° [5878]; IR [5878], UV [5878].

1,1′-[Methylenebis(2,4,6-trihydroxy-3,1-phenylene)]bis-[2-phenoxyethanone

[243465-50-1] $C_{29}H_{24}O_{10}$ mol.wt. 532.50



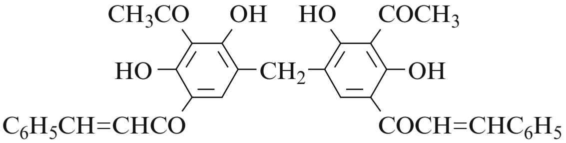
Synthesis

- The Mannich reaction of α-phenoxy-2,4,6-trihydroxy-acetophenone with aminoacids led to the exclusive formation of bis-(α-phenoxy-2,4,6-trihydroxy-acetophenon-3-yl)methane (26%) [5022].

m.p. 239° [5022]; ^{1}H NMR [5022].

1,1′-[Methylenebis(5-acetyl-4,6-dihydroxy-3,1-phenylene)]bis-[3-phenyl-2-propen-1-one

[84422-51-5] *(E,E)* $C_{35}H_{28}O_8$ mol.wt. 576.60



Synthesis

- Obtained by reaction of benzaldehyde with 3,3′,5,5′-tetra-acetyl-2,2′,4,4′-tetrahydroxy-diphenylmethane in ethanol in the presence of aqueous potassium hydroxide, first with shaking for 30 min, then the reaction mixture was kept in the refrigerator for 48 h (10%) [5661].

m.p. 220° [5661]; ^{1}H NMR [5661]; TLC [5661], column chromatography [5661].

Part IX
Addendum to Volume 3

Chapter 22
Addendum 2005–2008

Monoketones Substituted on the Acetyl Groups

Chapter 11. Compounds Derived from Halogenoacetic Acids [5958] p. 1201

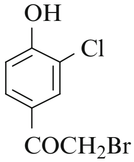
11.1 Compounds Derived from Bromoacetic Acids [5958] p. 1201

11.1.1 From Monobromoacetic Acid [5958] p. 1201

2-Bromo-1-(3-chloro-4-hydroxyphenyl)ethanone

[41877-19-4] $C_8H_6BrClO_2$ mol.wt. 249.49

Described [5958] **p. 1202**



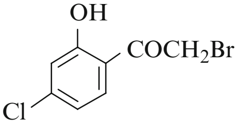
Methyl ether $C_9H_8BrClO_2$ mol.wt. 263.52

- Preparation by reaction of bromine with 3-chloro-4-methoxyacetophenone in acetic acid in the presence of concentrated hydrochloric acid at r.t. for 3.5 h (88%) [5959].

  Yellow solid [5959]; ^{1}H NMR [5959].

2-Bromo-1-(4-chloro-2-hydroxyphenyl)ethanone

[157068-00-3] $C_8H_6BrClO_2$ mol.wt. 249.49



Described [5958] **p. 1203**

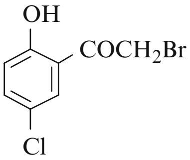
Synthesis

- Also refer to: [5960].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_22, © Springer Science+Business Media B.V. 2011

2-Bromo-1-(5-chloro-2-hydroxyphenyl)ethanone

[52727-99-8] $C_8H_6BrClO_2$ mol.wt. 249.49



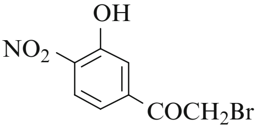
Described [5958] **p. 1203**

Syntheses

- Also refer to: [5961,5962].

2-Bromo-1-(3-hydroxy-4-nitrophenyl)ethanone

[5086-77-1] $C_8H_6BrNO_4$ mol.wt. 260.04



New compound

Syntheses

- Refer to: [5963,5964].

m.p. 89° [5963]; IR [5964].

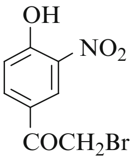
Methyl ether [90725-63-6] $C_9H_8BrNO_4$ mol.wt. 274.07

- Obtained by reaction of bromine with 3-methoxy-4-nitroacetophenone in acetic acid [5965].
- Also obtained from 3-methoxy-4-nitrotoluene (multi-step reaction) [5965].
- Also obtained from 3-methoxy-4-nitrobenzoic acid (multi-step reaction) [5965].
- Also obtained from 3-methoxy-4-nitrobenzoyl chloride (multi-step reaction) [5965].
- Also refer to: [5966,5967].

m.p. 90–91.5° [5965].

2-Bromo-1-(4-hydroxy-3-nitrophenyl)ethanone

[5029-61-8] $C_8H_6BrNO_4$ mol.wt. 260.04



Described [5958] **p. 1204**

Synthesis

- Also refer to: [5968].

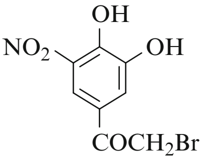
Methyl ether [65447-49-6] $C_9H_8BrNO_4$ mol.wt. 274.07

- Obtained by reaction of bromine with 4-hydroxy-3-nitroacetophenone in chloroform [5969,5970].
- Also obtained by reaction of pyridinium hydrobromide perbromide with 4-methoxy-3-nitro-acetophenone in THF at r.t. for 3 h [5971].
- Also refer to: [5972].

m.p. 96° [5970], 95–96° [5972]; ^{1}H NMR [5972], IR [5972].

2-Bromo-1-(3,4-dihydroxy-5-nitrophenyl)ethanone

[134610-95-0] $C_8H_6BrNO_5$ mol.wt. 276.04



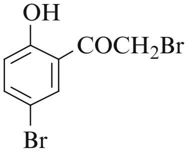
Described [5958] **p. 1205**

Synthesis

- Also refer to: [5973].

2-Bromo-1-(5-bromo-2-hydroxyphenyl)ethanone

[67029-74-7] $C_8H_6Br_2O_2$ mol.wt. 293.94



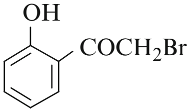
Described [5958] **p. 1206**

Methyl ether [67639-58-1] $C_9H_8Br_2O_2$ mol.wt. 307.97

- Refer to: [5974,5975,5976,5977].

2-Bromo-1-(2-hydroxyphenyl)ethanone

[2491-36-3] $C_8H_7BrO_2$ mol.wt. 215.05



Described [5958] **p. 1206**

Syntheses

- Also refer to: [5960,5978,5979].

Methyl ether [31949-21-0] $C_9H_9BrO_2$ mol.wt. 229.07

- Also obtained according to the procedure of Buckman (80%) [5980].
- Also refer to: [5981,5982,5983].

b.p.$_1$ 130° [5980]; ^{1}H NMR [5980], IR [5980].

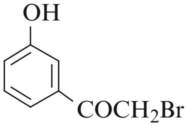
Benzyl ether $C_{15}H_{13}BrO_2$ mol.wt. 305.17

- Obtained by reaction of bromine with 2-(benzyloxy)acetophenone in diethyl ether (65%) [5984].

m.p. 79–81° [5984]; ^{1}H NMR [5984]; GC-MS [5984].

2-Bromo-1-(3-hydroxyphenyl)ethanone

[2491-37-4] $C_8H_7BrO_2$ mol.wt. 215.05



Described [5958] **p. 1207**

Syntheses

- Also refer to: [5985,5986,5987,5988].

Methyl ether [5000-65-7] $C_9H_9BrO_2$ mol.wt. 229.07

- Obtained by reaction of bromine with 3-methoxyacetophenone in chloroform at 0° for 3 h (88%) [5989].
- Also refer to: [5981,5983,5990,5991,5992,5993,5994,5995].

Benzyl ether $C_{15}H_{13}BrO_2$ mol.wt. 305.17

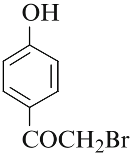
- Obtained by reaction of bromine with 3-(benzyloxy)acetophenone in diethyl ether (70%) [5984].

m.p. 58–60° [5984]; ^{1}H NMR [5984]; GC-MS [5984].

Benzoate $C_{15}H_{11}BrO_3$ mol.wt. 319.15 m.p. 104–105° [5996]

2-Bromo-1-(4-hydroxyphenyl)ethanone

[2491-38-5] $C_8H_7BrO_2$ mol.wt. 215.05



Described [5958] **p. 1207**

Syntheses

- Also obtained by reaction of pyridinium hydrobromide perbromide with 4-hydroxyacetophenone in THF at r.t. for 3 h [5971].
- Also refer to: [5977,5985,5997,5998,5999,6000,6001,6002,6003,6004,6005,6006,6007].

Methyl ether [2632-13-5] $C_9H_9BrO_2$ mol.wt. 229.07

- Obtained by reaction of N-bromosuccinimide (NBS) with 4-methoxyacetophenone in the presence of trimethylsilyl trifluoromethanesulfonate (TMS-OTf) in acetonitrile at r.t. for 24 h (87%) [6008].
- Also obtained by reaction of pyridinium hydrobromide perbromide with 4-methoxyacetophenone in THF at r.t. for 3 h [5971].
- Also refer to: [5994,5999,6009].

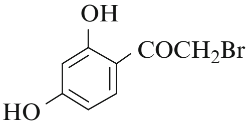
Benzyl ether [4254-67-5] $C_{15}H_{13}BrO_2$ mol.wt. 305.17

- Obtained by reaction of bromine with 4-benzyloxyacetophenone in methanol in the presence of concentrated hydrochloric acid at 0–5° for 1 h, then at r.t. for another 1 h (90%) [6010].
- Also refer to: [6011,6012,6013,6014,6015,6016,6017].

m.p. 83–84° [6011], 82° [6010], 68° [6012].
One of the reported melting points is obviously wrong.
^{1}H NMR [6010,6012,6015], IR [6010], MS [6010].

2-Bromo-1-(2,4-dihydroxyphenyl)ethanone

[2491-39-6] $C_8H_7BrO_3$ mol.wt. 231.05



Described [5958] **p. 1208**

Synthesis

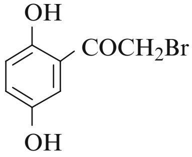
- Also refer to: [5985].

Dimethyl ether [60965-26-6] $C_{10}H_{11}BrO_3$ mol.wt. 259.10

- Refer to: [5981,6018,6019,6020].

2-Bromo-1-(2,5-dihydroxyphenyl)ethanone

[25015-91-2] $C_8H_7BrO_3$ mol.wt. 231.05



Described [5958] **p. 1208**

Synthesis

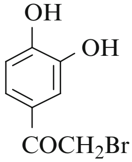
- Also refer to: [5985].

Dimethyl ether [1204-21-3] $C_{10}H_{11}BrO_3$ mol.wt. 259.10

- Obtained by bromination of 2,5-dimethoxyacetophenone using polymer-supported pyridinium bromide perbromide in chloroform at r.t. [6021].
- Also obtained by a halogen-exchange reaction [6022].
- Also refer to: [5981,5994,6018,6023,6024,6025,6026,6027].

2-Bromo-1-(3,4-dihydroxyphenyl)ethanone

[40131-99-5] $C_8H_7BrO_3$ mol.wt. 231.05



Described [5958] **p. 1209**

Syntheses

- Also refer to: [6028,6029,6030].

Dimethyl ether [1835-02-5] $C_{10}H_{11}BrO_3$ mol.wt. 259.10

- Obtained by reaction of bromine with acetoveratrone in chloroform at r.t. (84%) [6031].
- Also obtained by reaction of pyridinium bromide with 3,4-dimethoxyacetophenone in a mixture of chloroform and ethanol [6032].
- Also obtained by reaction of pyridinium hydrobromide perbromide with 3,4-dimethoxy-acetophenone in THF at r.t. for 3 h [5971].
- Also obtained by bromination of 3,4-dimethoxyacetophenone using polymer-supported pyridinium bromide perbromide in chloroform at r.t. [6021].
- Also obtained by reaction of bromine with 3,4-dimethoxyacetophenone in chloroform at r.t. for 1 h (58%) [6033].
- Also refer to: [6009,6020,6026,6027,6034,6035].

m.p. 80–81° [6031].

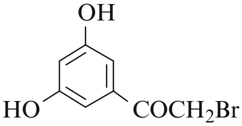
Diacetate $C_{12}H_{11}BrO_5$ mol.wt. 315.12

- Preparation by bubbling bromine vapours with nitrogen into a concentrated solution of 3,4-diacetoxyacetophenone in chloroform (71%) [6036].

m.p. 99.2–99.8° [6036].

2-Bromo-1-(3,5-dihydroxyphenyl)ethanone

[62932-92-7] $C_8H_7BrO_3$ mol.wt. 231.05



Described [5958] **p. 1210**

Syntheses

- Also obtained by treatment of 3,5-dihydroxyacetophenone with cupric bromide in boiling ethyl acetate for 2.5 h [6037].
- Also obtained by treatment of its diacetyl ester (**I**) with hydrobromic acid in boiling dilute ethanol for 30 min. **I** was prepared by reaction of bromine with 3,5-diacetoxyacetophenone in chloroform at r.t. for 30 min [6038].

Dimethyl ether [50841-50-4] $C_{10}H_{11}BrO_3$ mol.wt. 259.10

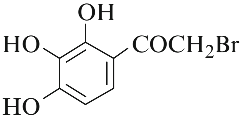
- Refer to: [6023,6026,6034,6039].

Dibenzyl ether [28924-18-7] $C_{22}H_{19}BrO_3$ mol.wt. 411.30

- Refer to: [6040,6041].

2-Bromo-1-(2,3,4-trihydroxyphenyl)ethanone

[105190-52-1] $C_8H_7BrO_4$ mol.wt. 247.05



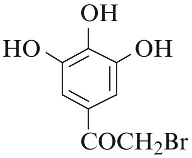
Described [5958] **p. 1210**

Trimethyl ether [103477-58-3] $C_{11}H_{13}BrO_4$ mol.wt. 289.13

- Refer to: [6042].

2-Bromo-1-(3,4,5-trihydroxyphenyl)ethanone

[111011-09-7] $C_8H_7BrO_4$ mol.wt. 247.05



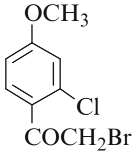
Described [5958] **p. 1210**

Trimethyl ether [51490-01-8] $C_{11}H_{13}BrO_4$ mol.wt. 289.13

- Refer to: [6009,6023,6034,6043].

2-Bromo-1-(2-chloro-4-methoxyphenyl)ethanone

[30095-51-3] $C_9H_8BrClO_2$ mol.wt. 263.52



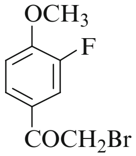
New compound

Synthesis

- Refer to: [6044].

2-Bromo-1-(3-fluoro-4-methoxyphenyl)ethanone

[350-27-6] $C_9H_8BrFO_2$ mol.wt. 247.06



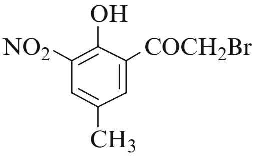
New compound

Synthesis

- Refer to: [6045].

2-Bromo-1-(2-hydroxy-5-methyl-3-nitrophenyl)ethanone

[685892-02-8] $C_9H_8BrNO_4$ mol.wt. 274.07



New compound

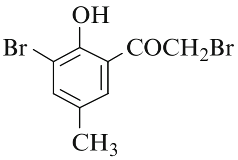
Syntheses

- Refer to: [6046,6047].

Benzoate [36695-28-0] $C_{16}H_{12}BrNO_5$ mol.wt. 378.18 (m.p. 20°) [6048].

2-Bromo-1-(3-bromo-2-hydroxy-5-methylphenyl)ethanone

[194226-50-1] $C_9H_8Br_2O_2$ mol.wt. 307.97



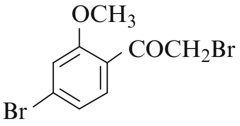
Described [5958] **p. 1212**

Syntheses

- Also refer to: [6046,6047].

2-Bromo-1-(4-bromo-2-methoxyphenyl)ethanone

[252561-75-4] $C_9H_8Br_2O_2$ mol.wt. 307.97



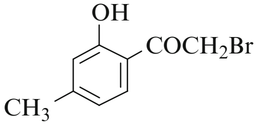
New compound

Synthesis

- Refer to: [6049].

2-Bromo-1-(2-hydroxy-4-methylphenyl)ethanone

[144219-74-9] $C_9H_9BrO_2$ mol.wt. 229.07



Described [5958] **p. 1213**

Synthesis

- Also obtained by reaction of cupric bromide with 2-hydroxy-4-methylacetophenone in boiling ethyl acetate/chloroform mixture for 8 h (82%) [6050].

^{1}H NMR [6050].

Methyl ether [145964-98-3] $C_{10}H_{11}BrO_2$ mol.wt. 243.10

- Obtained by bromination of 2-methoxy-4-methylacetophenone in ethyl ether [6051].
- Also refer to: [5966,6052].

b.p.$_{15}$ 190° [6051].

BIOLOGICAL DATA: Phytotoxic activity [6052]; phytogrow-inhibitory activity on seeds of *Amaranthus hypochondriacus*, *Echinochloa crus-galli* and *Medicago sativa* [6052].

Benzyl ether [860782-82-7] $C_{16}H_{15}BrO_2$ mol.wt. 319.20

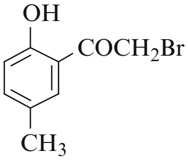
- Obtained by reaction of bromine with 2-benzyloxy-4-methylacetophenone in benzene, first at 0°, then at r.t. for 4 h (40%) [6052].

White solid; m.p. 84° [6052]; ^{1}H NMR [6052], ^{13}C NMR [6052], IR [6052], MS [6052].

BIOLOGICAL DATA: Phytotoxic activity [6052]; phytogrow-inhibitory activity on seeds of *Amaranthus hypochondriacus*, *Echinochloa crus-galli* and *Medicago sativa* [6052].

2-Bromo-1-(2-hydroxy-5-methylphenyl)ethanone

[51317-87-4] $C_9H_9BrO_2$ mol.wt. 229.07



Described [5958] **p. 1213**

Syntheses

- Also refer to: [6046,6047].

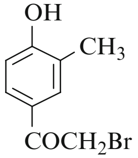
Benzyl ether $C_{16}H_{15}BrO_2$ mol.wt. 319.20

- Obtained by reaction of bromine with 2-benzyloxy-5-methylacetophenone in methanol in the presence of concentrated hydrochloric acid at 0–5° for 1 h, then at r.t. for another 1 h (89%) [6010].

m.p. 115° [6010]; ^{1}H NMR [6010], IR [6010], MS [6010].

2-Bromo-1-(4-hydroxy-3-methylphenyl)ethanone

[41877-17-2] $C_9H_9BrO_2$ mol.wt. 229.07



Described [5958] **p. 1214**

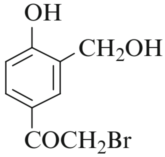
Benzyl ether $C_{16}H_{15}BrO_2$ mol.wt. 319.20

- Obtained by reaction of bromine with 4-benzyloxy-3-methylacetophenone in methanol in the presence of concentrated hydrochloric acid first at 0–5° for 1 h, then at r.t. for 1 h (71%) [6010].

m.p. 170° [6010]; ^1H NMR [6010], ^{13}C NMR [6010], IR [6010], MS [6010].

2-Bromo-1-[4-hydroxy-3-(hydroxymethyl)phenyl]ethanone

[62932-94-9] $C_9H_9BrO_3$ mol.wt. 245.07



Described [5958] **p. 1214**

Synthesis

- Obtained by treatment of its diacetate with 1 N hydrogen bromide in tetrahydrofuran at 75° [6053].

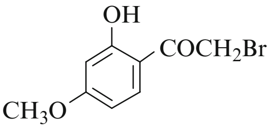
4-Benzyl ether [324556-80-1] $C_{16}H_{15}BrO_3$ mol.wt. 335.20

- Obtained by reaction of bromine with 4-benzyloxy-3-hydroxymethylacetophenone in methanol in the presence of concentrated hydrochloric acid first at 0–5° for 1 h, then at r.t. for 1 h (70%) [6010].
- Also refer to: [6013,6014,6054].

m.p. 130° [6010]; ^1H NMR [6010], ^{13}C NMR [6010], IR [6010], MS [6010].

2-Bromo-1-(2-hydroxy-4-methoxyphenyl)ethanone

[60965-24-4] $C_9H_9BrO_3$ mol.wt. 245.07



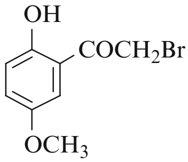
Described [5958] **p. 1214**

Synthesis

- Also refer to: [5960].

2-Bromo-1-(2-hydroxy-5-methoxyphenyl)ethanone

[203524-87-2] $C_9H_9BrO_3$ mol.wt. 245.07



Described [5958] **p. 1215**

Syntheses

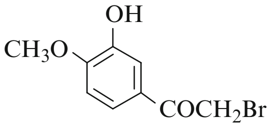
- Also refer to: [5961,6055].

Benzyl ether [857561-04-7] $C_{16}H_{15}BrO_3$ mol.wt. 335.20

– Obtained by bromination of 2-benzyloxy-5-methoxyacetophenone,
  - using polymer-supported pyridinium bromide perbromide in chloroform (μw, 300 sec) at 60° [6021];
  - with bromine in water at r.t. [6021].

2-Bromo-1-(3-hydroxy-4-methoxyphenyl)ethanone

[90971-90-7] $C_9H_9BrO_3$ mol.wt. 245.07



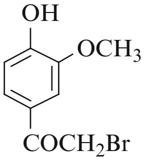
Described [5958] **p. 1215**

Benzyl ether $C_{16}H_{15}BrO_3$ mol.wt. 335.20

– Obtained by reaction of pyridinium hydrobromide perbromide with 3-benzyloxy-4-methoxy-acetophenone in THF at r.t. for 3 h [5971].

2-Bromo-1-(4-hydroxy-3-methoxyphenyl)ethanone

[69638-06-8] $C_9H_9BrO_3$ mol.wt. 245.07



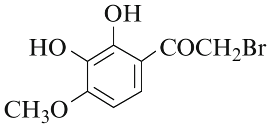
Described [5958] **p. 1215**

Acetate [50893-83-9] $C_{11}H_{11}BrO_4$ mol.wt. 287.11

– Refer to: [6056].

2-Bromo-1-(2,3-dihydroxy-4-methoxyphenyl)ethanone

[204648-67-9] $C_9H_9BrO_4$ mol.wt. 261.07



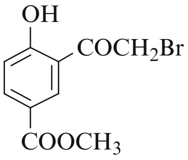
Described [5958] **p. 1216**

Synthesis

– Also refer to: [6057].

Methyl 3-(Bromoacetyl)-4-hydroxybenzoate

[71620-33-2] $C_{10}H_9BrO_4$ mol.wt. 273.08



New compound

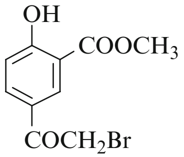
Synthesis

– Obtained by adding methyl 3-acetyl-4-hydroxybenzoate in a chloroform and ethyl acetate mixture to a suspension of $CuBr_2$ in the same solvent. Then the resulting mixture was refluxed for 6 h (49%) [6058].

m.p. 98–99° [6058].

Methyl 5-(Bromoacetyl)-2-hydroxybenzoate

[36256-45-8] $C_{10}H_9BrO_4$ mol.wt. 273.08



New compound

Synthesis

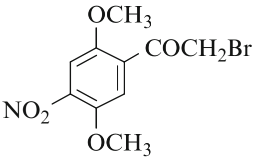
- Obtained by reaction of dioxane dibromide with methyl 5-acetyl-2-hydroxybenzoate in dioxane/ethyl ether mixture (76%) [6059].

Benzoate $C_{17}H_{13}BrO_5$ mol.wt. 377.19

- Refer to: [6054]

2-Bromo-1-(2,5-dimethoxy-4-nitrophenyl)ethanone

[851531-99-2] $C_{10}H_{10}BrNO_5$ mol.wt. 304.10



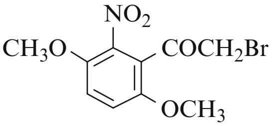
New compound

Synthesis

- Refer to: [6039].

2-Bromo-1-(3,6-dimethoxy-2-nitrophenyl)ethanone

[99057-95-1] $C_{10}H_{10}BrNO_5$ mol.wt. 304.10



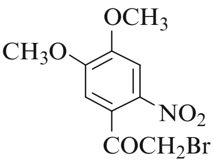
New compound

Synthesis

- Refer to: [6039].

2-Bromo-1-(4,5-dimethoxy-2-nitrophenyl)ethanone

[33245-76-0] $C_{10}H_{10}BrNO_5$ mol.wt. 304.10



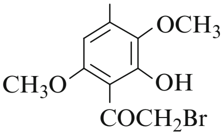
New compound

Synthesis

- Refer to: [6039].

2-Bromo-1-(2,4-dihydroxy-3,6-dimethoxyphenyl)ethanone

$C_{10}H_{11}BrO_5$ mol.wt. 291.10



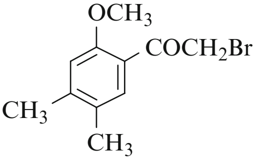
Described [5958] **p. 1220**

Synthesis

- Preparation by reaction of N-bromosuccinimide with 2,4-dihydroxy-3,6-dimethoxyacetophenone [6060].

m.p. 159–160° [6060].

2-Bromo-1-(2-methoxy-4,5-dimethylphenyl)ethanone



$C_{11}H_{13}BrO_2$ mol.wt. 257.13

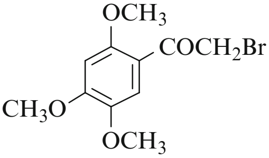
New compound

Synthesis

- Obtained by bromination of 2-methoxy-4,5-dimethyl-acetophenone [6051].

2-Bromo-1-(2,4,5-trimethoxyphenyl)ethanone

[7298-46-6]



$C_{11}H_{13}BrO_4$ mol.wt. 289.13

New compound

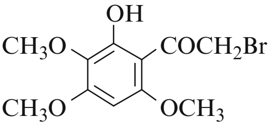
Synthesis

- Obtained by reaction of bromoacetonitrile with 1,2,4-trimethoxybenzene (Hoesch reaction) [6061].

USE: Intermediate in lamellarin-S synthesis [6061].

2-Bromo-1-(2-hydroxy-3,4,6-trimethoxyphenyl)ethanone

[91335-60-3]



$C_{11}H_{13}BrO_5$ mol.wt. 305.13

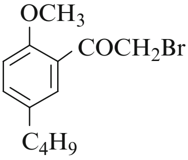
New compound

Synthesis

- Obtained by bromination under UV light of 2-acetoxy-3,4,6-trimethoxyacetophenone followed by hydrolysis of the product with methanolic hydrochloric acid [6060] according to the process [6062].

2-Bromo-1-(5-butyl-2-methoxyphenyl)ethanone

[784177-15-7]



$C_{13}H_{17}BrO_2$ mol.wt. 285.18

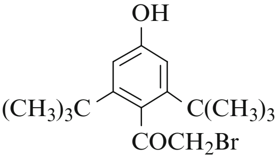
New compound

Synthesis

- Refer to: [6063].

1-[2,6-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-2-bromoethanone

[1019197-17-1]



$C_{16}H_{23}BrO_2$ mol.wt. 327.26

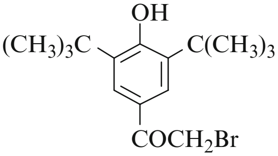
New compound

Synthesis

- Refer to: [6064].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-2-bromoethanone

[14386-64-2] $C_{16}H_{23}BrO_2$ mol.wt. 327.26



Described [5958] **p. 1223**

Syntheses

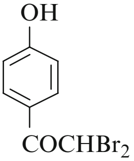
- Also obtained by reaction of bromine with 4-hydroxy-3,5-ditert-butylacetophenone in the presence of aluminium chloride in octane at 70° for 30 min (91%) [6065].
- Also obtained by reaction of bromine with 4-hydroxy-3,5-di-tert-butylacetophenone [6066] in octane or isooctane [6067].
- Also refer to: [6064,6068].

m.p. 95–97° [6065].

11.1.2 From Dibromoacetic Acid [5958] p. 1224

2,2-Dibromo-1-(4-hydroxyphenyl)ethanone

[92596-96-8] $C_8H_6Br_2O_2$ mol.wt. 293.94



Described [5958] **p. 1226**

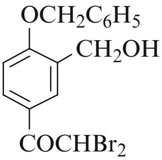
Synthesis

- Also refer to: [5958].

Methyl ether [13664-92-1] $C_9H_8Br_2O_2$ mol.wt. 307.97 [5990]

2,2-Dibromo-1-[3-(hydroxymethyl)-4-(phenylmethoxy)phenyl]ethanone

[324556-83-4] $C_{16}H_{14}Br_2O_3$ mol.wt. 414.09



New compound

Syntheses

- Obtained by reaction of bromine with 4-benzyloxy-3-hydroxymethylacetophenone in methanol in the presence of concentrated hydrochloric acid at 0–5° for 1 h, then at r.t. for another 1 h (25%) [6010].
- Also refer to: [6014].

m.p. 110° [6010]; ^1H NMR [6010], IR [6010], MS [6010].

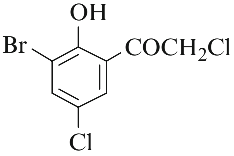
11.1.3 From Tribromoacetic Acid [5958] p. 1228

11.2 Compounds Derived from Chloroacetic Acids [5958] p. 1229

11.2.1 From Monochloroacetic Acid [5958] p. 1229

1-(3-Bromo-5-chloro-2-hydroxyphenyl)-2-chloroethanone

[1019854-96-6] $C_8H_5BrCl_2O_2$ mol.wt. 283.94



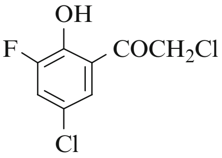
New compound

Synthesis

- Refer to: [6069].

2-Chloro-1-(5-chloro-3-fluoro-2-hydroxyphenyl)ethanone

[1019854-97-7] $C_8H_5Cl_2FO_2$ mol.wt. 223.03



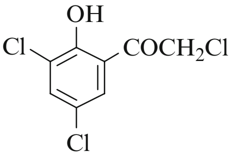
New compound

Synthesis

- Refer to: [6069].

2-Chloro-1-(3,5-dichloro-2-hydroxyphenyl)ethanone

[79214-30-5] $C_8H_5Cl_3O_2$ mol.wt. 239.48



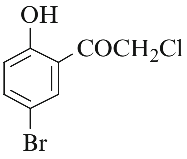
Described [5958] **p. 1229**

Synthesis

- Also refer to: [6069].

1-(5-Bromo-2-hydroxyphenyl)-2-chloroethanone

[100959-21-5] $C_8H_6BrClO_2$ mol.wt. 249.49



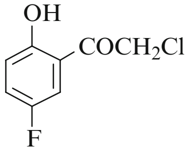
Described [5958] **p. 1230**

Synthesis

- Also refer to: [6070].

2-Chloro-1-(5-fluoro-2-hydroxyphenyl)ethanone

[2002-75-7] $C_8H_6ClFO_2$ mol.wt. 188.59



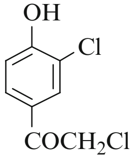
Described [5958] **p. 1230**

Synthesis

- Also refer to: [6070].

2-Chloro-1-(3-chloro-4-hydroxyphenyl)ethanone

[39066-18-7] $C_8H_6Cl_2O_2$ mol.wt. 205.04

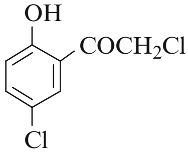


Described [5958] **p. 1231**

Methyl ether [79881-25-7] $C_9H_8Cl_2O_2$ mol.wt. 219.07 [6071]

2-Chloro-1-(5-chloro-2-hydroxyphenyl)ethanone

[24483-75-8] $C_8H_6Cl_2O_2$ mol.wt. 205.04



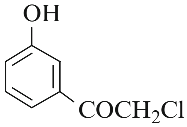
Described [5958] **p. 1232**

Syntheses

- Also refer to: [6069,6072].

2-Chloro-1-(3-hydroxyphenyl)ethanone

[62932-90-5] $C_8H_7ClO_2$ mol.wt. 170.60



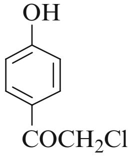
Described [5958] **p. 1233**

Synthesis

- Also refer to: [6073].

2-Chloro-1-(4-hydroxyphenyl)ethanone

[6305-04-0] $C_8H_7ClO_2$ mol.wt. 170.60



Described [5958] **p. 1233**

Syntheses

- Also obtained by reaction of acetyl chloride with anisole in the presence of aluminium chloride in n-heptane at 35° for 4 h 30 min (20%) [5961].
- Also refer to: [5962,6002,6074].

^{1}H NMR [5961], MS [5961].

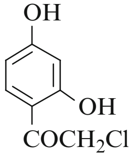
Methyl ether [2196-99-8] $C_9H_9ClO_2$ mol.wt. 184.62

- Obtained by treatment of 4-methoxyacetophenone with 1,3-dichloro-5,5-dimethylhydantoin (DCDMH) and p-toluenesulfonic acid in methanol at 30–35° (94%) [6075].
- Also refer to: [6074,6076,6077,6078].

m.p. 96–98° [6078], 92–94° [6075].

2-Chloro-1-(2,4-dihydroxyphenyl)ethanone

[25015-92-3] $C_8H_7ClO_3$ mol.wt. 186.59



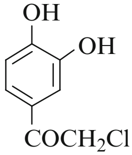
Described [5958] **p. 1234**

Dimethyl ether [4783-90-8] $C_{10}H_{11}ClO_3$ mol.wt. 214.65

- Refer to: [6079].

2-Chloro-1-(3,4-dihydroxyphenyl)ethanone

[99-40-1] $C_8H_7ClO_3$ mol.wt. 186.59



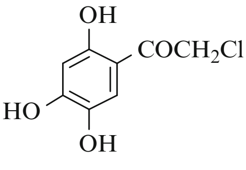
Described [5958] **p. 1235**

Syntheses

- Also refer to: [5985,6080,6081,6082,6083].

2-Chloro-1-(2,4,5-trihydroxyphenyl)ethanone

[14771-02-9] $C_8H_7ClO_4$ mol.wt. 202.59



Described [5958] **p. 1236**

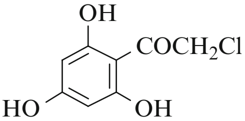
Trimethyl ether [19278-85-4] $C_{11}H_{13}ClO_4$ mol.wt. 244.67

- Obtained by reaction of chloroacetonitrile with 1,2,4-trimethoxybenzene (Hoesch reaction) [6061].

USE: Intermediate in lamellarin-S synthesis [6061].

2-Chloro-1-(2,4,6-trihydroxyphenyl)ethanone

[110865-03-7] $C_8H_7ClO_4$ mol.wt. 202.59



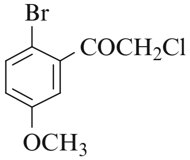
Described [5958] **p. 1236**

Syntheses

- Also refer to: [6084,6085].

1-(2-Bromo-5-methoxyphenyl)-2-chloroethanone

[949898-83-3] $C_9H_8BrClO_2$ mol.wt. 263.52



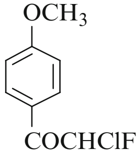
New compound

Synthesis

- Refer to: [6086].

2-Chloro-2-fluoro-1-(4-methoxyphenyl)ethanone

$C_9H_8ClFO_2$ mol.wt. 202.61



New compound

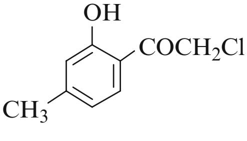
Syntheses

- Obtained by adding 2,2-dichloro-2-fluoromethyl 4-methoxyphenyl ketone in DMF to a solution of stannous chloride and aluminium powder in DMF at r.t. Then, the mixture was heated at 60° for 1.5 h (52%) [6087].
- Also obtained by reaction of ethyl chlorofluoroacetate with p-anisole magnesium bromide in ethyl ether at −78° for 2 h (20%) [6088].

b.p.$_{12}$ 145° [6088]; light-yellow crystals [6088];
^{1}H NMR [6087,6088], ^{19}F NMR [6087,6088], IR [6087,6088]; TLC [6087].

2-Chloro-1-(2-hydroxy-4-methylphenyl)ethanone

[20834-75-7] $C_9H_9ClO_2$ mol.wt. 184.62



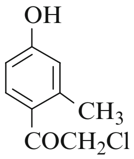
Described [5958] **p. 1239**

Synthesis

- Also obtained by Fries rearrangement of 3-methylphenyl chloroacetate in the presence of Fe- or Zn-contg. catalysts [6089].

2-Chloro-1-(4-hydroxy-2-methylphenyl)ethanone

[37904-71-5] $C_9H_9ClO_2$ mol.wt. 184.62



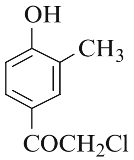
Described [5958] **p. 1240**

Synthesis

- Also obtained by Fries rearrangement of 3-methylphenyl chloroacetate in the presence of Fe- or Zn-contg. catalysts [6089].

2-Chloro-1-(4-hydroxy-3-methylphenyl)ethanone

[40943-24-6] $C_9H_9ClO_2$ mol.wt. 184.62



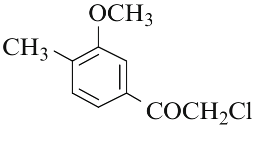
Described [5958] **p. 1240**

Methyl ether [62613-62-1] $C_{10}H_{11}ClO_2$ mol.wt. 198.65

- Obtained by reaction of chloroacetyl chloride with 2-methoxytoluene [6090]. m.p. 70° [6090].

2-Chloro-1-(3-methoxy-4-methylphenyl)ethanone

$C_{10}H_{11}ClO_2$ mol.wt. 198.65



New compound

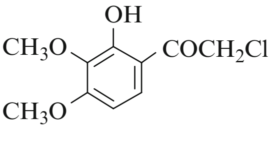
Synthesis

- Obtained from 3-methoxy-4-methylbenzoyl chloride by Nierenstein reaction [6051].

b.p.$_{10}$ 157–160° [6051].

2-Chloro-1-(2-hydroxy-3,4-dimethoxyphenyl)ethanone

[7507-92-8] $C_{10}H_{11}ClO_4$ mol.wt. 230.65



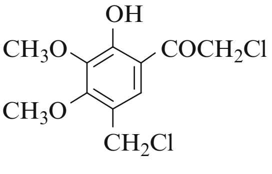
Described [5958] **p. 1248**

Synthesis

- Also refer to: [6091].

2-Chloro-1-[5-(chloromethyl)-2-hydroxy-3,4-dimethoxyphenyl]ethanone

[76439-46-8] $C_{11}H_{12}Cl_2O_4$ mol.wt. 279.12

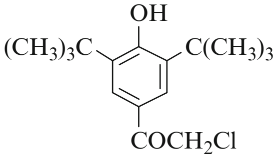


Described [5958] **p. 1250**

Synthesis

- Also refer to: [6091].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-2-chloroethanone



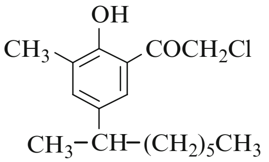
$C_{16}H_{23}ClO_2$ mol.wt. 282.81

New compound

Syntheses

- Refer to: [6065,6092,6093].

2-Chloro-1-[2-hydroxy-3-methyl-5-(1-methylheptyl)phenyl]ethanone



$C_{17}H_{25}ClO_2$ mol.wt. 296.84

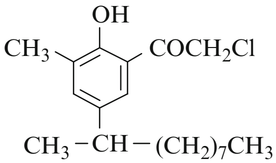
New compound

Synthesis

- Obtained by Friedel–Crafts acylation of 2-methyl-4-(1-methylheptyl)phenol with chloroacetyl chloride in the presence of zinc chloride [6094].

b.p.$_3$ 186–188.3° [6094]; d_{20} = 1.0486 [6094]; n_D^{20} = 1.5188 [6094].

2-Chloro-1-[2-hydroxy-3-methyl-5-(1-methylnonyl)phenyl]ethanone



$C_{19}H_{29}ClO_2$ mol.wt. 324.89

New compound

Synthesis

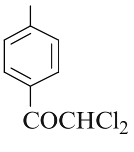
- Obtained by Friedel–Crafts acylation of 2-methyl-4-(1-methylnonyl)phenol with chloroacetyl chloride in the presence of zinc chloride [6094].

b.p.$_{1.8}$ 176–177° [6094]; d_{20} = 1.0301 [6094]; n_D^{20} = 1.5184 [6094].

11.2.2 From Dichloroacetic Acid [5958] p. 1254

2,2-Dichloro-1-(4-hydroxyphenyl)ethanone

[4974-60-1] $C_8H_6Cl_2O_2$ mol.wt. 205.04



Described [5958] **p. 1256**

Syntheses

- Also refer to: [6095,6097].

Methyl ether [29003-60-9] $C_9H_8Cl_2O_2$ mol.wt. 219.07 [6096]

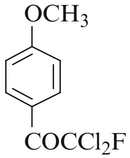
- Preparation by treatment of anisole with benzyltrimethylammonium tetrachloroiodate (BTMA ICl_4) in acetic acid at 70° for 5 h (78%) [6098].

- Also obtained by reaction of anisole with dichloroacetyl chloride in the presence of aluminium chloride [6095].

m.p. 77–78° [6098], 75–76° [6095]; ^{1}H NMR [6098], IR [6098].

2,2-Dichloro-2-fluoro-1-(4-methoxyphenyl)ethanone

[16629-88-2] $C_9H_7Cl_2FO_2$ mol.wt. 237.06



New compound

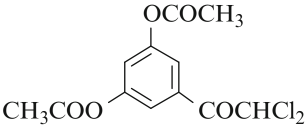
Syntheses

- To acetonitrile was added periodic acid and this mixture was stirred at r.t. for 15 min. Dichlorofluoromethyl 4-methoxyphenyl alcohol was added followed by addition of pyridinium chlorochromate in acetonitrile, then the reaction mixture was stirred at 0°, for 10 min and at r.t. for 2.5 h (62%) [6087].
- Also obtained by reaction of sodium fluorodichloroacetate with p-anisole magnesium bromide in ethyl ether [6099].

b.p.$_{2}$ 110–112° [6099]; $n_D^{18} = 1.5616$ [6099];
^{1}H NMR [6087], ^{19}F NMR [6087], IR [6087,6099].

1-(3,5-Diacetoxyphenyl)-2,2-dichloroethanone

[144660-11-7] $C_{12}H_{10}Cl_2O_5$ mol.wt. 305.11



New compound

Synthesis

- Preparation by treatment of 3,5-diacetoxyacetophenone with benzyltrimethylammonium tetrachloroiodate (BTMA ICl_4) in acetic acid at 70° for 6 h (93%) [6098].

m.p. 85–87° [6098]; ^{1}H NMR [6098], IR [6098].

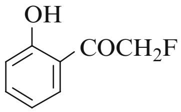
11.2.3 From Trichloroacetic Acid [5958] p. 1259

11.3 Compounds Derived from Fluoroacetic Acids [5958] p. 1264

11.3.1 From Monofluoroacetic Acid [5958] p. 1264

2-Fluoro-1-(2-hydroxyphenyl)ethanone

[83505-27-5] $C_8H_7FO_2$ mol.wt. 154.14



Described [5958] **p. 1264**

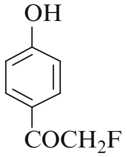
Methyl ether [2967-87-5] $C_9H_9FO_2$ mol.wt. 168.17

- Obtained by reaction of α-fluoroacetonitrile with 2-bromoanisole [6100].
- Also refer to: [6101].

m.p. 88–89° [6100]; IR [6100].

2-Fluoro-1-(4-hydroxyphenyl)ethanone

[295779-85-0] $C_8H_7FO_2$ mol.wt. 154.14



Described [5958] **p. 1265**

Synthesis

- Also refer to: [6101].

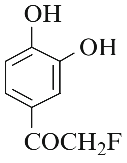
Methyl ether [73744-44-2] $C_9H_9FO_2$ mol.wt. 168.17

- Obtained by reaction of fluoroacetyl chloride with anisole in the presence of aluminium chloride in chloroform at r.t. [6102].
- Also obtained by reaction of pyridinium poly(hydrogen fluoride) with 4-methoxy-α-diazo-acetophenone in ethyl ether at r.t. for 24 h (54%) [6103].
- Also obtained by reaction of Selectfluor with 1-(4-methoxyphenyl)ethanone in boiling methanol for 24 h (71%) [6104].
- Preparation by adding Rongalite to a solution of dichlorofluoromethyl 4-methoxyphenyl ketone in ethanol under nitrogen. Then, the solution was refluxing for 45 min (65%) [6087].
- Also refer to: [6101,6105,6106,6107,6108].

m.p. 79.3–79.8° [6106], 78.5–79.4° [6102], 78–79° [6103,6104];
^{1}H NMR [6087,6102,6103,6104,6108], ^{13}C NMR [6102,6103,6106],
^{19}F NMR [6087,6102,6103,6106,6108], IR [6087,6102,6103,6106,6108],
MS [6103,6108]; TLC [6087].

1-(3,4-Dihydroxyphenyl)-2-fluoroethanone

[402-99-3] $C_8H_7FO_3$ mol.wt. 170.14



New compound

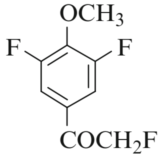
Synthesis

- Obtained by reaction of monofluoroacetyl chloride with pyrocatechol in the presence of phosphorous oxychloride in benzene (32%) [6109].

m.p. 185° [6109].

1-(3,5-Difluoro-4-methoxyphenyl)-2-fluoroethanone

[872968-14-4] $C_9H_7F_3O_2$ mol.wt. 204.15



New compound

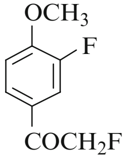
Synthesis

- Obtained by fluorination of 4-methoxyacetophenone with manganese (IV) tetrafluoride formed in situ using manganese (IV) dioxide and pyridinium polyhydrogeno-fluoride under very mild conditions [6110].

MS [6110].

2-Fluoro-1-(3-fluoro-4-methoxyphenyl)ethanone

[501426-62-6] $C_9H_8F_2O_2$ mol.wt. 186.16



New compound

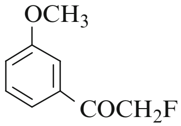
Synthesis

- Also obtained by reaction of Selectfluor with 1-(4-methoxyphenyl)ethanone in boiling methanol for 24 h (9%) [6104].

m.p. 82–84° [6104]; ^{1}H NMR [6104].

2-Fluoro-1-(3-methoxyphenyl)ethanone

$C_9H_9FO_2$ mol.wt. 168.17



New compound

Syntheses

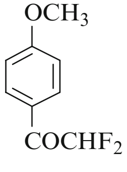
- Obtained by reaction of potassium bifluoride with α-bromo-3-methoxyacetophenone in triethyleneglycol at 100° for 3 h (54%) [5989].
- Also obtained by reaction of aqueous sodium hydroxide with 2,3,3-trifluoro-1-(3-methoxy-phenyl)-1-propenyl p-toluenesulfonate in DMSO at 80° for 3 h (69%) [6106].

m.p. 53–54° [5989].

11.3.2 From Difluoroacetic Acid [5958] p. 1265

2,2-Difluoro-1-(4-methoxyphenyl)ethanone

[114829-07-1] $C_9H_8F_2O_2$ mol.wt. 186.16



Described [5958] **p. 1266**

Syntheses

- Also obtained by treatment of butyl-[2,2-difluoro-1-(4-methoxyphenyl)-eth-*(Z)*-ylidene]amine with aqueous hydrochloric acid in acetonitrile [6111].

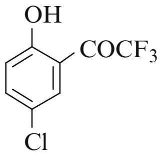
- Also obtained by reaction of p-anisole magnesium bromide with ethyl 2,2-difluoroacetate in THF at −78° (36%) [6112].
- Also obtained by treatment of 2,2-difluoro-1-(4-methoxyphenyl)vinyl benzoate with aqueous potassium hydroxide in THF at r.t. for 12 h (72%) [6113].
- Also obtained by reaction of Selectfluor with 1-(4-morpholinyl)-1-(4-methoxyphenyl)ethene in the presence of 4 Å molecular sieves in acetonitrile at −10° for 8 h (64%) [6107].
- Also refer to: [6108,6114,6115].

^{1}H NMR [6107,6108,6111,6112,6113], ^{13}C NMR [6111,6112],
^{19}F NMR [6107,6108,6111,6112,6113], IR [6108,6111,6112,6115], MS [6108,6111].

11.3.3 From Trifluoroacetic Acid [5958] p. 1266

1-(5-Chloro-2-hydroxyphenyl)-2,2,2-trifluoroethanone

$C_8H_4ClF_3O_2$ mol.wt. 224.57



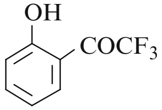
New compound

Synthesis

- Preparation in two steps: First, reaction of ethyl trifluoroacetate with 2-butoxy-5-chlorophenyllithium, itself obtained by reaction of butyllithium with 2-butoxy-5-chloro-1-bromo-benzene; then, addition of trifluoroacetic acid in the mixture so obtained (70%) [6116].

2,2,2-Trifluoro-1-(2-hydroxyphenyl)ethanone

[25666-51-7] $C_8H_5F_3O_2$ mol.wt. 190.12



Described [5958] **p. 1268**

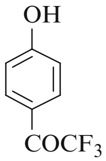
Synthesis

- Preparation in two steps: First, reaction of ethyl trifluoroacetate with 2-butoxyphenyllithium, itself obtained by reaction of butyllithium with 2-butoxy-1-bromobenzene; then, addition of trifluoroacetic acid in the mixture so obtained (69%) [6116].

Methylether [26944-43-4] $C_9H_7F_3O_2$ mol.wt. 204.15
Refer to: [6112,6117].

2,2,2-Trifluoro-1-(4-hydroxyphenyl)ethanone

[1823-63-8] $C_8H_5F_3O_2$ mol.wt. 190.12



Described [5958] **p. 1268**

Synthesis

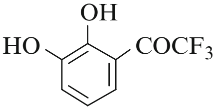
- Also refer to: [6118].

Methyl ether [711-38-6] $C_9H_7F_3O_2$ mol.wt. 204.15

- Refer to: [6112,6118,6119,6120,6121,6122].

1-(2,3-Dihydroxyphenyl)-2,2,2-trifluoroethanone

[874992-53-7] $C_8H_5F_3O_3$ mol.wt. 206.12



New compound

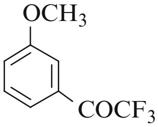
Syntheses

- Obtained by bioconversion of trifluoroacetophenone through living cells of *Escherichia coli* carrying,
  - plasmid pKF6256 expressing *todCI-bphA2A3A4* (1.9%) [6123];
  - plasmid pUC6256B expressing *todCI-bphA2A3A4* and *bphB* (4.4%) [6123].

^1H NMR [6123], ^{13}C NMR [6123], MS [6123].

2,2,2-Trifluoro-1-(3-methoxyphenyl)ethanone

[30724-22-2] $C_9H_7F_3O_2$ mol.wt. 204.15



New compound

Syntheses

- Obtained by adding successively 1.6 M n-BuLi in hexane, then after 10 min, N,N-diethyltrifluoroacetamide (5 min) to a solution of 1-bromo-3-methoxybenzene in THF at −78° under argon and stirring the mixture for 2 h (88%) [6124].
- Also refer to: [6112].

Pale yellow oil [6124];
^1H NMR [6124], ^{13}C NMR [6124], ^{19}F NMR [6124], IR [6124], MS [6124].

Oxime [154187-44-7] $C_9H_8F_3NO_2$ mol.wt. 219.05

- Preparation by reaction of hydroxylamine hydrochloride with 2,2,2-trifluoro-1-(3-methoxy-phenyl)ethanone in refluxing methanol for 2 h (97%) [6124].
- Also refer to: [6125].

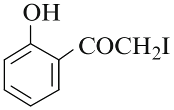
Clear viscous oil [6124];
^1H NMR [6124], ^{13}C NMR [6124], ^{19}F NMR [6124], IR [6124], MS [6124].

11.4 Compounds Derived from Iodoacetic Acids [5958] p. 1288

11.4.1 From Monoiodoacetic Acid [5958] p. 1288

1-(2-Hydroxyphenyl)-2-iodoethanone

[99233-30-4] $C_8H_7IO_2$ mol.wt. 262.05



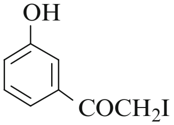
Described [5958] **p. 1289**

Synthesis

- Also refer to: [6126].

1-(3-Hydroxyphenyl)-2-iodoethanone

[1000375-32-5] $C_8H_7IO_2$ mol.wt. 262.05



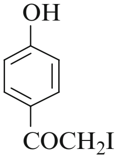
New compound

Synthesis

- Obtained by reaction of iodine with 3-hydroxyacetophenone in the presence of cupric oxide in refluxing methanol for 2 h (84%) [6127].

1-(4-Hydroxyphenyl)-2-iodoethanone

[99233-31-5] $C_8H_7IO_2$ mol.wt. 262.05



Described [5958] **p. 1289**

Synthesis

- Also obtained by reaction of iodine with 4-hydroxy-acetophenone in the presence of cupric oxide in refluxing methanol for 2 h (91%) [6124].

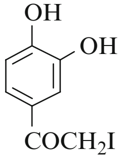
Methyl ether [80336-72-7] $C_9H_9IO_2$ mol.wt. 276.07

- To a solution of 4-methoxyacetophenone (2 mmol) in methanol, containing iodine (1 mmol) and a 30% aqueous solution of hydrogen peroxide (1.2 mmol), was added concentrated sulfuric acid (0.2 mmol) and the mixture stirred at 60° for 1–3 h (94%) [6128].
- To a solution of 4-methoxyacetophenone (1 mmol) in methanol, containing iodine (0.5 mmol) and a 30% aqueous solution of hydrogen peroxide (0.6 mmol), was added $H_4SiO_4.12WO_3$ (0.03–0.033 mmol) and the mixture stirred at 65° for 1.5–3 h (95%) [6128].
- Also obtained by reaction of iodine with 4-methoxyacetophenone in the presence of cupric oxide in methanol at 65° for 1 h (99%) [6127].
- Also obtained by reaction of potassium iodide with ω-chloro-p-methoxyacetophenone by prolonged heating in aqueous alcohol or in nitromethane [6076].
- Also refer to: [6129].

m.p. 61° [6076]; ^{1}H NMR [6128], MS [6128]; TLC [6128].

1-(3,4-Dihydroxyphenyl)-2-iodoethanone

[105174-59-2] $C_8H_7IO_3$ mol.wt. 278.05



Described [5958] **p. 1290**

Dimethyl ether [569352-21-2] $C_{10}H_{11}IO_3$ mol.wt. 306.10

- To a solution of 3,4-dimethoxyacetophenone (2 mmol) in methanol, containing iodine (1 mmol) and a 30% aqueous solution of hydrogen peroxide (1.2 mmol), was added concentrated sulfuric acid (0.2 mmol) and the mixture stirred at 60° for 1–3 h (93%) [6128].
- To a solution of 3,4-dimethoxyacetophenone (1 mmol) in methanol, containing iodine (0.5 mmol) and a 30% aqueous solution of hydrogen peroxide (0.6 mmol), was added $H_4SiO_4.12WO_3$ (0.03–0.033 mmol) and the mixture stirred at 65° for 1.5–3 h (95%) [6128].

^{1}H NMR [6128], MS [6128]; TLC [6128].

11.4.2 From Diiodoacetic Acid [5958] p. 1291

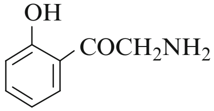
11.4.3 From Triiodoacetic Acid [5958] p. 1291

Chapter 12. Compounds Derived from Aminoacetic Acids [5958] p. 1293

12.1 Compounds Derived from Aminoacetic Acid [5958] p. 1293

2-Amino-1-(2-hydroxyphenyl)ethanone

[72481-17-5] $C_8H_9NO_2$ mol.wt. 151.17



Described [5958] **p. 1293**

Syntheses

- Also obtained by reaction of hexamethylenetetramine with 2-chloro-1-(2-hydroxyphenyl)ethanone in trichloroethylene [6126,6130].
- Also obtained by reaction of Hexamethylenetetramine with 1-(2-hydroxyphenyl)-2-iodoethanone in trichloroethylene [6126,6130].
- Also obtained by treatment of 2-phthalimido-1-(2-methoxyphenyl)ethanone with hydrogen iodide in methyl acetate [6131].

Hydrochloride [505094-69-9] $C_8H_9NO_2$, HCl mol.wt. 187.63

- Refer to: [6126,6132].

  m.p. 235° [6126].

Methyl ether $C_9H_{11}NO_2$ mol.wt. 165.19 [6133].

Hydrochloride (of the methyl ether) [34589-97-4] $C_9H_{11}NO_2$, HCl mol.wt. 201.65

- Refer to: [6132,6134], (72%) [6133].

  m.p. 165° [6133]; ^{1}H NMR [6133], IR [6133].

2-Amino-1-(3-hydroxyphenyl)ethanone *(Hydrochloride)*

[14665-75-9] $C_8H_9NO_2$, HCl mol.wt. 187.63

Described [5958] **p. 1294**

Synthesis

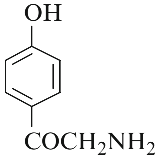
- Also refer to: [6132].

Methyl ether [24037-72-7] $C_9H_{11}NO_2$, HCl mol.wt. 201.65

- Refer to: [6027,6132,6134].

2-Amino-1-(4-hydroxyphenyl)ethanone

[77369-38-1] $C_8H_9NO_2$ mol.wt. 151.17



Described [5958] **p. 1294**

Synthesis

- Also refer to: [6135].

Hydrochloride [19745-72-3] $C_8H_9NO_2$, HCl mol.wt. 187.63

- Obtained by treatment of α-bromo-4-hydroxyacetophenone with hexamethylenetetramine in chloroform at r.t. for 1 h [5971].
- Also refer to: [6132].

Methyl ether [40513-43-7] $C_9H_{11}NO_2$ mol.wt. 165.19

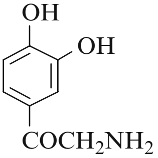
- Refer to: [6027,6135,6136].

Hydrochloride (of the methyl ether) [3883-94-1] $C_9H_{11}NO_2$, HCl mol.wt. 201.65

- Obtained by treatment of α-bromo-4-methoxyacetophenone with hexamethylenetetramine in chloroform at r.t. for 1 h [5971].
- Also refer to: [6127,6132,6134,6137,6138,6139].

2-Amino-1-(3,4-dihydroxyphenyl)ethanone

[499-61-6] $C_8H_9NO_3$ mol.wt. 167.16



Described [5958] **p. 1296**

Synthesis

- Also obtained by reaction of 3,4-dihydroxyphenacyl chloride and α-N-acetyllysine in 15% potassium borate and r.t. overnight, then adding acetic acid to adjust pH to 5 [6140].

Isolation from natural sources

- Obtained by hydrolysis of sclerotized cuticles from locust *Scistocerca gregaria* and the beetle *Tenebrio molitor* by dilute hydrochloric acid [6140].

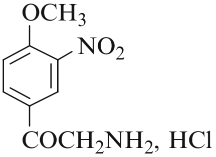
MS [6140]; HPLC [6140].

Dimethyl ether *(Hydrochloride)* [61416-34-0] $C_{10}H_{13}NO_3$, HCl mol.wt. 231.69

- Also obtained by treatment of α-bromo-3,4-dimethoxyacetophenone with hexamethylenetetramine in chloroform at r.t. for 1 h [5971].
- Also refer to: [6132].

2-Amino-1-(4-methoxy-3-nitrophenyl)ethanone *(Hydrochloride)*

[877395-19-2] $C_9H_{10}N_2O_4$, HCl mol.wt. 246.65



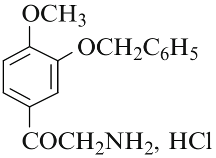
New compound

Synthesis

- Obtained by treatment of α-bromo-4-methoxy-3-nitro-acetophenone with hexamethylenetetramine in chloroform at r.t. for 1 h [5971].

2-Amino-1-[-4-methoxy-3-(phenylmethoxy)phenyl]ethanone *(Hydrochloride)*

$C_{16}H_{17}NO_3$, HCl mol.wt. 307.78



New compound

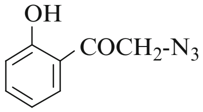
Synthesis

- Obtained by treatment of α-bromo-3-(benzyloxy)-4-methoxyacetophenone with hexamethylenetetramine in chloroform at r.t. for 1 h [5971].

12.2 Compounds Derived from Substituted Aminoacetic Acids [5958] p. 1298

2-Azido-1-(2-hydroxyphenyl)ethanone

[67139-49-5] $C_8H_7N_3O_2$ mol.wt. 177.16



New compound

Syntheses

- Preparation by reaction of sodium azide with 2-bromo-1-(2-hydroxyphenyl)ethanone (89%) [6141].
- Also refer to: [6142,6143].

m.p. 73–74° [6141].

Methyl ether $C_9H_9N_3O_2$ mol.wt. 191.19

- Obtained by reaction of sodium azide with 2-bromo-1-(2-methoxyphenyl)ethanone in dilute ethanol at r.t. for 30 min (89%) [6133].

m.p. 45–46° [6133]; ^1H NMR [6133], IR [6133].

Acetate $C_{10}H_9N_3O_3$ mol.wt. 219.20

- Preparation by adding dropwise triethylamine and acetyl chloride to a solution of o-hydroxy-phenacyl azide in THF at 0–5°, then at r.t. for 1 h (95%) [6143].

m.p. 54–55° [6143]; ^1H NMR [6143].

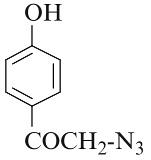
Benzoate $C_{15}H_{11}N_3O_3$ mol.wt. 281.27 [6143]

- Preparation by adding dropwise triethylamine and benzoyl chloride to a solution of o-hydroxy-phenacyl azide in THF at 0–5°, then at r.t. for 1.5 h (90%) [6143].

m.p. 59–60° [6143]; ^1H NMR [6143].

2-Azido-1-(4-hydroxyphenyl)ethanone

[169315-44-0] $C_8H_7N_3O_2$ mol.wt. 177.16



New compound

Syntheses

- Preparation by reaction of sodium azide with α-bromo-4-hydroxyacetophenone [6144], (80%) [6141].
- Also obtained by reaction of sodium azide with α-chloro-4-hydroxyacetophenone in alcohol [6145].
- Also refer to: [6146,6147].

m.p. 139–140° [6141], 136° [6145]; ^1H NMR [6146], ^{13}C NMR [6146].

Methyl ether [6595-28-4] $C_9H_9N_3O_2$ mol.wt. 191.19

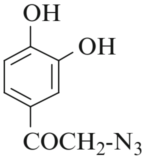
- Obtained by reaction of sodium azide with 4-methoxyphenacyl bromide in DMSO at r.t. for at least 30 min (93%) [6148].

- Also refer to: [6149,6150].

Light yellow powder [6148]; ^{1}H NMR [6148].

2-Azido-1-(3,4-dihydroxyphenyl)ethanone

[165947-83-1] $C_8H_7N_3O_3$ mol.wt. 193.16



New compound

Syntheses

- Preparation by reaction of sodium azide with 3,4-dihydroxy-phenacyl chloride in water (80%) [5996] or in DMF (90%) [6080].

m.p. 132° (d) [5996].

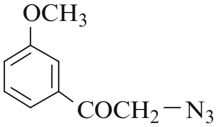
Dimethyl ether [187101-52-6] $C_{10}H_{11}N_3O_3$ mol.wt. 221.22

- Obtained by reaction of sodium azide with 3,4-dimethoxyphenacyl bromide in DMSO at r.t. for at least 30 min (87%) [6148].

Yellow powder [6148]; ^{1}H NMR [6148].

2-Azido-1-(3-methoxyphenyl)ethanone

[194787-89-8] $C_9H_9N_3O_2$ mol.wt. 191.19



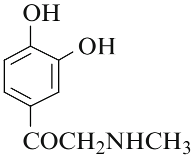
New compound

Syntheses

- Refer to: [6027,6151,6152] (Chinese patent).

1-(3,4-Dihydroxyphenyl)-2-(methylamino)ethanone

[99-45-6] $C_9H_{11}NO_3$ mol.wt. 181.19



Described [5958] **p. 1300**

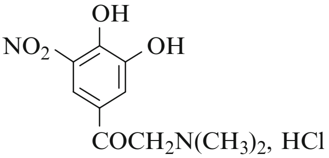
Syntheses

- Also refer to: [6153,6154,6155,6156].

Diacetate *(Hydrochloride)* $C_{13}H_{15}NO_5$, HCl mol.wt. 301.73 [6157]

1-(3,4-Dihydroxy-5-nitrophenyl)-2-(dimethylamino)ethanone *(Hydrochloride)*

$C_{10}H_{12}NO_5$, HCl mol.wt. 262.67



New compound

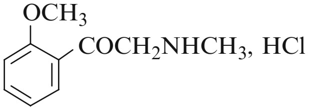
Synthesis

- Obtained by adding a solution of 3,4-dihydroxy-5-nitro-α-bromoacetophenone in DMF to a solution of 40% aqueous dimethylamine for 1 h. at r.t. Then, treatment with concentrated hydrochloric acid (pH 1) (96%) [5973].

Monohydrate; yellow solid [5973]; ^{1}H NMR [5973], ^{13}C NMR [5973], IR [5973].

1-(2-Methoxyphenyl)-2-(methylamino)ethanone *(Hydrochloride)*

[958292-64-3] $C_{10}H_{13}NO_2$, HCl mol.wt. 215.68



New compound

Synthesis

– A solution of α-bromo-2-methoxyacetophenone in acetonitrile was added to a solution of dimethylamine (8 M solution in ethanol) in acetonitrile at 0°. The solution was stirred at 0° for 5 min. Ether was added and the resulting precipitate after drying, was dissolved in ether and 1 M hydrogen chloride in ether was added at 0° [6027].

1-(3-Methoxyphenyl)-2-(methylamino)ethanone *(Hydrochloride)*

[958292-65-4] $C_{10}H_{13}NO_2$, HCl mol.wt. 215.68

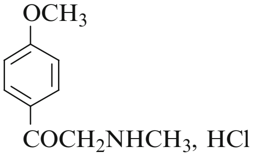
New compound

Synthesis

– A solution of α-bromo-3-methoxyacetophenone in acetonitrile was added to a solution of dimethylamine (8 M solution in ethanol) in acetonitrile at 0°. The solution was stirred at 0° for 5 min. Ether was added and the resulting precipitate after drying, was dissolved in ether and 1 M hydrogen chloride in ether was added at 0° [6027].

1-(4-Methoxyphenyl)-2-(methylamino)ethanone *(Hydrochloride)*

[29705-80-4] $C_{10}H_{13}NO_2$, HCl mol.wt. 215.68



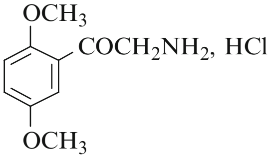
New compound

Synthesis

– A solution of α-bromo-4-methoxyacetophenone in acetonitrile was added to a solution of dimethylamine (8 M solution in ethanol) in acetonitrile at 0°. The solution was stirred at 0° for 5 min. Ether was added and the resulting precipitate after drying, was dissolved in ether and 1 M hydrogen chloride in ether was added at 0° [6027].

2-Amino-1-(2,5-dimethoxyphenyl)ethanone *(Hydrochloride)*

[671224-08-1] $C_{10}H_{13}NO_3$, HCl mol.wt. 231.69



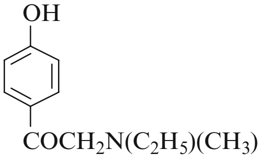
New compound

Synthesis

– Refer to: [6027].

2-(Ethylmethylamino)-1-(4-hydroxyphenyl)ethanone

[1009636-07-0] $C_{11}H_{15}NO_2$ mol.wt. 193.25



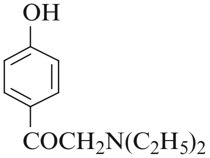
New compound

Synthesis

- Refer to: [6158] (Chinese patent).

2-(Diethylamino)-1-(4-hydroxyphenyl)ethanone

[87154-81-2] $C_{12}H_{17}NO_2$ mol.wt. 207.27



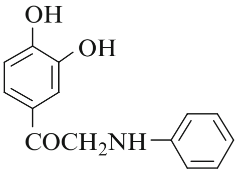
New compound

Synthesis

- Refer to: [6158] (Chinese patent).

1-(3,4-Dihydroxyphenyl)-2-(phenylamino)ethanone

$C_{14}H_{13}NO_3$ mol.wt. 243.26



New compound

Syntheses

- Obtained by reaction of 3,4-dihydroxy-α-chloroacetophenone in ethanol with aniline [6159,6160].
- Also refer to: [6161,6162].

m.p. 160° [6159], 149° [6160,6161].

Sulfate $C_{14}H_{13}NO_3, H_2SO_4$ mol.wt. 584.60 [6162].

m.p. 208° [6162].

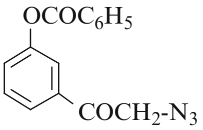
Dimethyl ether $C_{16}H_{17}NO_3$ mol.wt. 271.31

- This compound was synthesized [6163] from 2-bromo-3′, 4′-dimethoxyacetophenone by using a known procedure [6164].

m.p. 121° [6163]; ^{1}H NMR [6163], ^{13}C NMR [6163], MS [6163].

2-Azido-1-[3-(benzoyloxy)phenyl]ethanone

$C_{15}H_{11}N_3O_3$ mol.wt. 281.27



New compound

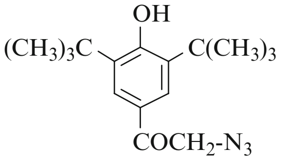
Synthesis

- Obtained by reaction of sodium azide with 2-bromo-1-[(3-benzoyloxy)phenyl]ethanone in dilute ethanol (95%) [5996].

m.p. 115° [5996].

2-Azido-1-[3,5-bis(1,1-dimethylethyl)-4-hydroxyphenyl]ethanone

[870789-66-5] $C_{16}H_{23}N_3O_2$ mol.wt. 289.38



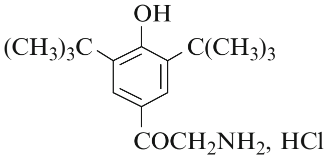
New compound

Syntheses

- Preparation by reaction of sodium azide with 2-bromo-1-(3,5-di-tert-butyl-4-hydroxyphenyl) ethanone in dilute acetone at r.t. for 1 h [6066].
- Also refer to: [6165].

2-Amino-1-[3,5-(1,1-dimethylethyl)-4-hydroxyphenyl]ethanone *(Hydrochloride)*

[84203-40-7] $C_{16}H_{25}NO_2$, HCl mol.wt. 299.84



New compound

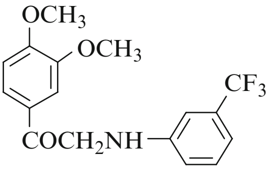
Synthesis

- Obtained by hydrogenation of 2-azido-1-[3,5-bis-(1,1-dimethylethyl)-4-hydroxyphenyl] ethanone in the presence of Pd/C in methanol, then treatment of the amine so obtained with hydrochloric acid (67%) [6066].

1-(3,4-Dimethoxyphenyl)-2-(3-trifluoromethylphenylamino)ethanone

α-(m-Trifluoromethylphenylamino)acetoveratrone

$C_{17}H_{16}F_3NO_3$ mol.wt. 339.31



New compound

Synthesis

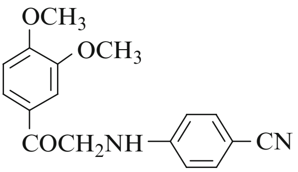
- This compound was synthesized [6163] from 2-bromo-3′,4′-dimethoxyacetophenone by using a known procedure [6164].

m.p. 127° [6163]; ^{1}H NMR [6163], ^{13}C NMR [6163], MS [6163].

2-(4-Cyanophenylamino)-1-(3,4-dimethoxyphenyl)ethanone

α-(p-Cyanophenylamino)acetoveratrone

$C_{17}H_{16}N_2O_3$ mol.wt. 296.33



New compound

Synthesis

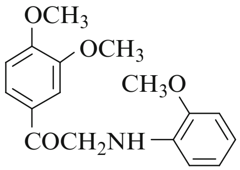
- This compound was synthesized [6163] from 2-bromo-3′,4′-dimethoxyacetophenone by using a known procedure [6164].

m.p. 200–201° [6163]; ^{1}H NMR [6163], ^{13}C NMR [6163], MS [6163].

1-(3,4-Dimethoxyphenyl)-2-[(2-methoxyphenyl)amino]ethanone

α-(o-Methoxyphenylamino)acetoveratrone

[252655-15-5] $C_{17}H_{19}NO_4$ mol.wt. 301.34



New compound

Synthesis

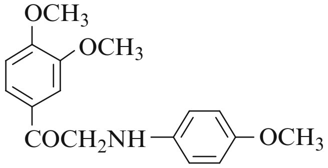
- This compound was synthesized [6163] from 2-bromo-3′,4′-dimethoxyacetophenone by using a known procedure [6164].

m.p. 117–118° [6163]; ^{1}H NMR [6163], ^{13}C NMR [6163], MS [6163].

1-(3,4-Dimethoxyphenyl)-2-[(4-methoxyphenyl)amino]ethanone

α-(p-Methoxyphenylamino)acetoveratrone

[252655-16-6] $C_{17}H_{19}NO_4$ mol.wt. 301.34



New compound

Synthesis

- This compound was synthesized [6163] from2-bromo-3′,4′-dimethoxyacetophenone by using a known procedure [6164].

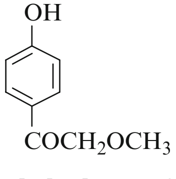
m.p. 117° [6163]; ^{1}H NMR [6163], ^{13}C NMR [6163], MS [6163].

Chapter 13. Compounds Derived from Alkoxyacetic Acids [5958] p. 1321

13.1 Compounds Derived from Methoxyacetic Acids [5958] p. 1321

1-(4-Hydroxyphenyl)-2-methoxyethanone

[32136-81-5] $C_9H_{10}O_3$ mol.wt. 166.18



Described [5958] **p. 1322**

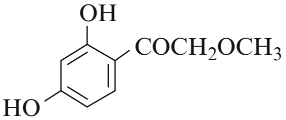
Methyl ether [21160-26-9] $C_{10}H_{12}O_3$ mol.wt. 180.20

- Obtained by adding with cooling an ethereal solution of methoxyacetonitrile to a solution of magnesium anisyl bromide in ethyl ether [6166], (30%) [6167].
- Also refer to: [6168].

b.p.$_{35}$ 185–190° [6167].

1-(2,4-Dihydroxyphenyl)-2-methoxyethanone

[57280-75-8] $C_9H_{10}O_4$ mol.wt. 182.18



Described [5958] **p. 1322**

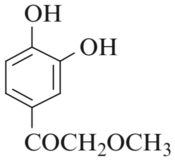
Synthesis

- Also obtained by reaction of methoxyacetonitrile with resorcinol and subsequent hydrolysis of the ketimine hydrochloride so formed (Hoesch reaction) (48%) [6169].

White solid [6169]; m.p. 108–110° [6169];
^{1}H NMR [6169], ^{13}C NMR [6169], IR [6169], MS [6169].

1-(3,4-Dihydroxyphenyl)-2-methoxyethanone

[64349-40-2] $C_9H_{10}O_4$ mol.wt. 182.18



Described [5958] **p. 1323**

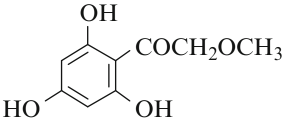
Dimethyl ether [22341-22-6] $C_{11}H_{14}O_4$ mol.wt. 210.23
α-methoxyacetoveratrone

- This compound was synthesized by [6163] according to: [6170].
- Also refer to: [6166,6167,6171].

b.p.$_{15}$ 190° [6166]; m.p. 66–67° [6163], 64–65° [6171], 62° [6166];
^{1}H NMR [6163], ^{13}C NMR [6163], IR [6171], MS [6163].

2-Methoxy-1-(2,4,6-trihydroxyphenyl)ethanone

[55317-02-7] $C_9H_{10}O_5$ mol.wt. 198.18



Described [5958] **p. 1323**

Syntheses

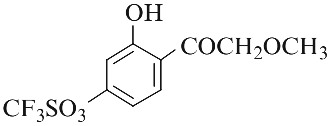
- Preparation by reaction of methoxyacetonitrile with phloroglucinol (Houben-Hoesch reaction) (76%) [6172].

- Also refer to: [6173].

1-[2-Hydroxy-4-[(trifluoromethanesulfonyl)oxy]phenyl]-2-methoxyethanone

(Trifluoromethane sulfonic acid, 3-hydroxy-4-(methoxyacetyl)phenyl ester) (Chem. Abstr., Formula Index Vol. **141**, 2004, 1496F)

[649551-91-7]



$C_{10}H_9F_3O_6S$ mol.wt. 314.24

New compound

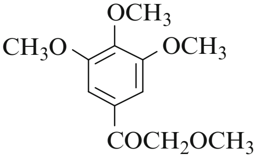
Synthesis

- Obtained by reaction of trifluoromethanesulfonic anhydride (triflic anhydride) with2′,4′-dihydroxy-2-methoxy-acetophenone in the presence of 2,6-lutidine in methylene chloride at 0° for 2 h under argon atmosphere (87%) [6169].

N.B.: The product was contaminated with 5% of its ditriflate.

Purple oil [6169]; ^{1}H NMR [6169].

2-Methoxy-1-(3,4,5-trimethoxyphenyl)ethanone



$C_{12}H_{16}O_5$ mol.wt. 240.26

New compound

Synthesis

- Obtained by adding an etheral solution of 3,4,5-trimethoxy-benzoyl chloride to a solution of sodio-derivative of ethyl α,γ-dimethoxyacetoacetate in toluene on a steam bath for 8 h (36%) [6166].

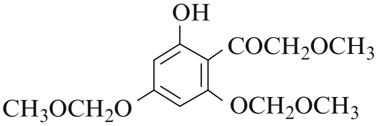
b.p.$_{15}$ 212° [6166]; m.p. 54° [6166].

Semicarbazone $C_{13}H_{19}N_3O_5$ mol.wt. 255.23

m.p. 158° [6166].

1-[2-Hydroxy-4,6-bis(methoxymethoxy)phenyl]-2-methoxyethanone

[943827-52-9]



$C_{13}H_{18}O_7$ mol.wt. 198.18

New compound

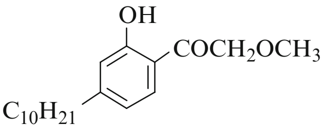
Synthesis

- Preparation by reaction of chloromethyl methyl ether with 2-methoxy-1-(2,4,6-trihydroxyphenyl)ethanone in the presence of potassium carbonate in acetone at 23° (68%) [6172].

^{1}H NMR [6172], ^{13}C NMR [6172].

1-(4-Decyl-2-hydroxyphenyl)-2-methoxyethanone

2′-Hydroxy-4′-decyl-2-methoxyacetophenone



$C_{19}H_{30}O_3$ mol.wt. 306.44

New compound

Synthesis

- Refer to: [6169] (complex procedure) (76%).

Pale yellow solid; m.p. <25° [6169];
^{1}H NMR [6169], ^{13}C NMR [6169], MS [6169].

2,4,5-Trimethoxybenzoate $C_{29}H_{40}O_7$ mol.wt. 500.28

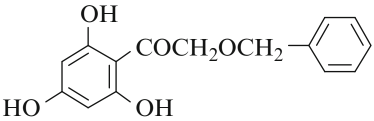
- Preparation: Refer to [6169] (68%).

Yellow solid; m.p. 80–81° [6169];
^{1}H NMR [6169], ^{13}C NMR [6169], IR [6169], MS [6169]; TLC [6169].

13.2 Compounds Derived from Phenylmethoxyacetic Acids [5958] p. 1345

2-(Phenylmethoxy)-1-(2,4,6-trihydroxyphenyl)ethanone

[322405-72-1]



$C_{15}H_{14}O_5$ mol.wt. 274.27

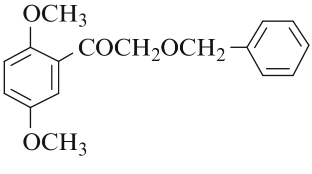
Described [5958] **p. 1345**

Synthesis

- Also refer to: [6174].

1-(2,5-Dimethoxyphenyl)-2-(phenylmethoxy)ethanone

[736933-09-8]



$C_{17}H_{18}O_4$ mol.wt. 286.33

New compound

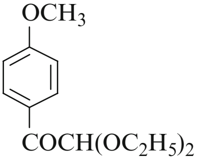
Synthesis

- Refer to: [6175].

13.3 Compounds Derived from Ethoxyacetic Acids [5958] p. 1346

2,2-Diethoxy-1-(4-methoxyphenyl)ethanone

[66186-69-4] $C_{13}H_{18}O_4$ mol.wt. 238.28



New compound

Synthesis

- Refer to: [6176].

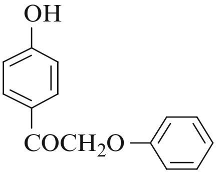
13.4 Miscellaneous [5958] p. 1351

Chapter 14. Compounds Derived from Aryloxyacetic Acids [5958] p. 1353

14.1 Compounds Derived from Phenoxyacetic Acid [5958] p. 1353

1-(4-Hydroxyphenyl)-2-phenoxyethanone

[41978-29-4] $C_{14}H_{12}O_3$ mol.wt. 228.25

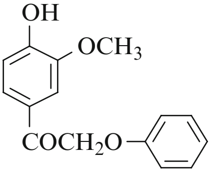


Described [5958] **p. 1353**

Methyl ether [19513-78-1] $C_{15}H_{14}O_3$ mol.wt. 242.27 Refer to: [6177].

1-(4-Hydroxy-3-methoxyphenyl)-2-phenoxyethanone

[41978-28-3] $C_{15}H_{14}O_4$ mol.wt. 258.27



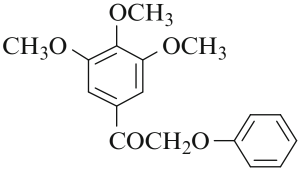
Described [5958] **p. 1354**

Methyl ether [140455-40-9] $C_{16}H_{16}O_4$ mol.wt. 272.30

- Preparation by reaction of 3′,4′-dimethoxy-α-bromoacetophenone with phenol in the presence of potassium carbonate in acetone [6163,6178,6179,6180].
- Also refer to: [6177,6181].

m.p. 95–96° [6179], 91–92° [6180];
^{1}H NMR [6177,6179,6180,6181], ^{13}C NMR [6179], MS [6177,6179].

2-Phenoxy-1-(3,4,5-trimethoxyphenyl)ethanone



$C_{17}H_{18}O_5$ mol.wt. 303.33

New compound

Synthesis

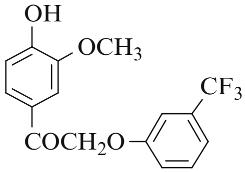
- Refer to: [6177].

14.2 Compounds Derived from Substituted Phenoxyacetic Acids [5958] p. 1355

1-(4-Hydroxy-3-methoxyphenyl)-2-[3-(trifluoromethyl)phenoxy]ethanone

[107584-69-0] $C_{16}H_{13}F_3O_4$ mol.wt. 326.27

Described [5958] **p. 1362**



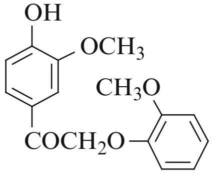
Methyl ether $C_{17}H_{15}F_3O_4$ mol.wt. 340.30

- Preparation by [6163] according to: [6178,6179].

m.p. 144–145° [6163]; ^{1}H NMR [6163], ^{13}C NMR [6163], MS [6163].

1-(4-Hydroxy-3-methoxyphenyl)-2-(2-methoxyphenoxy)ethanone
α-(2-Methoxyphenoxy)acetoveratrone
[22317-35-7] $C_{16}H_{16}O_5$ mol.wt. 288.30

Described [5958] **p. 1363**



Methyl ether [22675-96-3] $C_{17}H_{18}O_5$ mol.wt. 302.33

- Preparation by reaction of 3′,4′-dimethoxy-α-bromoacetophenone with guaiacol in the presence of potassium carbonate in acetone [6163,6178,6179], (94%) [6182].

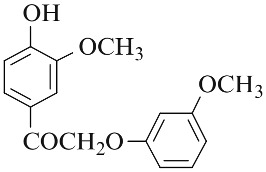
- Preparation [6183], according to the procedure [6178].
- Also refer to: [6180,6184,6185,6186,6187,6188,6189,6190].

m.p. 94–95.4° [6191], 94° [6180], 93–94° [6182], 92–93° [6183], 90–92° [6178];
^{1}H NMR [6180,6182,6191], UV [6190]; circular dichroism [6188].

1-(4-Hydroxy-3-methoxyphenyl)-2-(3-methoxyphenoxy)ethanone

[107584-68-9] $C_{16}H_{16}O_5$ mol.wt. 288.30

Described [5958] **p. 1364**



Methyl ether [72327-16-3] $C_{17}H_{18}O_5$ mol.wt. 302.33

- Preparation by reaction of 3′,4′-dimethoxy-α-bromoacetophenone with 3-methoxyphenol in the presence of potassium carbonate in boiling acetone [6163,6178,6179] for 6 h [6192].

m.p. 90° [6179,6192];
^{1}H NMR [6179], ^{13}C NMR [6179], IR [6192], MS [6179].

2,4-Dinitrophenylhydrazone (of the methyl ether) $C_{23}H_{22}N_4O_8$ mol.wt. 482.30

m.p. 177° [6192].

3-[2-(3,4-Dimethoxy)-2-oxoethoxy]benzonitrile

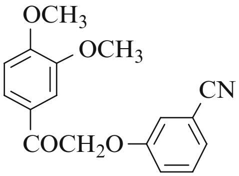
α-(m-Cyanophenoxy)acetoveratrone

$C_{17}H_{15}NO_4$ mol.wt. 297.31

New compound

Synthesis

- Preparation by [6163] according to: [6178,6179].

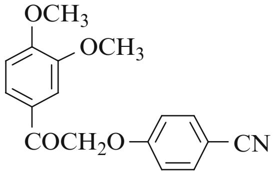


m.p. 144–145° [6163].
^{1}H NMR [6163], ^{13}C NMR [6163], MS [6163].

4-[2-(3,4-Dimethoxy)-2-oxoethoxy]benzonitrile

α-(p-Cyanophenoxy)acetoveratrone

[151425-43-3] $C_{17}H_{15}NO_4$ mol.wt. 297.31



New compound

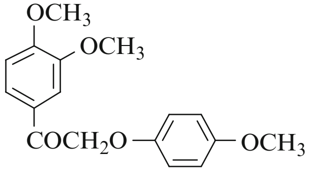
Syntheses

- Preparation by reaction of α-bromoacetoveratrone with 4-hydroxybenzonitrile in the presence of potassium carbonate in refluxing acetone [6163,6178,6179].

m.p. 131–132° [6179]; ^{1}H NMR [6179], ^{13}C NMR [6179], MS [6179].

1-(3,4-Dimethoxyphenyl)-2-(4-methoxyphenoxy)ethanone

[129229-41-0] $C_{17}H_{18}O_5$ mol.wt. 302.33



New compound

Syntheses

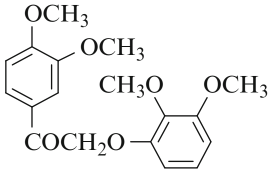
- Preparation by reaction of 3′,4′-dimethoxy-α-bromo-acetophenone with 4-m.ethoxyphenol in the presence of potassium carbonate in acetone [6163,6178,6179].

m.p. 104° [6179]; ^{1}H NMR [6179], ^{13}C NMR [6179], MS [6179].

2-(2,3-Dimethoxyphenoxy)-1-(3,4-dimethoxyphenyl)ethanone

ω-(2′,3′-Dimethoxyphenoxy)acetoveratrone

[72327-23-2] $C_{18}H_{20}O_6$ mol.wt. 332.35



New compound

Synthesis

- Obtained by reaction of ω-bromoacetoveratrone with 2,3-dimethoxyphenol in the presence of potassium carbonate in refluxing acetone for 8 h (70%) [6192].

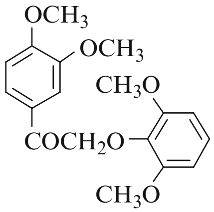
m.p. 94–95° [6192]; IR [6192].

2,4-Dinitrophenylhydrazone $C_{24}H_{24}N_4O_9$ mol.wt. 512.48

m.p. 198–199° [6192].

2-(2,6-Dimethoxyphenoxy)-1-(3,4-dimethoxyphenyl)ethanone
α-(2,6-Dimethoxyphenoxy)acetoveratrone

[29389-04-6] $C_{18}H_{20}O_6$ mol.wt. 332.35



New compound

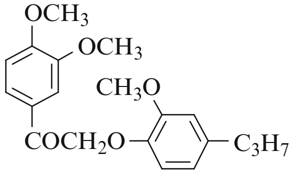
Syntheses

- Preparation by reaction of α-bromoacetoveratrone with 2,6-dimethoxyphenol in the presence of potassium carbonate in refluxing acetone [6179,6180].
- Also obtained by reaction of 3′,4′-dimethoxy-α-bromo-acetophenone with the sodium salt of 2,6-dimethoxyphenol in DMF at r.t. for 1 h (93%) [6193].

m.p. 103–104° [6179], 100–103° [6193], 97° [6180];
^{1}H NMR [6179,6180,6193], ^{13}C NMR [6179], MS [6179].

1-(3,4-Dimethoxyphenyl)-2-(2-methoxy-4-propylphenoxy)ethanone
ω-(2-Methoxy-4-propylphenoxy)acetoveratrone

$C_{20}H_{24}O_5$ mol.wt. 344.41



New compound

Synthesis

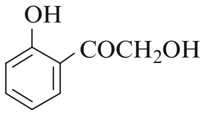
- Refer to: [6182].

^{1}H NMR [6182].

Chapter 15. Compounds Derived from Hydroxyacetic Acids [5958] p. 1369

2-Hydroxy-1-(2-hydroxyphenyl)ethanone

[17375-96-1] $C_8H_8O_3$ mol.wt. 152.15



Described p. [5958] **1370**

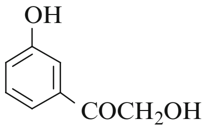
Syntheses

- Also refer to: [6194,6195,6196].

o-Methyl ether [224321-19-1] $C_9H_{10}O_3$ mol.wt. 166.18 Refer to: [6100,6197]

2-Hydroxy-1-(3-hydroxyphenyl)ethanone

[131341-58-7] $C_8H_8O_3$ mol.wt. 152.15



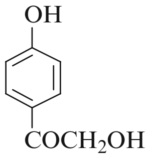
Described [5958] **p. 1370**

m-Methyl ether [87428-52-2] $C_9H_{10}O_3$ mol.wt. 166.18

- Refer to: [6198].

2-Hydroxy-1-(4-hydroxyphenyl)ethanone

[5706-85-4] $C_8H_8O_3$ mol.wt. 152.15



Described [5958] **p. 1370**

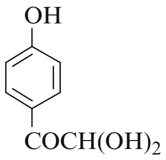
p-Methyl ether [4136-21-4] $C_9H_{10}O_3$ mol.wt. 166.18

- Obtained by photocatalytic oxidation of 2-(4-methoxyphenyl)-1,2-ethanediol using silica-encapsulated $H_3PW_{12}O_{40}$ as photocatalyst in acetonitrile for 1 h at r.t. under oxygen as the sole reoxidant of the catalyst, (90%) [6199].
- Also obtained by treatment of 4-methoxyacetophenone with iodosobenzene in methanolic sodium hydroxide (50%) [6200].
- Also refer to: [6201].

^1H NMR [6199], IR [6199], MS [6199]; TLC [6199].

2,2-Dihydroxy-1-(4-hydroxyphenyl)ethanone

[197447-05-5] $C_8H_8O_4$ mol.wt. 168.15



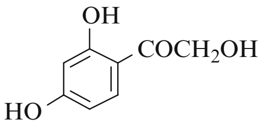
Described [5958] **p. 1372**

Synthesis

- Also obtained by adding a saturated aqueous solution of sodium carbonate to a solution of ω-dichloro p-hydroxyacetophenone in ethanol and heating to 40–120° [6097].

1-(2,4-Dihydroxyphenyl)-2-hydroxyethanone *(Fisetol)*

[487-47-8] $C_8H_8O_4$ mol.wt. 168.15



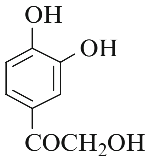
Described [5958] **p. 1372**

Synthesis

- Also refer to: [6202].

1-(3,4-Dihydroxyphenyl)-2-hydroxyethanone (DOPKET)

[29477-54-1] $C_8H_8O_4$ mol.wt. 168.15



Described [5958] **p. 1373**

Syntheses

- Also refer to: [6203,6204,6205].

Isolation from natural sources

- From the aerial parts of *Jasminum grandiflorum* collected in Jessore (Bangladesh) [6206].

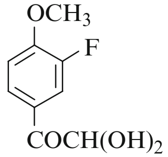
^{1}H NMR [6207], ^{13}C NMR [6207], IR [6207], UV [6207].

BIOLOGICAL ACTIVITY: In vitro antioxidative activity with the linoleic acid system [6207].

Dimethyl ether $C_{10}H_{12}O_4$ mol.wt. 196.20

- Obtained by treatment of 3,4-dimethoxyacetophenone with iodosobenzene in methanolic sodium hydroxide (40%) [6200].

1-(3-Fluoro-4-methoxyphenyl)-2,2-dihydroxyethanone



$C_9H_9FO_4$ mol.wt. 200.17

New compound

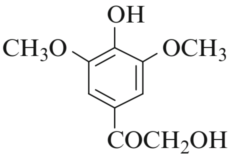
Synthesis

- Obtained by hydrolysis of (3-fluoro-4-methoxyphenyl)-oxo-acetaldehyde [6208].

m.p. 95° [6208]; ^{1}H NMR [6208], IR [6208].

2-Hydroxy-1-(4-hydroxy-3,5-dimethoxyphenyl)ethanone

[90426-22-5]



$C_{10}H_{12}O_5$ mol.wt. 212.20

Described [5958] **p. 1377**

Synthesis

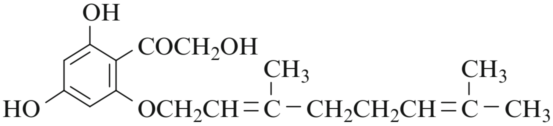
- Also refer to: [6209].

1-[2-[[(2*E*)-3,7-Dimethyl-2,6-octadien-1-yl]oxy]-4,6-dihydroxy]-2-hydroxyethanone

[149492-42-2]

$C_{18}H_{24}O_5$ mol.wt. 320.38

Described [5958] **p. 1380**



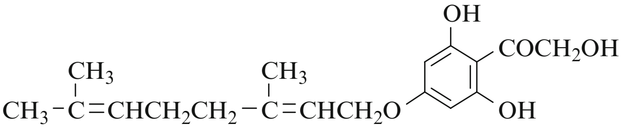
Isolation from natural sources

- From the fruits of *Melicope*
- *semecarpifolia* (Merr.) T. G. Hartley (Rutaceae) compound **8** [6210].

BIOLOGICAL ACTIVITY: Antiinflammatory [6210].

1-[4-[[(2*E*)-3,7-Dimethyl-2,6-octadien-1-yl]oxy]-2,6-dihydroxy]-2-hydroxyethanone

[142905-41-7] $C_{18}H_{24}O_5$ mol.wt. 320.38



Described [5958] **p. 1380**

Isolation from natural sources

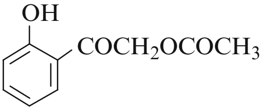
- From the fruits of *Melicope semecarpifolia* (Merr.) T. G. Hartley (Rutaceae) compound **9** [6210].BIOLOGICAL ACTIVITY: Antiinflammatory [6210].

Chapter 16. Compounds Derived from Acyloxy- and Aroyloxyacetic Acids [5958] p. 1383

16.1 Compounds Derived from Acetoxyacetic Acids [5958] p. 1383

2-(Acetyloxy)-1-(2-hydroxyphenyl)ethanone

[40231-09-2] $C_{10}H_{10}O_4$ mol.wt. 194.19



Described [5958] **p. 1383**

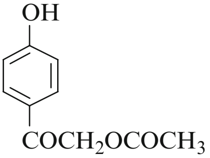
Methyl ether [74786-55-3] $C_{11}H_{12}O_4$ mol.wt. 208.21

- Obtained from 2-methoxyacetophenone and [hydroxy(tosyloxy)iodo]benzene (HTIB)/polymer-supported [hydroxy(tosyloxy)iodo] benzene (PSHTIB) in N,N-dimethylformamide/dimethyl-acetamide (72%) [6211].

m.p. 55° [6211]; ^{1}H NMR [6211], ^{13}C NMR [6211].

2-(Acetyloxy)-1-(4-hydroxyphenyl)ethanone

[20816-46-0] $C_{10}H_{10}O_4$ mol.wt. 194.19



Described [5958] **p. 1383**

Syntheses

- Also obtained by adding, step by step, triethylamine in a solution of ω-chloro-4-hydroxy acetophenone in acetic acid/acetonitrile at 0°, then refluxing for 3 h (40%) [5961].
- Also refer to: [6055,6212].

^{1}H NMR [5961], MS [5961].

Methyl ether [58518-78-8] $C_{11}H_{12}O_4$ mol.wt. 208.21

- Obtained by reaction of sodium acetate with 2-bromo-4′-methoxyacetophenone in boiling methanol for 48 h (43%) [6213].
- Also obtained by reaction of potassium thioacetate with 4-methoxy-α-chloroacetophenone [6214].
- Also obtained from 4-methoxyacetophenone and [hydroxy(tosyloxy)iodo]benzene (HTIB)/polymer-supported [hydroxy(tosyloxy)iodo]benzene (PSHTIB) in N,N-dimethyl-formamide/dimethylacetamide (74%) [6211].
- Also refer to: [5962].

b.p.$_{2.5}$ 140–141.5° [6215];
m.p. 69° [6211], 59° [6214], 56° [6213], 55–56° [6216].
One of the reported melting points is obviously wrong.
^{1}H NMR [6211,6213,6216], ^{13}C NMR [6211,6216], IR [6213,6216], MS [6216].

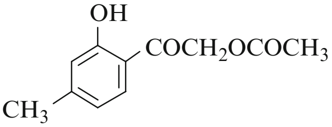
Acetate $C_{12}H_{12}O_5$ mol.wt. 236.22

- Obtained by reaction of acetic anhydride with ω-acetoxy-4-hydroxyacetophenone at reflux (100°) for 2 h (75%) [5961].

^{1}H NMR [5961], MS [5961].

2-(Acetyloxy)-1-(2-hydroxy-4-methylphenyl)ethanone

[860806-61-7] $C_{11}H_{12}O_4$ mol.wt. 208.21



New compound

Synthesis

- Obtained by hydrogenation of α-acetoxy-2-benzyloxy-4-methylacetophenone in ethyl acetate with hydrogen (60 bar) in the presence of 10% Pd/C at 60° for 5 h (57%) [6052].

Colourless crystalline needles; m.p. 87° [6052];
^{1}H NMR [6052], ^{13}C NMR [6052], IR [6052], UV [6052], MS [6052].

BIOLOGICAL DATA: Phytotoxic activity [6052]; phytogrow-inhibitory activity on seeds of *Amaranthus hypochondriacus*, *Echinochloa crus-galli* and *Medicago sativa* [6052].
Isolation from natural sources

- From *Hofmeisteria schaffneri* (A. Gray) R.M. King and H. Robinson (Asteraceae) [6052].

Methyl ether [860782-84-9] $C_{12}H_{14}O_4$ mol.wt. 222.24

- Obtained by adding successively DBU, then after 10 min, α-bromo-2-methoxy-4-methyl-acetophenone in THF to an acetic acid/THF mixture and stirring at r.t. for 2.5 h (76%) [6052].

Colourless oil [6052]; ^{1}H NMR [6052], ^{13}C NMR [6052], IR [6052], MS [6052].

BIOLOGICAL DATA: Phytotoxic activity [6052]; phytogrowth-inhibitory activity on seeds of *Amaranthus hypochondriacus*, *Echinochloa crus-galli* and *Medicago sativa* [6052].

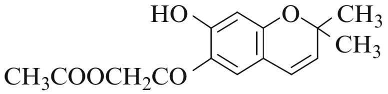
Benzyl ether [860782-83-8] $C_{18}H_{18}O_4$ mol.wt. 298.34

- Obtained by adding α-bromo-2-benzyloxy-4-methylacetophenone in THF to a mixture of DBU in ethyl acetate/THF at r.t. for 10 min, then stirring for 2.5 h (82%) [6052].

White solid; m.p. 73° [6052];
^{1}H NMR [6052], ^{13}C NMR [6052], IR [6052], UV [6052], MS [6052].

BIOLOGICAL DATA: Phytotoxic activity [6052]; phytogrowth-inhibitory activity on seeds of *Amaranthus hypochondriacus*, *Echinochloa crus-galli* and *Medicago sativa* [6052].

2-Acetoxy-1-(7-hydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)ethanone
(Ripariochromene B)
[27045-16-5]



$C_{15}H_{16}O_5$ mol.wt. 276.29

New compound

Synthesis

- Refer to: [6217].

Isolation from natural sources

- From the roots of *Eupatorium riparium* Regel [6218].
- From the aerial parts of *Ageratina riparia* [6219].

m.p. 146–147° [6217,6220];
^{1}H NMR [6217,6220], IR [6217,6220], UV [6217,6220].

BIOLOGICAL DATA: Antifungic activity [6218].

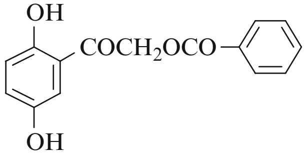
16.2 Compounds Derived from Other Acyloxy- and Phenacyloxyacetic Acids [5958] p. 1386

16.3 Compounds Derived from Benzoyloxyacetic Acids [5958] p. 1389

2-(Benzoyloxy)-1-(2,5-dihydroxyphenyl)ethanone

[117421-24-6] $C_{15}H_{12}O_5$ mol.wt. 272.26

Described [5958] **p. 1389**



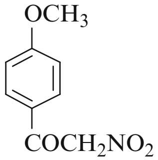
Dimethyl ether [478972-03-1] $C_{17}H_{16}O_5$ mol.wt. 300.31

– Obtained from 2-bromo-1-(2,5-dimethoxyphenyl)ethanone (Chinese paper) [6022].

Chapter 17. Compounds Derived from Nitroacetic Acids [5958] p. 1395

1-(4-Methoxyphenyl)-2-nitroethanone

[46318-58-5] $C_9H_9NO_4$ mol.wt. 195.17



New compound

Synthesis

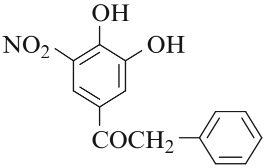
– Refer to: [6221].

Chapter 18. Compounds Derived from Arylacetic Acids [5958] p. 1399

18.1 Compounds Derived from Phenylacetic Acid [5958] p. 1399

1-(3,4-Dihydroxy-5-nitrophenyl)-2-phenylethanone *(BIA-3-202)*

[274925-86-9] $C_{14}H_{11}NO_5$ mol.wt. 273.25



Described [5958] **p. 1403**

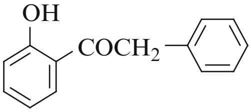
Syntheses

– Also refer to: [5973,6222,6223,6224].

BIOLOGICAL ACTIVITY: [5973].

1-(2-Hydroxyphenyl)-2-phenylethanone

[2491-31-8] $C_{14}H_{12}O_2$ mol.wt. 212.25



Described [5958] **p. 1404**

Acetate $C_{16}H_{14}O_3$ mol.wt. 254.29

– Obtained by reaction of acetic anhydride with 2-hydroxybenzoin [6225].

Methyl ether [33470-10-9] $C_{15}H_{14}O_2$ mol.wt. 226.27

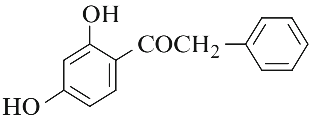
2-Methoxydeoxybenzoin

- Also obtained by deamination of 2-amino-1-(2-methoxyphenyl)-1-phenylethanol with sodium nitrite in 50% aqueous acetic acid at 0° [6133].
- Also obtained by reaction of 2-methoxybenzamide with benzylmagnesium bromide [6133].

^{1}H NMR [6133], IR [6133], UV [6133].

1-(2,4-Dihydroxyphenyl)-2-phenylethanone

[3669-41-8] $C_{14}H_{12}O_3$ mol.wt. 228.25



Described [5958] **p. 1407**

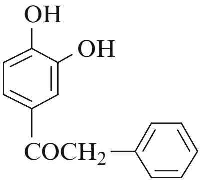
Synthesis

- Also refer to: [6226].

1-(3,4-Dihydroxyphenyl)-2-phenylethanone

[107410-02-6] $C_{14}H_{12}O_3$ mol.wt. 228.25

Described [5958] **p. 1409**

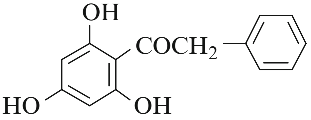


Dimethyl ether [3141-93-3] $C_{16}H_{16}O_3$ mol.wt. 256.30

- Refer to: [6227] (Japanese patent).

2-Phenyl-1-(2,4,6-trihydroxyphenyl)ethanone

[727-71-9] $C_{14}H_{12}O_4$ mol.wt. 244.25



Described [5958] **p. 1411**

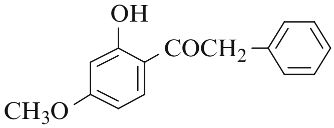
Syntheses

- Preparation by reaction of phenylacetonitrile with phloroglucinol (Hoesch reaction) in the presence of zinc chloride [6084].
- Also refer to: [6225,6228], (20%) [6229].

White crystals; m.p. 162° [6229];
^{1}H NMR [6229], ^{13}C NMR [6229], IR [6229], UV [6229], MS [6229];
TLC [6229].

1-(2-Hydroxy-4-methoxyphenyl)-2-phenylethanone

[18439-96-8] $C_{15}H_{14}O_3$ mol.wt. 242.27



Described [5958] **p. 1419**

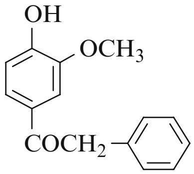
Synthesis

- Also refer to: [6230].

N.B.: Terbium complex (luminescent properties) [6230].

1-(4-Hydroxy-3-methoxyphenyl)-2-phenylethanone

[66476-02-6] $C_{15}H_{14}O_3$ mol.wt. 242.27



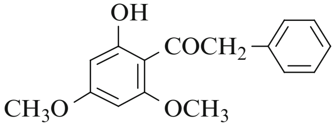
Described [5958] **p. 1422**

Synthesis

- Also obtained by cleavage of 1-(4-benzyloxy-3-methoxyphenyl)-2-phenylethanone with 30% HBr in acetic acid/methylene chloride (91%) [6231].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-phenylethanone

[39604-66-5] $C_{16}H_{16}O_4$ mol.wt. 272.30



Described [5958] **p. 1430**

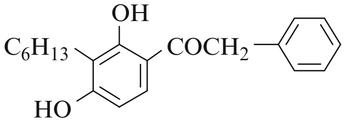
Synthesis

- Also refer to: [6230].

N.B.: Terbium complex (luminescent properties) [6230].

1-(3-Hexyl-2,4-dihydroxyphenyl)-2-phenylethanone

$C_{20}H_{24}O_3$ mol.wt. 312.41



New compound

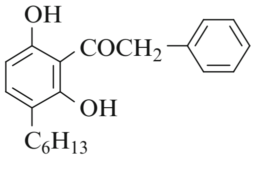
Synthesis

- Obtained by reaction of phenylacetonitrile with 2-hexyl-resorcinol in the presence of zinc chloride (31%) (Hoesch reaction) [6225].

m.p. 104–105° [6225].

1-(3-Hexyl-2,6-dihydroxyphenyl)-2-phenylethanone

[110146-61-7] $C_{20}H_{24}O_3$ mol.wt. 312.41



New compound

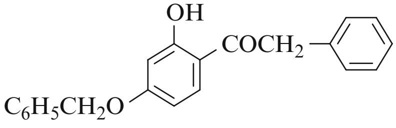
Synthesis

- Obtained by reaction of phenylacetonitrile with 4-hexyl-resorcinol in the presence of zinc chloride (17%) (Hoesch reaction) [6225].

m.p. 210–211° [6225].

1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-2-phenylethanone

[39604-80-3]



$C_{21}H_{18}O_3$ mol.wt. 318.37

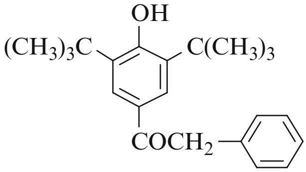
Described [5958] **p. 1441**

Synthesis

- Also obtained by reaction of benzyl alcohol with 1-(2,4-dihydrox-yphenyl)-2-phenylethanone in the presence of triphenylphosphine and DIAD (diisopropyl diazodicarboxylate) in THF (87%) [6226].

1-[3,5-Bis(1,1-dimethyl)-4-hydroxyphenyl]-2-phenylethanone

[14035-39-3]



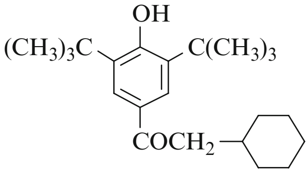
$C_{22}H_{28}O_2$ mol.wt. 324.46

Described [5958] **p. 1443**

Synthesis

- Preparation by reaction of phenylacetyl chloride with 2,6-di-tert-butylphenol in the presence of titanium tetrachloride [6066].

1-[3,5-Bis(1,1-dimethyl)-4-hydroxyphenyl]-2-cyclohexylethanone



$C_{22}H_{34}O_2$ mol.wt. 330.51

New compound

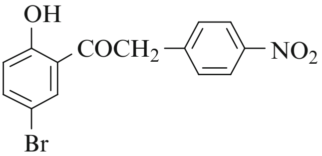
Synthesis

- Preparation by reaction of cyclohexylacetyl chloride with 2,6-di-tert-butylphenol in the presence of titanium tetrachloride [6066].

18.2 Compounds Derived from Substituted Phenylacetic Acids [5958] p. 1449

1-(5-Bromo-2-hydroxyphenyl)-2-(4-nitrophenyl)ethanone

[340959-83-3]



$C_{14}H_{10}BrNO_4$ mol.wt. 322.14

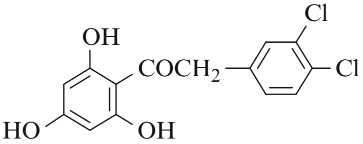
New compound

Synthesis

- Refer to: [6232].

2-(3,4-Dichlorophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[1005418-07-4] $C_{14}H_{10}Cl_2O_4$ mol.wt. 313.14



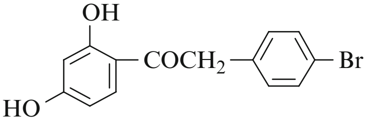
New compound

Synthesis

– Preparation by reaction of 3,4-dichlorophenylacetonitrile with phloroglucinol (Hoesch reaction) in the presence of zinc chloride [6084].

2-(4-Bromophenyl)-1-(2,4-dihydroxyphenyl)ethanone

[92152-60-5] $C_{14}H_{11}BrO_3$ mol.wt. 307.14



Described [5958] **p. 1450**

Synthesis

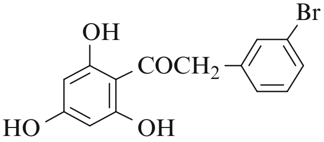
– Also obtained by Friedel–Crafts acylation of resorcinol with 4-bromophenylacetic acid in the presence of boron trifluoride (good yield) [6233,6234].

^{1}H NMR [6233], ^{13}C NMR [6233], MS [6233].

BIOLOGICAL DATA: Potent selective estrogen receptor modulator [6233].

2-(3-Bromophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[879559-91-8] $C_{14}H_{11}BrO_4$ mol.wt. 323.15



New compound

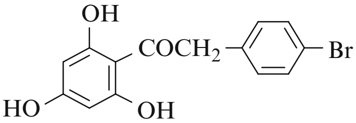
Synthesis

– Obtained by reaction of 3-bromophenylacetonitrile with phloroglucinol (Hoesch reaction) [6235].

BIOLOGICAL ACTIVITY: In vitro activity against the coccidian parasite *Cryptosporidium parvum* [6235].

2-(4-Bromophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[147220-80-2] $C_{14}H_{11}BrO_4$ mol.wt. 323.15



Described [5958] **p. 1451**

Syntheses

– Also obtained by reaction of 4-bromophenylacetonitrile with phloroglucinol (Hoesch reaction) [6235].

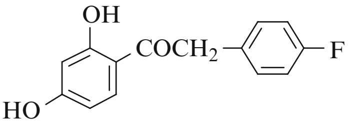
– Also refer to: [6236].

m.p. 231° [6236], 222–224° [6235]; ^{1}H NMR [6235,6236], ^{13}C NMR [6235], IR [6236].

BIOLOGICAL ACTIVITY: In vitro activity against the coccidian parasite *Cryptosporidium parvum* [6235].

1-(2,4-Dihydroxyphenyl)-2-(4-fluorophenyl)ethanone

[15485-70-8] $C_{14}H_{11}FO_3$ mol.wt. 246.24



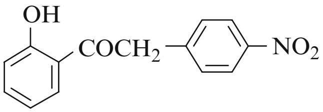
Described [5958] **p. 1453**

Dimethyl ether [315233-59-1] $C_{16}H_{15}FO_3$ mol.wt. 274.29

- Refer to: [6237].

1-(2-Hydroxyphenyl)-2-(4-nitrophenyl)ethanone

[340959-86-6] $C_{14}H_{11}NO_4$ mol.wt. 257.25



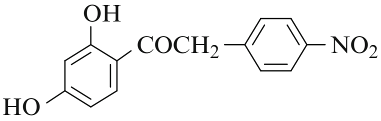
New compound

Synthesis

- Refer to: [6238] (70%).

1-(2,4-Dihydroxyphenyl)-2-(4-nitrophenyl)ethanone

[15485-63-9] $C_{14}H_{11}NO_5$ mol.wt. 273.25



Described [5958] **p. 1454**

Syntheses

- Also obtained by reaction of p-nitrophenylacetic acid with resorcinol in the presence of boron trifluoride etherate at 80° for 5 h [6239].
- Also refer to: [6240,6241].

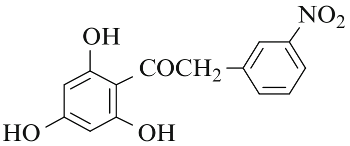
m.p. 206–208° [6241]; ^{1}H NMR [6241].

BIOLOGICAL ACTIVITY: Effect on enzyme activity [6241].

2,4-Dinitrophenylhydrazone $C_{20}H_{15}N_5O_8$ mol.wt. 453.37 m.p. 238° [6242]

2-(3-Nitrophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[876487-63-7] $C_{14}H_{11}NO_6$ mol.wt. 289.25



Described [5958] **p. 1454**

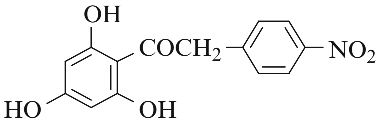
Synthesis

- Also obtained by reaction of 3-nitrophenylacetonitrile with phloroglucinol (Hoesch reaction) [6235].

BIOLOGICAL ACTIVITY: In vitro activity against the coccidian parasite *Cryptosporidium parvum* [6235].

2-(4-Nitrophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[15485-67-3]



$C_{14}H_{11}NO_6$ mol.wt. 289.25

Described [5958] **p. 1455**

Syntheses

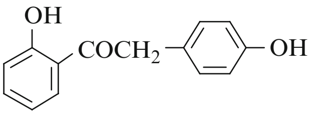
- Also obtained by reaction of 4-nitrophenylacetonitrile with phloroglucinol (Hoesch reaction) (51%) [6235].
- Also refer to: [6240,6243,6244].

m.p. 212–214° [6235]; ^{1}H NMR [6235], ^{13}C NMR [6235], MS [6235].

BIOLOGICAL ACTIVITY: In vitro activity against the coccidian parasite *Cryptosporidium parvum* [6235].

1-(2-Hydroxyphenyl)-2-(4-hydroxyphenyl)ethanone

[109561-92-4]



$C_{14}H_{12}O_3$ mol.wt. 228.25

Described [5958] **p. 1456**

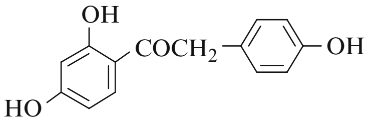
Synthesis

- Also obtained by demethylation of 1-(2-hydroxyphenyl)-2-(4-methoxyphenyl)ethanone [6245].

m.p. 102° [6245].

1-(2,4-Dihydroxyphenyl)-2-(4-hydroxyphenyl)ethanone

[17720-60-4]



$C_{14}H_{12}O_4$ mol.wt. 244.25

Described [5958] **p. 1457**

Synthesis

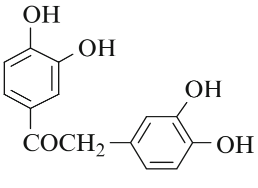
- Also obtained by demethylation of 1-(2,4-dihydroxy-phenyl)-2-(4-methoxyphenyl)ethanone [6245].

m.p. 186° [6245].

1,2-Bis(3,4-dihydroxyphenyl)ethanone

3,3′,4,4′-Tetrahydroxydeoxybenzoin

[100622-09-1]



$C_{14}H_{12}O_5$ mol.wt. 260.25

New compound

Synthesis

- Obtained by treatment of 3,3′,4,4′-tetrahydroxybenzil dihydrate (m.p. 125–130°) with granulated tin in dilute hydrochloric acid. The mixture was heated on a steam bath 1.5 h (49%) (compound **VIII**) [6246].

m.p. 196–198° (monohydrate) [6246]; UV [6246].

Tetramethyl ether [4927-55-3] $C_{18}H_{20}O_5$ mol.wt. 316.35

(Desoxyveratroin), (3,3′,4,4′-Tetramethoxydesoxybenzoin)

- Preparation by the reduction of veratril with tin and hydrochloric acid [6247].
- Also obtained by treatment of veratroin with zinc powder in methanol and acetic acid mixture on a boiling water bath for 1–2 h [6248].
- Also obtained by treatment of veratril in ethanol with granulated tin and a saturated copper sulfate solution in the presence of concentrated hydrochloric acid on a boiling water bath for 4 h [6247].
- Also obtained by reaction of homoveratroyl chloride (3,4-dimethoxyphenylacetyl chloride) with veratrole in the presence of aluminium chloride,
  - in refluxing carbon disulfide (31%) [6249] for 2 h [6247];
  - in refluxing methylene chloride for 2 h (98%) [6250].
- Also refer to: [6251,6252].

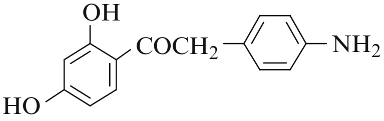
b.p.$_{0.01}$ 190–210° [6248], b.p.$_1$ 240–270° [6249];
m.p. 107° [6247], 106° [6248], 105–107° [6250], 104–106° [6249], 100–101° [6253];
^{1}H NMR [6250,6252], ^{13}C NMR [6250], IR [6252], MS [6251].

Tetraacetate [102599-72-4] $C_{22}H_{20}O_9$ mol.wt. 428.39

- Obtained by reaction of acetic anhydride with 3,3′,4,4′-tetrahydroxydeoxybenzoin in the presence of pyridine [6246].

m.p. 127–128° [6246]; UV [6246].

2-(4-Aminophenyl)-1-(2,4-dihydroxyphenyl)ethanone



$C_{14}H_{13}NO_3$ mol.wt. 243.26

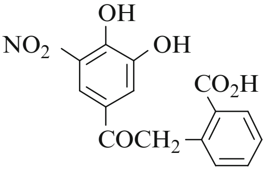
New compound

Synthesis

- Obtained by reduction of 1-(2,4-dihydroxyphenyl)-2-(4-nitrophenyl) ethanone with iron powder in the presence of ammonium chloride in water at 60–70° for 2 h (56%) [6225].

m.p. 241° [6225].

2-(2-Carboxyphenyl)-1-(3,4-dihydroxy-5-nitrophenyl)ethanone



$C_{15}H_{11}NO_7$ mol.wt. 2255.27

New compound

Synthesis

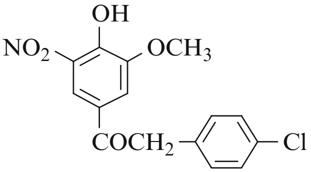
- Obtained by demethylation of 2-(2-carboxyphenyl)-1-(4-hydroxy-3-methoxy-5-nitrophenyl)ethanone with aluminium chloride by refluxing an ethyl acetate/pyridine mixture for 2 h (72%) [6254].

m.p. 244–247°; ^{1}H NMR [6254], ^{13}C NMR [6254], IR [6254].

BIOLOGICAL ACTIVITY: [6254].

1-(4-Hydroxy-3-methoxy-5-nitrophenyl)-2-(4-chlorophenyl)ethanone

$C_{15}H_{12}ClNO_5$ mol.wt. 321.72



New compound

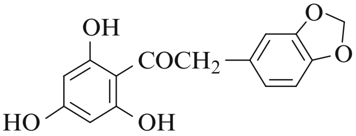
Syntheses

- Preparation by reaction of 70% nitric acid with 2-(4-chloro-phenyl)-1-(4-hydroxy-3-methoxyphenyl)ethanone in acetic acid at 20° for 30 min [6254].
- Also refer to: [6231].

2-(1,3-Benzodioxol-5-yl)-1-(2,4,6-trihydroxyphenyl)ethanone

[39548-98-6]

$C_{15}H_{12}O_6$ mol.wt. 288.26



Described [5958] **p. 1462**

Synthesis

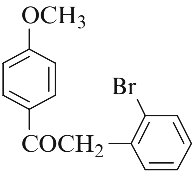
- Preparation by reaction of (3,4-methylenedioxy)phenyl-acetonitrile with phloroglucinol (Hoesch reaction) in the presence of zinc chloride (95%) [6084].

White powder [6084]; ^{1}H NMR [6084], ^{13}C NMR [6084], MS [6084].

2-(2-Bromophenyl)-1-(4-methoxyphenyl)ethanone

[655244-07-8]

$C_{15}H_{13}BrO_2$ mol.wt. 305.17



New compound

Syntheses

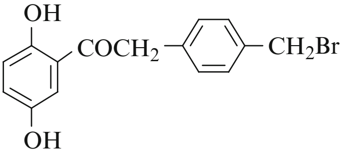
- Preparation by Friedel–Crafts acylation of anisole with 2-bromophenylacetyl chloride in the presence of aluminium chloride in tetrahydrofuran for 2 h at 20° (81%) [6255].
- Also refer to: [6256].

m.p. 91–92° [6255]; ^{1}H NMR [6255], ^{13}C NMR [6255].

2-[4-(Bromomethyl)phenyl]-1-(2,5-dihydroxyphenyl)ethanone

[340960-50-1]

$C_{15}H_{13}BrO_3$ mol.wt. 321.17



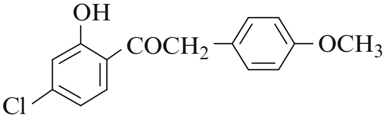
New compound

Synthesis

- Refer to: [6257].

1-(4-Chloro-2-hydroxyphenyl)-2-(4-methoxyphenyl)ethanone

[189289-99-4]



$C_{15}H_{13}ClO_3$ mol.wt. 276.72

New compound

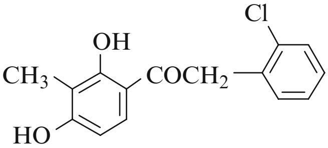
Synthesis

- Obtained by Fries rearrangement of 3-chlorophenyl 4-methoxyphenylacetate [6245].

m.p. 107° [6245].

2-(2-Chlorophenyl)-1-(2,4-dihydroxy-3-methylphenyl)ethanone

[328019-93-8]



$C_{15}H_{13}ClO_3$ mol.wt. 276.72

New compound

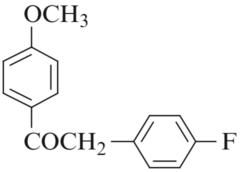
Synthesis

- Refer to: [6258].

BIOLOGICAL DATA: Protein binding [6258].

2-(4-Fluorophenyl)-1-(4-methoxyphenyl)ethanone

[2729-19-3]



$C_{15}H_{13}FO_2$ mol.wt. 244.27

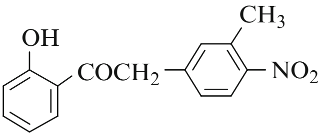
New compound

Synthesis

- Refer to: [6237].

1-(2-Hydroxyphenyl)-2-(3-methyl-4-nitrophenyl)ethanone

[1018669-07-2]



$C_{15}H_{13}NO_4$ mol.wt. 272.27

New compound

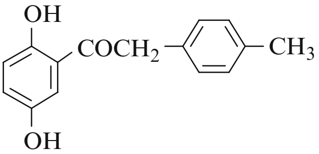
Synthesis

- Refer to: [6238] (50%).

^1H NMR [6238], ^{13}C NMR [6238], IR [6238], MS [6238].

1-(2,5-Dihydroxyphenyl)-2-(4-methylphenyl)ethanone

[340959-90-2]



$C_{15}H_{14}O_3$ mol.wt. 242.27

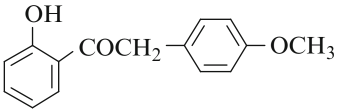
New compound

Synthesis

- Refer to: [6257].

1-(2-Hydroxyphenyl)-2-(4-methoxyphenyl)ethanone

[79744-47-1]



m.p. 85° [6245].

$C_{15}H_{14}O_3$ mol.wt. 242.27

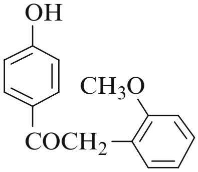
Described [5958] **p. 1465**

Synthesis

- Also obtained by Fries rearrangement of phenyl 4-methoxyphenylacetate [6245].

1-(4-Hydroxyphenyl)-2-(2-methoxyphenyl)ethanone

[341526-37-2]



$C_{15}H_{14}O_3$ mol.wt. 242.27

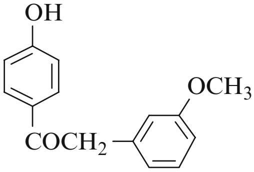
New compound

Synthesis

- Refer to: [6259].

1-(4-Hydroxyphenyl)-2-(3-methoxyphenyl)ethanone

[341526-35-0]



$C_{15}H_{14}O_3$ mol.wt. 242.27

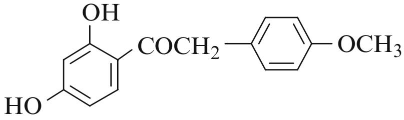
New compound

Synthesis

- Refer to: [6259].

1-(2,4-Dihydroxyphenyl)-2-(4-methoxyphenyl)ethanone

[487-49-0]



$C_{15}H_{14}O_4$ mol.wt. 258.27

Described [5958] **p. 1468**

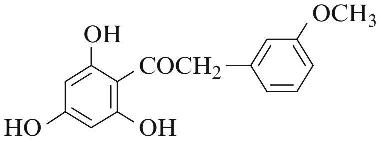
Syntheses

- Also obtained by reaction of resorcinol with 4-methoxyphenylacetic acid in the presence of PPA under microwave irradiation and solvent-free conditions [6260].
- Also obtained by Fries rearrangement of 3-hydroxyphenyl 4-methoxyphenylacetate [6245].
- Also refer to: [6261].

m.p. 155° [6245].

2-(3-Methoxyphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[111474-27-2]



$C_{15}H_{14}O_5$ mol.wt. 274.27

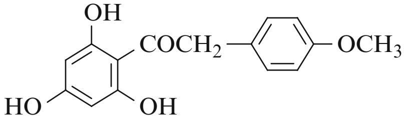
Described [5958] **p. 1473**

Synthesis

- Preparation by reaction of 3-methoxyphenylacetonitrile with phloroglucinol (Hoesch reaction) in the presence of zinc chloride [6084].

2-(4-Methoxyphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone

[15485-66-2]



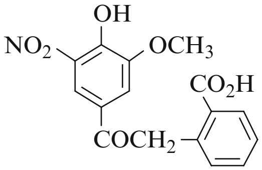
$C_{15}H_{14}O_5$ mol.wt. 274.27

Described [5958] **p. 1474**

Syntheses

- Preparation by reaction of 4-methoxyphenyl-acetonitrile with phloroglucinol (Hoesch reaction) in the presence of zinc chloride [6084].
- Also obtained by reaction of phloroglucinol with 4-methoxyphenylacetic acid in the presence of PPA under microwave irradiation [6260].

2-(2-Carboxyphenyl)-1-(4-hydroxy-3-methoxy-5-nitrophenyl)ethanone



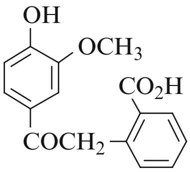
$C_{16}H_{13}NO_7$ mol.wt. 331.28

New compound

Synthesis

- Obtained by adding dropwise 70% nitric acid to a stirred suspension of 2-(2-carboxyphenyl)-1-(4-hydroxy-3-methoxyphenyl)ethanone in acetic acid at room temperature, then stirring for 30 min (62%) [6254].

2-(2-Carboxyphenyl)-1-(4-hydroxy-3-methoxyphenyl)ethanone



$C_{16}H_{14}O_5$ mol.wt. 286.28

New compound

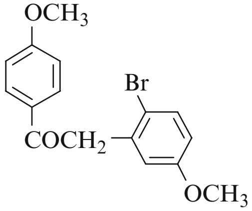
Synthesis

- To a stirred an cooled (−78°) solution of diisopropylamine in THF under nitrogen was added 2 M n-butyllithium solution in hexane. After stirring the mixture for 30 min, o-toluic acid in THF was added dropwise, and the resulting deep red solution was stirred at −78° for 1 h. Thereupon, a solution of methyl vanillate in THF was added dropwise, and the temperature was gradually allowed to reach room temperature. The mixture was then poured on ice/2 N HCl (66%) [6254].

m.p. 166–168° [6254].

2-(2-Bromo-5-methoxyphenyl)-1-(4-methoxyphenyl)ethanone

[1019652-01-7]



$C_{16}H_{15}BrO_3$ mol.wt. 335.20

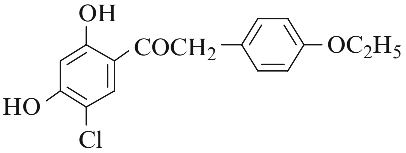
New compound

Synthesis

- Obtained by reaction of 2-bromo-5-methoxyphenylacetyl chloride with anisole in the presence of aluminium chloride in methylene chloride (89%) [6262].

1-(5-Chloro-2,4-dihydroxyphenyl)-2-(ethoxyphenyl)ethanone

[708259-71-6]



$C_{16}H_{15}ClO_4$ mol.wt. 306.75

New compound

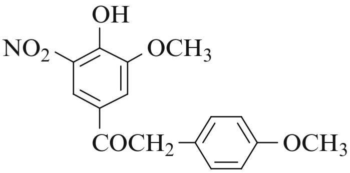
Synthesis

- Obtained by Friedel–Crafts acylation of 4-chloro-resorcinol with 4-ethoxyphenylacetic acid in the presence of boron trifluoride (good yield) [6233,6234].

^{1}H NMR [6233], ^{13}C NMR [6233], MS [6233].

BIOLOGICAL DATA: Potent selective estrogen receptor modulator [6233].

1-(4-Hydroxy-3-methoxy-5-nitrophenyl)-2-(4-methoxyphenyl)ethanone



$C_{16}H_{15}NO_6$ mol.wt. 317.30

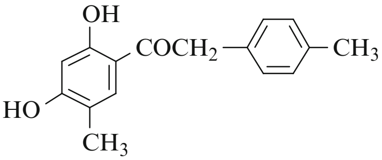
New compound

Syntheses

- Obtained by reaction of 70% nitric acid with 1-(4-hydroxy-3-methoxyphenyl)-2-(4-methoxyphenyl)ethanone [6254].
- Also refer to: [6231].

1-(2,4-Dihydroxy-5-methylphenyl)-2-(4-methylphenyl)ethanone

[1002716-64-4]



$C_{16}H_{16}O_3$ mol.wt. 256.30

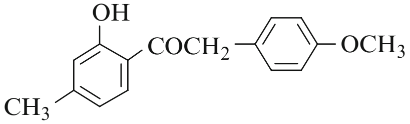
New compound

Synthesis

- Obtained by reaction of 4-methylresorcinol with 4-methylphenylacetyl compound at molar ratio 1:1 in solvent in the presence of Lewis acid catalyst [6263] (Chinese paper).

1-(2-Hydroxy-4-methylphenyl)-2-(4-methoxyphenyl)ethanone

[189289-98-3]



$C_{16}H_{16}O_3$ mol.wt. 256.30

New compound

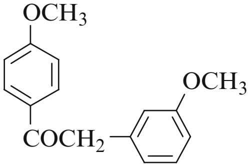
Synthesis

- Also obtained by Fries rearrangement of 3-methyl-phenyl 4-methoxyphenylacetate [6245].

m.p. 124° [6245].

2-(3-Methoxyphenyl)-1-(4-methoxyphenyl)ethanone

[98540-26-2]



$C_{16}H_{16}O_3$ mol.wt. 256.30

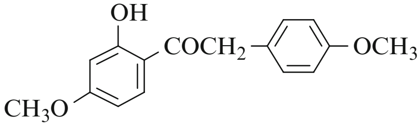
New compound

Synthesis

- Refer to: [6262].

1-(2-Hydroxy-4-methoxyphenyl)-2-(4-methoxyphenyl)ethanone

[39604-64-3]



$C_{16}H_{16}O_4$ mol.wt. 272.30

Described [5958] **p. 1483**

Syntheses

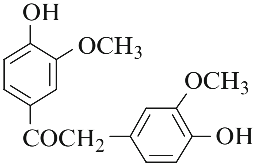
- Also obtained by Fries rearrangement of 3-methoxyphenyl 4-methoxyphenylacetate [6245].
- Also refer to: [6230].

m.p. 136° [6245].

N.B.: Terbium complex (luminescent properties) [6230].

1,2-Bis(4-hydroxy-3-methoxyphenyl)ethanone *(Melicopone, Desoxyvanilloin)*

[5438-67-5]



$C_{16}H_{16}O_5$ mol.wt. 288.30

New compound

Syntheses

- Obtained by treatment of 4,4′-dihydroxy-3,3′-dimethoxy-benzil with zinc and ammonium chloride in boiling dilute ethanol [6253], (84%) [6253].
- Also obtained from 3,3′-dimethoxy-4,4′-dihydroxy-*trans*-stilbene (27%) [6251].

- Also refer to: [6264,6265].

Isolation from natural sources

- From the root wood *Melicope semecarpifolia* Merr. T. Hartley [6266].

m.p. 154–155° [6253,6265];
^{1}H NMR [6266], ^{13}C NMR [6266], IR [6266], UV [6253,6266], MS [6266].
BIOLOGICAL DATA: Cytotoxicity [6266].

Diacetate $C_{20}H_{20}O_7$ mol.wt. 372.37

- Obtained by reaction of acetic anhydride with Melicopone in the presence of pyridine [6253].

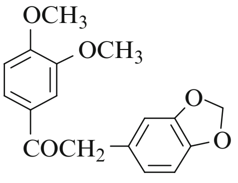
m.p. 172–173° [6253].

Dibenzyl ether [3122-36-9] $C_{30}H_{28}O_5$ mol.wt. 468.55

- Obtained by reaction of benzyl chloride with melicopone in the presence of potassium carbonate in boiling ethanol for 12 h (74%) [6264].
- Also obtained by reduction of 4,4′-dibenzyloxy-3,3′-dimethoxybenzoin with zinc powder in acetic acid [6267].

m.p. 146.5–147.5° [6267], 145–146° [6264].

2-(1,3-Benzodioxol-5-yl)-1-(3,4-dimethoxyphenyl)ethanone



$C_{17}H_{16}O_5$ mol.wt. 300.31

New compound

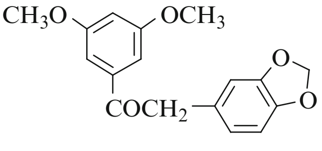
Syntheses

- Obtained by reaction of 3,4-dimethoxyphenylacetyl chloride with 1,3-benzodioxole in the presence of stannic chloride in methylene chloride, first at −10°, then at r.t. for 2 h (66%) [6250].
- Also obtained from 1,3-benzodioxole-5-carbaldehyde and [(3,4-dimethoxyphenyl)-(tetrahydropyran-2-yloxy)methyl]phosphonic acid dimethyl ester [6268].
- Also obtained from polymer-$PPh_2(1+)$-CH_3*I(1-) (multi-step reaction) [6268].

m.p. 110–111° [6250], 88–89° [6268]. One of the reported melting points is obviously wrong.
^{1}H NMR [6250,6268], ^{13}C NMR [6250], IR [6250,6268].

2-(1,3-Benzodioxol-5-yl)-1-(3,5-dimethoxyphenyl)ethanone

[1003857-18-8]



$C_{17}H_{16}O_5$ mol.wt. 300.31

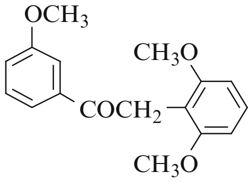
New compound

Synthesis

- Refer to: [6269].

2-(2,6-Dimethoxyphenyl)-1-(3-methoxyphenyl)ethanone

[960591-80-4] $C_{17}H_{18}O_4$ mol.wt. 286.33



New compound

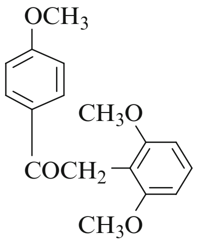
Synthesis

- Obtained by reaction of 1-chloro-2,6-dimethoxybenzene with 3-methoxyacetophenone in the presence of (DtBPF)PdCl$_2$ (2 mol%) and NaOtBu (1.1 equiv) in dioxane at 100° (98%) [6270].

GC [6270].

2-(2,6-Dimethoxyphenyl)-1-(4-methoxyphenyl)ethanone

[960591-81-5] $C_{17}H_{18}O_4$ mol.wt. 286.33



New compound

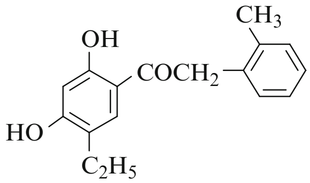
Synthesis

- Obtained by reaction of 1-chloro-2,6-dimethoxybenzene with 4-methoxyacetophenone in the presence of (DtBPF)PdCl$_2$ (2 mol%) and NaOtBu (1.1 equiv) in dioxane at 100° (99%) [6270].

GC [6270].

1-(5-Ethyl-2,4-dihydroxyphenyl)-2-(2-methylphenyl)ethanone

[302918-18-9] $C_{17}H_{18}O_3$ mol.wt. 270.33



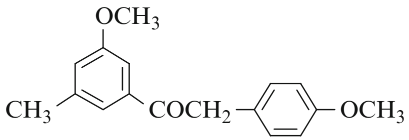
New compound

Synthesis

- Refer to: [6258].

1-(3-Methoxy-5-methylphenyl)-2-(4-methoxyphenyl)ethanone

[1019984-29-2] $C_{17}H_{18}O_3$ mol.wt. 270.33

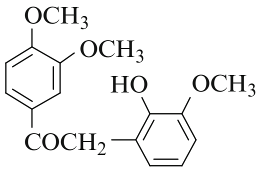


New compound

Synthesis

- Refer to: [6271].

1-(3,4-Dimethoxyphenyl)-2-(2-hydroxy-3-methoxyphenyl)ethanone



$C_{17}H_{18}O_5$ mol.wt. 302.33

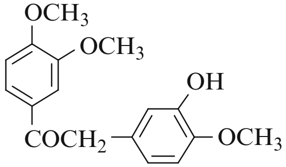
New compound

Synthesis

- Obtained by photochemical rearrangement of α-guaiacoxy-acetoveratrone on solid supports [6179].

1-(3,4-Dimethoxyphenyl)-2-(3-hydroxy-4-methoxyphenyl)ethanone

[18929-89-0]



$C_{17}H_{18}O_5$ mol.wt. 302.33

New compound

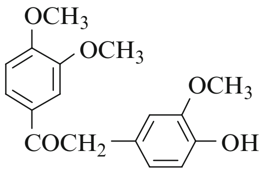
Synthesis

- Obtained by reaction of 3-hydroxy-4-methoxyphenylacetic acid with pyrocatechol dimethyl ether in the presence of phosphoric acid [6272].

m.p. 112–113° [6272]; ^{1}H NMR [6272], IR [6272], UV [6272].

1-(3,4-Dimethoxyphenyl)-2-(4-hydroxy-3-methoxyphenyl)ethanone

[135625-64-8]



$C_{17}H_{18}O_5$ mol.wt. 302.33

New compound

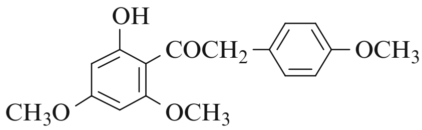
Syntheses

- Obtained by photochemical rearrangement of α-guaiacoxyl-acetoveratrone [6183],
  - on solid supports [6179];
  - in tetradeuteriomethanol at 25° [6180];
  - in acetonitrile [6190].
- Also obtained by desilylation of 1-(3,4-dimethoxyphenyl)-2-[4-[(tert-butyldimethylsilyl)oxy]-phenyl]ethanone ($C_{23}H_{32}O_5S$; m.p. 129–130°) (60%) [6183], according to the procedure [6273].

m.p. 119–119.5° [6274], 118–120° [6183];
^{1}H NMR [6180,6183], ^{13}C NMR [6183], IR [6183], MS [6183].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-(4-methoxyphenyl)ethanone

[39604-68-7]



$C_{17}H_{18}O_5$ mol.wt. 302.33

Described [5958] **p. 1504**

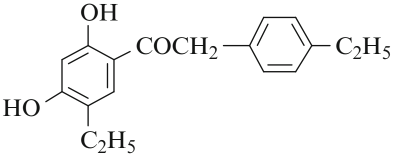
Synthesis

- Refer to: [6230].

N.B.: Terbium complex (luminescent properties) [6230].

1-(5-Ethyl-2,4-dihydroxyphenyl)-2-(4-ethylphenyl)ethanone

[1002716-66-6] $C_{18}H_{20}O_3$ mol.wt. 284.36



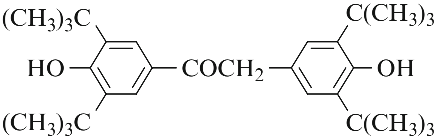
New compound

Synthesis

- Refer to: [6263] (Chinese patent).

1,2-Bis(3,5-di-tert-butyl-4-hydroxyphenyl)ethanone

[17055-19-5] $C_{30}H_{44}O_3$ mol.wt. 452.68



New compound

Syntheses

- Obtained by adding 2,6-di-tert-butylphenol (0.01 mol) in bromoacetyl chloride (10 ml) to a mixture of bromoacetyl chloride (15 ml) and aluminium chloride (0.5g) at −10° and allowed to stand for 15 min (23%) [6065].

  N.B.: The same compound was obtained by reaction of chloroacetyl chloride with 2,6-di-tert-butylphenol.
- Also refer to: [6275].

m.p. 240–241° [6275], 95–97° (recrystallized from octane) [6065].
One of the reported melting points is obviously wrong. IR [6065,6275].

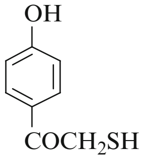
18.3 Compounds Derived from Di- and Triphenylacetic Acids [5958] p. 1538

18.4 Compounds Derived from Cycloalkylacetic Acids [5958] p. 1541

Chapter 19. Compounds Derived from S-Substituted Mercaptoacetic acids [5958] p. 1543

1-(4-Hydroxyphenyl)-2-mercaptoethanone

[23081-13-2] $C_8H_8O_2S$ mol.wt. 168.22



New compound

Synthesis

- Refer to: [6276].

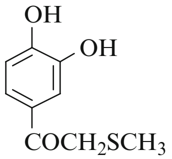
m.p. 133–134° [6276].

Methyl ether [139488-44-1] $C_9H_{10}O_2S$ mol.wt. 182.24

- Facile displacement of the bromide in 2-bromo-1-(4-methoxyphenyl)ethanone with potassium thioacetate delivered the corresponding thioacyl analog, which was easily hydrolyzed to the free thiol [5981]. Obtained in two steps: First, reaction of potassium thioacetate with 2-bromo-1-(4-methoxyphenyl)ethanone in THF at 40° for 24 h (99%), then treatment of the thioacyl derivative so obtained with 1 M sodium hydroxide in methanol at r.t. for 1 h (84%) [5981].

1-(3,4-Dihydroxyphenyl)-2-(methylthio)ethanone

[104692-98-0] $C_9H_{10}O_3S$ mol.wt. 198.24



New compound

Synthesis

- Obtained by reaction of methanethiol sodium salt and 2-chloro-1-(3,4-dihydroxyphenyl)ethanone in methanol (85%) [6277], (77%) [6278].

m.p. 110° [6277], 107–108° [6278]; ^{1}H NMR [6278].

Dimethyl ether [67489-10-5] $C_{11}H_{14}O_3S$ mol.wt. 226.30

- Obtained by reaction of α-(methylthio)acetyl chloride with veratrole in the presence of aluminium chloride in methylene chloride for 30 min at 0° (82%) [6279].
- Also obtained by reaction of methanethiol sodium salt and 2-chloro-1-(3,4-dimethoxyphenyl)ethanone in methanol (53%) [6277].
- Also refer to: [6279,6280,6281].

m.p. 53° [6277]; ^{1}H NMR [6279,6281].

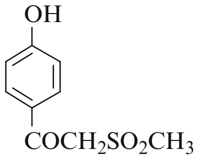
Diacetate $C_{13}H_{14}O_5S$ mol.wt. 282.32

- Obtained by reaction of acetic anhydride with the titled compound in the presence of concentrated sulfuric acid [6277].

m.p. 97–98° [6277].

1-(4-Hydroxyphenyl)-2-(methylsulfonyl)ethanone

[52945-18-3] $C_9H_{10}O_4S$ mol.wt. 214.24



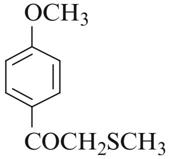
Described [5958] **p. 1544**

Synthesis

- Also refer to: [6282].

1-(4-Methoxyphenyl)-2-(methylthio)ethanone

[46188-84-5] $C_{10}H_{12}O_2S$ mol.wt. 196.27



New compound

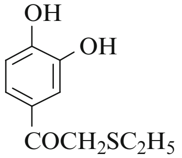
Syntheses

- Obtained by reaction of methanethiol sodium salt and ω-chloro-p-methoxyacetophenone in methanol (94%) [6277], (72%) [6283].
- Also obtained by adding a 20% aqueous solution of the sodium salt of methylmercaptan to a solution of α-bromo-4-methoxyacetophenone in ethyl ether at r.t. for 14 h [6284], (89%) [6285].
- Also obtained by reaction of α-(methylthio)acetyl chloride with anisole in the presence of aluminium chloride in methylene chloride at 0° for 30 min (83%) [6279].
- Also refer to: [6280,6284,6286].

b.p.$_{1}$ 132–136° [6285], b.p.$_{10}$ 175° [6284]; m.p. 33° [6277,6283];
^{1}H NMR [6279,6284], ^{13}C NMR [6284], IR [6284], MS [6284].

1-(3,4-Dihydroxyphenyl)-2-(ethylthio)ethanone

$C_{10}H_{12}O_3S$ mol.wt. 212.26



New compound

Synthesis

- Obtained by reaction of ethanethiol sodium salt and 2-chloro-1-(3,4-dihydroxyphenyl)ethanone in methanol [6277].

m.p. 94–94.5° [6277].

Dimethyl ether [100257-47-4] $C_{12}H_{16}O_3S$ mol.wt. 240.32

- Obtained by reaction of ethanethiol with 2-bromo-1-(3,4-dimethoxyphenyl) ethanone in the presence of ethanolic sodium ethylate [6287].

b.p.$_{0.1}$ 170° [6287].

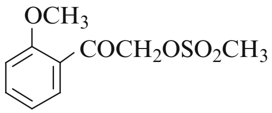
Dibenzoate $C_{24}H_{20}O_5S$ mol.wt. 420.49

- Obtained by reaction of benzoyl chloride with the titled compound in the presence of pyridine [6277].

m.p. 107.5–108.5° [6277].

1-(2-Methoxyphenyl)-2-[(methylsulfonyl)oxy]ethanone

[1023921-91-6] $C_{10}H_{12}O_5S$ mol.wt. 244.27



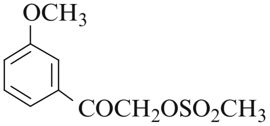
New compound

Synthesis

- Refer to: [6288].

1-(3-Methoxyphenyl)-2-[(methylsulfonyl)oxy]ethanone

[1023921-93-8] $C_{10}H_{12}O_5S$ mol.wt. 244.27



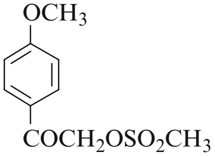
New compound

Synthesis

- Refer to: [6288].

1-(4-Methoxyphenyl)-2-[(methylsulfonyl)oxy]ethanone

[142608-19-3] $C_{10}H_{12}O_5S$ mol.wt. 244.27



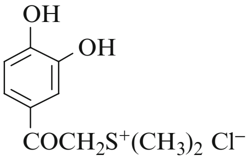
New compound

Synthesis

- Refer to: [6288].

[2-(3,4-Dihydroxyphenyl)-2-(oxoethyl)]dimethylsulfonium chloride

$C_{10}H_{13}ClO_3S$ mol.wt. 249.26



New compound

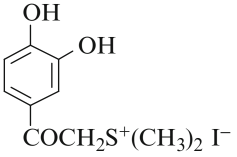
Synthesis

- Obtained by reaction of silver chloride with dimethyl-(3,4-dihydroxyphenacyl)sulfonium iodide [6277].

m.p. 151.5–152.5° [6277].

[2-(3,4-Dihydroxyphenyl)-2-(oxoethyl)]dimethylsulfonium iodide

[77263-39-9] $C_{10}H_{13}IO_3S$ mol.wt. 340.18



New compound

Syntheses

- Obtained from 1-(3,4-dihydroxyphenyl)-2-methylsulfanyl ethanone [6277].
- Also refer to: [6289].

m.p. 135° [6289], 130–130.5° [6277]; ^{1}H NMR [6289].

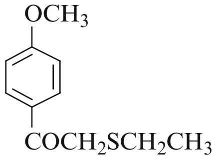
BIOLOGICAL ACTIVITY: Inhibition of <3H> dopamine binding [6289]; ability to produce circling behavior in rats lesioned unilaterally in the substantia nigra with 6-hydroxydopamine and antagonism by haloperidol of circling behavior [6289].

Dimethyl ether $C_{12}H_{17}IO_3S$ mol.wt. 368.24

- Refer to: [6277]. m.p. 107–108° [6277].

2-(Ethylthio)-1-(4-methoxyphenyl)ethanone

[115505-09-4] $C_{11}H_{14}O_2S$ mol.wt. 210.30



New compound

Syntheses

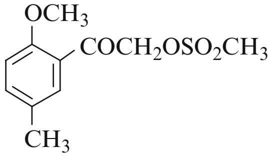
- Obtained by reaction of ethanethiolate sodium salt with 2′-chloro-4-methoxyacetophenone in methanol (50%) [6277].
- Also refer to: [5981,6290].

b.p.$_{11}$ 181° [6277];
^{1}H NMR [6291], ^{13}C NMR [6290], IR [6291], UV [6291,6292].

BIOLOGICAL DATA: Inhibition of enzyme activity [5981]; effect on cell growth [5981].

1-(2-Methoxy-5-methylphenyl)-2-[(methylsulfonyl)oxy]ethanone

[1023022-17-9] $C_{11}H_{14}O_5S$ mol.wt. 258.30



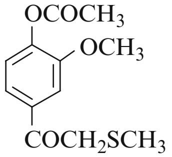
New compound

Synthesis

- Refer to: [6288].

1-[4-(Acetyloxy)-3-methoxyphenyl]-2-(methylthio)ethanone

[1018948-88-3] $C_{12}H_{14}O_4S$ mol.wt. 228.27



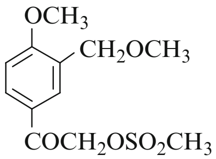
New compound

Synthesis

- Refer to: [6056].

1-[4-Methoxy-3-(methoxymethyl)phenyl]-2-[(methylsulfonyl)oxy]ethanone

[1023922-21-5] $C_{12}H_{16}O_6S$ mol.wt. 288.32



New compound

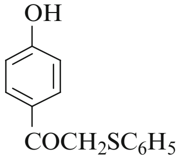
Synthesis

- Refer to: [6288].

1-(4-Hydroxyphenyl)-2-(phenylthio)ethanone

[137524-65-3] $C_{14}H_{12}O_2S$ mol.wt. 244.31

Described [5958] **p. 1550**



Methyl ether $C_{15}H_{14}O_2S$ mol.wt. 258.34

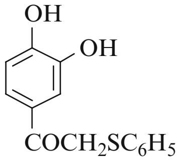
- Obtained by reaction of α-(phenylthio)acetyl chloride with anisole in the presence of aluminium chloride in methylene chloride for 30 min at 0° (59%) [6279].
- Also refer to: [6293].

m.p. 85–85.5° [6279], 79–81.5° [6293]; ^{1}H NMR [6279].

1-(3,4-Dihydroxyphenyl)-2-(phenylthio)ethanone

[131985-77-8] $C_{14}H_{12}O_3S$ mol.wt. 260.31

Described [5958] **p. 1551**



Dimethyl ether $C_{16}H_{16}O_3S$ mol.wt. 288.37

- Obtained by reaction of α-(phenylthio)acetyl chloride with veratrole in the presence of aluminium chloride in methylene chloride at 0° for 30 min (65%) [6279].

^{1}H NMR [6279].

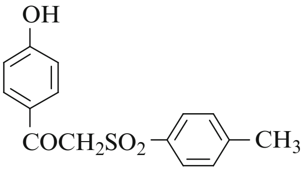
1-(4-Hydroxyphenyl)-2-[(4-methylphenyl)sulfonyl]ethanone

[896109-71-0] $C_{15}H_{14}O_4S$ mol.wt. 290.34

New compound

Synthesis

- Refer to: [6282].



Part VIII Di- and Polyketones

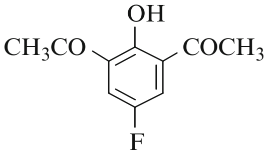
Chapter 20. Aromatic Ketones Containing Only Acetyl Groups [5958] p. 1559

20.1 Acetyl Groups Located on One Ring [5958] p. 1559

20.1.1 Unsubstituted Acetyl Groups [5958] p. 1559

1,1′-(5-Fluoro-2-hydroxy-1,3-phenylene)bis-ethanone

[106823-62-5] $C_{10}H_9FO_3$ mol.wt. 196.18



Described [5958] **p. 1560**

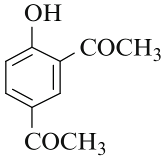
Synthesis

– Also obtained by Fries rearrangement of 2-acetyl-4-fluorophenyl acetate by heating at 170–180° for 3 h with aluminium chloride first heated at 140° for 20 min (19%) [6294].

Yellow solid; m.p. 64–66° [6294]; ^{1}H NMR [6294].

1,1′-(4-Hydroxy-1,3-phenylene)bis-ethanone

[30186-16-4] $C_{10}H_{10}O_3$ mol.wt. 178.19



Described [5958] **p. 1562**

Isolation from natural sources

– From capitulum of a head rot-resistant sunflower genotype [6295].
BIOLOGICAL ACTIVITY: Antifungal [6295].

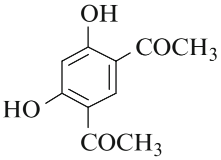
Acetate [1007089-42-0] $C_{12}H_{12}O_4$ mol.wt. 220.22

Isolation from natural sources

– From capitulum of a head rot-resistant sunflower genotype [6295].
BIOLOGICAL ACTIVITY: Antifungal [6295].

1,1′-(4,6-Dihydroxy-1,3-phenylene)bis-ethanone

[2161-85-5] $C_{10}H_{10}O_4$ mol.wt. 194.19



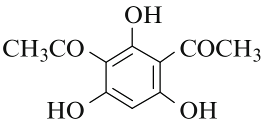
Described [5958] **p. 1566**

Synthesis

- Also refer to: [6296].

1,1′-(2,4,6-Trihydroxy-1,3-phenylene)bis-ethanone

[2161-86-6] $C_{10}H_{10}O_5$ mol.wt. 210.19



Described [5958] **p. 1568**

Syntheses

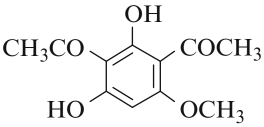
- Obtained by phloroglucin acetylation with acetic anhydride catalyzed by boron trifluoride etherate at 95° [6297].
- Also obtained by reaction of acetonitrile on phloroglucinol (Houben-Hoesch reaction) (14%) [6298].
- Also refer to: [6299,6300,6301,6302,6303,6304.6305].

m.p. 167–169° [6298].

BIOLOGICAL ACTIVITY: Antifungal compound [6299,6306,6307,6308]; antimicrobial antibiotic [6309]; an active ingredient in plant protection against soil-borne pathogens [6297]; role of 2,4-diacetylphloroglucinol-producing fluorescent Pseudomonas spp. in the defense of plant roots [6310]; phenotypic, genotypic and colonization properties of 2,4-diacetylphloroglucinol-producing Pseudomonas spp. isolated from roots of wheat [6311].

1,1′-(2,4-Dihydroxy-6-methoxy-1,3-phenylene)bis-ethanone

[3098-38-2] $C_{11}H_{12}O_5$ mol.wt. 224.21



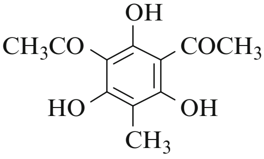
Described [5958] **p. 1575**

Synthesis

- Also obtained by reaction of acetonitrile with 2,4-dihydroxy-6-methoxyacetophenone (Houben-Hoesch reaction) (23%) [6298].

1,1′-(2,4,6-Trihydroxy-5-methyl-1,3-phenylene)bis-ethanone

[2999-42-0] $C_{11}H_{12}O_5$ mol.wt. 224.21



Described [5958] **p. 1576**

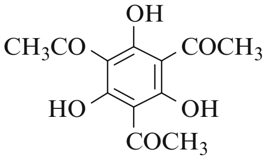
Trimethyl ether [3133-39-9] $C_{14}H_{18}O_5$ mol.wt. 266.29

- Obtained by total methylation of 2,4-diacetyl-6-methylphloroglucinol with dimethyl sulfate in the presence of potassium carbonate in boiling acetone [6312].
- Also obtained by methylation of 4,6-diacetyl-5-methoxy-2-methylresorcinol with dimethyl sulfate in the presence of potassium carbonate in boiling acetone [6312].
- Also obtained by methylation of 2,4-diacetyl-5-methoxy-6-methylresorcinol with dimethyl sulfate in the presence of potassium carbonate in boiling acetone [6312].
- Also refer to: [6313].

m.p. 67–68° [6313], 66–67° [6312]; ^{1}H NMR [6313], IR [6313].

1,1′,1″-(2,4,6-Trihydroxy-1,3,5-benzenetriyl)tris-ethanone

[2161-87-7] $C_{12}H_{12}O_6$ mol.wt. 252.22



Described [5958] **p. 1578**

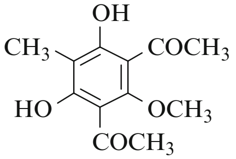
Syntheses

- Also obtained by Fries rearrangement of phloroglucinol triacetate [6314].
- Also obtained by reaction of acetic acid with phloroglucinol in the presence of PPA on a boiling water bath for 5–20 min (12%) [6315].

m.p. 154–155° [6315].

1,1′-(4,6-Dihydroxy-2-methoxy-5-methyl-1,3-phenylene)bis-ethanone

$C_{12}H_{14}O_5$ mol.wt. 238.24



New compound

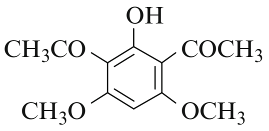
Synthesis

- Obtained by reaction of acetic anhydride with 2,4-dihydroxy-6-methoxy-3-methylacetophenone in the presence of boron trifluoride at 80° for 1 h (47%) [6312].

m.p. 94° [6312].

1,1′-(2-Hydroxy-4,6-dimethoxy-1,3-phenylene)bis-ethanone

$C_{12}H_{14}O_5$ mol.wt. 238.24



New compound

Syntheses

- Obtained by reaction of acetonitrile with 2-hydroxy-4,6-dimethoxyacetophenone (Houben-Hoesch reaction) [6298].

- Also obtained by reaction of acetic anhydride with phloroglucinol dimethyl ether in the presence of boron trifluoride [6312].
- Also refer to: [6316].

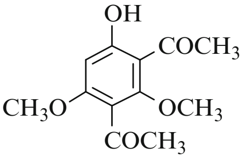
b.p.$_{0.005}$ 140–160° [6298]; m.p. 128° [6312], 127° [6298].

Benzyl ether $C_{19}H_{20}O_5$ mol.wt. 328.34

- Obtained by reaction of benzyl bromide with the titled diketone [6312].

1,1′-(4-Hydroxy-2,6-dimethoxy-1,3-phenylene)bis-ethanone

[72221-04-6] $C_{12}H_{14}O_5$ mol.wt. 238.24



Described [5958] **p. 1581**

Synthesis

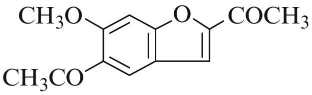
- Also obtained by reaction of acetic acid with phloroglucinol dimethyl ether in the presence of PPA on a boiling water bath for 5–20 min (46%) [6315].

m.p. 126° [6315].

1-(2-Acetyl-6-methoxy-5-benzofuranyl)ethanone

(2,5-Diacetyl-6-methoxybenzofuran) (Euparone methyl ether)

[23840-15-5] $C_{13}H_{12}O_4$ mol.wt. 232.14



New compound

Synthesis

- Obtained by adding a solution of 6-methoxycoumarone-2,5-dicarboxylic acid in THF to a solution of methyllithium in ethyl ether. After 3 h, the reaction mixture was decomposed with aqueous ammonium chloride [6317].

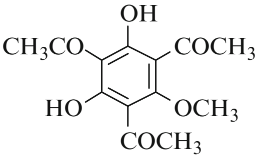
Isolation from natural source

- From *Encelia california* Nutt. (Compositae, tribe Heliantheae) [6317].

m.p. 140–141° [6317]; ^{1}H NMR [6317], IR [6317], UV [6317], MS [6317].

1′,1″,1‴-(2,4-Dihydroxy-6-methoxy-1,3,5-benzenetriyl)tris-ethanone

$C_{13}H_{14}O_6$ mol.wt. 266.25



New compound

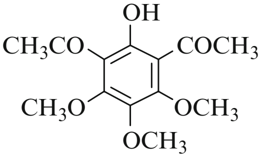
Synthesis

- Obtained by reaction of acetic acid with phloroglucinol monomethyl ether in the presence of PPA on a boiling water bath for 5–20 min (34%) [6315].

m.p. 109° [6315].

1,1′-(2-Hydroxy-4,5,6-trimethoxy-1,3-phenylene)bis-ethanone

[870480-47-0] $C_{13}H_{16}O_6$ mol.wt. 268.27



New compound

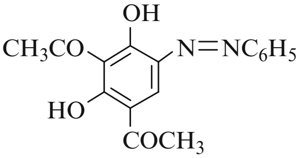
Synthesis

- Preparation by reaction of acetic anhydride (4 equiv) with 3,4,5-trimethoxyphenol (1 equiv) in the presence of aluminium chloride (3 equiv) in nitromethane, first at 0°, then at r.t. for 20 h (74%) [6318].

Colourless crystals [6318]; m.p. 85–86° [6318]; ^{1}H NMR [6318], MS [6318].

1-[3-Acetyl-2,6-dihydroxy-5-(phenylazo)phenyl]ethanone

$C_{16}H_{14}N_2O_4$ mol.wt. 298.30



New compound

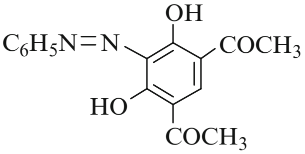
Synthesis

- Obtained by coupling 2,4-diacetylresorcinol with benzenediazonium chloride [6319].

m.p. 144° [6319].

1-[5-Acetyl-2,4-dihydroxy-3-(phenylazo)phenyl]ethanone

$C_{16}H_{14}N_2O_4$ mol.wt. 298.30



New compound

Synthesis

- Obtained by coupling 4,6-diacetylresorcinol with benzenediazonium chloride [6319].

m.p. 202° [6319].

20.1.2 *Diversely Substituted Acetyl Groups [5958] p. 1590*

20.2 Acetyl Groups Located on Different Rings [5958] p. 1590

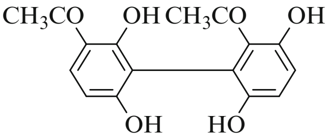
20.2.1 *Diphenyl Derivatives [5958] p. 1590*

Symmetrical ketones [5958] p. 1590

Asymmetrical ketones [5958] p. 1595

1-1′-(2′,3,6,6′-Tetrahydroxy[1,1′-biphenyl]-2,3′-diyl)bis-ethanone *(Cynandione A)*

[168706-29-4] $C_{16}H_{14}O_6$ mol.wt. 302.28



Described [5958] **p. 1596**

Isolation from natural sources

- Refer to: [6320] (Chinese patent).

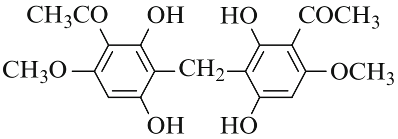
  BIOLOGICAL DATA: For treating nervous degenerative diseases [6320].

20.2.2. Diphenylmethane Derivatives [5958] p. 1598

20.2.2.1 Unsubstituted Acetyl Groups [5958] p. 1598

1,1′-[Methylenebis(2,4-dihydroxy-6-methoxy-3,1-phenylene)]bis-ethanone

[142382-28-3] $C_{19}H_{20}O_8$ mol.wt. 376.36



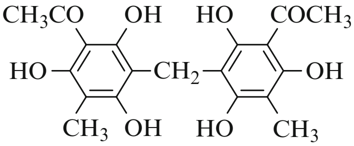
Described [5958] **p. 1603**

Isolation from natural sources

- Also from the roots of *Euphorbia ebracteolata* Hayata (Euphorbiaceae) [6321].

1,1′-[Methylenebis(2,4,6-trihydroxy-5-methyl-3,1-phenylene)]bis-ethanone

$C_{19}H_{20}O_8$ mol.wt. 376.36



Described [5958] **p. 1603**

Hexamethyl ether $C_{25}H_{32}O_8$ mol.wt. 460.53

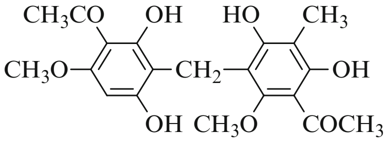
- Refer to: [6322]; UV [6322].

1-[3-[(3-Acetyl-4,6-dihydroxy-2-methoxy-5-methylphenyl)methyl]-2,4-dihydroxy-6-methoxyphenyl]ethanone

*2,2',4,4'-Tetrahydroxy-6,6'-dimethoxy-3,3'-dimethyl-7,5'-bis-acetophenone (compound **2**)*

[883886-03-1] $C_{20}H_{22}O_8$ mol.wt. 390.39

New compound



Isolation from natural sources

- From the roots of *Euphorbia ebracteolata* Hayata (Euphorbiaceae) [6321].

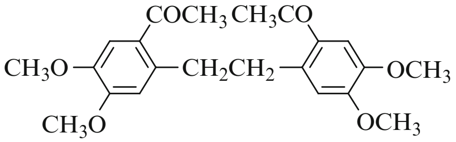
m.p. 228–230° [6321];
^{1}H NMR [6321], ^{13}C NMR [6321], IR [6321], MS [6321];
X-ray crystal data [6321].

20.2.2.2 Halogenated Acetyl Groups [5958] p. 1606

20.2.3 Diphenylalkanes Derivatives and Homologues [5958] p. 1610

1,1′-[1,2-Ethanediylbis(4,5-dimethoxy-1,2-phenylene)]bis-ethanone

2,2'-Diacetyl-4,5,3',4'-tetramethoxydibenzyl



$C_{22}H_{26}O_6$ mol.wt. 386.44

New compound

Synthesis

- Obtained by reaction of acetyl chloride with *3,4,3′,4′-tetramethoxydibenzyl in the presence of aluminium chloride in benzene at r.t for 1 h, then at reflux for 2.5 h (28%) [6323].

- Colourless rods; m.p. 173–175° [6323]; UV [6323].

*1,2-(3,4-Dimethoxyphenyl)ethane

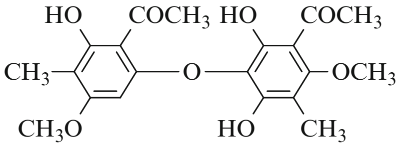
20.2.4 Diphenyl Ethers and Related Compounds [5958] p. 1614

1-[6-(3-Acetyl-2,6-dihydroxy-4-methoxy-5-methylphenoxy)-2-hydroxy-4-methoxy-3-methylphenyl]ethanone

3'-(2"-Acetyl-3"-hydroxy-5"-methoxy-4"-methylphenoxy)-2',4'-dihydroxy-6'-methoxy-5'-methyl-acetophenone (Leprolomin)

[68984-67-8] $C_{20}H_{22}O_8$ mol.wt. 390.39

New compound



Isolation from natural sources

- From *Psoroma leprolomum* (Nyl.) Räs (Lichen collected from the bark of eucalypts) (compound **12**) (0.6%) [6324].
- From *Psoroma anthrophyllum* [6325].

Pale yellow prisms [6324]; m.p. 238–239° [6324];
^{1}H NMR [6324], ^{13}C NMR [6324], UV [6324], MS [6324]; TLC [6324].

Triacetate (compound **15**) $C_{26}H_{28}O_{11}$ mol.wt. 516.50

- Obtained by reaction of acetic anhydride with leprolomin in the presence of sulfuric acid (2 drops) at 40° for 30 min (46%) [6324].

m.p. 121.5–122.5° [6324]; ^{1}H NMR [6324], MS [6324];

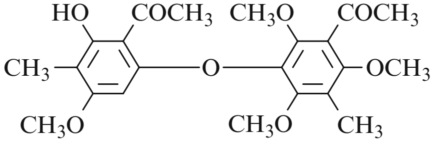
X-ray crystallography [6324].

1-[3-(2-Acetyl-3-hydroxy-5-methoxy-4-methylphenoxy)-2,4,6-trimethoxy-5-methyl-phenyl]ethanone

3'-(2"-Acetyl-3"-hydroxy-5"-methoxy-4"-methylphenoxy)-2',4',6'-trimethoxy-5'-methyl-acetophenone (Di-O-methyllepromin)

[68984-68-9] $C_{22}H_{26}O_8$ mol.wt. 418.16

New compound



Synthesis

- Obtained by treatment of a solution of *leprolomin* in ethyl acetate with an excess of etheral diazomethane at r.t. for 2 h (compound **13**) (93%) [6324].

Pale yellow crystals; m.p. 122–123° [6324];
^{1}H NMR [6324], MS [6324]; TLC [6324].

Methyl ether $C_{23}H_{28}O_8$ mol.wt. 432.18

*(Tri-O-methylleprolomin) (compound **14**)*

- Obtained by reaction of dimethyl sulfate with di-O-methylleprolomin in the presence of potassium carbonate in refluxing acetone for 16 h (77%) [6324].

oil [6324]; ^{1}H NMR [6324], MS [6324]; TLC [6324].

20.2.5 Diphenyl Sulfide Derivatives and Related Compounds [5958] p. 1620

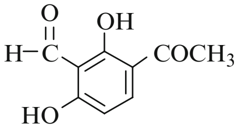
20.2.5.1 Diphenyl Sulfide Derivatives [5958] p. 1620

20.2.5.2 Diphenyl Sulfone Derivatives [5958] p. 1623

Chapter 21. Aromatic Polyketones Containing At Least One Acetyl Group and One Other Acyl Group [5958] p. 1627

21.1 Acyl Groups Located on One Ring [5958] p. 1627

2,4-Dihydroxy-3-(1-oxoethyl)benzaldehyde



$C_9H_8O_4$ mol.wt. 180.16

New compound

Syntheses

- Obtained by adding successively zinc cyanide (2 mol) and aluminium chloride (2 mol) to asolution of resacetophenone in ethyl ether, then passing hydrogen chloride for 4.5 h into the stirred mixture [6326], (45%) [6327].

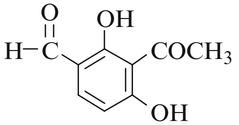
m.p. 112–114° [6327].

2,4-Dinitrophenylhydrazone $C_{15}H_{12}N_4O_7$ mol.wt. 360.49

m.p. 283–285° (d) [6327]

Dioxime $C_9H_{10}N_2O_4$ mol.wt. 210.19 m.p. 218–219° (d) [6327]

2,6-Dihydroxy-3-(1-oxoethyl)benzaldehyde



$C_9H_8O_4$ mol.wt. 180.16

New compound

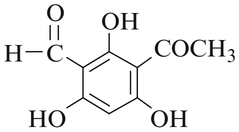
Synthesis

- Obtained by adding zinc cyanide to a solution of 2,6-dihydroxyacetophenone in ethyl ether; hydrogen chloride was passed into the solution to saturation (6–7 h). Then, the reaction mixture was allowed to stand overnight (35%) [6326].

m.p. 101–102° [6326].

2,4,6-Trihydroxy-3-(1-oxoethyl)benzaldehyde

[62018-55-7] $C_9H_8O_5$ mol.wt. 196.16



New compound

Syntheses

- Obtained by reaction of acetonitrile with 2,4,6-trihydroxy-benzaldehyde (Houben-Hoesch reaction) [6298].
- Also obtained by reaction of ethyl orthoformate with phloracetophenone in the presence of aluminium chloride [6328].
- Preparation by adding successively zinc cyanide and aluminium chloride to a solution of phloracetophenone in ethyl ether. The reaction mixture was cooled in ice and saturated with hydrogen chloride [6298,6329], (73%) [6326].
- Also obtained by reaction of DMF with phloracetophenone in the presence of phosphorous oxychloride at 20° for 1 h (70%) [6330,6331,6332].
- Also refer to: [6326,6333,6334,6335] (compound **10**).

m.p. 182–183° [6334], 180–182° [6329], 176° [6326], 173–174° [6298], 163–165° [6336].

One of the reported melting points is obviously wrong.
^{1}H NMR [6335,6336], ^{13}C NMR [6335], IR [6333,6336].

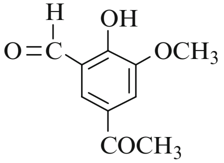
BIOLOGICAL ACTIVITY: Cytotoxicity [6332].

- No effect: Antifungal [6332]; antimalarial [6332]; antibacterial [6332].

Monohydrate $C_9H_8O_{5,}\ H_2O$ mol.wt. 214.18 [6326]

5-Acetyl-2-hydroxy-3-methoxybenzaldehyde

[112579-47-2] $C_{10}H_{10}O_4$ mol.wt. 194.19



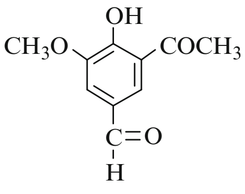
New compound

Synthesis

- Obtained by Fries rearrangement of o-vanillin acetate (small amount) [6337].

5-Acetyl-3-hydroxy-2-methoxybenzaldehyde *(5-Acetylvanillin)*

$C_{10}H_{10}O_4$ mol.wt. 194.19



New compound

Synthesis

- Obtained by treatment of 2-hydroxy-3-methoxy-acetophenone by chloroform in the presence of potassium hydroxide (19%) [6337].
  Pale yellow needles; m.p. 120–121° [6337].

Bis-semicarbazone $C_{12}H_{16}N_6O_4$ mol.wt. 308.30

m.p. 335–338° [6337].

Bis-phenylhydrazone $C_{22}H_{22}N_4O_2$ mol.wt. 374.44

m.p. 186–188° [6337].

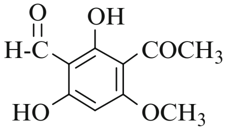
Benzenesulfonate $C_{16}H_{14}O_6S$ mol.wt. 334.35

- Obtained by reaction of benzenesulfonyl chloride with 5-acetylvanillin in the presence of pyridine (84%) [6337].

m.p. 123–125° [6337].

3-Acetyl-2,6-dihydroxy-4-methoxybenzaldehyde

[52117-67-6] $C_{10}H_{10}O_5$ mol.wt. 210.19



New compound

Synthesis

- Obtained by reaction of hydrogen cyanide with 2,4-dihydroxy-6-methoxyacetophenone [6338].
- Also refer to: [6339]

Isolation from natural sources

- From the roots of *Euphorbia ebracteolata* Hayata (Euphorbiaceae) [6321].
- From the roots of *Euphorbia kansui* (Euphorbiaceae) [6340].
- From *Euphorbia decipiens* Boiss and Buhse (Euphorbiaceae) [6341].
- Also obtained by heating 2,4-dihydroxy-6-methoxy-3-methylacetophenone 4-O-α-L-arabinofuranosyl-(1—>6)-β-D-glucopyranoside with 10% HCl under reflux for 5 h [6321].

Plates [6340]; m.p. 137–138° [6340,6341], 134° [6338];

^{1}H NMR [6321,6340,6341], ^{13}C NMR [6321,6340], IR [6339,6340,6341], MS [6340, 6341].

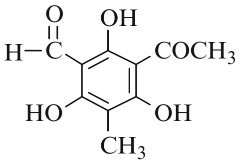
BIOLOGICAL ACTIVITY: [6341].

2,4-Dinitrophenylhydrazone $C_{16}H_{14}N_4O_8$ mol.wt. 390.31

m.p. 291–293° (d) [6338]

3-Acetyl-2,4,6-trihydroxy-5-methylbenzaldehyde

[59677-81-5] $C_{10}H_{10}O_5$ mol.wt. 210.19



New compound

Syntheses

- Obtained by reaction of methyl iodide with 2,4,6-trihydroxy-3-(1-oxoethyl)benzaldehyde in the presence of potassium hydroxide in methanol at 70° (50%) [6330,6331,6332].

- Also prepared by interaction of C-methylphloroacetophenone, zinc cyanide and hydrogen cyanide in ethyl ether saturated at 0°, for 24 h followed by rapid heating to boiling of the aldimine salt with water [6334].
- Also obtained by reaction of acetyl chloride with 2-formyl-4-methylphloroglucinol in the presence of aluminium chloride at 0° and at r.t. for 48 h, then at 60° for 1 h [6342].
- Also refer to: [6339].

m.p. 155° [6342], 134° [6334]. One of the reported melting points is obviously wrong.

^{1}H NMR [6342], IR [6339], MS [6342].

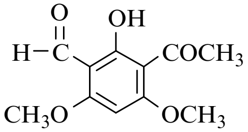
BIOLOGICAL ACTIVITY: Antifungal (Candida albicans) [6332]; antibacterial (no effect*) [6332],*Aspergillus fumigatus, mycobactetium intracellulaire, methicillin-resistant Staphylococcus aureus; antimalarial (no effect) [6332]; antiprotozoal (no effect) [6332]; cytotoxicity [6332].

2,4-Dinitrophenylhydrazone $C_{16}H_{14}N_4O_8$ mol.wt. 390.31

m.p. 294–295° [6334].

2-Hydroxy-4,6-dimethoxy-3-(1-oxoethyl)benzaldehyde

[99866-01-0] $C_{11}H_{12}O_5$ mol.wt. 224.21



New compound

Syntheses

- Obtained by treatment of 2-hydroxy-4,6-dimethoxyacetophenone with zinc cyanide in ethyl ether in the presence of hydrogen chloride (84%) [6298].
- Preparation by reaction of acetonitrile with 4,6-dimethoxysalicylaldehyde (Houben-Hoesch reaction) (76%) [6298].
- Also refer to: [6343,6344].

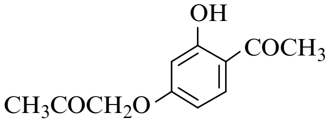
Sublimation 180–200°/0.005 mm [6298]; m.p. 170° [6343], 168–170° [6298].

21.1.1 Diphenyl Ketone Derivatives [5958] p. 1627

21.1.2 Miscellaneous [5958] p. 1631

1-(4-Acetyl-3-hydroxyphenoxy)-2-propanone

[248595-17-7] $C_{11}H_{12}O_4$ mol.wt. 208.21



New compound

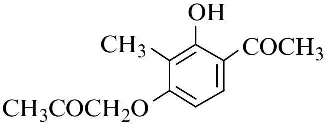
Synthesis

- Obtained by reaction of chloroacetone with resacetophenone in the presence ofpotassium carbonate in refluxing acetone for 12 h (75%) [6345].

m.p. 102–103° [6345]; ^{1}H NMR [6345], IR [6345].

1-(4-Acetyl-3-hydroxy-2-methylphenoxy)-2-propanone

[948045-85-0] $C_{12}H_{14}O_4$ mol.wt. 222.24



New compound

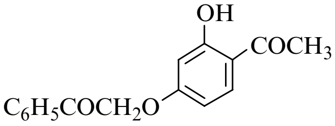
Synthesis

- Obtained by reaction of chloroacetone with 2,4-dihydroxy-3-methylacetophenone in the presence of potassium carbonate in refluxing acetone for 12 h (79%) [6345].

m.p. 126–128° [6345]; ^{1}H NMR [6345], IR [6345].

2-(4-Acetyl-3-hydroxyphenoxy)-1-phenylethanone

[948045-82-7] $C_{16}H_{14}O_4$ mol.wt. 270.28



New compound

Synthesis

- Obtained by reaction of phenacyl bromide with resacetophenone in the presence of potassium carbonate in refluxing acetone for 12 h (53%) [6345].

m.p. 132–133° [6345]; ^{1}H NMR [6345], IR [6345].

21.2 Acyl Groups Located on Different Rings [5958] p. 1636

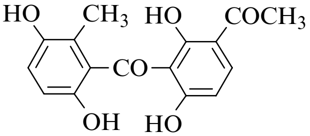
21.2.1 Diphenyl Ketone Derivatives [5958] p. 1636

Symmetrical ketones [5958] p. 1636

Asymmetrical ketones [5958] p. 1638

1-[3-(3,6-Dihydroxy-2-methylbenzoyl)-2,4-dihydroxyphenyl]ethanone

[115834-34-9] $C_{16}H_{14}O_6$ mol.wt. 302.28

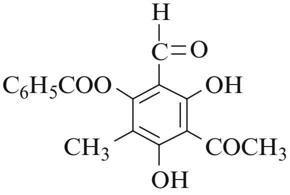


Described [5958] **p. 1639**

Isolation from natural sources

- From the ethyl acetate extract of the rhizome of *Cynanchum otophyllum* (Schneid Asclepiadaceae) [6346].

3-Acetyl-(6-benzoyloxy)-2,4-dihydroxy-5-methylbenzaldehyde



$C_{17}H_{14}O_6$ mol.wt. 314.29

New compound

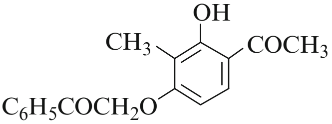
Synthesis

- Obtained by heating a mixture of benzoyl chloride and 3-acetyl-2,4,6-trihydroxy-5-methylbenzaldehyde in pyridine on a water bath for 15 min and left at r.t. overnight (73%) [6342].

m.p. 165–166° [6342]; ^{1}H NMR [6342].

2-(4-Acetyl-3-hydroxy-2-methylphenoxy)-1-phenylethanone

[948045-84-9]



$C_{17}H_{16}O_4$ mol.wt. 284.30

New compound

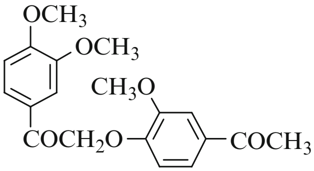
Synthesis

- Obtained by reaction of phenacyl bromide with 2,4-dihydroxy-3-methylacetophenone in the presence of potassium carbonate in refluxing acetone for 12 h (56%) [6345].

m.p. 124–125° [6345]; ^{1}H NMR [6345], IR [6345].

2-(4-Acetyl-2-methoxyphenoxy)-1-(3,4-dimethoxyphenyl)ethanone

[7249-35-6]



$C_{19}H_{20}O_6$ mol.wt. 344.37

New compound

Syntheses

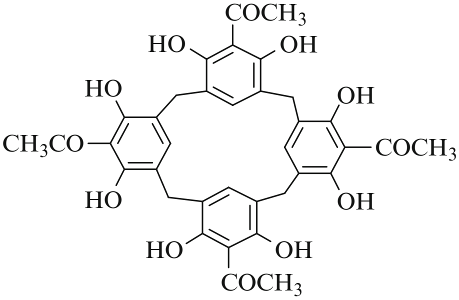
- Preparation by reaction of 3,4-dimethoxyphenacyl bromide with the sodium salt of 4-hydroxy-3-methoxyacetophenone in ethanol [6031,6347].
- Also refer to: [6182].

m.p. 138–139° and 120.5–121° (allotropic varieties) [6031,6347];
^{1}H NMR [6182], UV [6031,6347].

1,1′,1″,1‴-(4,6,10,12,16,18,22,24-Octahydropentacyclo[19.3.1.1^{3,7}.1$^{9.13}$.1$^{15.19}$]octacosa-1(25),3,5,7(28),9,11,13(27),15,17,19(26),21,23-dodecane-5,11,17,23-tetrayl)-tetrakis[ethanone]

5,11,17,23-Tetraacetylresorcinol

[872681-08-8]



m.p. > 300° [6348];
^{1}H NMR [6348], ^{13}C NMR [6348],
IR [6348], MS [6348].

$C_{36}H_{32}O_{12}$ mol.wt. 656.64

New compound

Synthesis

- Obtained by reaction of 2-acetylresorcinol with formaldehyde in dilute sodium hydroxide for 2 h at 0° under nitrogen, then 24 h at r.t. (37%) [6348].

Part X

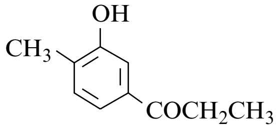
Hydroxypropiophenones, Hydroxyisobutyrophenones, Hydroxypivalophenones and Derivatives

Chapter 23
Aromatic Ketones Containing One Propionyl Group

23.1 Benzene Derivatives

1-(3-Hydroxy-4-methylphenyl-$^{14}C_6$)-1-propanone

[106697-21-6] $C_4{}^{14}C_6H_{12}O_2$ mol.wt. 176.16

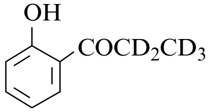


Synthesis

- Preparation by treatment of ^{14}C-3-amino-4-methyl-propiophenone in dilute hydrochloric acid with sodium nitrite in water, first at 0°, then at 50° (71%) [6349].

1-(2-Hydroxyphenyl)-1-propanone-*2,2,3,3,3-d*$_5$

$C_9H_5D_5O_2$ mol.wt. 155.21



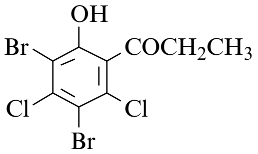
Synthesis

- Refer to: [6350].

ion 1^-, radical ion 1^- [72051-68-4], ESR spectrum [6350].

1-(3,5-Dibromo-4,6-dichloro-2-hydroxyphenyl)-1-propanone

$C_9H_6Br_2Cl_2O_2$ mol.wt. 376.86



Synthesis

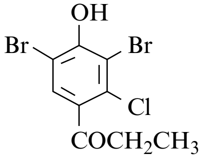
- Preparation by bromination of 2,4-dichloro-6-hydroxy-propiophenone [6351] according to the method [6352].

m.p. 97° [6351];
^1H NMR (Sadtler: standard n° 9475M),
IR (Sadtler: standard n° 38941); TLC [6353].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_23, © Springer Science+Business Media B.V. 2011

1-(3,5-Dibromo-2-chloro-4-hydroxyphenyl)-1-propanone

[2892-20-8] $C_9H_7Br_2ClO_2$ mol.wt. 342.41



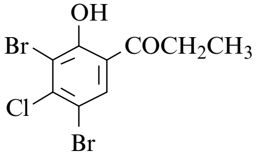
Syntheses

- Preparation by bromination of 2-chloro-4-hydroxypropiophenone, according to [6354], (good yield) [6355, 6356].

m.p. 80° [6355,6356];
^{1}H NMR (Sadtler: standard n° 8917M),
IR (Sadtler: standard n° 38080) [6355,6356], UV [6356,6357];
TLC [6353,6358].

1-(3,5-Dibromo-4-chloro-2-hydroxyphenyl)-1-propanone

[17765-21-8] $C_9H_7Br_2ClO_2$ mol.wt. 342.41



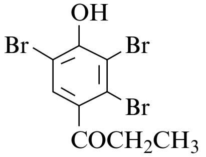
Syntheses

- Preparation by bromination of 4-chloro-2-hydroxypropiophenone, according to [6354], (good yield) [6355,6356].

m.p. 87° [6355,6356];
IR [6355,6356], UV [6356,6359]; TLC [6353,6358].

1-(2,3,5-Tribromo-4-hydroxyphenyl)-1-propanone

[17765-09-2] $C_9H_7Br_3O_2$ mol.wt. 386.87



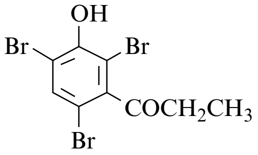
Synthesis

- Preparation by reaction of potassium bromate and potassium bromide on 2-bromo-4-hydroxypropiophenone according to the procedure [6352], (96%) [6360].

m.p. 84–85° [6360];
IR (Sadtler: standard n° 38935) [6360],
UV [6357,6360]; TLC [6353,6358].

1-(2,4,6-Tribromo-3-hydroxyphenyl)-1-propanone

[23689-31-8] $C_9H_7Br_3O_2$ mol.wt. 386.87



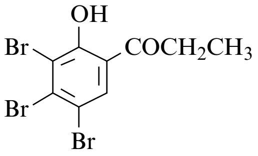
Synthesis

- Preparation by bromination of m-hydroxypropiophenone in acetic acid using potassium bromate-potassium bromide mixture [6352] in the presence of hydrochloric acid at r.t. for 24 h (90%) [6360].

m.p. 72–73° [6360];
^{1}H NMR (Sadtler: standard 9471M), IR [6360], UV [6360].

1-(3,4,5-Tribromo-2-hydroxyphenyl)-1-propanone

[17765-22-9] $C_9H_7Br_3O_2$ mol.wt. 386.87



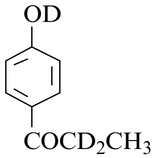
Synthesis

- Preparation by bromination of 4-bromo-2-hydroxypropiophenone according to [6354], (89%) [6360].

m.p. 111° [6360];
IR (Sadtler: standard n° 38936) [6360], UV [6359,6360];
TLC [6353,6358].

1-[4-(Hydroxy-*d*)phenyl]-1-propanone-*2,2-d*$_2$

[91889-34-8] $C_9H_7D_3O_2$ mol.wt. 153.20



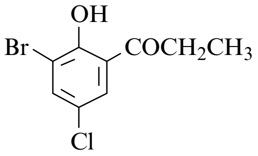
Synthesis

- Preparation from p-hydroxypropiophenone in a refluxing mixture of dioxane/D_2O in nitrogen atmosphere with p-toluenesulfonyl chloride overnight (94%) [6361].

^{1}H NMR [6361].

1-(3-Bromo-5-chloro-2-hydroxyphenyl)-1-propanone

[2892-15-1] $C_9H_8BrClO_2$ mol.wt. 263.52



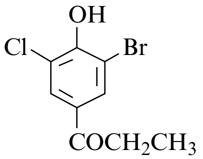
Syntheses

- Preparation by bromination of 5-chloro-2-hydroxypropiophenone, according to [6354], (good yield) [6355,6356].

m.p. 126–127° [6355,6356];
^{1}H NMR (Sadtler: standard n° 9089M),
IR (Sadtler: standard n° 38077) [6355,6356], UV [6356,6359];
TLC [6353,6358].

1-(3-Bromo-5-chloro-4-hydroxyphenyl)-1-propanone

[2892-25-3] $C_9H_8BrClO_2$ mol.wt. 263.52



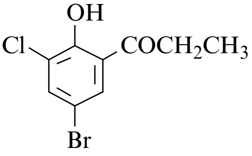
Syntheses

- Preparation by bromination of 3-chloro-4-hydroxy-propiophenone in aqueous acetic acid [6362] or according to the procedure [6354], (good yield) [6355,6356].
- Also refer to: [6363].

m.p. 125° [6362], 121–122° [6355,6356];
^{1}H NMR (Sadtler: standard n° 8729M), IR (Sadtler: standard n° 38074) [6355,6356], UV [6356,6357]; TLC [6353,6358].

1-(5-Bromo-3-chloro-2-hydroxyphenyl)-1-propanone

[2892-22-0] $C_9H_8BrClO_2$ mol.wt. 263.52



Syntheses

- Preparation by bromination of 3-chloro-2-hydroxypropiophenone, according to the procedure [6354], (good yield) [6355,6356].
- Also refer to: [6364].

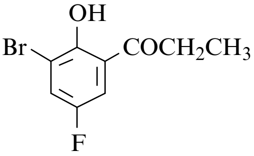
m.p. 115° [6355,6356];
^{1}H NMR (Sadtler: standard n° 8730M), IR (Sadtler: standard n° 38075) [6355,6356], UV [6356,6359]; TLC [6353,6358].

Thiosemicarbazone $C_{10}H_{11}BrClN_3OS$ mol.wt. 336.64 (m.p. 213°).

USE: Fungicide [6364].
BIOLOGICAL ACTIVITY: Antituberculotic [6364].

1-(3-Bromo-5-fluoro-2-hydroxyphenyl)-1-propanone

[342-16-5] $C_9H_8BrFO_2$ mol.wt. 247.06



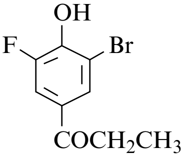
Syntheses

- Preparation by bromination of 5-fluoro-2-hydroxypropiophenone [6351] according to the method [6352], in aqueous acetic acid medium [6365].

m.p. 85° [6351,6365];
^{1}H NMR (Sadtler: standard n° 9473M), IR (Sadtler: standard n° 38939), UV [6359];
TLC [6353,6358].

1-(3-Bromo-5-fluoro-4-hydroxyphenyl)-1-propanone

[350-14-1] $C_9H_8BrFO_2$ mol.wt. 247.06



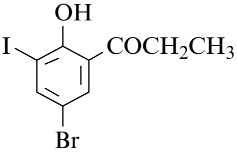
Synthesis

- Preparation by bromination of 3-fluoro-4-hydroxy-propiophenone in aqueous acetic acid [6362].

m.p. 114° [6362].

1-(5-Bromo-2-hydroxy-3-iodophenyl)-1-propanone

[868606-10-4] $C_9H_8BrIO_2$ mol.wt. 354.97

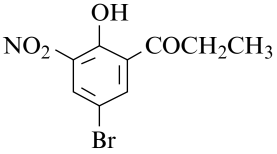


Synthesis

- Preparation by reaction of iodine and iodic acid with 5-bromo-2-hydroxypropiophenone in dilute ethanol for 1.5 h at 35–40° (82%) [6366].

1-(5-Bromo-2-hydroxy-3-nitrophenyl)-1-propanone

[90725-67-0] $C_9H_8BrNO_4$ mol.wt. 274.07



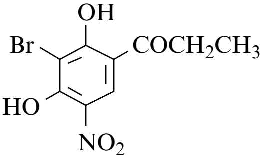
Synthesis

– Preparation by reaction of nitric acid (d = 1.52) with 5-bromo-2-hydroxypropiophenone in concentrated sulfuric acid (d = 1.80) between −2° and 0° (73%) [6367].

m.p. 101° [6367].

1-(3-Bromo-2,4-dihydroxy-5-nitrophenyl)-1-propanone

[100246-22-8] $C_9H_8BrNO_5$ mol.wt. 290.07



Syntheses

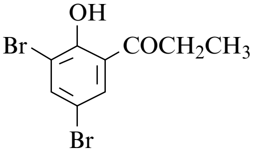
– Obtained by treatment of 3,3′-dipropionyl-4,4′,6,6′-tetra-hydroxy-5,5′-dibromophenyl thioether (m.p. 212–213°) with nitric acid (d = 1.4) at 5° for 2 h [6368].

– Also obtained by bromination of 5-nitrorespropiophenone in acetic acid with bromine [6368].

m.p. 230–231° [6368].

1-(3,5-Dibromo-2-hydroxyphenyl)-1-propanone

[2887-68-5] $C_9H_8Br_2O_2$ mol.wt. 307.97



Syntheses

– Preparation by bromination of various o-hydroxypropiophenones,

- from 2-hydroxypropiophenone, according to the procedure [6354], (71%) [6360] or with bromine in 80% aqueous acetic acid (54%) [6369];
- from 3-bromo-2-hydroxypropiophenone, according to the procedure [6354], (98%) [6360];
- from 5-bromo-2-hydroxypropiophenone, according to the procedure [6354], (98%) [6360].

– Preparation by Fries rearrangement of 2,4-dibromophenyl propionate (b.p. 275–280°) [6370] with aluminium chloride at 100° for 2 h (88%) [6360], at 160–165° for 30 min (66%) [6371] or at 150° for 2 h (33%) [6370].

– Also obtained by Friedel–Crafts acylation of 2,4-dibromophenol with propionic anhydride in nitrobenzene in the presence of aluminium chloride at 120° [6370].

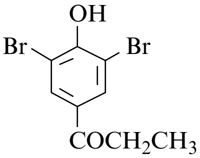
– Also refer to: [6363,6372,6373].

m.p. 121° [6370], 119–120° [6360], 118° [6369], 117–118° [6355,6356], 116.5–117° [6371];

^{1}H NMR (Sadtler: standard n° 9699M), IR (Sadtler: standard n° 38934) [6355,6356,6360], UV [6356,6359,6360]; TLC [6353,6358].

1-(3,5-Dibromo-4-hydroxyphenyl)-1-propanone

[2887-65-2] $C_9H_8Br_2O_2$ mol.wt. 307.97



Syntheses

- Preparation by treatment of p-hydroxypropiophenone with bromine,
  - in dilute acetic acid [6374], at r.t. for 5 h (80%) [6375], (63%) [6362] or in an acetic acid and sodium acetate mixture [6375];
  - in dilute ethanol [6376,6377] or in methanol (90%) [6378];
  - in acetone (97%) [6355,6356,6360].
- Preparation by treatment of 3-bromo-4-hydroxypropiophenone with bromine in acetone (97%) [6360].
- Also obtained by demethylation of 3,5-dibromo-4-methoxypropiophenone (m.p. 59°) [6362], (m.p. 99°) [6379] with pyridinium chloride at reflux for 10 min [6379].

N.B.: One of the reported melting points is obviously wrong.

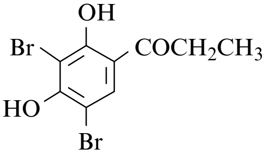
- Also refer to: [6363].

m.p. 130° [6379], 115–116° [6360], 115° [6355,6356], 114–115° [6378], 114° [6362,6375,6377], 100° [6376];
IR (Sadtler: standard n° 38933) [6360], UV [6357,6360]; TLC [6358].

Acetate $C_{11}H_{10}Br_2O_3$ mol.wt. 350.01 (m.p. 79.5–80.5°) [6378].

1-(3,5-Dibromo-2,4-dihydroxyphenyl)-1-propanone

[64603-55-0] $C_9H_8Br_2O_3$ mol.wt. 323.97



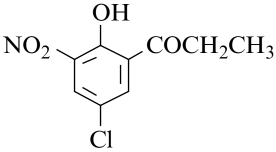
Syntheses

- Preparation by bromination of respropiophenone (40%) [6380], with bromine in chloroform [6381], in acetic acid [6382], in 80% acetic acid (51%) [6383] or in 50% acetic acid [6369].
- Also obtained by hydrolysis of 6,8-dibromo-2,3-dimethyl-7-hydroxychromone (m.p. 243°) [6381].

m.p. 158° [6369], 151–152° [6380,6383], 148° [6382], 73–74° [6381].
One of the reported melting points is obviously wrong.
^{1}H NMR [6380], IR [6380]; TLC [6380].

1-(5-Chloro-2-hydroxy-3-nitrophenyl)-1-propanone

[90537-41-0] $C_9H_8ClNO_4$ mol.wt. 229.62



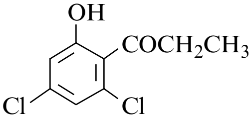
Synthesis

- Preparation by reaction of nitric acid (d = 1.52) with 5-chloro-2-hydroxypropiophenone in concentrated sulfuric acid (d = 1.80) between −2° and 0° (72%) [6367].

m.p. 98° [6367].

1-(2,4-Dichloro-6-hydroxyphenyl)-1-propanone

[22362-84-1] $C_9H_8Cl_2O_2$ mol.wt. 219.07



Syntheses

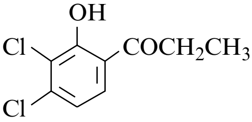
- Preparation by Fries rearrangement of 3,5-dichlorophenyl propionate with aluminium chloride [6351].
- Also refer to: [6384].

m.p. 85° [6351];
^1H NMR (Sadtler: standard n° 9474M), IR (Sadtler: standard n°38940), UV [6359];
TLC [6353].

Oxime [75408-94-5] $C_9H_9Cl_2NO_2$ mol.wt. 234.58 [6384].

1-(3,4-Dichloro-2-hydroxyphenyl)-1-propanone

[777067-72-8] $C_9H_8Cl_2O_2$ mol.wt. 219.07



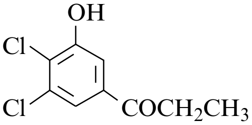
Synthesis

- Obtained by Fries rearrangement of 2,3-dichlorophenyl propionate with aluminium chloride at 120–130° for 2 h (38%) [6385].

m.p. 97–98° [6385]; ^1H NMR [6385].

1-(3,4-Dichloro-5-hydroxyphenyl)-1-propanone

[113730-39-5] $C_9H_8Cl_2O_2$ mol.wt. 219.07



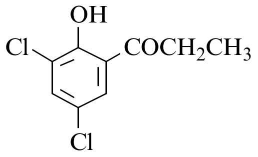
Synthesis

- Preparation by demethylation of 3,4-dichloro-5-methoxy-propiophenone (m.p. 85°) with 30% hydrogen bromide in acetic acid at reflux for 48 h (88%) [6386].

m.p. 133–134° [6386]; ^1H NMR [6386].

1-(3,5-Dichloro-2-hydroxyphenyl)-1-propanone

[18430-74-5] $C_9H_8Cl_2O_2$ mol.wt. 219.07



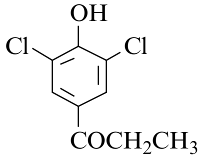
Syntheses

- Preparation by Fries rearrangement of 2,4-dichlorophenyl propionate (b.p.$_{14}$ 148°) [6387] with aluminium chloride [6351,6388], at 155°, following Hartung's procedure [6389], (90%) [6390] or at 165–175° for 40 min (76%) [6387].
- Preparation by Friedel–Crafts reaction of propionyl chloride with 2,4-dichlorophenol in the presence of aluminium chloride [6391].
- Also refer to: [6392–6396].

m.p. 117° [6351], 116.5–117.5° [6390], 115–116° [6387];
^{1}H NMR (Sadtler: standard n° 9698M) [6397], IR (Sadtler: standard n° 38937) [6398],
UV [6359,6397,6399,6400], fluorescence spectrum [6397];
TLC [6353,6358].

1-(3,5-Dichloro-4-hydroxyphenyl)-1-propanone

[51335-40-1] $C_9H_8Cl_2O_2$ mol.wt. 219.07



Syntheses

- Preparation by Fries rearrangement of 2,6-dichlorophenyl propionate (b.p.$_{0.5}$ 113–115°) with aluminium chloride at 134–145° for 2 h (87%) [6401].

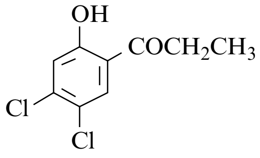
N.B.: Not obtained by Fries rearrangement of the same ester with boron trifluoride instead of aluminium chloride at 195–210° for 3 h.

- Also obtained by heating a suspension of sodium propionate and 2,6-dichlorophenol in neat triflic acid [6402].
- Also obtained by demethylation of 3,5-dichloro-4-methoxypropiophenone (m.p. 90°) with pyridinium chloride at reflux for 10 min [6379].
- Also obtained by chlorination of p-hydroxypropiophenone [6403].
- Also refer to: [6404–6408].

m.p. 118° [6379], 110–111° [6401].

1-(4,5-Dichloro-2-hydroxyphenyl)-1-propanone

[288401-09-2] $C_9H_8Cl_2O_2$ mol.wt. 219.07



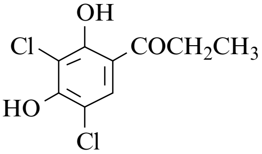
Synthesis

- Preparation by Fries rearrangement of 3,4-dichlorophenyl propionate with aluminium chloride at 160° for 3 h [6409].

white solid [6409];
^{1}H NMR [6409], ^{13}C NMR [6409], MS [6409].

1-(3,5-Dichloro-2,4-dihydroxyphenyl)-1-propanone

[66021-81-6] $C_9H_8Cl_2O_3$ mol.wt. 235.07



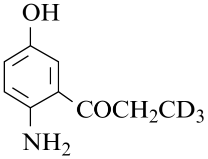
Syntheses

- Preparation by chlorination of respropiophenone [6410] in 80% acetic acid [6383].

m.p. 146–147° [6383], 142–145° [6410].

1-(2-Amino-5-hydroxyphenyl)-1-propanone-*3,3,3-d*$_3$

[718613-15-1] $C_9H_8D_3NO_2$ mol.wt. 168.21

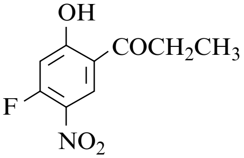


Synthesis

- Preparation [6411].

1-(4-Fluoro-2-hydroxy-5-nitrophenyl)-1-propanone

[123450-85-1] $C_9H_8FNO_4$ mol.wt. 213.17

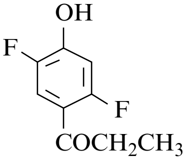


Synthesis

- Obtained by reaction of fuming nitric acid with 4-fluoro-2-hydroxypropiophenone in acetic acid at 0° for 30 min [6412].

1-(2,5-Difluoro-4-hydroxyphenyl)-1-propanone

[188435-69-0] $C_9H_8F_2O_2$ mol.wt. 186.16

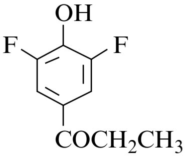


Synthesis

- Refer to: [6413].

1-(3,5-Difluoro-4-hydroxyphenyl)-1-propanone

[178374-78-2] $C_9H_8F_2O_2$ mol.wt. 186.16



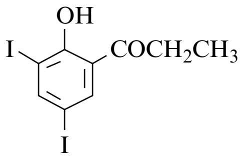
Syntheses

- Obtained by Fries rearrangement of 2,6-difluorophenyl propionate (b.p.$_5$ 61–65°) with aluminium chloride for 2 h at 90–95° under nitrogen (50%) [6414].
- Also refer to: [6405,6406,6415–6417].

m.p. 127–129° [6414]; ^{1}H NMR [6414].

1-(2-Hydroxy-3,5-diiodophenyl)-1-propanone

[32559-06-1] $C_9H_8I_2O_2$ mol.wt. 401.97



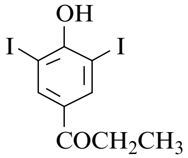
Syntheses

– Preparation by reaction of iodine with o-hydroxypropiophenone in 10% aqueous sodium carbonate at r.t. for some hours (75%) [6418,6419].

m.p. 110–112° [6418].

1-(4-Hydroxy-3,5-diiodophenyl)-1-propanone

[7091-08-9] $C_9H_8I_2O_2$ mol.wt. 401.97



Syntheses

– Preparation by reaction of iodine with 4-hydroxypropiophenone,

- in the presence of an aqueous sodium carbonate solution at r.t. (97%) [6419];
- in ammonia in the presence of potassium iodide at r.t. overnight (94%) [6420];
- in ethanol in the presence of mercuric oxide yellow [6362].

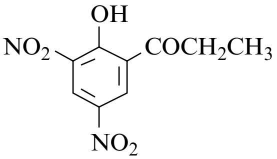
– Also refer to: [6363,6421].

Note: Effect on pituitary-thyroid relation [6421].

m.p. 124° [6362], 122° [6420]; UV [6422]; TLC [6422].

1-(2-Hydroxy-3,5-dinitrophenyl)-1-propanone

[91211-02-8] $C_9H_8N_2O_6$ mol.wt. 240.17



Syntheses

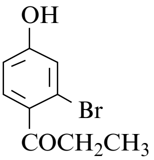
– Obtained (by-product) by reaction of fuming nitric acid (d = 1.52) with o-hydroxypropiophenone in concentrated sulfuric acid at 0° for 2 h [6423,6424].

– Also obtained by reaction of nitric acid with 2-hydroxy-5-nitropropiophenone in acetic acid, first at 0°, then at r.t. [6425].

m.p. 115–117° [6423,6424]; IR [6425]; HPLC [6426]; TLC [6426].

1-(2-Bromo-4-hydroxyphenyl)-1-propanone

[18430-73-4] $C_9H_9BrO_2$ mol.wt. 229.07



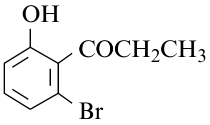
Synthesis

– Obtained by Fries rearrangement of m-bromophenyl propionate with aluminium chloride at 50° for 3 h (16%) [6360].

m.p. 101–102° [6360];
IR (Sadtler: standard n° 38931) [6360], UV [6357,6360]; TLC [6353,6358].

1-(2-Bromo-6-hydroxyphenyl)-1-propanone

$C_9H_9BrO_2$ mol.wt. 229.07



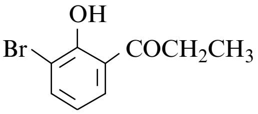
Synthesis

– Obtained by alkaline degradation of 5-bromo-2,3-dimethyl-chromone (m.p. 163°) [6427].

m.p. 82° [6427].

1-(3-Bromo-2-hydroxyphenyl)-1-propanone

[17764-91-9] $C_9H_9BrO_2$ mol.wt. 229.07



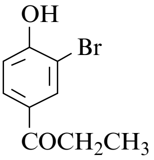
Synthesis

– Obtained by Fries rearrangement of o-bromophenyl propionate with titanium tetrachloride at 110° for 30 min (29%) [6360].

m.p. 55–56° [6360];
^{1}H NMR (Sadtler: standard n° 9531M), IR (Sadtler: standard n° 38930) [6360], UV [6359,6360]; TLC [6353,6358].

1-(3-Bromo-4-hydroxyphenyl)-1-propanone

[18430-72-3] $C_9H_9BrO_2$ mol.wt. 229.07



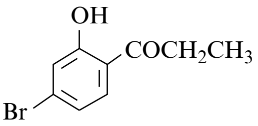
Syntheses

– Preparation by Fries rearrangement of o-bromophenyl propionate with aluminium chloride at 100° for 2 h (95%) [6360] or 120° for 20–30 min [6428].
– Also obtained (by-product) by Friedel–Crafts acylation of o-bromoanisole or o-bromophenetole with propionyl chloride in the presence of aluminium chloride [6429,6430].
– Also refer to: [6431,6432].

m.p. 132° [6360], 130° [6428], 125° [6429,6430];
IR (Sadtler: standard n° 38929) [6360], UV [6357,6360]; TLC [6353,6358].
USE: Fungicide [6432].

1-(4-Bromo-2-hydroxyphenyl)-1-propanone

[17764-92-0] $C_9H_9BrO_2$ mol.wt. 229.07



Syntheses

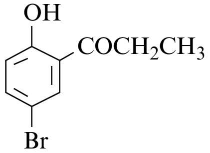
– Preparation by Friedel–Crafts acylation of m-bromophenol with propionyl chloride in the presence of aluminium chloride in refluxing ethylene dichloride for 2 h (78%) [6433].
– Also obtained by Fries rearrangement of 3-bromophenyl propionate in the presence of aluminium chloride at 50° for 3 h (47%) [6360].

- Also refer to: [6434].

b.p.$_{17}$ 156–158° [6360];
m.p. 61–62° [6360], 49–52° [6433]. One of the reported melting points is obviously wrong.
^{1}H NMR (Sadtler: standard n° 9532M), [6433], ^{13}C NMR [6433],
IR (Sadtler: standard n° 38932), [6360,6433], UV [6359,6360];
TLC [6353,6358].

1-(5-Bromo-2-hydroxyphenyl)-1-propanone

[17764-93-1] $C_9H_9BrO_2$ mol.wt. 229.07



Syntheses

- Preparation by Fries rearrangement of 4-bromophenyl propionate with aluminium chloride [6435] at 165° for 1 h (97%) [6360], at 150–160° for 30 min [6436] or at 120° for 30–40 min (80%) [6428].
- Preparation by bromination of o-hydroxypropiophenone with bromine in 80% aqueous acetic acid at r.t. (66%) [6369].
- Also obtained by acylation of p-bromophenol with propionic acid in the presence of boron trifluoride in a sealed tube at 120° for 1 h (59%) [6437].
- Also refer to: [6364,6438–6445].

b.p.$_{14}$ 143–153° [6437], b.p.$_{21}$ 154–156° [6360];
m.p. 78° [6428], 77° [6369], 76° [6360,6436], 75° [6437];
^{1}H NMR (Sadtler: standard n° 9533M); IR [6360], UV [6359,6360];
TLC [6353,6358].

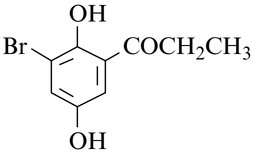
Notes: Acidity of methylene proton [6446]; C-deuteration [6447].

Thiosemicarbazone $C_{10}H_{12}BrN_3OS$ mol.wt. 302.19
(m.p. 220°) [6364].

USE: Fungicide [6364].
BIOLOGICAL ACTIVITY: Antituberculotic [6364].

1-(3-Bromo-2,5-dihydroxyphenyl)-1-propanone

[139590-48-0] $C_9H_9BrO_3$ mol.wt. 245.07

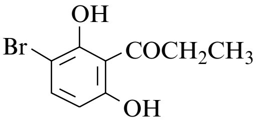


Syntheses

- Refer to: [6448,6449].

1-(3-Bromo-2,6-dihydroxyphenyl)-1-propanone

[99548-74-0] $C_9H_9BrO_3$ mol.wt. 245.07



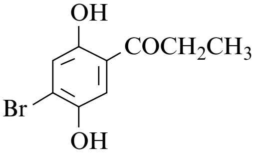
Synthesis

- Obtained by decarboxylation of 5-bromo-2,4-dihydroxy-3-propionylbenzoic acid (36%) [6450].

m.p. 115° [6450].

1-(4-Bromo-2,5-dihydroxyphenyl)-1-propanone

[52376-21-3] $C_9H_9BrO_3$ mol.wt. 245.07



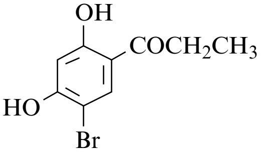
Synthesis

- Obtained by Fries rearrangement of 2-bromohydroquinone dipropionate (m.p. 58–59°) with aluminium chloride at 170–180° for 2 h [6451].

m.p. 110–111° [6451].

1-(5-Bromo-2,4-dihydroxyphenyl)-1-propanone

[157732-52-0] $C_9H_9BrO_3$ mol.wt. 245.07

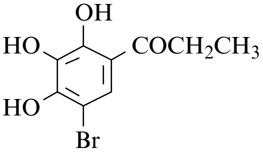


Syntheses

- Refer to: [6452] (compound **1h**), [6453] (compound **1c**).

1-(5-Bromo-2,3,4-trihydroxyphenyl)-1-propanone

$C_9H_9BrO_4$ mol.wt. 261.07



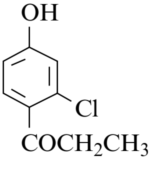
Synthesis

- Preparation by bromination of 4-propionylpyrogallol in acetic acid [6454].

m.p. 131° [6454].

1-(2-Chloro-4-hydroxyphenyl)-1-propanone

[2892-21-9] $C_9H_9ClO_2$ mol.wt. 184.62



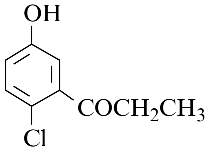
Syntheses

- Obtained by Fries rearrangement of m-chlorophenyl propionate with aluminium chloride for 3 h at 50° (19%) [6355,6356].
- Also obtained by demethylation of 2-chloro-4-methoxypropiophenone with aluminium chloride in refluxing heptane for 3 h [6455].
- Also refer to: [6456].

m.p. 93° [6355,6356], 92–93° [6455];
IR (Sadtler: standard n° 37036) [6355,6356], UV [6356,6357,6457];
TLC [6353,6358].

1-(2-Chloro-5-hydroxyphenyl)-1-propanone

[1127-96-4] $C_9H_9ClO_2$ mol.wt. 184.62



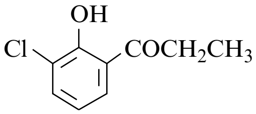
Syntheses

- Preparation by diazotization of 5-amino-2-chloropropiophenone (b.p.$_{0.5}$ 143–162°), followed by hydrolysis of the diazonium salt obtained [6458–6460], (27%) [6455,6461].
- Also refer to: [6456,6462].

b.p.$_{0.4}$ 135–140° [6459], b.p.$_{0.5}$ 135–140° [6455,6458,6461–6463].

1-(3-Chloro-2-hydroxyphenyl)-1-propanone

[938-67-0] $C_9H_9ClO_2$ mol.wt. 184.62



Syntheses

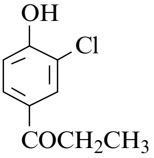
- Obtained by Fries rearrangement of o-chlorophenyl propionate,
  - with aluminium chloride at 110° for 2 h (35%) [6464], (25%) [6465];
  - with titanium tetrachloride at 100° for 7 h (17%) [6355,6356,6466];
  - with zirconium chloride in o-dichlorobenzene over 1 h at 120° [6467].
- Also obtained by isomerization of 3-chloro-4-hydroxypropiophenone with aluminium chloride at 180–200° for 1 h (24%) [6466,6468].
- Also obtained by tert-butyl group elimination of 5-tert-butyl-3-chloro-2-hydroxypropiophenone with aluminium chloride at 190° for 15 min (80%) [6466].
- Also refer to: [6469–6471].

m.p. 72° [6464], 43–44° [6355,6356], 41–42° [6465]. One of the reported melting points is obviously wrong.
^{1}H NMR (Sadtler: standard n° 8375M) [6472],
IR (Sadtler: standard n° 37031) [6355,6356,6465],
UV [6356,6359,6457]; TLC [6353,6358].

USE: Preparation of tetrahydronaphthalines as antiinflammatory [6469].

1-(3-Chloro-4-hydroxyphenyl)-1-propanone

[2892-27-5] $C_9H_9ClO_2$ mol.wt. 184.62



Syntheses

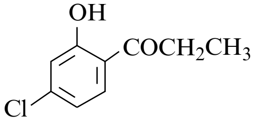
- Preparation by Fries rearrangement of o-chlorophenyl propionate with aluminium chloride [6428], at 165° for 1 h (98%) [6355,6356] or at 110° for 2 h (63%) [6465].

- Preparation by demethylation of 3-chloro-4-methoxy-propiophenone (m.p. 88°) [6429] (m.p. 89–90°) [6377] with refluxing pyridinium chloride for 15 min (75%) [6362].
- Preparation by diazotization of 3-amino-4-hydroxypropiophenone, followed by decomposition of the diazonium salt obtained (52%) [6473].
- Preparation by chlorination of p-hydroxypropiophenone with chlorine in cooled acetic acid (57%) [6362].
- Also refer to: [6363,6471,6474–6476].

m.p. 118–119° [6355,6356], 114.5–115.5° [6465], 114° [6362], 80° [6428], 79° [6473]. Some reported melting points are obviously wrong.
^{1}H NMR (Sadtler: standard n° 8376M), IR (Sadtler: standard n° 37032) [6355,6356], UV [6356,6357,6457]; TLC [6353,6358].

1-(4-Chloro-2-hydroxyphenyl)-1-propanone

[1127-97-5] $C_9H_9ClO_2$ mol.wt. 184.62



Syntheses

- Preparation by Fries rearrangement of m-chlorophenyl propionate (b.p.$_{0.8}$ 90°) [6477] with aluminium chloride,
  - without solvent at 100° for 2 h (92%) [6355], at 140° for 2 h (89%) [6477], at 140–150° for 3 h [6478], (77%) [6479], at 130° for 2 h (77%) [6480], at 165° for 1 h (72%) [6355], at 50° for 3 h (55%) [6356] or at 30–40° (53%) [6461];
  - in nitrobenzene at 25° for 6 h (87%) [6480].
- Preparation by Fries rearrangement of m-chlorophenyl propionate with titanium tetrachloride at 100° for 2 h (75%) [6355].
- Also obtained by Friedel–Crafts reaction of propionyl chloride with m-chlorophenol in the presence of aluminium chloride [6481].
- Also obtained by isomerization of 2-chloro-4-hydroxypropiophenone on heating with aluminium chloride between 165° and 200° (30–70%) [6482].
- Also refer to: [6483] (compound **1e**), [6484] (compound **1b**) and [6485] (compound **1a**) and [6407] (*12i*).

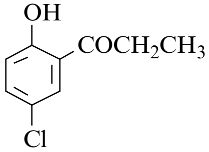
b.p.$_1$ 103° [6479], b.p.$_{60}$ 130–140° [6460,6461,6463];
colourless prisms [6477];
m.p. 49–51° [6355,6356], 49° [6479,6480], 45–47° [6459–6461,6463];
^{1}H NMR (Sadtler: standard n° 8380M) [6477,6479],
IR (Sadtler: standard n° 37035) [6355,6356], [6477,6479],
UV [6356,6359,6457]; TLC [6353,6358].

Note: C-Deuteration [6447].

Oxime [75408-96-7] $C_9H_{10}ClNO_2$ mol.wt. 199.64 [6384].

1-(5-Chloro-2-hydroxyphenyl)-1-propanone

[2892-16-2] $C_9H_9ClO_2$ mol.wt. 184.62



Syntheses

- Preparation by Fries rearrangement of 4-chlorophenyl propionate without solvent,
  - with aluminium chloride [6486] according to the procedure [6487], at 130–140° for 90 min (almost quantitative yield) [6488,6489], at 140° for 2 h [6477], at 155° [6490], at 165° for 1 h (95%) [6355,6356] or at 170° for 45 min (87%) [6491] or for 1–2 h (80–96%) [6492];
  - with titanium tetrachloride at 50° for 3 h (59%) [6355,6356].
- Preparation by acylation of p-chlorophenol with propionic acid in the presence of boron trifluoride in a sealed tube at 150° for 5 h (82%) [6437].
- Also refer to: [6364,6493–6499].

b.p.$_9$ 124–126° [6355], b.p.$_{15}$ 135–136° [6437];
m.p. 62–63° [6355], 59.7° [6490], 59–60° [6437], 56.5–57.5° [6489];
^{1}H NMR [6397,6500], IR (Sadtler: standard n° 8981) [6355,6356,6398],
UV [6356,6359,6397,6399,6400,6457], fluorescence spectrum [6397];
ionization [6501]; TLC [6353,6358].

Note: C-Deuteration [6447].
BIOLOGICAL ACTIVITY: Antimicrobial [6486].

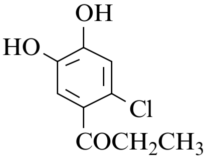
Oxime [29725-94-8] $C_9H_{10}ClNO_2$ mol.wt. 199.64 [6502].

Thiosemicarbazone $C_{10}H_{12}ClN_3OS$ mol.wt. 257.74 (m.p. 218°) [6364].

USE: Fungicide [6364].
BIOLOGICAL ACTIVITY: Antituberculotic [6364].

1-(2-Chloro-4,5-dihydroxyphenyl)-1-propanone

[25069-71-0] $C_9H_9ClO_3$ mol.wt. 200.62



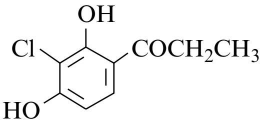
Synthesis

- Refer to: [6503].

m.p. 100° [6503].

1-(3-Chloro-2,4-dihydroxyphenyl)-1-propanone

[888968-47-6] $C_9H_9ClO_3$ mol.wt. 200.62

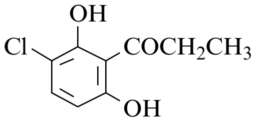


Syntheses

- Refer to: [6504–6506].

1-(3-Chloro-2,6-dihydroxyphenyl)-1-propanone

[99055-11-5] $C_9H_9ClO_3$ mol.wt. 200.62



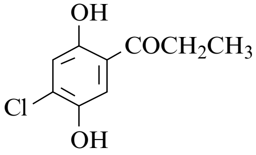
Syntheses

- Obtained by decarboxylation of 5-chloro-2,4-dihydroxy-3-propionylbenzoic acid [6450].
- Also obtained by hydrolysis of 6-chloro-7-hydroxy-4-methyl-8-propionylcoumarin [6450].

m.p. 107° [6450].

1-(4-Chloro-2,5-dihydroxyphenyl)-1-propanone

[75859-14-2] $C_9H_9ClO_3$ mol.wt. 200.62



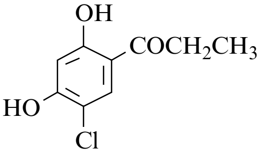
Syntheses

- Obtained by reaction of propionic acid with 2-chloro-hydroquinone [6507] by known methods [6508–6510].

m.p. 127–129° [6507].

1-(5-Chloro-2,4-dihydroxyphenyl)-1-propanone

[85131-64-2] $C_9H_9ClO_3$ mol.wt. 200.62



Syntheses

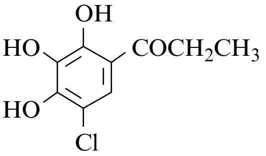
- Obtained by reaction of propionic acid with 4-chloro-resorcinol in the presence of zinc chloride at reflux for 3 min (Nencki reaction) (29%) [6511].
- Also refer to: [6453,6512].

m.p. 90° [6511].
Note: Ionization [6513].

Oxime [82667-80-9] $C_9H_{10}ClNO_3$ mol.wt. 215.63.

USE: In iron spectrophotometric determination in pharmaceuticals [6512,6514]; in spectrometric determination of palladium [6515].

1-(5-Chloro-2,3,4-trihydroxyphenyl)-1-propanone



$C_9H_9ClO_4$ mol.wt. 216.62

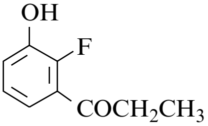
Synthesis

- Preparation by chlorination of 4-propionylpyrogallol in acetic acid [6454].

m.p. 126° [6454].

1-(2-Fluoro-3-hydroxyphenyl)-1-propanone

[164072-22-4] $C_9H_9FO_2$ mol.wt. 168.17

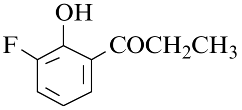


Synthesis

- Refer to: [6516].

1-(3-Fluoro-2-hydroxyphenyl)-1-propanone

[879339-86-3] $C_9H_9FO_2$ mol.wt. 168.17



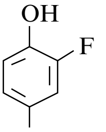
Syntheses

- Refer to: [6469,6517].

USE: Preparation of heteroaryl alkylidenetetrahydro-naphthalenamines as antiinflammatories [6517]; preparation of tetrahydronaphthalines as antiinflammatory [6469].

1-(3-Fluoro-4-hydroxyphenyl)-1-propanone

[586-16-3] $C_9H_9FO_2$ mol.wt. 168.17



Syntheses

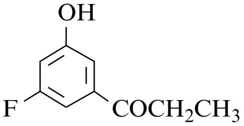
- Obtained by demethylation of 3-fluoro-4-methoxy-propiophenone with pyridinium chloride at reflux for 15 min [6362], (59%) [6518].
- Also refer to: [6363,6405,6406,6517,6519].

m.p. 109–110° [6518].

USE: Preparation of heteroaryl alkylidenetetrahydronaphthalenamines as antiinflammatories [6517].

1-(3-Fluoro-5-hydroxyphenyl)-1-propanone

[179113-58-7] $C_9H_9FO_2$ mol.wt. 168.17

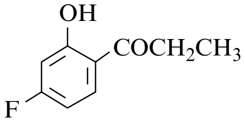


Synthesis

- Refer to: [6520].

1-(4-Fluoro-2-hydroxyphenyl)-1-propanone

[247230-94-0] $C_9H_9FO_2$ mol.wt. 168.17



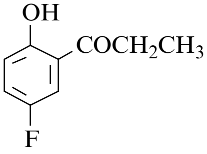
Syntheses

- Preparation by Fries rearrangement of 3-fluorophenyl propionate [6521] with aluminium chloride at 150° for 10 min (88%) [6522,6523].
- Also refer to: [6524–6526].

^{1}H NMR [6522,6523].

1-(5-Fluoro-2-hydroxyphenyl)-1-propanone

[443-09-4] $C_9H_9FO_2$ mol.wt. 168.17



Syntheses

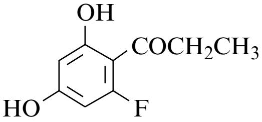
- Preparation by Fries rearrangement of p-fluorophenyl propionate (b.p.$_{18–20}$ 118–120°) [6527] with aluminium chloride at 150° for 1 h (80–81%) [6351,6528] or at reflux for 30 min (57%) [6527].
- Also obtained by demethylation of 5-fluoro-2-methoxy-propiophenone (b.p.$_{13}$ 132°) with refluxing pyridinium chloride for 15 min (76%) [6365].

b.p.$_{6–7}$ 97–99° [6527], b.p.$_{13}$ 111–112° [6365], b.p.$_{13}$ 113° [6351], b.p.$_{22}$ 117–121° [6528];
m.p. 36–37° [6351], 32° [6365], 30.5° [6528];
^{1}H NMR (Sadtler: standard n° 9472M), IR (Sadtler: standard n° 38938), UV [6359];
TLC [6353,6358].

Notes: Deuteration [6447]; acidity of methylene proton [6446].

1-(2-Fluoro-4,6-dihydroxyphenyl)-1-propanone

[864866-62-6] $C_9H_9FO_3$ mol.wt. 184.17

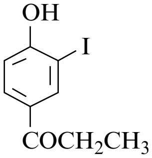


Synthesis

- Refer to: [6529].

1-(4-Hydroxy-3-iodophenyl)-1-propanone

$C_9H_9IO_2$ mol.wt. 276.07



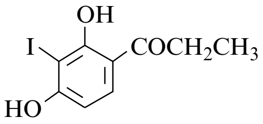
Synthesis

- Refer to: [6432] (compound **F/54**).

USE: Fungicide [6432].

1-(2,4-Dihydroxy-3-iodophenyl)-1-propanone

[117844-79-8] $C_9H_9IO_3$ mol.wt. 292.07



Synthesis

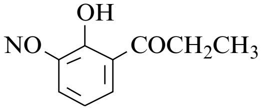
– Obtained by adding an aqueous solution of iodine and periodic acid to an ethanolic solution of respropiophenone, then stirring the mixture for 2 h (49%) [6530].

m.p. 97–98° [6530]; ^{1}H NMR [6530].

1-(2-Hydroxy-3-nitrosophenyl)-1-propanone

2-Propionyl-6-nitrosophenol

$C_9H_9NO_3$ mol.wt. 179.18



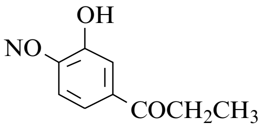
Synthesis

– Preparation from o-propionylphenol [6531].

1-(3-Hydroxy-4-nitrosophenyl)-1-propanone

3-Propionyl-6-nitrosophenol

$C_9H_9NO_3$ mol.wt. 179.18



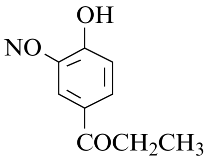
Synthesis

– Preparation from propiophenone (no precision) [6531].

1-(4-Hydroxy-3-nitrosophenyl)-1-propanone

4-Propionyl-2-nitrosophenol

$C_9H_9NO_3$ mol.wt. 179.18

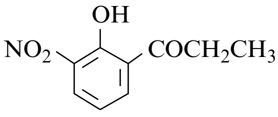


Synthesis

– Preparation from p-propionylphenol [6531].

1-(2-Hydroxy-3-nitrophenyl)-1-propanone

[91991-98-9] $C_9H_9NO_4$ mol.wt. 195.17



Syntheses

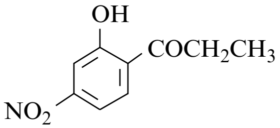
– Obtained by degradation of 3-methyl-8-nitrochromone (m.p. 148–150°) with boiling 5% potassium hydroxide for 2 h (84%) [6423].

- Also obtained by reaction of fuming nitric acid (d = 1.52) with o-hydroxypropiophenone in concentrated sulfuric acid at 0° for 2 h (38%) [6423], (8%) [6532].
- Also refer to: [6424,6533,6534].

m.p. 97–98° [6423], 95–97° [6423], 92–94° [6532];
TLC [6426]; HPLC [6426]; conformation and H bonds [6425].

1-(2-Hydroxy-4-nitrophenyl)-1-propanone

[79925-34-1] $C_9H_9NO_4$ mol.wt. 195.17



Syntheses

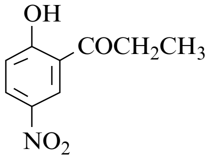
- Obtained by Fries rearrangement of 3-nitrophenyl propionate* (m.p. 49°) [6535] with aluminium chloride [6536], in nitrobenzene at 125–130° for 25 h (20%) or without solvent at 125° for 20 h (14%) [6537].
- Also obtained by Friedel–Crafts acylation of 3-nitrophenol with propionyl chloride in the presence of aluminium chloride [6536].
- Also refer to: [6483] (compound **1f**), [6446].

m.p. 68–69° [6537].

Notes: C-Deuteration [6447]; *temperature of decomposition of the mixture $AlCl_3$/ester (2:1) 183° [6535].

1-(2-Hydroxy-5-nitrophenyl)-1-propanone

[55805-95-3] $C_9H_9NO_4$ mol.wt. 195.17



Syntheses

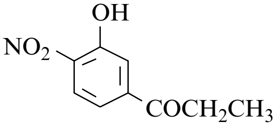
- Preparation by Friedel–Crafts acylation of p-nitrophenol with propionyl chloride in nitrobenzene in the presence of aluminium chloride (36%) [6538].
- Also obtained by Fries rearrangement of p-nitrophenyl propionate (m.p. 63–64°) [6535] in nitrobenzene in the presence of aluminium chloride at 125° for 5 h (23%) [6539].
- Preparation by reaction of boron trichloride with 2-methoxy-5-nitropropiophenone in methylene chloride, first at −78°, then at r.t. for 14 h (92%) [6409].
- Also obtained by nitration of o-hydroxypropiophenone (23%) [6532], in acetic acid with fuming nitric acid, first at 0°, then at 60° [6540].
- Also obtained by alkaline hydrolysis of 3-methyl-6-nitrochromone (m.p. 147–148°) (85%) [6540].
- Also refer to: [6534,6541].

white solid [6409];
m.p. 114° [6437], 99° [6540], 94.5–95.0° [6538], 93–94° [6539], 91–93° [6532]. One of the reported melting points is obviously wrong.
^{1}H NMR [6409,6540], ^{13}C NMR [6409], IR [6538], MS [6409];
TLC [6426]; HPLC [6426].

Notes: Temperature of decomposition of the mixture $AlCl_3$/ester (2:1) 135° [6535]; C-deuteration [6447]; chelates with cobalt (II), copper (II), nickel (II), and zinc (II) [6542,6543]; acidity of methylene proton [6446].

Oxime [58402-96-3] $C_9H_{10}N_2O_4$ mol.wt. 210.18 [6544].

1-(3-Hydroxy-4-nitrophenyl)-1-propanone



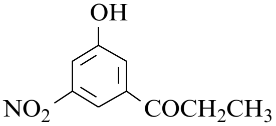
$C_9H_9NO_4$ mol.wt. 195.17

Synthesis

- Obtained by reaction of 3-hydroxypropiophenone with nitric acid in acetic acid at 70° [6545].

1-(3-Hydroxy-5-nitrophenyl)-1-propanone

[193693-96-8]



$C_9H_9NO_4$ mol.wt. 195.17

Synthesis

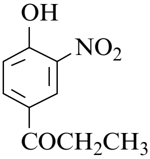
- Obtained in two steps: First, a mixture of 3-hydroxy-propiophenone and dysprosium nitrate in ethyl acetate was refluxed (85–105°) for 30 min. Then, the isolated intermediate $(C_9H_8NO_4)_3Dy$ (60%) was dissolved in 6 N hydrochloric acid (52%) [6546].

m.p. 130–132° [6546]; MS [6546].

Dysprosium salt [193693-93-5] [6546].

1-(4-Hydroxy-3-nitrophenyl)-1-propanone

[50916-44-4]



$C_9H_9NO_4$ mol.wt. 195.17

Syntheses

- Preparation by treatment of 4-hydroxypropiophenone with fuming nitric acid [6473,6547] or with nitric acid in concentrated sulfuric acid between −2° and 0° (80%) [6367].
- Preparation by Friedel–Crafts acylation of o-nitrophenol with propionyl chloride in nitrobenzene in the presence of aluminium chloride at 55–60° for 2.5 h, then at r.t. overnight [6547,6548], (41%) [6549].
- Also obtained (poor yield) by Fries rearrangement of 2-nitrophenyl propionate in nitrobenzene with aluminium chloride at 90° for 8 h, then at r.t. overnight [6539].
- Also obtained by reaction of concentrated nitric acid with 2,2′-dihydroxy-5,5′-dipropionyldiphenyl sulfide (m.p. 103°) in concentrated sulfuric acid at r.t. overnight [6550].
- Also refer to: [6534,6551].

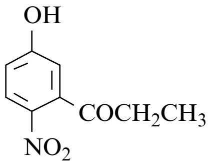
m.p. 67° [6367], 66° [6473], 65.4–66.2° [6549], 65–66° [6548], 58–61° [6539], 58–60° [6550].

USE: Fungicide [6552].

BIOLOGICAL ACTIVITY: Sympathomimetic [6548].

1-(5-Hydroxy-2-nitrophenyl)-1-propanone

[453518-19-9] $C_9H_9NO_4$ mol.wt. 195.17

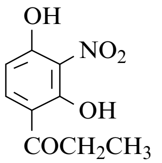


Syntheses

- Obtained by reaction of nitric acid with 3-hydroxypropiophenone in acetic acid at 70° (30–35%) [6545].
- Also refer to: [6553].

1-(2,4-Dihydroxy-3-nitrophenyl)-1-propanone

[103441-87-8] $C_9H_9NO_5$ mol.wt. 211.17



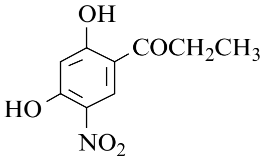
Synthesis

- Obtained by Friedel–Crafts acylation of 2-nitroresorcinol with propionic anhydride (1 mol) in the presence of aluminium chloride (3.3 mol) in nitrobenzene at 120–130° for 3 h [6554].

m.p. 83° [6554].

1-(2,4-Dihydroxy-5-nitrophenyl)-1-propanone

[63411-89-2] $C_9H_9NO_5$ mol.wt. 211.17



Syntheses

- Preparation by adding respropiophenone in small portions with constant stirring to nitric acid (d = 1.42) cooled in an ice bath (<10°), (77%) [6555], (43%) [6380].
- Preparation by reaction of fuming nitric acid with respropiophenone in glacial acetic acid at 0–5° [6556].
- Also obtained by treatment of 3,3′-dipropionyl-4,4′,6,6′-tetrahydroxydiphenyl thioether (m.p. 161–162°) with nitric acid (d = 1.4) at 5° for 2 h [6368].
- Also refer to: [6452] (compound **1g**).

m.p. 137–138° [6380], 131° [6368,6555];
^{1}H NMR [6380], IR [6380]; TLC [6380].

Diacetate $C_{13}H_{13}NO_7$ mol.wt. 295.25 (m.p. 89°) [6555].

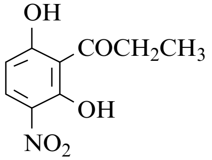
- Preparation by reaction of acetic anhydride with 2,4-dihydroxy-5-nitropropiophenone in the presence of sodium acetate [6555].

Oxime [473807-71-5] $C_9H_{10}N_2O_5$ mol.wt. 226.18.

USE: As a gravimetric reagent for Cu (II) determination [6557] and Ni (II) and Pd (II) determination [6558].

1-(2,6-Dihydroxy-3-nitrophenyl)-1-propanone

[103440-67-1] $C_9H_9NO_5$ mol.wt. 211.17



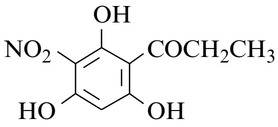
Syntheses

- Obtained by reaction of propionic anhydride with 4-nitroresorcinol in nitrobenzene in the presence of aluminium chloride, first at 0°, then heating in a steam bath for 3 h [6559].
- Also obtained by reaction of nitric acid (d = 1.42) with 2,6-dihydroxypropiophenone at 0° for 10 min [6559].
- Also obtained by Fries rearrangement of 4-nitroresorcinol dipropionate with aluminium chloride (3.3 equiv) in nitrobenzene, first at 95–100° for 2 h, then at r.t. for 72 h (42%) or without solvent at 100° for 3 h (25%) [6560].

m.p. 115–116° [6560], 114–115° [6559].

1-(2,4,6-Trihydroxy-3-nitrophenyl)-1-propanone

[119691-92-8] $C_9H_9NO_6$ mol.wt. 227.17



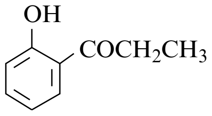
Synthesis

- Preparation by addition of a concentrated sulfuric acid and fuming nitric acid mixture to a solution of 2,4,6-trihydroxy-propiophenone in concentrated sulfuric acid and hexane mixture under cooling with an ice bath (70–80%) [6561].

m.p. 108–109° [6561]; ^{1}H NMR [6561], IR [6561], MS [6561].

1-(2-Hydroxyphenyl)-1-propanone

[610-99-1] $C_9H_{10}O_2$ mol.wt. 150.18



Syntheses

- Obtained by Fries rearrangement of phenyl propionate,
  - with aluminium chloride in refluxing carbon disulfide [6562], then at 130–150° for 2–3 h after solvent elimination (32–35%) [6389,6563,6564], (43%) [6565];
  - with aluminium chloride in chlorobenzene using microwave irradiation for 3 min at 106° (28%) [6566];
  - with aluminium chloride in nitrobenzene at 50° for 18 h (16%) [6567] or at 20° for 48 h (10%) [6568];
  - with aluminium chloride in nitromethane at 20° for 7 days (20%) [6569,6570];
  - with aluminium chloride in heptane at 80–90° for 7 h (53%) or in tetrachloroethane at 95° for 5 h (43%) [6571];
  - with aluminium chloride without solvent at 250° for 5 min or at 180–200° for 15 min (76–78%) [6355], at 140–160° (32–35%) [6572,6573], at 140° (48%) [6574], at 120° (30%) [6575] or at 50° for 10 h (30%) [6355,6356,6576].

N.B.: Industrial manufacturing (S.P.C.A. in 1956), with aluminium chloride without solvent at 180–200° for 15 min (62%) [6351].

  - with zirconium chloride in o-dichlorobenzene at 120° for 3 h (83%) [6467];
  - with titanium tetrachloride in o-dichlorobenzene at 150° for 1 h (62%) [6577], in nitromethane at 20° for 7 days (13%) [6569] or without solvent at 50° for 10 h (30%) [6355,6356,6576];
  - with titanium tetrabromide in o-dichlorobenzene at 150° for 1 h (60%) [6577];
  - with stannic chloride at 50° for 3 h (25%) [6355,6356];
  - with boron trifluoride at 120–135° for 15 min (15%) [6355,6356];
  - with zinc chloride at 180–200° for 15 min (10%) [6355];
  - with polyphosphoric acid at 100° (13%) [6578].

- Also obtained by Friedel–Crafts acylation of phenol with propionyl chloride in the presence of aluminium chloride (40%) [6579], at 120–130° for 1 h (45%) [6580,6581] according to the method [6582].
- Also obtained by acylation of phenol with propionic acid,
  - in the presence of boron trifluoride at 165° for 1 h (45%) [6583] or at 80° for 2 h (8%) [6572];
  - in the presence of polyphosphoric acid at 100° for 10 min (by-product) (5%) [6578];
  - in the presence of zinc chloride at 160° for 1 h [6584].
- Also obtained by isomerization of 4-hydroxypropiophenone with aluminium chloride (1.5 equiv) at 165° for 1 h (40%) or at 180–200° for 15 min (55%) [6356,6468,6482].
- Also obtained by demethylation of 2-propionylanisole with fuming hydrochloric acid (d = 1.19) in a sealed tube at 110° for 6 h [6585].
- Also obtained (by-product) by heating 3-tert-butyl-4-hydroxypropiophenone with aluminium chloride at 170° for 15 min (13%) [6586].
- Also obtained by treatment of 2,3-dimethylchromone with sodium ethoxide in boiling ethanol for 30 h [6587] according to the method [6588].
- Also obtained from 2-allylphenol by treatment with perbenzoic acid in ethyl ether, first at 0°, then between 0° and 25° for 24 h (78%) [6589].
- Also obtained by reaction of 2-bromophenyl propionate in a ethyl ether/hexane/THF mixture at low temperature (−78 to −95°) with sec-butyllithium to give, after hydrolysis, the titled ketone (metal-promoted Fries rearrangement) (17%) [6590].
- Also obtained by reaction of ethylmagnesium bromide,
  - with 2-hydroxybenzamide in boiling benzene, followed by hydrolysis (30%) [6591];
  - with 2-hydroxy-N,N-diethylbenzamide in boiling benzene, followed by hydrolysis (82–84%) [6591].
- Also isolated by heating of 1-(o-hydroxyphenyl)cyclopropyltrimethylammonium iodide for 1 h at 140° with 1.5 equiv of N,N-diisopropylethylamine (not water-free) (38%) [6592].
- Also obtained by hydrolysis of 2-(1-methyliminopropyl)phenol (4aa) with aqueous acetic acid/THF at 40° for 4 h (86%) [6593].

- Also obtained by photolysis of phenyl propionate [6594] in water or in solution containing β-cyclodextrine (254 nm) at 25° for 2 h [6595].
- Also obtained by treatment of 2-(1-hydroxypropyl)phenol with MnO_2 in methylene chloride for 7 h at r.t. [6596].
- Also refer to: [6439,6597–6619].

b.p.$_{0.1}$ 54° [6574], b.p.$_1$ 72–74° [6583,6620], b.p.$_4$ 83° [6579],
b.p.$_6$ 110–114° [6621],
b.p.$_6$ 110–115° [6562,6573,6584], [6389,6563,6564]
b.p.$_{12}$ 110–115° [6580,6581], b.p.$_{15}$ 111° [6591], b.p.$_{12}$ 113° [6355],
b.p.$_{15}$ 115° [6575,6585], b.p.$_{26–28}$ 129–133° [6356], b.p.$_8$ 150° [6622],
b.p.$_{80}$ 150° [6587];
m.p. 20–22° [6591], 12–13° [6355,6356]; d_{17} = 1.106 [6356];
n_D^{20} = 1.550 [6356], n_D^{20} = 1.5490 [6579], n_D^{25} = 1.5485 [6574];
^{1}H NMR [6590,6592,6593], ^{13}C NMR [6590,6593],
^{17}O NMR [6623], IR (Sadtler: standard n° 833) [6593,6622,6624], [6355,6356,6590,6592], UV [6356,6359,6457,6625–6627],
MS [6592,6593,6628]; TLC [6353,6356,6358,6629];
GLC [6630]; paper chromatography [6631,6632]; polarography [6627]; ionization [6501].

Notes: Metal-ligand stability constants of complexes of o-hydroxypropiophenone with cobalt [41586-12-3], copper [18906-66-6] and iron [41586-11-2] [6633]; nickel complex [6634]; Uranium complexes [54204-29-4], [54204-30-7] and [54331-13-4] [6635]; zinc complex [37848-03-6] [6636]; boron complex [42593-40-8] [6637,6638]; cadmium complex [37848-04-7] [6636]; copper complex [60400-13-7] [6639].
USE: For preparation of 3-methylchromone *(Tricromyl)* [85-90-5] [6640].
BIOLOGICAL ACTIVITY: Antispasmodic; vasodilator (coronary) [6640,6641].

Oxime [18265-75-3] $C_9H_{11}NO_2$ mol.wt. 165.19 [6642–6645].

Hydrazone [70136-38-8] $C_9H_{12}N_2O$ mol.wt. 164.21 [6646].

β-D-Glucopyranoside [31826-79-6] $C_{15}H_{20}O_7$ mol.wt. 312.32.

- Preparation by reaction of sodium methoxide with its tetraacetate below in methanol at r.t. for 20 min [6647,6648].
- Also refer to: [6649].

m.p. 119–121° [6648], 105° [6647];

UV [6647]; $(\alpha)_D^{25}$ = −66.4° (water) [6647], n_D^{21} = −50.3° (water) [6648].

Tetra-O-acetyl-β-D-glucopyranoside

[31826-78-5] $C_{23}H_{28}O_{11}$ mol.wt. 480.47.

- Preparation by reaction of tetra-O-acetyl-αα-D-glucopyranosyl bromide with 2-hydroxy-propiophenone in the presence of silver oxide in quinoline at r.t. for 30 min [6647], (40–50%) [6648].
- Also refer to: [6650].

m.p. 167–168° [6648], 162–162.5° [6647];
UV [6647]; $(\alpha)_D^{20} = -41°$ (chloroform) [6648], $(\alpha)_D^{25} = -31°$ (1,2-dichloroethane) [6647].

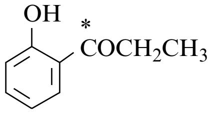
Acetate [97139-82-7] $C_{11}H_{12}O_3$ mol.wt. 192.21.

- Obtained by reaction of acetic anhydride with 2-hydroxypropiophenone [6591].
- Also obtained by reaction of ethylmagnesium bromide with 2-acetoxybenzoyl chloride (84%) [6651].
- Also refer to: [6565,6652].

b.p.$_{14}$ 147° [6591]; m.p. 26° [6591,6653];
^{1}H NMR [6651], ^{13}C NMR [6651].

1-(2-Hydroxyphenyl)-1-propanone labelled with carbon-14
1-[2-Hydroxy[phenyl-U-^{14}C]]-1-propanone

[132899-53-7] $C_9H_{10}O_2$ mol.wt. 152.16



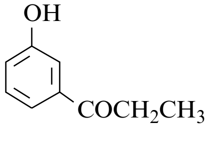
Synthesis

- Obtained by Fries rearrangement of [phenyl-U-^{14}C] propionate (232 μCi) with aluminium chloride at 110° for 30 min (48%) (112 μCi) [6654].

Colourless oil [6654].

1-(3-Hydroxyphenyl)-1-propanone

[13103-80-5] $C_9H_{10}O_2$ mol.wt. 150.18



Syntheses

- Preparation by diazotization of m-aminopropiophenone, followed by decomposition of the diazonium salt obtained [6562,6655], (76%) [6389], (71%) [6656].
- Also obtained by saponification of 3-acetoxypropiophenone (b.p.$_2$ 127–128°) with refluxing 10% sodium hydroxide for 2–3 h (83%) [6657].
- Also obtained by reductive deamination of 2-amino-5-hydroxypropiophenone (diazotization, followed by decomposition of the diazonium salt formed with copper powder in ethanol) [6658].
- Also obtained by reaction of ethylmagnesium bromide with 3-hydroxy-N, N-diethylbenzamide in refluxing n-butyl ether for 4 h (75%) [6591].
- Also obtained by treatment of 3-methoxypropiophenone (b.p.$_{0.05}$ 70–76°) with pyridinium chloride at 210° for 30 min (85%) [6659].

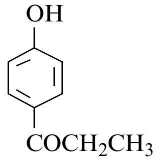
- Also obtained by treatment of 1-hydroxy-1-(3-hydroxyphenyl)propane (m.p. 105–107°) with DDQ in dioxane for 72 h (97%) [6659].
- Also refer to: [6545,6546,6659–6664].

b.p.$_{1.8}$ 131–133° [6657], b.p.$_{0.05}$ 150° [6656];
m.p. 82° [6389,6562,6591], 78° [6656], 77.5–78° [6658], 76–77° [6659], 76° [6657], 75–77° [6663];
MS [6628], UV [6457]; paper chromatography [6631,6632,6665].

BIOLOGICAL ACTIVITY: Tyrosine hydroxylase inhibition [6666].

1-(4-Hydroxyphenyl)-1-propanone *(Paroxypropione)*

[70-70-2] $C_9H_{10}O_2$ mol.wt. 150.18



Syntheses

- Preparation by Fries rearrangement of phenyl propionate,
  - with aluminium chloride in refluxing carbon disulfide [6562], (44%) [6473], then at 130–150° for 2–3 h after solvent elimination (45–50%) [6389,6563,6564];
  - with aluminium chloride in nitrobenzene at 50° for 18 h (72%) [6567] or at 20° for 48 h (60%) [6568];
  - with aluminium chloride in nitromethane at 20° for 7–8 days (80%) [6569,6570];
  - with aluminium chloride in chlorobenzene using microwave irradiation for 3 min at 106° (62%) [6566];
  - with aluminium chloride in ethylene dichloride at 95° for 5 h or in heptane at 80–90° for 7 h (36%) [6571], in benzene or in tetrachloroethane at 80° [6667];
  - with aluminium chloride in the presence of propionyl chloride at 50–60° for 6 h (49%) [6668];
  - with aluminium chloride without solvent at 50° for 10 h (60%) [6355,6356,6576], at 120° (43%) [6575] or at 140° (40%) [6574];
  - with titanium tetrachloride in nitromethane at 20° for 7 days (56%) [6569] or without solvent at 50° for 10 h (39%) [6355,6356,6576];
  - with polyphosphoric acid at 100° (61%) [6578];
  - with boron trifluoride at 50° for 3 h (46%) [6355];
  - with stannic chloride at 50° for 3 h (10%) [6355].
- Also obtained by acylation of phenol with propionic acid,
  - in the presence of boron trifluoride at 70° for 1 h (84%) [6583], at 70–75° (65–70%) [6362], at 73–75° for 3 h (88%) [6378] according to the method [6669] or at 80° for 2 h (40%) [6572];
  - in the presence of polyphosphoric acid at 100° for 10 min (81%) [6578] or in a boiling water bath for 5 min (58%) [6670];
  - in the presence of zinc chloride (Nencki reaction) at 160° for 1 h [6584] or at reflux for 5 min (155°) [6376], (10–12%) [6620,6667].

- Also obtained by Friedel–Crafts acylation of phenol with propionyl chloride in the presence of aluminium chloride (37%) [6579] or at 125–130° for 1 h (22%) [6580,6581] according to the method [6582]. **N.B.**: This same reaction in carbon disulfide, using propionyl chloride formed in situ by action of oxalyl chloride with sodium propionate (method B), for 1 h at 70°, yield 65% [6671].
- Also obtained by demethylation of 4-propionylanisole with refluxing pyridinium chloride for 10–15 min (90%) [6667], (78%) [6672].
- Also obtained by an ethyl group elimination in 4-propionylphenetole with aluminium chloride [6673] according to the method [6674] or with refluxing pyridinium chloride for 10 min (90%) [6667].
- Also obtained by tert-butyl group elimination in 3-tert-butyl-4-hydroxypropiophenone with aluminium chloride without solvent at 170° for 15 min (66%) [6586] or in nitromethane at 20° for 8 days (34%) [6570,6586].
- Also obtained by hydrolysis of 4-propionoxypropiophenone with potassium hydroxide in boiling ethanol [6675].
- Also obtained by reaction of ethylmagnesium bromide with 4-hydroxy-N, N-diethylbenzamide in boiling n-butyl ether (135°), followed by hydrolysis (65%) [6591].
- Also obtained by reaction of propionitrile with phenol (Hoesch reaction) [6667].
- Also obtained by pyrolysis of α-(4′-propionylphenoxy)propiophenone, first at 335–345° for 16 min, then at 430° for 8 min (26%) [6676].
- Also obtained by photolysis of phenyl propionate [6594] in water or in solution containing β-cyclodextrine (254 nm) at 25° for 2 h [6595].
- Also refer to: [6364,6403,6408,6431,6432,6598,6602,6607,6610,6611, 6677–6695].

b.p.$_{11}$ 135–150° [6563,6564], b.p.$_{0.5}$ 140–145° [6583], b.p.$_{1}$ 164° [6574].
Some boiling points are obviously wrong.

m.p. 152–153° [6696], 150° [6355,6356], 149–150° [6578], 149° [6473], 148–149° [6378,6579], 148.5° [6675], 148° [6376,6389,6562,6566,6575, 6697], 147–150° [6668],147–148° [6563,6564,6567,6584,6621], 147° [6670], 146° [6574], 145–147° [6583];

^{1}H NMR (Sadtler: standard n° 8373M) [6698],
IR (Sadtler: standard n° 8329) [6356,6698–6702],
UV [6422,6696,6703], [6356,6357,6457,6625,6698];
MS [6628,6704];
TLC [6353,6356,6358,6422], chromatography [6700,6705];
high-speed liquid chromatography [6706]; polarography [6707];
pK$_a$ [6707]; dielectric properties [6708]; cryoscopic study [6673].

Notes: Toxicity [6709,6710]; deuteration [6361]; bacterial degradation [6711].

BIOLOGICAL ACTIVITY: Pituitary gonadotropic hormone inhibitor (compound **7116**) [6640]; antimicrobial [6712]; antiinflammatory [6713]; antiviral [6714]; choleretic [6715]; melanoma metastasis chemotherapy [6716].

Oxime [133595-72-9] $C_9H_{11}NO_2$ mol.wt. 165.19 [6717,6718].

Potassium salt [138660-02-3] [6719].

β-D-Glucopyranoside [31826-80-9] $C_{15}H_{20}O_7$ mol.wt. 312.32.

- Preparation by reaction of sodium methoxide with the tetraacetate described below in methanol at r.t. for 20 min [6647,6648].
- Also refer to: [6720].

m.p. 173–175° [6648], 160° [6647]. One of the reported melting points is obviously wrong.

$(\alpha)_D^{25} = -95.9°$ (water) [6647], $(\alpha)_D^{15} = -84.4°$ [6648]; UV [6647].

Tetra-O-acetyl-β-D-glucopyranoside [31867-16-0] $C_{23}H_{28}O_{11}$ mol.wt. 480.47.

- Preparation by reaction of tetra-O-acetyl-α-D-glucopyranosyl bromide with 4-hydroxy-propiophenone,
  - in the presence of silver oxide in quinoline at r.t. for 30 min [6647];
  - in the presence of aqueous potassium hydroxide in acetone (40–50%) [6648].
- Also refer to: [6650,6720].

m.p. 158–159.5° [6647], 157–158° [6648];

UV [6647]; $(\alpha)_D^{24} = -27.8°$ (chloroform) [6648], $(\alpha)_D^{25} = -21.5°$ (1,2-dichloroethane) [6647].

Ethyl ether $C_{11}H_{14}O_2$ mol.wt. 178.23.

- Obtained by reaction of propionic anhydride with phenetole in the presence of iodine at reflux for 3 h (57%) [6721].
- Preparation by reaction of propionyl chloride with phenetole in the presence of aluminium chloride in carbon disulfide [6722].

m.p. 30° [6722], 29–30° [6721].

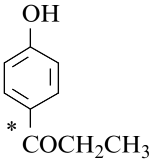
Propionate $C_{12}H_{14}O_3$ mol.wt. 206.24 (m.p. 63°) [6351].

^{1}H NMR (Sadtler: standard n° 57885M), IR (Sadtler: standard n° 84933K).

1-(4-Hydroxyphenyl)-1-propanone labelled with carbon-14

1-[4-Hydroxy[phenyl-U-^{14}C]]-1-propanone

[132899-54-8] $C_9H_{10}O_2$ mol.wt. 152.16



Syntheses

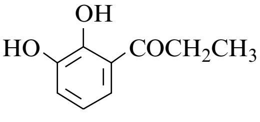
- Obtained by Fries rearrangement of [phenyl-U-^{14}C] propionate (232 mCi) with aluminium chloride at 110° for 30 min (39%) (90 mCi) [6654].

– Also obtained with a radioactivity of 27 microcuries/mg by condensing phenol with $Et^{14}CO_2H$ in the presence of BF_3 [6723].

m.p. 147–149° [6654].

1-(2,3-Dihydroxyphenyl)-1-propanone

[90536-26-8] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

– Obtained by total demethylation of 2,3-dimethoxypropiophenone (b.p.$_2$ 114°) (SM) with refluxing hydriodic acid (d = 1.96) and an equal volume of acetic acid for 6 h (41%) [6724]. SM was prepared by action of ethyl-magnesium iodide on 2,3-dimethoxybenzaldehyde (m.p. 51–52°), followed by oxidation of the obtained carbinol (b.p.$_2$ 108–110°) with potassium dichromate in dilute sulfuric acid.

– Obtained by total demethylation of 2,3-dimethoxypropiophenone (b.p.$_{0.2–0.3}$ 100–102°) with refluxing pyridinium chloride for 30 min (75%) [6725].

– Obtained by total demethylation of 2,3-dimethoxypropiophenone with boron tribromide in methylene chloride at r.t. overnight (78%) [6726].

– Also obtained (by-product) by Fries rearrangement of o-methoxyphenyl propionate (1 mol) or pyrocatechol dipropionate in the presence of pyrocatechol with aluminium chloride (2 mol) without solvent at 135–140° for 2–3.5 h (small amounts) [6727].

b.p.$_5$ 182–187° [6727];

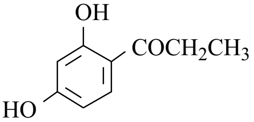
m.p. 102.5–103.5° [6727], 56–57° [6725], 56° [6726], 53° [6724]. One of the reported melting points is obviously wrong.

^{1}H NMR [6726], ^{13}C NMR [6726], IR [6726,6728], MS [6726].

Note: This leads [6724] to doubt the correctness of the structure given to the by-product obtained [6727].

1-(2,4-Dihydroxyphenyl)-1-propanone *(Respropiophenone)*

[5792-36-9] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

– Preparation by reaction of propionic acid with resorcinol,

- in the presence of zinc chloride (Nencki reaction) [6729], at reflux (160–165°) for 15 min [6376,6382,6389,6556,6730,6731], (86%) [6732], (76%) [6733], (73%) [6383], (62%) [6380,6734], at 150° for 20 min [6735];
- in the presence of boron trifluoride for 2 h at 70° (79%) [6736], at 80° (67%) [6572] or at 105–108° for 15 min (71%) [6737];
- in the presence of polyphosphoric acid for 10–20 min in a boiling water bath (65%) [6738];

- in the presence of 70% perchloric acid at reflux for 30 min (70%) [6739];
- in the presence of Amberlite IR-120 (cation exchange resin sulfonic acid type) at 160° for 2–3 h (76%) [6740].

N.B.: Zeokarb 225 was found to be as effective.

- Preparation by reaction of propionitrile with resorcinol (Hoesch reaction) [6741,6742], (75%) [6565], (65%) [6555], (46%) [6743], (35%) [6744].
- Preparation by reaction of propionic anhydride with resorcinol,
  - in the presence of Amberlite IR-120 (cation exchange resin sulfonic acid type) at 160° for 2–3 h (82%) [6740];
  - in the presence of concentrated sulfuric acid (small drops) at 130° for some min (60%) [6744].
- Also obtained by Fries rearrangement of resorcinol dipropionate,
  - with aluminium chloride at 180–185° for 90 min (25%) [6745];
  - with boron trifluoride, in the presence of resorcinol, at 75° for 1 h (83%) [6736].
- Also obtained by reaction of ethylmagnesium bromide with 2,4-dihydroxy-N, N-diethylbenzamide in refluxing benzene (12%) [6591].
- Also refer to: [6452,6524,6617,6746–6771].

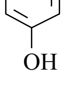
b.p.$_{0.8}$ 152–154° [6736];
m.p. (monohydrate) 57° [6743], 56° [6572,6591,6730,6731];
(anhydrous) 102–104° [6740], 101.5° [6743], 101° [6733,6736,6738],
100–101° [6737], 99° [6744], 98–100° [6565],
98° [6382,6772], 97.5° [6730,6731], 97–98° [6380],
97° [6572,6591,6739], 96° [6383], 95–96° [6732], 95° [6376];
^{1}H NMR [6380], IR [6380,6733,6737], UV [6733,6737];
paper chromatography [6773]; TLC [6380].

USE: For chelatometric determination of iron [6774,6775]; preserving agent for liquid foods [6776]; fungicide [6777].

BIOLOGICAL ACTIVITY: Antimelanoma and skin depigmentation [6778]; choleretic [6715]; anthelmintic [6779]; trypanosoma brucei brucei response [6780]; bactericide [6781].

1-(2,5-Dihydroxyphenyl)-1-propanone

[938-46-5] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

- Preparation by Fries rearrangement of quinol dipropionate (hydroquinone dipropionate),

  - with aluminium chloride without solvent at 130–140° for 4 h (75%) [6782], at 142° for 30 min (80%) [6783], at 160–165° for 3 h (good yields) [6508] or at 190–200° for 90 min (60%) [6745];
  - by using various quantities of aluminium chloride at 180° for 2 h (to see below) [6733];

$AlCl_3$ (equiv)	Yields (%)
1	21
2	21
3	45
4	78
5	89

  - with aluminium chloride (2 equiv) in nitromethane at 20° for a week (7%) [6733] or in nitrobenzene (24%) [6784];
  - with boron trifluoride complex (BF_3-OBu_2) at reflux for 1 h (42%) [6785];
  - by using titanium tetrachloride (2 equiv) at 130° for 2 h gave only 6% yield [6733].

- Also obtained by treatment of,
  - hydroquinone dipropionate with aluminium chloride in the presence of hydroquinone (61%) [6784];
  - 2-hydroxy-5-(propionyloxy)propiophenone with aluminium chloride (5 equiv) at 180° for 30 min in the presence of hydroquinone (57%) [6733].
- Also obtained by acylation of hydroquinone with propionic acid,
  - in the presence of boron trifluoride in ethylene dichloride at 50–55° (59%) [6509], in tetrachloroethane at 90–95° for 5 h (70%) [6786] or at 50° for 4 h (70%) [6787] or without solvent (67%) [6510], at 80° for 2 h (54%) [6572] or at 125° for 2 h (71%) [6736];
  - in the presence of zinc chloride at 190° few min (Nencki reaction) [6376];
  - in the presence of 70% perchloric acid at reflux for 1 h (12%) [6739].
- Also obtained by Friedel–Crafts acylation of hydroquinone with propionyl chloride in nitrobenzene in the presence of aluminium chloride (40%) [6784].
- Also obtained from 2-hydroxy-5-(propionyloxy)propiophenone; the ester group elimination on hydrolysis with 85% sulfuric acid at r.t. or by treatment with by aluminium chloride [6745].
- Also obtained by reaction of benzoquinone (2.5 equiv) with 2-oxobutanoic acid in aqueous acetonitrile or an acetonitrile/methylene chloride mixture (91%) [6788].
- Also refer to: [6761,6789–6798].

m.p. 99–99.5° [6787], 98° [6786], 97–98° [6509], 97° [6733], 96° [6510,6745], 92° [6376,6572,6736,6739,6784], 91–92° [6782], 80–82° [6783].

Some melting points are obviously wrong.

^{1}H NMR [6472,6783], IR [6733,6783];

TLC [6783]; capillary electrochromatography [6799];

paper chromatography [6632].

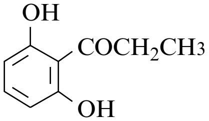
Notes: C-Deuteration [6447]; acidity of methylene proton [6446]; skin depigmentation [6778].

USE: Preservating agent for liquid foods [6776].

BIOLOGICAL ACTIVITY: Antimelanoma [6778].

1-(2,6-Dihydroxyphenyl)-1-propanone

[3361-72-6] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

– Preparation by alkaline degradation of two substituted coumarins,

- from 4-methyl-7-hydroxy-8-propionylcoumarin (m.p. 200°, [6800], 197–198° [6801], 187° [6802,6803]) (70%) [6800], (65%) [6380,6804], (60%) [6801,6803], (48%) [6805];
- from 7-hydroxy-8-propionylcoumarin (m.p. 168°) [6806].

– Also obtained by reaction of ethylmagnesium iodide on 2,6-dimethoxybenzonitrile, followed by cleavage of the ether linkages with aluminium chloride [6804].

– Preparation by dehydrogenation of 2-propionylcyclohexane-1,3-dione,

- in the presence of 10% Pd/C in refluxing cumene overnight under nitrogen (57%) [6807];
- in the presence of 5% Pd/C in tetraethyleneglycol dimethyl ether at 185° [6808].

– Also obtained by decarboxylation of 4,6-dihydroxy-5-propionyl-1,3-benzenedicarboxylic acid in refluxing water for 12 h (25%) [6809].

– Also obtained from 2-chloro-2-propionyl-1,3-cyclohexanedione (m.p. 89°) by rearrangement in 10–20% hydrogen chloride-DMF at 120–130° for 15–20 min, according to the procedure [6810], (79%) [6811].

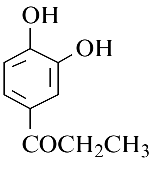
– Also refer to [6812–6817].

m.p. 139° [6800,6806], 137–138° [6380,6809], 136–137° [6801],
136° [6811], 135–137° [6807], 133.5° [6803], 130–132° [6802,6805];
^{1}H NMR [6380,6811], ^{13}C NMR [6811], IR [6380,6811], UV [6733]; TLC [6380]; sublimation 118–132°/0.01 mbar [6811].

Dibenzoate $C_{23}H_{18}O_5$ mol.wt. 374.39 (m.p. 95°) [6800].

1-(3,4-Dihydroxyphenyl)-1-propanone *(4-Propionylpyrocatechol)*

[7451-98-1] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

– Obtained by Fries rearrangement of pyrocatechol dipropionate with aluminium chloride in chlorobenzene at 80° for 2 h (84%) [6818] or in nitrobenzene at 100° for 15 min [6389], (39%) [6784].

- Obtained by Fries rearrangement of pyrocatechol dipropionate with aluminium chloride in the presence of pyrocatechol, in nitrobenzene at 80° for 1 h (37%) [6580,6581] or in nitromethane at r.t. for 72 h (76%) [6733].
- Also obtained by Fries rearrangement of o-methoxyphenyl propionate (1 mol) by aluminium chloride (2 mol) without solvent at 140° for 2 h (51–55%) [6727] or in nitrobenzene at 80° for 30–60 min [6620]. A demethylation occurs during the reaction.
- Also obtained by acylation of pyrocatechol with propionic acid,
  - in the presence of boron trifluoride [6819], at 150° for 2 h in a sealed tube (36%) [6736];
  - in the presence of polyphosphoric acid for 15 min in a boiling water bath (12%) [6738].
- Preparation by demethylation of 4-hydroxy-3-methoxypropiophenone with refluxing pyridinium chloride for 10 min (80%) [6820].
- Also obtained when propiovanillone was heated with an alkali sulfide under conditions used in the kraft cooking process [6821].
- Also refer to: [6685,6713,6822–6835].

Isolation from natural sources

- From sprucewood pulp cooking with sodium bisulfite [6836].
- From high-moorland peat extracts [6837].
- From components of the peat fulvic acid fraction [6838].

b.p.$_{15}$ 210–220° [6620];
m.p. 148° [6819], 146° [6580,6581,6620,6736,6738,6784],
146–148° [6818], 145° [6733], 142° [6820];
ESR spectrum [6839], IR [6733], UV [6733];
chromatography [6840–6842]; paper chromatography [6843]; chromatography on ion-exchange paper [6844]; TLC [6733];
dielectric constant and dipole moment in p-dioxane [6845].

Note: skin depigmentation [6778].
USE: Antioxidizing agent [6846,6847].
BIOLOGICAL ACTIVITY: β-Adrenergic receptor mutant [6848]; antiinflammatory [6713]; as trypanocide, mitochondria respiration response [6849]; trypanosoma brucei brucei inhibition [6780]; pharmacological [6828]; antimelanoma [6778].

Diethyl ether [720-66-1] $C_{13}H_{18}O_3$ mol.wt. 222.28.

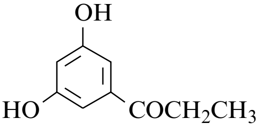
- Obtained by reaction of propionyl chloride with catechol diethyl ether in the presence of aluminium chloride in refluxing benzene for 40 min (49%) [6850].
- Also refer to: [6851–6854].

b.p.$_{0.5}$ 119–121° [6850], b.p.$_{30}$ 181° [6851];
m.p. 39–40° [6854], 38–39° [6851], 36–39° [6850].

BIOLOGICAL ACTIVITY: Antiinflammatory [6713].

1-(3,5-Dihydroxyphenyl)-1-propanone

[100523-86-2] $C_9H_{10}O_3$ mol.wt. 166.18



Syntheses

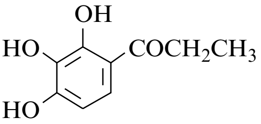
- Obtained by hydrolysis of its diacetate (SM) with boiling 10% sulfuric acid. SM was prepared by condensation of 3,5-diacetoxybenzoyl chloride (m.p. 88°) with diethyl cadmium in benzene (50%) [6855].
- Also refer to: [6856].

m.p. 175° [6855].

BIOLOGICAL ACTIVITY: Anthelmintic [6857].

1-(2,3,4-Trihydroxyphenyl)-1-propanone

[22760-98-1] $C_9H_{10}O_4$ mol.wt. 182.18



Syntheses

- Preparation by acylation of pyrogallol with propionic acid,
  - in the presence of boron trifluoride [6454] in ethyl ether for 1 h at 0° (80%) [6858,6859];
  - in the presence of Amberlite IR-120 (cation exchange resin sulfonic acid type) at 160° for 2–3 h (78%) [6740].

  N.B.: Zeokarb 225 was found to be as effective.

  - in the presence of zinc chloride [6860,6861], at 130–140° for 90 min (Nencki reaction) (35%) [6862];
  - in the presence of 70% perchloric acid at reflux for 1 h (33%) [6739].
- Preparation by reaction of propionyl chloride with pyrogallol and heating 4 h on a water bath (75%) [6454].
- Preparation by acylation of pyrogallol with propionic anhydride,
  - in the presence of Amberlite IR-120 (cation exchange resin sulfonic acid type) at 160° for 2–3 h (79%) [6740].

  N.B.: Zeokarb 225 was found to be as effective.

  - in the presence of concentrated sulfuric acid (small drops) at 130° for some min (65%) [6744];
  - in the presence of zinc chloride, but using a mixture of propionic acid/propionic anhydride (85/100) at 135–140° for 40 min, then at r.t. overnight (59%) [6863].
- Also refer to: [6757,6758,6864,6865].

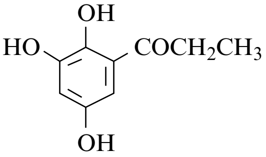
m.p. 129° [6454], 128–130° [6740], 128–129° [6862], 128° [6858,6859],
127° [6739,6860,6861], 126–127° [6744], 125–127° [6863];
^{1}H NMR [6863], IR [6863], ESR spectrum [6839], UV [6858].

Note: In determination of molybdenum (molybdenum complex) [6866] for determination of tungsten by spectrophotometry [6867].

USE: Matrix metalloprotease inhibitors and their application in cosmetic and pharmaceutical compositions [6868].

1-(2,3,5-Trihydroxyphenyl)-1-propanone

[870701-66-9] $C_9H_{10}O_4$ mol.wt. 182.18



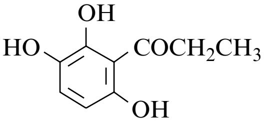
Syntheses

- Refer to: [6868,6869].

USE: Matrix metalloprotease inhibitors and their application in cosmetic and pharmaceutical compositions [6868].

1-(2,3,6-Trihydroxyphenyl)-1-propanone

[870701-67-0] $C_9H_{10}O_4$ mol.wt. 182.18



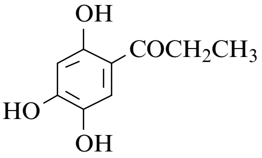
Syntheses

- Refer to: [6868,6869].

USE: Matrix metalloprotease inhibitors and their application in cosmetic and pharmaceutical compositions [6868].

1-(2,4,5-Trihydroxyphenyl)-1-propanone

[79744-62-0] $C_9H_{10}O_4$ mol.wt. 182.18



Syntheses

- Preparation from various methods [6870],
  - by Friedel–Crafts acylation of hydroxyhydroquinone with propionyl chloride in the presence of aluminium chloride;
  - by Fries rearrangement of hydroxyhydroquinone dipropionate with aluminium chloride;
  - by reaction of propionitrile with hydroxyhydroquinone (Hoesch reaction).
- Also refer to: [6871].

^1H NMR [6870], ^{13}C NMR [6870].

USE: Matrix metalloprotease inhibitors and their application in cosmetic and pharmaceutical compositions [6868].

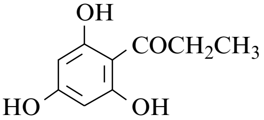
Triethyl ether [63213-30-9] $C_{15}H_{22}O_4$ mol.wt. 266.34.

– Preparation by condensation of 1,2,4-triethoxybenzene with propionic acid in the presence of PPA at 50–55° for 40 min (60–80%) [6872].

m.p. 66–67° [6872].

1-(2,4,6-Trihydroxyphenyl)-1-propanone *(Phloropropiophenone) (Flopropione)*

[2295-58-1] $C_9H_{10}O_4$ mol.wt. 182.18



Syntheses

– Preparation by reaction of propionitrile with phloroglucinol (Hoesch reaction) [6744,6873–6876], (85%) [6565], (73%) [6877], (64%) [6878].

N.B.: Shinoda [6875,6876] has previously described this experiment and gives the m.p. as 113° for the hydrated and 207° for the anhydrous product. These melting points correspond with those of hydrated and anhydrous phloroglucinol, and it would appear that Shinoda did not obtain phloropropiophenone [6744].

– Preparation by reaction of propionic anhydride with phloroglucinol,

- in the presence of concentrated sulfuric acid (one drop) at 130° for 5 min (65%) [6744];
- in the presence of Amberlite IR-120 (cation exchange resin sulfonic acid type) at 160° for 2–3 h (42%) [6740]. **N.B.**: Zeokarb 225 was found to be as effective.
- in the presence of boron trifluoride etherate at 10° (62–65%) [6879].

– Preparation by reaction of propionyl chloride with phloroglucinol,

- in the presence of aluminium chloride in nitrobenzene/carbon disulfide solution (76%) [6880], in nitrobenzene at 60° (55%) [6881] or without solvent at 50° (70%) [6882];
- in the presence of boron trifluoride etherate at 10° (compound **3**) (62–65%) [6879].

– Also refer to: [6743,6758,6765,6857,6883–6891].

Isolation from natural sources

– From *Inula viscosa* (L.) Ait (Tribus Inulea, Compositae) [6892].

m.p. (monohydrate) 114–115° [6565], 113° [6875,6876];
(anhydrous) 207° [6875,6876], 185–186° [6874], 177° [6565],
175–178° [6879], 175–176° [6877], 174–176° [6740,6890],
174–175° [6873,6885,6886], 174° [6880],
170–171° [6744];

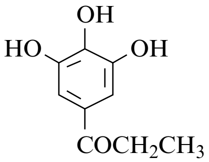
^{1}H NMR [6881,6893], ^{13}C NMR [6881], UV-visible spectrum [6894]; GLC [6895]; TLC [6629,6890,6895]; HPLC [6896];
paper chromatography [6895].

Note: Toxicity [6897].
USE: Fungicide [6880]; matrix metalloprotease inhibitors and their application in cosmetic and pharmaceutical compositions [6868].
BIOLOGICAL ACTIVITY: Antispasmodic [6640,6898,6899]; choleretic [6715,6900]; antischistosomal [6879]; antimicrobial [6901], for staphylococcus aureus [6891]; identification of candidate drugs for treatment of amyotrophic lateral sclerosis [6902]; ligand-based virtual screening and design of antimalarial compounds [6903]; pharmacological action [6904].

Compound with 4,4′-(1*E*)-1,2-ethenediylbis[pyridine] (1:1) [820990-87-2] [6905].

1-(3,4,5-Trihydroxyphenyl)-1-propanone

[267410-40-2] $C_9H_{10}O_4$ mol.wt. 182.18

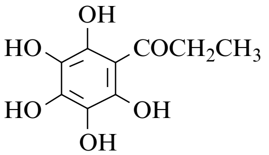


Syntheses
- Refer to: [6868,6869].

USE: Matrix metalloprotease inhibitors and their application in cosmetic and pharmaceutical compositions [6868].

1-(Pentahydroxyphenyl)-1-propanone

[881672-76-0] $C_9H_{10}O_6$ mol.wt. 214.27

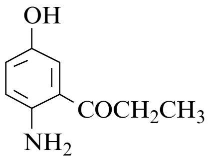


Synthesis
- Refer to: [6868].

USE: Matrix metalloprotease inhibitors and their application in cosmetic and pharmaceutical compositions [6868].

1-(2-Amino-5-hydroxyphenyl)-1-propanone

[35364-15-9] $C_9H_{11}NO_2$ mol.wt. 165.19

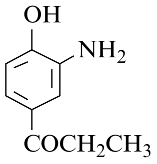


Syntheses
- Obtained by UV-irradiation of 3-ethyl-2,1-benzisoxazole in 66% sulfuric acid for 90 min at 80–90° [6906], (88–95%) [6658].
- Also prepared from 2-nitro-5-hydroxybenzaldehyde (five steps) or from p-anisidine (one step) [6907,6908].
- Also refer to: [6553,6908–6912].

m.p. 145° [6658].

1-(3-Amino-4-hydroxyphenyl)-1-propanone

[130521-20-9] $C_9H_{11}NO_2$ mol.wt. 165.19



Syntheses

- Preparation by hydrolysis of 3-acetamido-4-hydroxypropiophenone [6913] with refluxing 10 N hydrochloric acid for 30 min (74%) [6744,6913].
- Also obtained by treatment of 4-hydroxy-3-nitro-propiophenone suspended in hydrochloric acid with granulated tin [6473] or in the presence of Pd/C [6914].
- Preparation by reduction of the nitro group of 4-hydroxy-3-nitropropiophenone in boiling alkaline solution with sodium hydrosulfite [6915].
- Also refer to: [6916,6917].

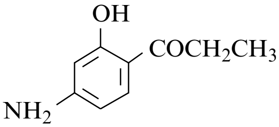
m.p. 145–146° [6913], 144–145° (d) [6915];
^{1}H NMR [6913], IR [6913].

BIOLOGICAL ACTIVITY: Bactericide [6915].

Hydrochloride $C_9H_{11}NO_2$, HCl mol.wt. 201.65 (m.p. 217°) [6473].

1-(4-Amino-2-hydroxyphenyl)-1-propanone

[83294-23-9] $C_9H_{11}NO_2$ mol.wt. 165.19



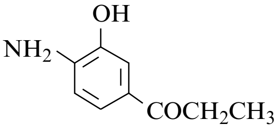
Syntheses

- Obtained by hydrolysis of 3-hydroxy-4-(1-oxopropyl)-acetanilide with boiling 50% hydrochloric acid [6918,6919].
- Also refer to: [6429].

Note: Acidity of methylene proton [6446].
m.p. 137° [6918].

1-(4-Amino-3-hydroxyphenyl)-1-propanone

[54903-52-5] $C_9H_{11}NO_2$ mol.wt. 165.19



Syntheses

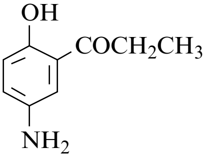
- Preparation by alkaline hydrolysis of 6-propionyl-2-(3*H*)-benzoxazolinone with boiling 10% aqueous sodium hydroxide solution for 4 h (90–100%) [6913,6920].
- Also refer to: [6921–6924].

m.p. 139° [6913], 138–139° [6920]; ^{1}H NMR [6913], IR [6913].

BIOLOGICAL ACTIVITY: Analgesic [6923].

1-(5-Amino-2-hydroxyphenyl)-1-propanone

[79925-35-2] $C_9H_{11}NO_2$ mol.wt. 165.19



Syntheses

- Obtained from 4-hydroxy-3-(1-oxopropyl)acetanilide by hydrolysis with boiling 50% hydrochloric acid [6429].
- Also refer to: [6925].

Notes: C-Deuteration [6447]; acidity of methylene proton [6446]; antidote against cyanide [6925].

m.p. 81° [6429].

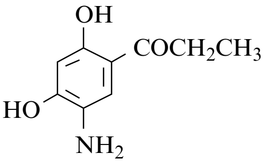
Hydrochloride [142301-96-0] $C_9H_{11}NO_2$, HCl mol.wt. 201.65.

- Refer to: [6925].

Note: Antidote against cyanide [6925].

1-(5-Amino-2,4-dihydroxyphenyl)-1-propanone

$C_9H_{11}NO_3$ mol.wt. 181.19



Syntheses

- Preparation by hydrogenation of 2,4-dihydroxy-5-nitro-propiophenone in acetone in the presence of Raney nickel [6555].
- Preparation from its hydrochloride, by addition of N sodium carbonate solution [6555].

m.p. 147–151° (d) [6555].

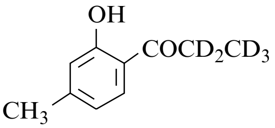
Hydrochloride $C_9H_{11}NO_3$, HCl mol.wt. 217.65.

- Preparation from the corresponding nitro compound by catalytic reduction in acetone solution using Raney nickel. Then, after filtration of the catalyst and half elimination of solvent by vacuum distillation, the hydrochloride was prepared by passing dry hydrogen chloride into the remaining solution [6555].

m.p. >300° [6555].

1-(2-Hydroxy-4-methylphenyl)-1-propanone-*2,2,3,3,3-d_5*

$C_{10}H_7D_5O_2$ mol.wt. 169.23



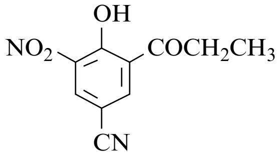
Synthesis

- Refer to: [6350].

ion (1^-), radical ion (1^-) [72051-77-5], ESR spectrum [6350].

4-Hydroxy-3-nitro-5-(1-oxopropyl)benzonitrile

[70978-55-1] $C_{10}H_8N_2O_4$ mol.wt. 220.18



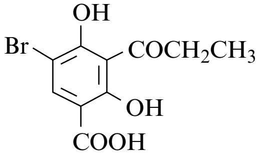
Synthesis

- Preparation by nitration of 4-hydroxy-3-(1-oxopropyl)-benzonitrile at –20° (40%) [6926].

m.p. 90° [6926].

5-Bromo-2,4-dihydroxy-3-(1-oxopropyl)benzoic acid

[101012-66-2] $C_{10}H_9BrO_5$ mol.wt. 289.08



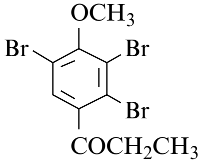
Syntheses

- Obtained by Fries rearrangement of methyl 5-bromo-2,4-di-propionoxybenzoate (m.p. 122°) with aluminium chloride (3.3 mol) at 125–130° for 1 h (25%) or at 160–170° [6450]. The above reaction, when carried out using nitrobenzene at r.t. for 24 h or at 100° for 1 h gave the same keto acid.
- Also obtained by acylation of methyl 5-bromo-β-resorcylate with propionic anhydride under the conditions of the Friedel–Crafts reaction [6450].
- Also obtained by treatment of methyl 5-bromo-2,4-dihydroxy-3-propionylbenzoate with 10% sodium hydroxide solution at r.t. for 24 h [6450].
- Also obtained by Fries rearrangement of 2-hydroxy-4-propionoxy-5-bromobenzoic acid (m.p. 166°) with aluminium chloride (3.3 mol) at 165–170° for 1 h (33%) [6450].
- Also obtained by Friedel–Crafts propionylation of 5-bromo-β-resorcylic acid [6450].

m.p. 219–220° [6450].

1-(2,3,5-Tribromo-4-methoxyphenyl)-1-propanone

[23689-25-0] $C_{10}H_9Br_3O_2$ mol.wt. 400.89



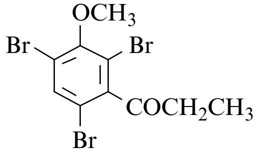
Synthesis

- Preparation by reaction of dimethyl sulfate with 2,3,5-tribromo-4-hydroxypropiophenone [6360].

m.p. 90° [6360]; IR [6360], UV [6360].

1-(2,4,6-Tribromo-3-methoxyphenyl)-1-propanone

[23689-32-9] $C_{10}H_9Br_3O_2$ mol.wt. 400.89



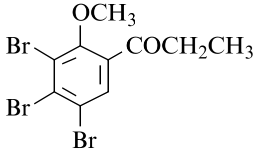
Synthesis

– Preparation by reaction of dimethyl sulfate with 2,4,6-tribromo-3-hydroxypropiophenone [6360].

b.p.$_{15}$ 192° [6360]; m.p. 36–37° [6360]; IR [6360], UV [6360].

1-(3,4,5-Tribromo-2-methoxyphenyl)-1-propanone

[23600-68-2] $C_{10}H_9Br_3O_2$ mol.wt. 400.89



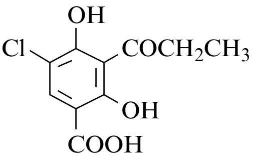
Synthesis

– Preparation by reaction of dimethyl sulfate with 3,4,5-tribromo-2-hydroxypropiophenone [6360].

m.p. 64° [6360]; IR [6360], UV [6360].

5-Chloro-2,4-dihydroxy-3-(1-oxopropyl)benzoic acid

[102541-32-2] $C_{10}H_9ClO_5$ mol.wt. 244.63



Syntheses

– Obtained by Fries rearrangement of methyl 5-chloro-2,4-dipropionoxybenzoate (m.p. 114°) with aluminium chloride at 125–130° for 1 h (21%) [6450].

– Also obtained by alkaline hydrolysis of methyl 5-chloro-2,4-dihydroxy-3-propionylbenzoate [6450].

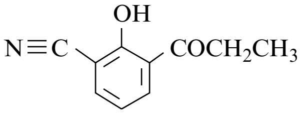
– Also obtained by Friedel–Crafts acylation of 5-chloro-2,4-dihydroxybenzoic acid with propionic anhydride in the presence of aluminium chloride [6450].

– Also obtained by Fries rearrangement of 5-chloro-2-hydroxy-4-propionoxybenzoic acid (m.p. 155°) with aluminium chloride at 165° for 1 h (32%) [6450].

m.p. 206–207° [6450].

2-Hydroxy-3-(1-oxopropyl)benzonitrile

[99184-81-3] $C_{10}H_9NO_2$ mol.wt. 175.19

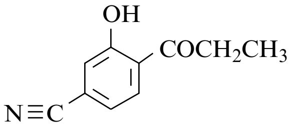


Synthesis

– Preparation by treatment of 2-methoxy-3-propionyl-benzonitrile with aluminium chloride in refluxing benzene for 2 h (72%) [6533].

m.p. 82–85° [6533]; UV [6533].

3-Hydroxy-4-(1-oxopropyl)benzonitrile



$C_{10}H_9NO_2$ mol.wt. 175.19

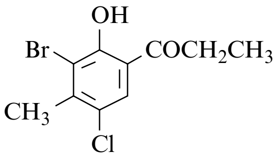
Synthesis

- Obtained by demethylation of its methyl ether with alumunium chloride in refluxing benzene for 1 h [6927].

m.p. 170–171° [6927].

1-(3-Bromo-5-chloro-2-hydroxy-4-methylphenyl)-1-propanone

[22362-76-1]



$C_{10}H_{10}BrClO_2$ mol.wt. 277.54

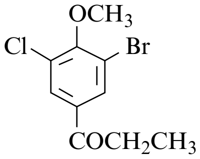
Synthesis

- Preparation by bromination of 5-chloro-2-hydroxy-4-methylpropiophenone [6351] according to the method [6352].

m.p. 100° [6351];

^{1}H NMR (Sadtler: standard n° 8734M), IR (Sadtler: standard n° 38082), UV [6359].

1-(3-Bromo-5-chloro-4-methoxyphenyl)-1-propanone



$C_{10}H_{10}BrClO_2$ mol.wt. 277.54

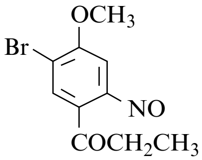
Synthesis

- Obtained by reaction of bromine with 3-chloro-4-methoxypropiophenone in dilute acetic acid [6362].

m.p. 57° [6362].

1-(5-Bromo-4-methoxy-2-nitrosophenyl)-1-propanone

[81484-62-0]



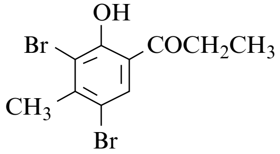
$C_{10}H_{10}BrNO_3$ mol.wt. 272.10

Synthesis

- Refer to: [6928].

1-(3,5-Dibromo-2-hydroxy-4-methylphenyl)-1-propanone

[2892-33-3] $C_{10}H_{10}Br_2O_2$ mol.wt. 322.00



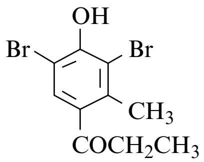
Syntheses

– Preparation by bromination of 2-hydroxy-4-methyl-propiophenone with bromine, according to the procedure [6354], (good yield) [6355,6356].

m.p. 105° [6355,6356];
^{1}H NMR (Sadtler: standard n° 8733M), IR (Sadtler: standard n° 38079) [6355,6356],
UV [6356,6359]; TLC [6353,6358].

1-(3,5-Dibromo-4-hydroxy-2-methylphenyl)-1-propanone

[3023-09-4] $C_{10}H_{10}Br_2O_2$ mol.wt. 322.00



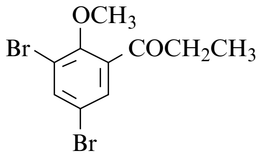
Syntheses

– Preparation by bromination of 4-hydroxy-2-methyl-propiophenone with bromine, according to the procedure [6354], (good yield) [6355,6356].

m.p. 97–98° [6355,6356];
^{1}H NMR (Sadtler: standard n° 8732M), IR (Sadtler: standard n° 38078) [6355,6356],
UV [6353,6356–6358].

1-(3,5-Dibromo-2-methoxyphenyl)-1-propanone

[24876-08-2] $C_{10}H_{10}Br_2O_2$ mol.wt. 322.00



Syntheses

– Preparation from 3,5-dibromo-2-hydroxypropiophenone,

- by reaction with dimethyl sulfate in the presence of potassium carbonate in acetone [6370] or in the presence of sodium hydroxide in methanol [6360];
- by reaction with methyl iodide in the presence of potassium hydroxide in ethanol [6363].

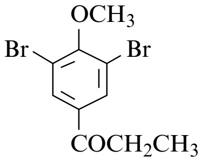
b.p.$_{17}$ 170° [6360];
m.p. 66° [6363], 54° [6360,6370]; IR [6360], UV [6360].

2,4-Dinitrophenylhydrazone [23600-74-0] $C_{16}H_{14}Br_2N_4O_5$ mol.wt. 502.12

(m.p. 131–132°) [6360].

1-(3,5-Dibromo-4-methoxyphenyl)-1-propanone

[24876-03-7] $C_{10}H_{10}Br_2O_2$ mol.wt. 322.00



Syntheses

- Preparation by reaction of dimethyl sulfate with 3,5-dibromo-4-hydroxypropiophenone [6360,6378].
- Also refer to: [6379].

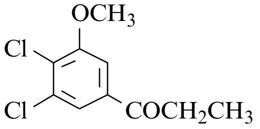
b.p.$_{17}$ 193° [6360];
m.p. 63–65° [6360], 62–63° [6378]; IR [6360], UV [6360].

2,4-Dinitrophenylhydrazone [23600-56-8] $C_{16}H_{14}Br_2N_4O_5$ mol.wt. 502.12

(m.p. 63–65°) [6360].

1-(3,4-Dichloro-5-methoxyphenyl)-1-propanone

[113730-37-3] $C_{10}H_{10}Cl_2O_2$ mol.wt. 233.09



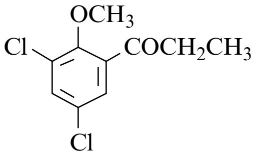
Synthesis

- Obtained by adding a solution of 45% nitrosylsulfuric acid in sulfuric acid to a solution of 2-amino-3,4-dichloro-5-methoxypropiophenone in THF between −25° and −16° over 7 min, then to keep at 0° for 1.5 h. Next, 45–50% aqueous hypophosphorous acid was added at <10° and to keep like this for 2.5 h (93%) [6386].

m.p. 85° [6386]; ^{1}H NMR [6386].

1-(3,5-Dichloro-2-methoxyphenyl)-1-propanone

$C_{10}H_{10}Cl_2O_2$ mol.wt. 233.09



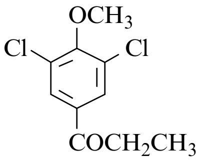
Synthesis

- Obtained by reaction of dimethyl sulfate with 3, 5-dichloro-2-hydroxypropiophenone in the presence of aqueous sodium hydroxide in refluxing acetone (80%) [6390].

m.p. 37–38° [6390].

1-(3,5-Dichloro-4-methoxyphenyl)-1-propanone

[213470-65-6] $C_{10}H_{10}Cl_2O_2$ mol.wt. 233.09



Syntheses

- Obtained by reaction of propionyl chloride with 2,6-di-chloroanisole in the presence of aluminium chloride in carbon disulfide for 2 h at 0° [6379,6407].

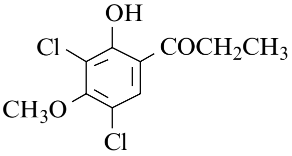
- Also obtained by oxidation of 1-(3,5-dichloro-4-methoxy-phenyl)-1-propanol with PDC in methylene chloride at 25° overnight (97%) [6929,6930].

m.p. 90° [6379], 65–66° [6929,6930]. One of the reported melting points is obviously wrong.

^{1}H NMR [6930], ^{13}C NMR [6930], MS [6930].

1-(3,5-Dichloro-2-hydroxy-4-methoxyphenyl)-1-propanone

[66021-80-5] $C_{10}H_{10}Cl_2O_3$ mol.wt. 249.09

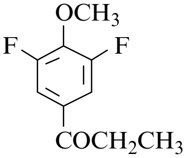


Synthesis

- Preparation by chlorination of 2-hydroxy-4-methoxy-propiophenone [6410].

1-(3,5-Difluoro-4-methoxyphenyl)-1-propanone

[71292-82-5] $C_{10}H_{10}F_2O_2$ mol.wt. 200.19

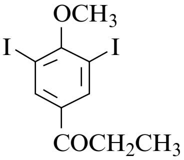


Synthesis

- Refer to: [6415].

1-(3,5-Diiodo-4-methoxyphenyl)-1-propanone

[31827-85-7] $C_{10}H_{10}I_2O_2$ mol.wt. 416.00



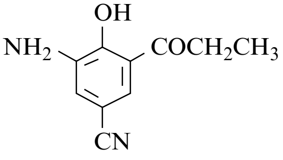
Syntheses

- Preparation by methylation of 4-hydroxy-3,5-diiodo-propiophenone with,
  - methyl iodide in the presence of potassium hydroxide in ethanol [6362] or methanol [6931];
  - dimethyl sulfate in the presence of sodium hydroxide in dilute methanol [6420].

m.p. 106° [6931], 104° [6362], 83–84° [6420]. One of the reported melting points is obviously wrong.

3-Amino-4-hydroxy-5-(1-oxopropyl)benzonitrile

[70977-86-5] $C_{10}H_{10}N_2O_2$ mol.wt. 190.20



Synthesis

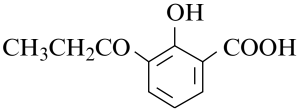
- Preparation by hydrogenation of 4-hydroxy-3-nitro-5-(1-oxopropyl)benzonitrile using 5% Pd/C as catalyst in ethanol (70%) [6932].

m.p. 144–146° [6926].

2-Hydroxy-3-(1-oxopropyl)benzoic acid

3-Propionylsalicylic acid

[35888-92-7] $C_{10}H_{10}O_4$ mol.wt. 194.19



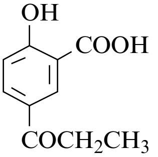
Syntheses

- Obtained by ozonolysis of 2-hydroxy-3-propenyl-propiophenone [6933].
- Also obtained by hydrolysis of 2-acetoxy-3-propionyl-benzoic acid (SM) with 10% sodium hydroxide (65%). SM was prepared by treatment of 2-acetoxy-3-propenylpropiophenone with potassium permanganate in acetic acid at 15° for 30 min (80%) [6934].
- Also obtained by heating 2-hydroxy-3-propionylbenzaldehyde with potassium hydroxide at 110° (85%) [6935].
- Also refer to: [6936].

2-Hydroxy-5-(1-oxopropyl)benzoic acid

5-Propionylsalicylic acid

[78417-99-9] $C_{10}H_{10}O_4$ mol.wt. 194.19



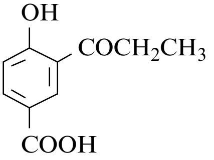
Syntheses

- Obtained by hydrolysis of methyl propionylsalicylate [6937], in boiling 20% aqueous potassium hydroxide [6938].
- Also obtained by alkaline hydrolysis of 5-propionyl-salicylamide (m.p. 216°) [6939].

m.p. 179° [6939], 177–179° [6938], 176–177° [6940].

4-Hydroxy-3-(1-oxopropyl)benzoic acid

[25065-13-8] $C_{10}H_{10}O_4$ mol.wt. 194.19



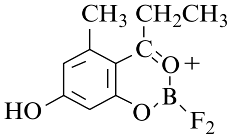
Syntheses

- Preparation by reaction of propionyl chloride with ethyl p-hydroxybenzoate in the presence of aluminium chloride in tetrachloroethane at 120° for 3–4 h (70–80%) [6941].
- Also obtained by Fries rearrangement of 4-propionyl-oxybenzoic acid (m.p. 185–188°) with aluminium chloride in nitrobenzene (47%) [6942], (25%) [6943].

m.p. 225–227° [6943], 220–221° [6942], 220° [6941].

1-[2-[(Difluoroboryl)oxy]-4-hydroxy-6-methylphenyl]-1-propanone

[154921-37-6] $C_{10}H_{11}BF_2O_3$ mol.wt. 228.00

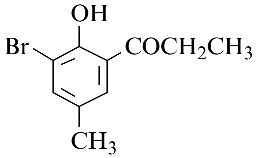


Synthesis

– Obtained by reaction of propionic acid with 5-methylresorcinol in the presence of boron trifluoride etherate [6944].

1-(3-Bromo-2-hydroxy-5-methylphenyl)-1-propanone

[2892-30-0] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



Syntheses

– Preparation by bromination of 2-hydroxy-5-methylpropiophenone,

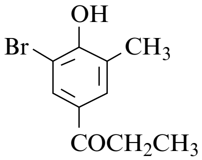
- with bromine, according to the procedure [6354], (good yield) [6355,6356];
- with N-bromosuccinimide in DMF, first at 0°, then at r.t. for 18 h (86%) [6945,6946].

– Also refer to: [6947] (compound **I**), [6948].

m.p. 133° [6355,6356], 130° [6945,6946];
^{1}H NMR (Sadtler: standard n° 8731M), IR (Sadtler: standard n° 38076) [6355,6356],
UV [6359]; TLC [6353,6358].

1-(3-Bromo-4-hydroxy-5-methylphenyl)-1-propanone

[2904-86-1] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



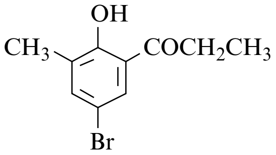
Syntheses

– Preparation by bromination of 4-hydroxy-3-methylpropiophenone with bromine, according to the procedure [6354], (good yield) [6355,6356].

m.p. 97° [6355,6356];
^{1}H NMR (Sadtler: standard n° 8728M), IR (Sadtler: standard n° 38072) [6355,6356],
UV [6356,6357]; TLC [6353,6358].

1-(5-Bromo-2-hydroxy-3-methylphenyl)-1-propanone

[2887-56-1] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



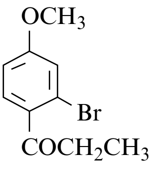
Syntheses

- Preparation by bromination of 2-hydroxy-3-methyl-propiophenone with bromine, according to the procedure [6354], (good yield) [6355,6356].

m.p. 69° [6355,6356];
^{1}H NMR (Sadtler: standard n° 8920M), IR (Sadtler: standard n° 38073) [6355,6356],
UV [6356,6359]; TLC [6353,6358].

1-(2-Bromo-4-methoxyphenyl)-1-propanone

[24876-04-8] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



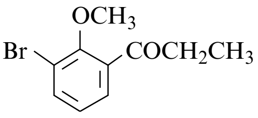
Syntheses

- Obtained by reaction of propionic anhydride with 3-bromoanisole in the presence of aluminium chloride in carbon disulfide (65%) [6949].
- Also obtained by reaction of dimethyl sulfate with 2-bromo-4-hydroxypropiophenone [6360].
- Also refer to: [6950].

b.p.$_6$ 146–147° [6949], b.p.$_{12}$ 167° [6360];
m.p. 33° [6360]; IR [6360], UV [6360].

1-(3-Bromo-2-methoxyphenyl)-1-propanone

[23689-23-8] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



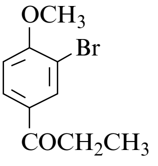
Synthesis

- Preparation by reaction of dimethyl sulfate with 3-bromo-2-hydroxypropiophenone [6360].

b.p.$_{14}$ 143° [6360];
m.p. 2–3° [6360]; $n_D^{20} = 1.551$ [6360]; IR [6360], UV [6360].

1-(3-Bromo-4-methoxyphenyl)-1-propanone

[4374-36-1] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



Syntheses

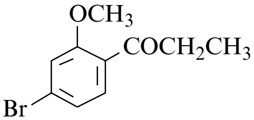
- Preparation by methylation of 3-bromo-4-hydroxy-propiophenone with dimethyl sulfate [6360].
- Also obtained by treatment of 4-methoxypropiophenone with bromine in acetic acid/potassium acetate [6375].

- Also obtained by reaction of sodium methoxide with 2-bromo-4-(2-bromo-1-methoxypropyl)-1-methoxy-benzene in boiling methanol for 2 h [6951].
- Also obtained by reaction of zinc powder with 2-bromo-1-(3-bromo-4-methoxyphenyl)-1-propanone in boiling ethanol for 2–3 h [6952].
- Also obtained by distillation of 4-(1-ethoxypropenyl)-2-bromo-1-methoxybenzene or by treatment of this with acid [6953].
- Also refer to: [6362,6377,6430,6954,6955].

m.p. 101° [6360,6375], 100–101° [6951], 100.5° [6953], 99–101° [6954,6956]; IR [6360], UV [6360].

1-(4-Bromo-2-methoxyphenyl)-1-propanone

[23600-63-7] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



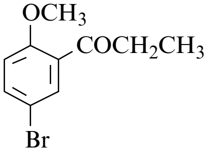
Synthesis

- Preparation by reaction of dimethyl sulfate with 4-bromo-2-hydroxypropiophenone [6360].

b.p.$_{11}$ [6360], b.p.$_{19}$ 171° [6360];
m.p. 42° [6360]; IR [6360], UV [6360].

1-(5-Bromo-2-methoxyphenyl)-1-propanone

[502924-41-6] $C_{10}H_{11}BrO_2$ mol.wt. 243.10



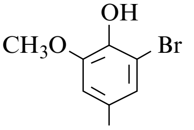
Syntheses

- Preparation by reaction of dimethyl sulfate with 5-bromo-2-hydroxypropiophenone in the presence of sodium hydroxide in methanol [6360].
- Obtained by ozonolysis of 4-bromo-2-(1-ethyl-1-propenyl)-anisole [6957].
- Also refer to: [6958].

b.p.$_{25}$ 145–147° [6957], b.p.$_{46}$ 192–200° [6360];
m.p. 74° [6957], 39° [6360]. One of the reported melting points is obviously wrong.
IR [6360], UV [6360].

1-(3-Bromo-4-hydroxy-5-methoxyphenyl)-1-propanone

[103653-15-2] $C_{10}H_{11}BrO_3$ mol.wt. 259.10



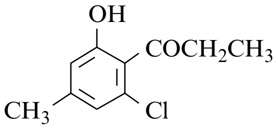
Synthesis

- Preparation by reaction of bromine with propiovanillone in dilute acetic acid (67%) [6959].

m.p. 141–142.5° [6959].

1-(2-Chloro-6-hydroxy-4-methylphenyl)-1-propanone

[24490-26-4] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



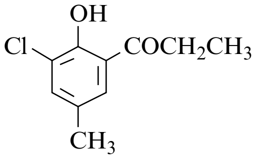
Synthesis

- Refer to: [6960].

Fluorescence spectrum data [6960].

1-(3-Chloro-2-hydroxy-5-methylphenyl)-1-propanone

$C_{10}H_{11}ClO_2$ mol.wt. 198.65



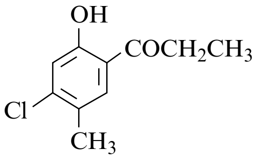
Syntheses

- Obtained by alkaline hydrolysis of 8-chloro-3,6-dimethyl-2-ethylchromone (m.p. 74–75°) or 8-chloro-3,6-dimethyl-2-propylchromone (m.p. 68–71°) with sodium ethoxide in ethanol at r.t. overnight [6428].
- Also obtained by Fries rearrangement of 2-chloro-4-methylphenyl propionate (b.p.$_{760}$ 248°) [6961] with aluminium chloride [6962], without solvent at 120° for 10 min (quantitative yield) [6961] or for 30 min [6428].
- Also refer to: [6963].

m.p. 115° [6961].

1-(4-Chloro-2-hydroxy-5-methylphenyl)-1-propanone

$C_{10}H_{11}ClO_2$ mol.wt. 198.65



Synthesis

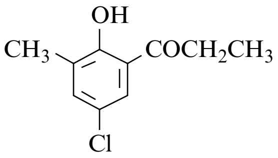
- Refer to: [6964].

Oxime [129078-81-5] $C_{11}H_{12}ClNO_2$ mol.wt. 213.66.

USE: In molybdenum determination by spectrophotometry [6964]; in uranium determination by spectrophotometry [6965].

1-(5-Chloro-2-hydroxy-3-methylphenyl)-1-propanone

[941-74-2] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



Syntheses

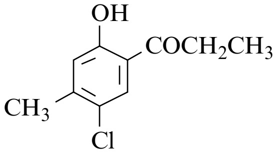
- Preparation by Friedel–Crafts acylation of 4-chloro-2-methylphenol with propionyl chloride in the presence of aluminium chloride at 80–90° for 3 h (quantitative yield) [6966].

- Also obtained by Fries rearrangement of 4-chloro-2-methyl-phenyl propionate (b.p. 249–252°) [6428] with aluminium chloride [6962], without solvent at 120° for 30 min (85%) [6428] or at 140° for 2 h [6477].
- Also obtained by alkaline hydrolysis of 6-chloro-3,8-dimethyl-2-ethylchromone (m.p. 85°) with sodium ethoxide in ethanol at r.t. overnight [6428].

m.p. 61° [6428];
^{1}H NMR [6472,6966], IR [6966], MS [6966], HRMS [6966].

1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-propanone

[22362-65-8] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



Syntheses

- Preparation by Fries rearrangement of 4-chloro-3-methyl-phenyl propionate (b.p.$_{760}$ 250°) [6961] in the presence of aluminium chloride [6351, 6488,6967], at 155° for 30 min [6490] or at 120° for 10 min (94%) [6961].
- Preparation by heating 4-chloro-3-methylphenol, propionic acid and boron trifluoride in a sealed tube 1 h at 100° (86%) [6437].
- Also refer to: [6968–6970].

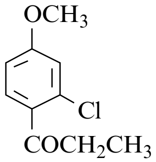
b.p.$_3$ 110–115° [6490];
m.p. 76° [6961], 67–68° [6351], 66° [6437,6967], 65.3° [6490].
^{1}H NMR (Sadtler: standard n° 8735M), IR (Sadtler: standard n° 8986),
UV [6359,6399], fluorescence spectrum data [6960]; TLC [6353].

Hydrazone [203301-19-3] $C_{10}H_{13}ClN_2O$ mol.wt. 212.68 (HMCPH).

USE: Gravimetric reagent for Cu (II), Ni (II), Co (II), Fe (II) and Fe (III) [6971].
BIOLOGICAL ACTIVITY: Antimicrobial [6968].

1-(2-Chloro-4-methoxyphenyl)-1-propanone

[13329-61-8] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



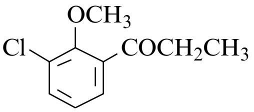
Syntheses

- Obtained from 3-chloroanisole by reaction,
  - with propionic anhydride in the presence of aluminium chloride in carbon disulfide (61%) [6949] or in the presence of ferric chloride (54%) [6972];
  - with propionyl chloride in the presence of aluminium chloride (63%) [6972].
- Also refer to: [6455,6950].

b.p.$_{12}$ 152° [6972], b.p.$_{12}$ 152–153° [6949], b.p.$_{16}$ 159–160° [6455];
m.p. 27–28° [6455].

1-(3-Chloro-2-methoxyphenyl)-1-propanone

[68597-43-3] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



Syntheses

- Preparation by reaction of ethylmagnesium bromide with 3-chloro-2-methoxybenzonitrile [6973].
- Preparation by oxidation of 1-(3-chloro-2-methoxyphenyl)-1-propanol with chromium trioxide in the presence of dilute sulfuric acid in acetone [6477].
- Also refer to: [6469,6974].

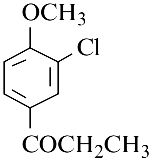
b.p.$_{15}$ 135–140° [6973], b.p.$_{50}$ 170° [6477].

USE: Preparation of tetrahydronaphthalines as antiinflammatory [6469].

2,4-Dinitrophenylhydrazone $C_{16}H_{15}ClN_4O_5$ mol.wt. 378.70 (m.p. 160°) [6973].

1-(3-Chloro-4-methoxyphenyl)-1-propanone

[4394-54-1] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



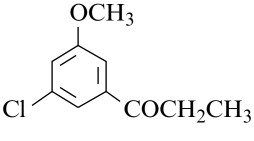
Syntheses

- Preparation by acylation of 2-chloroanisole,
  - with propionic anhydride in the presence of ferric chloride (62%) [6972];
  - with propionyl chloride in the presence of aluminium chloride [6430] in carbon disulfide (62%) [6975], (55%) [6972], in methylene chloride for 3 h at 0° [6976] or for 1.5 h at r.t. with addition of ammonium chloride (72%) [6977].
- Also obtained by oxidation of 1-(3-chloro-4-methoxyphenyl)-1-propanol with PDC in methylene chloride at 25° overnight (82%) [6930].
- Also refer to: [6363,6929,6978].

b.p.$_{12}$ 149° [6972];
m.p. 91–92° [6930], 88–90° [6978], 88–89° [6977], 88° [6430,6975].
^{1}H NMR [6930], ^{13}C NMR [6930], MS [6930].

1-(3-Chloro-5-methoxyphenyl)-1-propanone

[89106-39-8] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



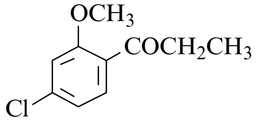
Synthesis

- Obtained (method P) by reaction of ethylmagnesium bromide with 3-chloro-5-methoxybenzoyl chloride in THF at −78° (83%) [6979].

oil [6979].

1-(4-Chloro-2-methoxyphenyl)-1-propanone

[36871-55-3] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



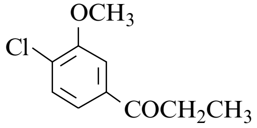
Syntheses

– Preparation by reaction of dimethyl sulfate with 4-chloro-2-hydroxypropiophenone in the presence of sodium hydroxide [6980,6981].

IR [6980].

1-(4-Chloro-3-methoxyphenyl)-1-propanone

[89106-18-3] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



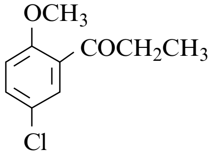
Synthesis

– Obtained (method O) by reaction of diethylcadmium with 4-chloro-3-methoxybenzoyl chloride (59%) [6979].

oil [6979].

1-(5-Chloro-2-methoxyphenyl)-1-propanone

[68597-44-4] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



Syntheses

– Preparation by acylation of 4-chloroanisole,

- with propionic anhydride in the presence of ferric chloride (62%) [6972];
- with propionyl chloride in the presence of aluminium chloride [6489,6975], (55%) [6972].

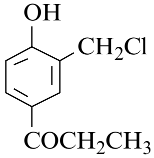
– Preparation by reaction of methyl iodide with 5-chloro-2-hydroxypropiophenone in the presence of ethanolic sodium ethoxide [6488] or methanolic sodium methoxide [6477].

– Also refer to: [6974].

b.p.$_6$ 135–140° [6488,6975], b.p.$_{16.5}$ 137° [6477], b.p.$_{12}$ 150° [6972];
m.p. 41–42° [6488,6975].

1-[3-(Chloromethyl)-4-hydroxyphenyl]-1-propanone

[106909-28-8] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



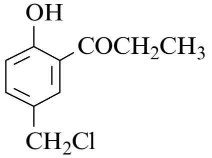
Syntheses

– Preparation by chloromethylation of 4-hydroxy-propiophenone [6982,6983] according to the procedure [6984].

– Also refer to: [6985] (Czech patent).

1-[5-(Chloromethyl)-2-hydroxyphenyl]-1-propanone

[99070-79-8] $C_{10}H_{11}ClO_2$ mol.wt. 198.65



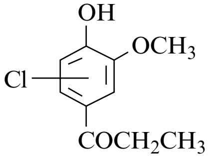
Syntheses

- Obtained by reaction of paraformaldehyde with o-hydroxy-propiophenone in the presence of hydrochloric acid at 30° for 7 h (33%) [6986].
- Also refer to: [6942].

b.p.$_4$ 145–150° [6986]; m.p. 54–55° [6986].

1-(X-Chloro-4-hydroxy-3-methoxyphenyl)-1-propanone

[67166-42-1] $C_{10}H_{11}ClO_3$ mol.wt. 214.65



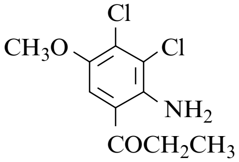
Natural sources

- In bleaching wastewater from pulp mill [6987].

GC-MS [6987].

1-(2-Amino-3,4-dichloro-5-methoxyphenyl)-1-propanone

[113730-35-1] $C_{10}H_{11}Cl_2NO_2$ mol.wt. 248.11



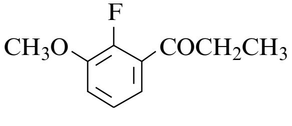
Synthesis

- Obtained by adding a solution of propionitrile and 2,3-dichloro-4-methoxyaniline in methylene chloride to a solution of boron trichloride in methylene chloride. Next, solid aluminium chloride was added to the mixture between 5° and 12°, then kept at r.t. for 30 min and at 75° for 90 h (34%) [6386].

m.p. 86° [6386]; ^{1}H NMR [6386].

1-(2-Fluoro-3-methoxyphenyl)-1-propanone

[934637-29-3] $C_{10}H_{11}FO_2$ mol.wt. 182.19

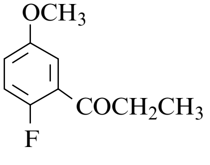


Synthesis

- Refer to: [6988].

1-(2-Fluoro-5-methoxyphenyl)-1-propanone

[89106-46-7] $C_{10}H_{11}FO_2$ mol.wt. 182.19



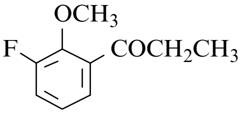
Synthesis

- Obtained (method P) by reaction of ethylmagnesium bromide with 2-fluoro-5-methoxybenzoyl chloride in THF at −78° (67%) [6979].

oil [6979].

1-(3-Fluoro-2-methoxyphenyl)-1-propanone

[879339-88-5] $C_{10}H_{11}FO_2$ mol.wt. 182.19



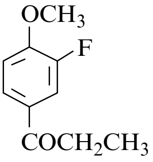
Syntheses

- Refer to: [6469,6517].

USE: Preparation of heteroaryl alkylidenetetrahydro-naphthalenamines as antiinflammatories [6517]; preparation of tetrahydronaphthalines as antiinflammatory [6469].

1-(3-Fluoro-4-methoxyphenyl)-1-propanone

[586-22-1] $C_{10}H_{11}FO_2$ mol.wt. 182.19



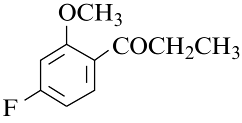
Syntheses

- Obtained by reaction of propionyl chloride [6978,6989] or propionic anhydride [6518] with 2-fluoroanisole in the presence of aluminium chloride in carbon disulfide.
- Also refer to: [6950,6954–6956].

b.p.$_{15}$ 160–162° [6518];
m.p. 86° [6518], 84° [6955], 83–85° [6978], 83–84° [6954,6956];
IR [6978].

1-(4-Fluoro-2-methoxyphenyl)-1-propanone

[231958-06-8] $C_{10}H_{11}FO_2$ mol.wt. 182.19



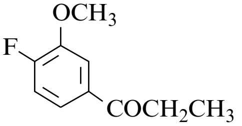
Syntheses

- Preparation by reaction of methyl iodide with 4-fluoro-2-hydroxypropiophenone in the presence of potassium carbonate in acetone at 40° for 12 h (97%) [6522].
- Also refer to: [6990].

^{1}H NMR [6522].

1-(4-Fluoro-3-methoxyphenyl)-1-propanone

[82846-20-6] $C_{10}H_{11}FO_2$ mol.wt. 182.19



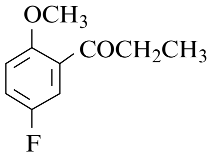
Synthesis

- Obtained (method O) by reaction of diethylcadmium with 4-fluoro-3-methoxybenzoyl chloride (68%) [6979].

m.p. 180–181° [6979].

1-(5-Fluoro-2-methoxyphenyl)-1-propanone

[653-64-5] $C_{10}H_{11}FO_2$ mol.wt. 182.19



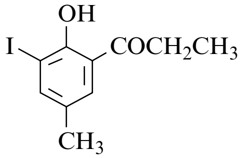
Syntheses

- Preparation by reaction of propionyl chloride with 4-fluoroanisole in the presence of aluminium chloride in carbon disulfide [6363,6365].
- Also refer to: [6954,6956].

b.p.$_{12}$ 100° [6954], b.p.$_{13}$ 132° [6365];
n_D^{23} = 1.5175 [6365].

1-(2-Hydroxy-3-iodo-5-methylphenyl)-1-propanone

[868606-11-5] $C_{10}H_{11}IO_2$ mol.wt. 290.10

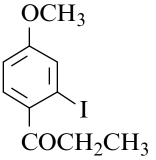


Synthesis

- Preparation by reaction of iodine and iodic acid with 2-hydroxy-5-methylpropiophenone in dilute ethanol for 1.5 h at 35–40° (86%) [6366].

1-(2-Iodo-4-methoxyphenyl)-1-propanone

$C_{10}H_{11}IO_2$ mol.wt. 290.10



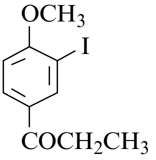
Synthesis

- Obtained by reaction of propionic anhydride with 3-iodoanisole in the presence of aluminium chloride in carbon disulfide (58%) [6949].

b.p.$_6$ 158–162° [6949]; m.p. 61–63° [6949].

1-(3-Iodo-4-methoxyphenyl)-1-propanone

$C_{10}H_{11}IO_2$ mol.wt. 290.10



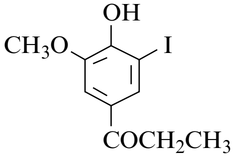
Syntheses

- Obtained by reaction of propionic anhydride with 2-iodoanisole in the presence of aluminium chloride in carbon disulfide [6518].
- Also refer to: [6672].

b.p.$_2$ 170° [6518]; m.p. 95° [6518].

1-(4-Hydroxy-3-iodo-5-methoxyphenyl)-1-propanone

[103653-13-0] $C_{10}H_{11}IO_3$ mol.wt. 306.10



Synthesis

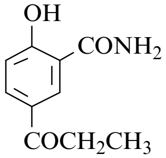
– Preparation by adding an aqueous solution of iodine and potassium iodide to a solution of 4-hydroxy-3-methoxy-propiophenone in aqueous sodium hydroxide (90%) [6959].

m.p. 136.5° [6959].

2-Hydroxy-5-(1-oxopropyl)benzamide

5-Propionylsalicylamide

$C_{10}H_{11}NO_3$ mol.wt. 193.20



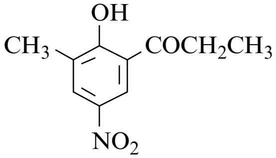
Synthesis

– Preparation by Friedel–Crafts acylation of salicylamide (m.p. 140°) (compound **8407**) [6640] with propionyl chloride in the presence of aluminium chloride in nitrobenzene at 20° for 12 h (90%) [6939].

m.p. 216° [6939].

1-(2-Hydroxy-3-methyl-5-nitrophenyl)-1-propanone

[90922-89-7] $C_{10}H_{11}NO_4$ mol.wt. 209.20



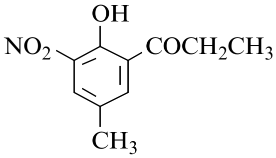
Synthesis

– Preparation by reaction of nitric acid (d = 1.52) with 2-hydroxy-3-methylpropiophenone in concentrated sulfuric acid (d = 1.80) between −2° and 0° (82%) [6367].

m.p. 113° [6367].

1-(2-Hydroxy-5-methyl-3-nitrophenyl)-1-propanone

[70978-40-4] $C_{10}H_{11}NO_4$ mol.wt. 209.20



Syntheses

– Preparation by nitration of 2-hydroxy-5-methyl-propiophenone,

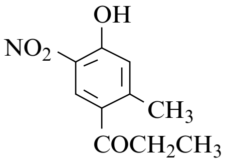
- with nitric acid in acetic acid [6991] at r.t. for 2.5 h (80%) [6992];
- with nitric acid (d = 1.52) in sulfuric acid (d = 1.8), between −2° and 0° for 20 min (75%) [6367].

– Also refer to: [6993].

m.p. 151° [6367], 136–137° [6992], 135–136° [6991]. One of the reported melting points is obviously wrong.

1-(4-Hydroxy-2-methyl-5-nitrophenyl)-1-propanone

[90922-90-0] $C_{10}H_{11}NO_4$ mol.wt. 209.20



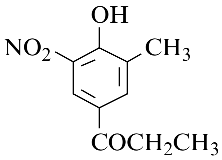
Syntheses

- Preparation by reaction of concentrated nitric acid (d = 1.42) with 4-hydroxy-2-methylpropiophenone in acetic acid [6994].
- Preparation by reaction of nitric acid (d = 1.52) with 4-hydroxy-2-methylpropiophenone in concentrated sulfuric acid (d = 1.80) between −2° and 0° (65%) [6367].

m.p. 95° [6994], 92° [6367].

1-(4-Hydroxy-3-methyl-5-nitrophenyl)-1-propanone

[141771-81-5] $C_{10}H_{11}NO_4$ mol.wt. 209.20

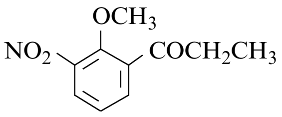


Synthesis

- Obtained by nitration of 4-hydroxy-3-methylpropiophenone (62%) [6995].

1-(2-Methoxy-3-nitrophenyl)-1-propanone

[103205-56-7] $C_{10}H_{11}NO_4$ mol.wt. 209.20



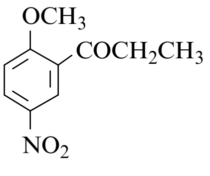
Synthesis

- Preparation by reaction of dimethyl sulfate with 2-hydroxy-3-nitropropiophenone in the presence of potassium carbonate in boiling acetone for 8–10 h (50%) [6533].

yellow oil [6533]; b.p.$_{10}$ 160–165° [6533];
d_{20} 1.2136 [6533];
n_D^{21} = 1.5379 [6533]; UV [6533].

1-(2-Methoxy-5-nitrophenyl)-1-propanone

[682320-25-8] $C_{10}H_{11}NO_4$ mol.wt. 209.20



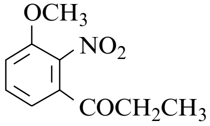
Synthesis

- Obtained by adding tetrabutylammonium nitrate to a solution of 2-methoxypropiophenone, trifluoroacetic anhydride and 18-crown-6 in methylene chloride under argon, then the solution mixed at r.t. for 3.5 h (91%) [6409].

white solid [6409];
^{1}H NMR [6409], ^{13}C NMR [6409], MS [6409]; TLC [6409].

1-(3-Methoxy-2-nitrophenyl)-1-propanone

[103204-36-0] $C_{10}H_{11}NO_4$ mol.wt. 209.20



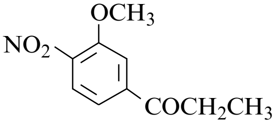
Synthesis

- Obtained by reaction of methyl iodide with ethyl 3-methoxy-2-nitrobenzoylmalonate in ethanolic sodium ethoxide solution [6996].

m.p. 96° [6996]; sublimation at 80°/0.001 mm [6996];
IR [6997], UV [6996].

1-(3-Methoxy-4-nitrophenyl)-1-propanone

[246041-90-7] $C_{10}H_{11}NO_4$ mol.wt. 209.20

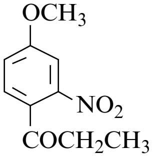


Synthesis

- Obtained by hydrolysis of 1-[1-(3-methoxy-4-nitrophenyl)-1-phenoxypropyl]-1*H*-benzotriazole in dioxane with dilute sulfuric acid [6998].

1-(4-Methoxy-2-nitrophenyl)-1-propanone

[37888-90-7] $C_{10}H_{11}NO_4$ mol.wt. 209.20

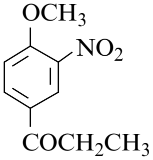


Synthesis

- Refer to: [6999].

1-(4-Methoxy-3-nitrophenyl)-1-propanone

[103204-39-3] $C_{10}H_{11}NO_4$ mol.wt. 209.20



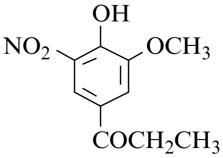
Syntheses

- Preparation by reaction of concentrated nitric acid with p-methoxypropiophenone in sulfuric acid between 0° and 5° for 30 min (64%) [7000], (77%) [6937], (96%) [7001,7002].
- Also obtained by treatment of 2,2′-dimethoxy-5,5′-di-propionyldiphenyl sulfide (m.p. 175°) with concentrated sulfuric acid/concentrated nitric acid mixture at r.t. overnight [7003].
- Also refer to: [7004].

m.p. 101° [7001], 100–101° [6937,7005], 99–101° [7002], 99–100° [7003], 86° [7000]. One of the reported melting points is obviously wrong.

1-(4-Hydroxy-3-methoxy-5-nitrophenyl)-1-propanone

$C_{10}H_{11}NO_5$ mol.wt. 225.20



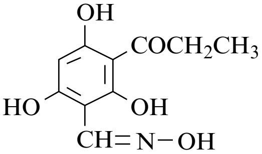
Synthesis

- Obtained (poor yield) by nitration of 1-(4-hydroxy-3-methoxyphenyl)-1-propanol with nitric acid (2 mol) in carbon tetrachloride at 5° [7006].

m.p. 160–161° [7006].

2,4,6-Trihydroxy-3-(1-oxopropyl)benzaldehyde 1-oxime

[96573-44-3] $C_{10}H_{11}NO_5$ mol.wt. 225.20



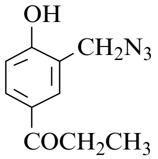
Synthesis

- Refer to: [7007] (compound **36**).

m.p. 168–170° [7007];
^1H NMR [7007], IR [7007], MS [7007].

1-[3-(Azidomethyl)-4-hydroxyphenyl]-1-propanone

[154603-70-0] $C_{10}H_{11}N_3O_2$ mol.wt. 205.22

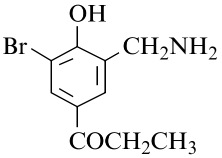


Synthesis

- Preparation by reaction of 3-(chloromethyl)-4-hydroxypropiophenone with sodium azide in DMF at 30° for 24 h [6985].

1-[3-(Aminomethyl)-5-bromo-4-hydroxyphenyl]-1-propanone

[104129-04-6] $C_{10}H_{12}BrNO_2$ mol.wt. 258.11



Syntheses

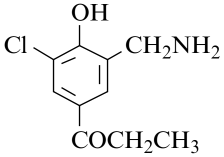
- Preparation by bromination of 3-(aminomethyl)-4-hydroxy-propiophenone [6476,7008].

Oxime [104129-05-7] $C_{10}H_{13}BrN_2O_2$ mol.wt. 273.12 [6476,7008].

BIOLOGICAL ACTIVITY: Diuretic and hypohypertensive [6476,7008].

1-[3-(Aminomethyl)-5-chloro-4-hydroxyphenyl]-1-propanone

[109314-57-0] $C_{10}H_{12}ClNO_2$ mol.wt. 213.66



Syntheses

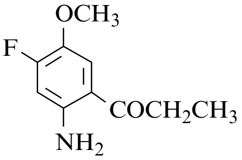
- Refer to: [6476,7008].

Oxime [104129-08-0] $C_{10}H_{13}ClN_2O_2$ mol.wt. 228.67 [6476,7008].

BIOLOGICAL ACTIVITY: Diuretic and antihypertensive [7008].

1-(2-Amino-4-fluoro-5-methoxyphenyl)-1-propanone

[124623-37-6] $C_{10}H_{12}FNO_2$ mol.wt. 197.21



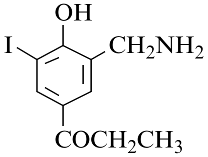
Syntheses

- Preparation by successively adding propionitrile and aluminium chloride to a solution of boron trichloride and 3-fluoro-4-methoxyaniline in benzene under nitrogen and refluxing the mixture obtained for 8 h (70%) [7009].
- Also refer to: [7010].

m.p. 96.5–97.5° [7009]; MS [7009].

1-[3-(Aminomethyl)-4-hydroxy-5-iodophenyl]-1-propanone

[109314-56-9] $C_{10}H_{12}INO_2$ mol.wt. 305.11



Syntheses

- Refer to: [6476,7008].

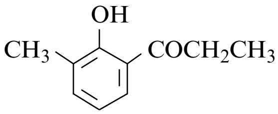
Oxime [104129-07-9] $C_{10}H_{13}IN_2O_2$ mol.wt. 320.12 [6476,7008].

Note: Isomerization [7011].

BIOLOGICAL ACTIVITY: Diuretic and antihypertensive [7008].

1-(2-Hydroxy-3-methylphenyl)-1-propanone

[3338-15-6] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

- Preparation by Fries rearrangement of o-cresyl propionate,

- with aluminium chloride [6389,6562], without solvent, at 50° for 3 h (16%) [6355], at 120° for 3 h (40%) [7012], (49%) [7013], at 130° (35%) [7014], at 160° for 2 h (60%) [7015] or at 165° for 1 h (41–45%) [6355,6466] or in nitromethane at 20° for a week (18%) [6569];
- with zirconium chloride in o-dichlorobenzene over 1 h at 120° [6467].

– Also obtained by Fries rearrangement of o-cresyl propionate with titanium tetrachloride at 50° for 3 h (38%) [6355].

– Also obtained by isomerization of 4′-hydroxy-3′-methylpropiophenone at 180–200° for 1 h in the presence of aluminium chloride (1.5 mol) (43%) [6468] or (2.5 mol) (59%) [6466].

– Also obtained from 5-tert-butyl-2-hydroxy-3-methylpropiophenone by tert-butyl group elimination with aluminium chloride (2 equiv) at 190° for 15 min (77%) [6466].

– Also obtained by acylation of o-cresol with propionic acid in the presence of zinc chloride at reflux for 30 min (25%) (Nencki reaction) [7016].

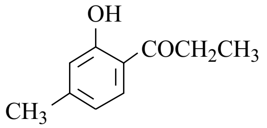
– Also refer to: [7017,7018].

b.p.$_{20}$ 115–117° [6355], b.p.$_{15}$ 127–129° [7012–7014,7016],
b.p.$_{3}$ 140° [6389,6562], b.p.$_{760}$ 222–224° [7015];
m.p. 22–23° [7012,7013]; d$_{20}$ 1.088 [6355];
n_D^{20} = 1.548 [6355];
^{1}H NMR (Sadtler: standard n° 8374M), IR (Sadtler: standard n° 37029) [6355], UV [6359,6457].

Note: Isomerization [7011].

1-(2-Hydroxy-4-methylphenyl)-1-propanone

[2886-52-4] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

– Preparation by Fries rearrangement of m-cresyl propionate with aluminium chloride [7019],

- in nitromethane at 20° for 7 days (81%) [6569];
- in nitrobenzene at r.t. [6620,7020], (79%) [6571], (63–67%) [7021,7022];
- first, in carbon disulfide, then at 60–70° for 1 h after solvent elimination and at r.t. for 24 h (76%) [7023];
- without solvent at 100° (86%) [7019], (71%) [6355,6576], 120° (85%) [7014], 120–150° [7024], (45%) [7025], (93%) [7021], (39%) [7013] or 165° (89%) [6355].

– Preparation by Fries rearrangement of m-cresyl propionate using various catalysts,

- with titanium tetrachloride without solvent at 100° for 7 h (96%) [6355,6576] or in nitromethane at 20° for 7 days (86%) [6569];
- with boron trifluoride in propionic acid at 70° for 2 h (90%) [6583];
- with stannic chloride without solvent at 100° for 7 h (56%) [6355];
- with zinc chloride at 150–160° for 1 h (20%) [6620].

- Also obtained by Fries rearrangement of 2-tert-butyl-5-methylphenyl propionate using various catalysts. In this reaction, the tert-butyl group elimination precedes the Fries rearrangement.
  - without solvent, in the presence of aluminium chloride at 120° for 30 min (45%) [7025];
  - with solvents, in the presence of various catalysts, at r.t. for 2 weeks [7025] (in the table below),

Catalyst	Solvent	Yield (%)
$AlCl_3$	Nitromethane	56
	Ethylene dichloride	17
$TiCl_4$	Nitromethane	22
	Ethylene dichloride	39
	Ethylene tetrachloride	32
$NbCl_5$	Nitromethane	26
	Ethylene dichloride	21
$TaCl_5$	Nitromethane	26
	Ethylene dichloride	25

- Also obtained (by-product) by Fries rearrangement of 2-isopropyl-5-methylphenyl propionate with aluminium chloride (1.4 equiv) without solvent at 100° for 2 h (14%) [7026].
- Also obtained by acylation of m-cresol with propionic acid,
  - in the presence of boron trifluoride for 2 h at 70° (93%) [6583] or 80° (83%) [6572] and for 30 min at 125–130° (50%) [7027];
  - in the presence of zinc chloride (Nencki reaction) at reflux for 20 min (good yield) [6667], (11%) [6620];
  - in the presence of polyphosphoric acid (low yield) [7028].
- Also obtained from 2-allyl-5-methylphenol by treatment with perbenzoic acid in ethyl ether, first at 0°, then between 0° and 25° for 24 h (74%) [6589].
- Also obtained by treatment of 5-tert-butyl-4-hydroxy-2-methylpropiophenone with aluminium chloride at 200° for 15 min (40%) [7025]. There are first a tert-butyl group elimination, followed by an acyl group migration from *para* to *ortho* position.
- Also obtained by reduction of 2-chloro-2′-hydroxy-4′-methylpropiophenone with zinc powder in acetic acid [7019].
- Also obtained by degradation of 3,7-dimethylchromone with sodium ethoxide in boiling ethanol for 30 min [7019].
- Also obtained by heating various ketones (SM) with a large excess of aluminium chloride (5.2 equiv) in chlorobenzene at 100° for 2 h [7026] (to see table below):

SM	Yield (%)
2-Hydroxy-4-methyl-3-isopropylpropiophenone	100
2-Hydroxy-4-methyl-5-isopropylpropiophenone	85
2-Hydroxy-6-methyl-3-isopropylpropiophenone	37
2-Hydroxy-4-methyl-6-isopropylpropiophenone	22

- Also obtained by isomerization of 4-hydroxy-2-methylpropiophenone with aluminium chloride (3 mol) for 1 h at 180–200° (43%) [6482].
- Also refer to: [6364,6614,6639,7011,7029–7032].

b.p.$_4$ 101–102° [7014], b.p.$_{10}$ 115–120° [6572,7019], b.p.$_{12}$ 124° [6355],
b.p.$_{15}$ 125–135° [6620], b.p.$_{13}$ 133–135° [6583];
m.p. 45–46° [6355,6620,7023], 44–46° [7022], 44–45° [7027], 44° [6583,6667], 43–44° [7013,7014,7020,7021], 43° [7024], 41.5–42.5° [7019], 41° [6572];
^{1}H NMR (Sadtler: standard n° 8377M),
IR (Sadtler: standard n° 37033), [6355,6356,7027],
UV [6356,6359,6457,7027]; ESR spectrum [6350]; TLC [6353,6358].
Notes: Acidity of methylene proton [6446]; radical ion (1^-) [72051-76-4]; C-deuteration [6447].
USE: Metal-ligand stability constants of complexes of 2-hydroxy-4-methylpropiophenone,

- with cobalt [41653-56-9], iron [41586-13-4] and nickel [41586-14-5] [6633];
- with cadmium [37848-06-9], copper [37848-05-8] and zinc complexes [6636].

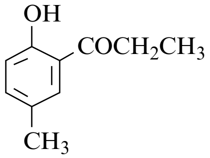
BIOLOGICAL ACTIVITY: Nematocide [7030].

Thiosemicarbazone $C_{11}H_{15}N_3OS$ mol.wt. 237.33 (m.p. 181°)

USE: Fungicide [6364].
BIOLOGICAL ACTIVITY: Antituberculotic [6364]

1-(2-Hydroxy-5-methylphenyl)-1-propanone

[938-45-4] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

- Preparation by acylation of p-cresol with propionic acid using various catalysts,
    - in the presence of boron trifluoride for 2 h at 80° (87%) [6572] or 70° (80%) [6583];
    - in the presence of zinc chloride (Nencki reaction) for 1 h at reflux (55%) [7016].
- Preparation by Fries rearrangement of 4-methylphenyl propionate [7033–7035], (80%) [7036] using various conditions,
    - with aluminium chloride, without solvent, at 110° for 90 min [6409], at 120° (90%) [7014] for 40 min (93%) [6992], at 165° for 1 h (88%) [6355,6356], at 170° for 1–2 h (80–96%) [6492] or heating on a water bath for 1 h (80%) [7037];
    - with aluminium chloride (1 equiv), without solvent, at 100° for 1 h (50%) [7013] or in nitromethane at 20° for 7 days (58%) [6569].

- Preparation by dealkylation of phenolic ethers,
    - from 2-ethoxy-5-methylpropiophenone by treatment with aluminium chloride in refluxing carbon disulfide, then heating at 60–70° for 8 h after solvent elimination [6673,7038] according to the Gattermann procedure [6674];
    - from 2-methoxy-5-methylpropiophenone by refluxing with pyridinium chloride for 4 h (77%) [7023];
    - from direct acylation of 4-methylanisole with propionyl chloride in the presence of aluminium chloride in methylene chloride (82%) [6946], in petroleum ether heating for 30 h in a water bath (42%) [7039] or in refluxing carbon disulfide for 5 h, dealkylation of the obtained 2-methoxy-5-methylpropiophenone occuring in situ [7033,7037,7040].
- Preparation by Friedel–Crafts acylation of p-cresol,
    - with propionyl chloride in the presence of aluminium chloride in ethylene dichloride at 110–120° for 8 h (70%) [7041];
    - with propionic anhydride in the presence of aluminium chloride (2 mol) and a trace of magnesium perchlorate at 170° for 1 h (96%) [6491].
- Also obtained by adding aluminium chloride, followed by addition over 6 h of propionyl chloride to aluminium p-cresylate in benzene. After standing 15 h, the mixture was refluxed 2 h (82%) [7042].
- Preparation from 3-iodo-2′-hydroxy-5′-methylpropiophenone by treatment with zinc powder in 80% acetic acid and heating on a water bath [7043].
- Also obtained by treatment of 2-hydroxy-3-isopropyl-6-methylpropiophenone with aluminium chloride (1.4 equiv) without solvent at 100° for 2 h (25%) [7026].
- Also refer to: [6496,7011,7044] (compound **3**), [7045] (compound **3b**) and [6364,7046–7051].
- Also obtained by treatment of 3,6-dimethylchromone with sodium methoxide in boiling methanol for 30 min [7052].

yellow liquid [6409], oil [7052,7053];
$n_D^{14.5}$ = 1.5480 [7042];
b.p.$_2$ 97° [7035], b.p.$_4$ 108–109° [7041], b.p.$_{10}$ 115–117° [6992], b.p.$_5$ 115–117° [7042],
b.p.$_{11}$ 123–124° [7033,7037], b.p.$_{13}$ 126–128° [6583], b.p.$_{15}$ 128° [6572],
b.p.$_{15}$ 128.5–129° [7040], b.p.$_{16-16.5}$ 129–130° [7013,7014], b.p.$_{15}$ 130–132° [7023],
b.p.$_{15}$ 131° [6356], b.p.$_{22}$ 135–140° [7038],
b.p.$_{40}$ 153° [7016,7039,7054], b.p.$_{30}$ 160° [7036], b.p. 241° [7034];
m.p. 2° [7039,7054], −2° [7038], −10° [7040];
^{1}H NMR (Sadtler: standard n° 8918M) [6409,6472,7053],
IR (Sadtler: standard n° 38070) [6355,6356,7035,7053],
UV [6356,6359,6457,7033,7035], MS [6409];
cryoscopic study [6673]; TLC [6353,6358]; pK_a [7034].

Notes: Acidity of methylene proton [6446]; deuteration [6447];
USE: Herbicide-antidote [6384].

Ethyl ether $C_{12}H_{16}O_2$ mol.wt. 192.26 (m.p. 50–51°) [7055].

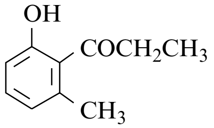
- Preparation by reaction of diethyl sulfate with 2-hydroxy-5-methylpropiophenone in the presence of sodium hydroxide [7055].

Acetate		$C_{12}H_{14}O_3$	mol.wt. 206.24 (m.p. 58°) [7056].
Oxime	[10080-44-1]	$C_{10}H_{13}NO_2$	mol.wt. 179.21 [6384,6502,7057].
Hydrazone	[70136-39-9]	$C_{10}H_{14}N_2O$	mol.wt. 178.23 [6646].
Thiosemicarbazone		$C_{11}H_{15}N_3OS$	mol.wt. 237.33 (m.p. 178°).

USE: Fungicide [6364].
BIOLOGICAL ACTIVITY: Antituberculotic [6364].

1-(2-Hydroxy-6-methylphenyl)-1-propanone

[51451-26-4] $C_{10}H_{12}O_2$ mol.wt. 164.20



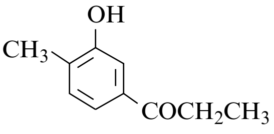
Syntheses

- Preparation from 3-tert-butyl-2-hydroxy-6-methylpropiophenone by tert-butyl group elimination with aluminium chloride in nitromethane at 20° for a week (89–90%) [7011,7025].
- Preparation from 2-allyl-3-methylphenol by treatment with perbenzoic acid in ethyl ether, first at 0°, then between 0° and 25° for 24 h (69%) [6589].
- Also obtained by demethylation of 2-methoxy-6-methylpropiophenone with aluminium chloride in refluxing carbon disulfide for 6 h (14%) [7058].
- Also obtained from 2-hydroxy-3-isopropyl-6-methylpropiophenone by heating with aluminium chloride (5.2 equiv) in chlorobenzene at 100° for 2 h (40%) [7026].
- Also obtained by alkaline degradation of 2,3,5-trimethylchromone with boiling aqueous sodium hydroxide [6427,7059] or potassium hydroxide [7060].

b.p.$_{11}$ 129–130° [7011], b.p.$_5$ 140° [7025,7058];
m.p. 28.5° [6427,7059], 27° [7011], 25–27° [7025,7058];
^{1}H NMR (Sadtler: standard n° 57891M) [7011],
IR (Sadtler: standard n° 84939K) [7011,7025], UV [7011,7025], MS [7011].

1-(3-Hydroxy-4-methylphenyl)-1-propanone

[18158-56-0] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

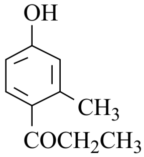
- Preparation by diazotization of 3-amino-4-methyl-propiophenone, followed by hydrolysis of the diazonium salt formed [6389, 6562, 7061, 7062], (79%) [7063], (67%) [7064].

m.p. 123° [6389,6562], 121° [7064], 120–122° [7062], 119–121° [7063];
^{1}H NMR [7063], IR [7063].

Sodium salt [7062].

1-(4-Hydroxy-2-methylphenyl)-1-propanone

[2887-55-0] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

- Obtained (poor yield) by Fries rearrangement of m-cresyl propionate (b.p.$_{10}$ 108–110°) [7019] with aluminium chloride,
  - in nitrobenzene at 2° for 24 h (10%) [7021] or at 0° for 5 days [7020];
  - without solvent at 50° for 3 h (21%) [6355,6576], at 100° for 90 min [6994], (11%) [7019] or for 7 h (12%) [6355,6356] or at 120° (6%) [7014].
- Also obtained (poor yield) by Fries rearrangement of m-cresyl propionate with boron trifluoride in propionic acid at 70° for 2 h (8%) [6583].
- Also obtained (poor yield) by Fries rearrangement of p-thymyl propionate with aluminium chloride at 100° for 2 h (11%) [7026].
- Also obtained by reaction of propionyl chloride with m-cresol in the presence of aluminium chloride in nitrobenzene at 15° for 42 h (39%) [7065].
- Also obtained (poor yields) by reaction of propionic acid with m-cresol,
  - in the presence of boron trifluoride at 80° for 2 h [6572] or at 70° for 2 h (6%) [6583];
  - in the presence of polyphosphoric acid [7028].
- Preparation by heating 4-methoxy-2-methylpropiophenone with pyridinium chloride at reflux for 10–15 min [7066], (90%) [6667], (78%) [6672].
- Also obtained by treatment of 2-methoxy-6-methylpropiophenone (b.p.$_{16}$ 137°; m.p. 8°) with aluminium chloride in refluxing carbon disulfide for 6 h (68%) [7058].
- Preparation by treatment of 4-hydroxy-2-methyl-5-isopropylpropiophenone with aluminium chloride in chlorobenzene, first at r.t. for 24 h, then at 50° for 3 h (63%) [7067].
- Also obtained by reaction of propionyl chloride with 3-methylanisole in the presence of aluminium chloride in refluxing methylene chloride for 3 h [6976].
- Also refer to: [7022,7031].

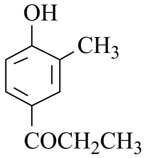
m.p. 127° [7014], 120° [6583,7067], 118° [7065], 117–119° [6355,6356], 117–118° [7021], 116° [6667,7066], 114–115° [7019], 114° [7058], 110–111° [7020]. There is a large dispersion of melting points pointed out in literature.

^{1}H NMR (Sadtler: standard n° 8378M) [7020],
IR (Sadtler: standard n° 37034) [6355,6356,7020],
UV [6356,6357,6457]; TLC [6353,6358].

BIOLOGICAL ACTIVITY: Nematocide [7030].

1-(4-Hydroxy-3-methylphenyl)-1-propanone

[940-04-5] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

– Preparation by Fries rearrangement of o-cresyl propionate with aluminium chloride,

- in nitrobenzene at r.t. [7068,7069], (75%) [7070];
- in nitromethane at 20° for a week (58%) [6569];
- first in gently refluxing carbon disulfide, then at 135–140° for 2 h after solvent elimination [6562], (43%) [6389];
- without solvent at 50° for 3 h (55%) [6355], at 120° for 3 h [7012], (15%) [7013], at 130° (65%) [7014], or at 165° for 1 h (22%) [6355].

– Also obtained by demethylation of its methyl ether by treatment with aluminium chloride in light petroleum (b.p. 100–120°), first at 100° for 4 h, then at 78–80° overnight [7071].

– Also obtained by isopropyl group elimination from 4-hydroxy-5-methyl-2-isopropylpropiophenone with aluminium chloride in chlorobenzene at r.t. for 24 h, then at 50° for 3 h (55%) [7067].

– Also obtained by acylation of o-cresol with propionic acid in the presence of zinc chloride at reflux for 5 min (15%) [6667], (11%) [7016].

– Also refer to: [6519,6995,7072–7077].

b.p.$_1$ 150–155° [7070], b.p.$_{16}$ 185–190° [7014], b.p.$_{12}$ 190–195° [6389,6562], b.p.$_{15}$ 205–210° [6667];

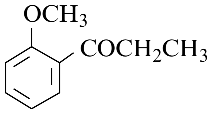
m.p. 86.5° [6389,6562], 86° [6355,6667,7067,7070], 85° [7013], 84–85° [7071], 83.5° [7012], 83–84° [7016], 83° [7014]; 63° (monohydrate) [6355];

^{1}H NMR [6472], IR (Sadtler: standard n° 37030) [7078], UV [6357,6457].

USE: Fungicide [7075].

1-(2-Methoxyphenyl)-1-propanone

[5561-92-2] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

– Preparation by reaction of ethylmagnesium bromide with o-methoxybenzonitrile (90%) [7079,7080].

– Also obtained (by-product) by Pd(II)-catalyzed aerobic dialkoxylation of 2-propenylanisole (22%) [7081].

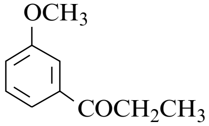
– Also obtained by propionylation of anisole with propionyl chloride and propionic anhydride over solid-acid catalysts [7082].

– Also refer to: [6980,7083–7086].

b.p.$_{12}$ 125° [7079], b.p.$_{16}$ 133° [7079].

1-(3-Methoxyphenyl)-1-propanone

[37951-49-8] $C_{10}H_{12}O_2$ mol.wt. 164.20



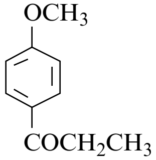
Syntheses

- Preparation by reaction of ethylmagnesium bromide,
  - with m-methoxybenzonitrile (m.p. 60°), (91–94%) [7079,7080];
  - with 3-methoxybenzaldehyde and oxidation of the resulting 1-(3-methoxyphenyl)-1-propanol with sodium dichromate in sulfuric acid (70%) [6979].
- Also refer to: [7083,7087,7088].

oil [6979]; b.p.$_{14}$ 129° [7079].

1-(4-Methoxyphenyl)-1-propanone

[121-97-1] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

- Preparation by reaction of ethylmagnesium bromide with p-methoxybenzonitrile (95%) [7079,7080].
- Preparation by acylation of anisole with propionic anhydride,
  - in the presence of chloroacetic acid for 48 h at 170–180° (86–88%) [7089];
  - in the presence of iodine for 3 h at reflux (50%) [6721].
- Preparation by acylation of anisole with propionic acid,
  - on the solid surface of alumina in the presence of trifluoroacetic anhydride (95%) [7090];
  - in the presence of $HNTf_2$ (20 mol %) in refluxing toluene for 24 h (64%) [7091];
  - and propionic anhydride in the presence of scandium triflate [7092].
- Formation from diethylpropionyloxonium chloroantimonate (V) reaction with anisole (41%) [7073].
- Also obtained by oxidation of 1-(4-methoxyphenyl)propane with DDQ in wet dioxane/silica gel under sonication (61%) [7093].
- Also refer to: [6605,6980,7033,7094–7100].

Note: Deuteration: Obtention of 1-(4-methoxyphenyl)-1-propanone-*2,2-d*$_2$ [91889-35-9] [6361].

Isolation from natural sources

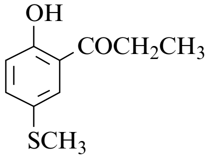
- From the essential oil of *Crithmum maritimum* L. (Umbelliferae) [7101].
- From *Pimpinella anisum* (Umbelliferae) [6704].
- From the essential oil of *Anethum graveolens* L. [7102].

- From volatile components and key odorants of Fennel (*Foeniculum vulgare* Mill.) and Thyme (*Thymus vulgaris* L.) [7103].
- From the essential oil of *Zanthoxylum rhetsa* (Roxb.) DC [7104].
- From volatile metabolites of marine *fungus Hypoxylon* sp. [7105].
- Also refer to: [6672,7083].

b.p.$_2$ 108–110° [7095], b.p.$_{12}$ 137° [7079], b.p.$_{12}$ 136–139° [7100], b.p.$_{12}$ 143–144° [7033], b.p.$_{15}$ 148–149° [7089];
m.p. 35° [7079], 27° [7089], 26–27° [6721,7100], 25–27° [7073], 25–26° [7033];
^{1}H NMR [7093], ^{13}C NMR [7093,7095], UV [7033].

1-[2-Hydroxy-5-(methylthio)phenyl]-1-propanone

[75060-89-8] $C_{10}H_{12}O_2S$ mol.wt. 196.27

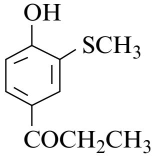


Synthesis

- Refer to: [7106].

1-[4-Hydroxy-3-(methylthio)phenyl]-1-propanone

[66265-14-3] $C_{10}H_{12}O_2S$ mol.wt. 196.27



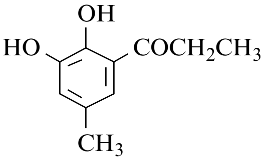
Syntheses

- Obtained by reaction of propionyl chloride with 2-(methyl-thio)phenol in nitrobenzene in the presence of aluminium chloride, first at 45–50°, then at 60–70° for 3 h (35%) [7107].
- Also obtained by reaction of propionic acid with 2-(methyl-thio)phenol in the presence of boron trifluoride [7108].
- Also refer to: [7109].

crystalline product [7107].

1-(2,3-Dihydroxy-5-methylphenyl)-1-propanone

[91061-55-1] $C_{10}H_{12}O_3$ mol.wt. 180.20



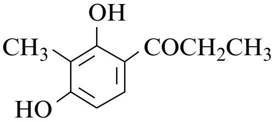
Syntheses

- Obtained by reaction of propionic acid with creosol in the presence of boron trifluoride at 160° for 90 min (60%) [7110].
- Also obtained by total demethylation of 2,3-dimethoxy-5-methylpropiophenone (b.p.$_{0.2}$ 103–105°) with boiling pyridinium chloride for 30 min (51%) [7110].

m.p. 84–85° [7110], 73° [6351]. One of the melting points is obviously wrong.
^{1}H NMR (Sadtler: standard n° 49337M), IR (Sadtler: standard n° 76410K), UV [7110].

1-(2,4-Dihydroxy-3-methylphenyl)-1-propanone

[63876-46-0] $C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

– Obtained by demethylation of 2-hydroxy-4-methoxy-3-methylpropiophenone,

- with hydriodic acid (d = 1.7) in acetic anhydride at 130–140° for 2 h (65%) [6769];
- with aluminium chloride at 135–140° for 3 h [6769].

– Preparation by reaction of propionic anhydride with 1,3-dihydroxy-2-methylbenzene in the presence of boron trifluoride etherate at 75° for 6 h (84%) [7111].

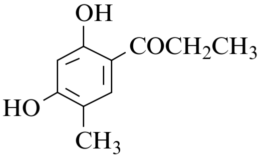
– Also obtained by reaction of propionitrile with 2-methylresorcinol (Hoesch reaction) [6768,6769].

– Also refer to: [6453,7112–7114].

m.p. 128–130° [6768,6769], 124–125° [7111];
^{1}H NMR [7111], IR [7111], UV [7111], MS [7111].

1-(2,4-Dihydroxy-5-methylphenyl)-1-propanone

[117952-43-9] $C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

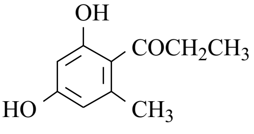
– Preparation by reaction of propionitrile with 4-methyl-resorcinol (Hoesch reaction) (66%) [7115].

– Also refer to: [7116,7117].

m.p. 109–110° [7115].

1-(2,4-Dihydroxy-6-methylphenyl)-1-propanone *(Orcpropiophenone)* *(β-Orcpropiophenone)*

[5880-41-1] $C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

– Obtained by reaction of propionitrile with orcinol [6744], (23%) [7118].

– Also obtained (by-product) by acylation of orcinol with propionic anhydride in nitrobenzene in the presence of aluminium chloride, first in a water bath for 1 h, then at r.t. overnight (5%) [7119].

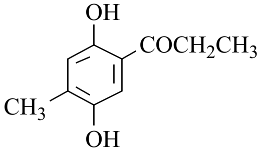
– Also obtained by reaction of propionic acid with orcinol in the presence of boron trifluoride etherate, then heating the chelate formed with dilute ethanol [6944].

– Also refer to: [7120] (compound **7**), [7121] (compound **XII**), [7122,7123] (compound **IX**).

m.p. 127–128° [7118], 122° [6744].

1-(2,5-Dihydroxy-4-methylphenyl)-1-propanone

[75859-13-1] $C_{10}H_{12}O_3$ mol.wt. 180.20



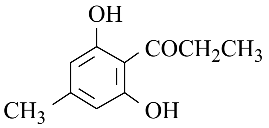
Syntheses

- Preparation by reaction of propionic acid with 2-methylhydroquinone [6507], by known methods [6509,6510].
- Also obtained (by-product) by irradiation (490 nm) of 5-methyl-2-propanoyl-1,4-benzoquinone in acetonitrile in the presence of Rose bengal for 12 h (5–10%) [7124].

m.p. 110–116° [6507].

1-(2,6-Dihydroxy-4-methylphenyl)-1-propanone *(p-Orcipropiophenone) (γ-Orcpropiophenone)*

[5792-37-0] $C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

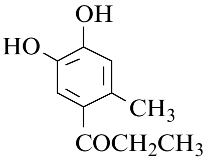
- Preparation by action of 85% sulfuric acid on di-propionylorcinol 4 h at r.t. (80%) [7125].
- Also obtained by reaction of propionic anhydride with orcinol,
  - in the presence of concentrated sulfuric acid (one drop) at 130° (60%) [6744].

  N.B.: Crystallized in yellow needles with one molecule of water.

  - in the presence of aluminium chloride in nitrobenzene, heating for 1 h on a water bath, then keeping the mixture at r.t. overnight (40%) [7119];
  - in the presence of Amberlite IR-120 (cation exchange resin sulfonic acid type) at 160° for 2–3 h (19%) [6740]. **N.B.**: Zeokarb 225 was found to be as effective.
- Also obtained (by-product) by Fries rearrangement of orcinol dipropionate with aluminium chloride (3 mol) at 140–150° for 90 min (20%) [6745].
- Also obtained by acylation of pyrogallol with propionic acid in the presence of Amberlite IR-120 (cation exchange resin sulfonic acid type) at 160° for 2–3 h (14%) [6740].
- Also refer to: [6609,7126].

m.p. 135° [7125], 132–133° [7119], 130–132° [6740], 129° [6744].

1-(4,5-Dihydroxy-2-methylphenyl)-1-propanone



$C_{10}H_{12}O_3$ mol.wt. 180.20

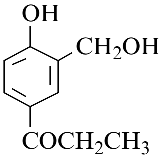
Synthesis

- Obtained by Fries rearrangement of 4-methylcatechol dipropionate with aluminium chloride [6351].

m.p. 95° [6351];
^{1}H NMR (Sadtler: standard n° 49335M),
IR (Sadtler: standard n° 76408K).

1-[4-Hydroxy-3-(hydroxymethyl)phenyl]-1-propanone

[106909-29-9] $C_{10}H_{12}O_3$ mol.wt. 180.20

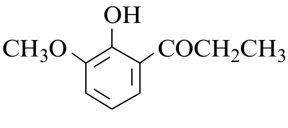


Synthesis

- Obtained by hydrolysis of 3-(chloromethyl)-4-hydroxy-propiophenone [6983].

1-(2-Hydroxy-3-methoxyphenyl)-1-propanone

[78094-43-6] $C_{10}H_{12}O_3$ mol.wt. 180.20



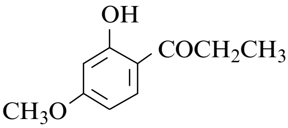
Syntheses

- Obtained by Fries rearrangement of 2-methoxyphenyl propionate with zirconium chloride in o-dichlorobenzene 1 h at 120° (good yield) [6467] or with aluminium chloride in nitrobenzene at 20° for 24 h (28%) [7127].
- Also obtained by partial demethylation of 2,3-dimethoxypropiophenone with aluminium chloride for 3 h at 0° (75%) [7128].
- Also obtained (by-product) by reaction of ethylmagnesium iodide with 2,3-dimethoxybenzonitrile (16%) [7129].
- Also obtained by reaction of ethylmagnesium bromide with 2-(benzyloxy)-3-methoxybenzonitrile in ethyl ether at r.t. overnight (11%) [7130].
- Also refer to: [7131].

m.p. 73–74° [7130], 72–73° [7128] 72° [7129].

1-(2-Hydroxy-4-methoxyphenyl)-1-propanone

[6270-44-6] $C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

- Preparation by reaction of propionyl chloride with resorcinol dimethyl ether in ethyl ether at 0° in the presence of aluminium chloride [7132,7133].

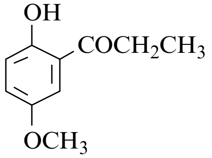
- Also obtained by reaction of propionic acid with resorcinol monomethyl ether in the presence of polyphosphoric acid for 10 min in a boiling water bath (31%) [6738].
- Also obtained by reaction of methyl iodide with 2,4-dihydroxypropiophenone in methanol in the presence of potassium hydroxide [7134] or potassium carbonate [6747].
- Also obtained by partial methylation of respropiophenone with methyl iodide [7135] or a methyl halogenide (unspecified) [6380] in the presence of potassium carbonate in refluxing acetone for 2.5 h (55%) [7135] or for 5 h (40%) [6380].
- Also obtained by partial methylation of respropiophenone [7136], with dimethyl sulfate in the presence of potassium carbonate in boiling benzene for 10 h (72%) [6762].
- Also obtained by treatment of 2-hydroxy-4-methoxy-α-(hydroxymethyl)propiophenone (pale yellow oil) with 4% aqueous sodium carbonate in refluxing ethanol for 1 h (31%) [6757].
- Preparation by Birch reduction of methoxychromone [7137].
- Also refer to: [6483,6617,6755,6757,6758,7138–7142].

m.p. 60° [6738], 58° [7134,7135], 57–58° [6757], 56° [6762], 55–57° [6380]; ^{1}H NMR (Sadtler: standard n° 30286M) [6380], IR (Sadtler: standard n° 57331) [6380]; TLC [6380].

Notes: Acidity of methylene proton [6446]; C-deuteration [6447].

1-(2-Hydroxy-5-methoxyphenyl)-1-propanone

[49710-99-8] $C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

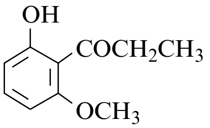
- Preparation by Fries rearrangement of p-methoxyphenyl propionate,
  - with titanium tetrachloride without solvent at 120° for 1 h (74%) [7143];
  - with aluminium chloride in nitrobenzene at 80–100° for 2 h (41%) [6465].
- Also obtained from 2-hydroxy-5-methoxy-α-chloropropiophenone by treatment with zinc in acetic acid [7144,7145].
- Also obtained (by-product) by reaction of propionyl chloride with p-dimethoxybenzene in methylene chloride in the presence of aluminium chloride at r.t. [7146,7147].
- Also refer to: [7148–7150].

b.p.$_{0.9}$ 140–142° [6465], b.p.$_{19}$ 158–160° [7144];
m.p. 85–88° [6465], 48.5° [7143], 47–49° [7144], 47–48° [7147]. One of the reported melting points is obviously wrong.
^{1}H NMR (Sadtler: standard n° 30291M), IR (Sadtler: standard n° 57336) [7143], UV [7143].

Notes: C-Deuteration [6447]; acidity of methylene proton [6446].

1-(2-Hydroxy-6-methoxyphenyl)-1-propanone

[3839-97-2] $C_{10}H_{12}O_3$ mol.wt. 180.20



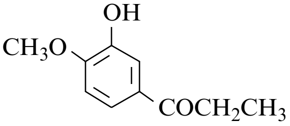
Syntheses

- Obtained by partial methylation of 2,6-dihydroxypropiophenone with dimethyl sulfate,
  - in the presence of potash in refluxing acetone (70–75%) [6801];
  - in the presence of 2 N sodium hydroxide at r.t. for 2 h (9%) [6800]. **N.B.**: In this reaction, the dimethyl ether was the main product ($C_{11}H_{14}O_3$, 37%, m.p. 45°).
- Also obtained by partial methylation of 2,6-dihydroxypropiophenone with methyl iodide (51%) [7151].
- Preparation by oxidative aromatization of 2-propionylcyclohexane-1,3-dione with iodine (2 equiv) in refluxing methanol for 13 h (79%) [7152].
- Also refer to: [7153].

vacuum sublimation [7151];
m.p. 55° [6800], 53–54° [6801], 52–53° [7151].

1-(3-Hydroxy-4-methoxyphenyl)-1-propanone

[829-76-5] $C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

- Preparation by hydrolysis of various esters,
  - of 4-methoxy-3-propionyloxypropiophenone with potassium hydroxide in refluxing methanol (92%) [7154];
  - of 3-chloroacetoxy-4-methoxypropiophenone (m.p. 77.5–78.5°) with sodium acetate in refluxing methanol for 3 h [7155].
- Also obtained by direct acylation of guaiacol with propionic acid in the presence of phosphorous oxychloride on a steam bath for 3 h (25%) [6620,7156].
- Also obtained (by-product) by Fries rearrangement of 2-methoxyphenyl propionate with various catalysts at 20° for 24 h: Aluminium chloride and titanium tetrachloride (10–13%), stannic chloride and antimony pentachloride (6–7%) [7154].
- Compound derived from secondary products of isoeugenol synthesis [7157].
- Also obtained by treatment of 2-methoxy-5-propionylphenyl methoxyphenylphosphonate with dilute hydrochloric acid in aqueous ethanol [6822].

m.p. 93–94° [6620], 93° [7154], 90–91° [6822], 89–91° [7155];
^{1}H NMR (Sadtler: standard n° 28212M), IR (Sadtler: standard n° 55284) [6822,7154],
UV [7154].

Propionate $C_{13}H_{16}O_4$ mol.wt. 236.27 (m.p. 44°) [6351];

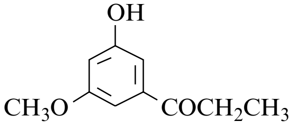
^{1}H NMR (Sadtler: standard n° 30296M), IR (Sadtler: standard n° 57341).

Benzoate $C_{17}H_{16}O_4$ mol.wt. 284.31 (m.p. 98°) [6351];

^{1}H NMR (Sadtler: standard n° 28223M), IR (Sadtler: standard n° 55295).

1-(3-Hydroxy-5-methoxyphenyl)-1-propanone

[502924-49-4] $C_{10}H_{12}O_3$ mol.wt. 180.20

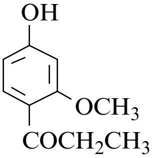


Synthesis

– Refer to: [6958].

1-(4-Hydroxy-2-methoxyphenyl)-1-propanone

$C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

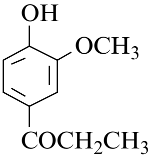
– Obtained by reaction of propionic acid with resorcinol monomethyl ether in the presence of polyphosphoric acid for 10 min in a boiling water bath (28%) [6738].

– Also obtained by debenzylation of 4′-(benzyloxy)-2′-methoxypropiophenone (m.p. 59–60°) with concentrated hydrochloric acid in acetic acid at 100° for 30 min [7135].

b.p.$_{0.001}$ 140° [7135];
m.p. 117° [6738], 114–116° [7135];
^{1}H NMR (Sadtler: standard n° 28220M), IR (Sadtler: standard n° 55292).

1-(4-Hydroxy-3-methoxyphenyl)-1-propanone *(Propiovanillone) (Propioguaiacone) (Guaiacylpropanone) (Ethyl guaiacyl ketone)*

[1835-14-9] $C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

– Preparation by Fries rearrangement of 2-methoxyphenyl propionate [7158], (b.p.$_{20}$ 140–143°) [7159],

  - with aluminium chloride in nitrobenzene, first at 60–80° for 1 h, then at r.t. overnight [7160], at 60° for 1 h [7161], (65%) [7159], (50%) [6620] or at 50° for 5 h (44%) [7127];
  - with aluminium chloride (2 mol) in nitromethane at r.t. for 24 h (65%) [7154];
  - with aluminium chloride in carbon disulfide [6832];
  - with titanium tetrachloride (2 mol) in nitromethane at r.t. for 24 h (62%) [7154];
  - with stannic chloride (2 mol) in nitromethane at r.t. for 24 h (69%) [7154];
  - with antimony pentachloride (2 mol) in nitromethane at r.t. for 24 h (28%) with formation of important tars [7154].

– Also obtained by acylation of guaiacol with propionic acid,

  - in the presence of boron trifluoride in xylene at 60–70° (almost theoretical yield) [6820], boron trifluoride at 70° for 90 min (77%) [7162], (50%)

  [7163], boron trifluoride (60%) [7164] or boron trifluoride etherate for 170 h at r.t. in a sealed tube (30%) [7165];
  - by heating in the presence of polyphosphoric acid (70%) [7166], for 15 min in a boiling water bath (61%) [6738].

- Also obtained by acylation of veratrole with propionyl chloride in the presence of aluminium chloride in carbon disulfide at r.t. for 10 h (50–60%) [7167].
- Also obtained by oxidation of 1-(4-hydroxy-3-methoxyphenyl)propane with DDQ in wet dioxane/silica gel under sonication (53%) [7093].
- Also obtained by reaction of propionyl chloride (SM) with guaiacol in carbon disulfide in the presence of aluminium chloride for 1 h at 70° (70%) [6671]. SM was formed in situ by action of oxalyl chloride with sodium propionate (method B).
- Also obtained by oxidation of 1-(4-hydroxy-3-methoxyphenyl)-1-propanol (m.p. 69°) [7168],
  - with silver nitrate in aqueous sodium hydroxide, first at 60°, then at reflux for 2 h [6700], (90%) [7169] or at reflux for 5 h (51%) [7170];
  - with DDQ in dioxane for 18 h at r.t. [7171] or for 3 days [7172] or in benzene [7168].
- Also obtained by hydrogenation of 1-(4-hydroxy-3-methoxyphenyl)-2-propen-1-one in ethyl acetate over Pd-$BaSO_4$, followed by chromatography on silica gel [7173].
- Also obtained by reduction of β-hydroxypropioguaiacone (VI) in the presence of sulfide ions [7174].
- Also refer to: [6821,7175–7194].

Isolation from natural sources

- From Brazilian propolis, *Myrceugenia euosma* (O. Berg) Legrand (Myrtaceae) (compound **5**) [7195].
- From leaves and stems of *Rhinacanthus nasutus* (L.) Kurz (Acanthaceae) [7196].
- From bamboo stems, *Phyllostachys edulis* Makino (Gramineae) (compound **11**) [7197].
- From the aerial parts of sage (*Salvia lavandulifolia*) [7198].
- By enzymatic hydrolysis of bound aroma constituents from raspberry fruit pulp [7199].
- By enzymatic hydrolysis of bound aroma constituents from strawberry fruit (*Fragaria vesca f. semperflorens*) [7200].
- Obtained by oxidation of Pepper *dioxane lignin* [8068-03-9] with hydrogen peroxide in 1 N sodium hydroxide [7201].
- Of raw cane sugar flavour [7202].
- Also obtained by oxidation of lignin [9005-53-2] [7203].
- Identification in softwood lignin [7164].
- The sodium bisulfite digestion of sprucewood yields about 1.5% of propiovanillone. Similar yield of this compound was obtained from sodium borohydride-reduced sprucewood [7204].
- Determination, in wastewater from kraft pulping [7205].
- Formation during lignin degradation with “white liquor” [7174].

- Formation in spruce lignin degradation in wood digestion with sodium bisulfide [7206].
- Also obtained by alkaline cooking of lignin model compounds (veratrylglycerol β-guaiacyl ether, α-(2-methoxyphenoxy)-β-hydroxypropioveratrone and ω-(2-methoxyphenoxy)acetoveratrone) with a solution containing sodium hydroxide and sodium sulfide or sodium bisulfide [7207].
- Identification in lignin and sprucewood ethanolysis products [7208].
- Identification on the aromatic composition of *Vitis vinifera* L. var. *treixadura* Wines [7209].
- Identification in Spanish oak heartwood of *Quercus robur*, *Quercus petraea*, *Quercus pyrenaica* and *Quercus faginea* [7210].
- Identification in *Quercus rubra* L. oak heartwood (Fagaceae) [7211].

b.p.$_{0.15}$ 110–125° [7163], b.p.$_{11}$ 162–168° [6738], b.p.$_{5-7}$ 165–175° [7162],
b.p.$_{3}$ 165–180° [7165], b.p.$_{11}$ 168–172° [7167], b.p.$_{8}$ 175–180° [7166],
b.p.$_{15}$ 180–185° [6620], b.p.$_{12}$ 182° [6820], b.p.$_{16}$ 182–187° [6832];
m.p. 63° [7168], 62–63° [7159,7169,7173], 61–62° [6620,7093,7160,7194], 59–60° [7161,7170], 58–60° [7172], 58° [7164,7167], 54° [6820,7166], 48–50° [7162,7165];
^{1}H NMR (Sadtler: standard n° 28216M), [7093,7165,7168,7195],
^{13}C NMR [7093,7195],
IR (Sadtler: standard n° 55288) [6699,6700,6822,7161,7194],
photoelectron spectrum [7212], UV [7161,7213],
MS [7174,7195,7214];
HPLC [7200,7215–7217]; GC [7174,7200,7218];
GC-MS [7198,7200,7210,7219–7223];
paper chromatography [7224,7225]; TLC [7169,7174];
chromatography [6700,6841,6843,7226–7228];
column chromatography [7174]; gel chromatography [7229];
pK$_a$ [7203,7230]; potentiometric titration in DMSO [7231];
ionization potential [7232].

Notes: Thermal decomposition [7233]; basicity [7234]; ion (1$^-$) [116235-78-0] [7235]; of sunflower, honeybee olfaction in relation to, [7236].

BIOLOGICAL ACTIVITY: Choleretic [7237].

Ethyl ether [833-67-0] $C_{12}H_{16}O_3$ mol.wt. 208.26.

- Preparation by reaction of diethyl sulfate with 4-hydroxy-3-methoxypropiophenone in the presence of potassium hydroxide in dilute methanol at 50° for 10 min (91%) [6850].
- Also obtained by treatment of α-(4-ethoxy-3-methoxybenzoyl)propionic acid ethyl ester (b.p.$_{3}$ 202–205°) with sodium hydroxide in boiling dilute ethanol for 1.5 h (81–94%) [7238] or in aqueous methanol [7239].
- Also obtained by reaction of sodium methoxide with 1-ethoxy-4-(1, 2-dibromopropyl)-2-methoxybenzene in methanol [7100,7240].

- Also obtained by treatment of 1-ethoxy-4-(1-ethoxypropenyl)-2-methoxybenzene with diluted acid [7100,7240,7241].
- Preparation from 4-hydroxy-3-methoxypropiophenone [7242].
- Also refer to: [7239].

b.p.$_{13}$ 155° [7100];
m.p. 62° [7100,7238,7239], 60–61° [6850], 56–57° [7241].

Propyl ether $C_{13}H_{18}O_3$ mol.wt. 222.28.

- Obtained by treatment of propyl isoeugenol dibromide (m.p. 53–54°) with sodium hydroxide in refluxing methanol for 4 h (quantitative yield) [7243].

b.p. 284–287° [7243]; m.p. 63–64° [7243].

Benzyl ether $C_{17}H_{18}O_3$ mol.wt. 270.33 [7244,7245].

Sodium salt [7167].

β-D-Glucopyranoside (*Baihuaqianhuoside*)

[155969-61-2] $C_{16}H_{22}O_8$ mol.wt. 342.35 [6720,7246].

- Isolated from *Kokuto*, non-centrifuged Cane Sugar (*Saccharum officinarum*) [7247] and from *Glehnia littoralis* root and rhizoma (Umbelliferae) [7248].

Note: Effect on lignification in potato and spruce bark cultures [7249] and in tissue cultures in vitro [7250].

Tetra-O-acetyl-β-D-glucopyranoside

[432547-82-5] $C_{24}H_{30}O_{12}$ mol.wt.510.49 [6720].

Acetate $C_{12}H_{14}O_4$ mol.wt. 222.40 GC-MS [7251].

Propionate $C_{13}H_{16}O_4$ mol.wt. 236.27 (b.p.$_{26}$ 202°) [6351];

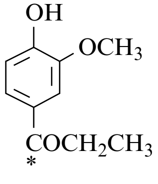
^{1}H NMR (Sadtler: standard n° 30294M), IR (Sadtler: standard n° 57339).

Benzoate $C_{17}H_{16}O_4$ mol.wt. 284.31 (m.p. 109°) [6351];

^{1}H NMR (Sadtler: standard n° 28222M), IR (Sadtler: standard n° 55294).

1-(4-Hydroxy-3-methoxyphenyl)-1-propanone-1-^{14}C

[15212-07-4] $C_{10}H_{12}O_3$ mol.wt. 182.20



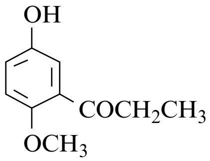
Syntheses

- Obtained by reaction of propionyl chloride-3-^{14}C (EtCOCl-1-^{14}C) (SM) with veratrole in the presence of aluminium chloride in refluxing carbon disulfide (40%) [7252], then at 70° for 1 h (60%) [7253]. SM was prepared by action of oxalyl chloride with sodium propionate-(carboxyl-^{14}C) [7253].

b.p.$_{0.001}$ 110–120°; dark red oil which slowly crystallized [7253].

1-(5-Hydroxy-2-methoxyphenyl)-1-propanone

[80427-31-2] $C_{10}H_{12}O_3$ mol.wt. 180.20



Synthesis

– Preparation by hydrolysis of the corresponding propionic ester (SM) with sodium hydroxide in refluxing methanol for 1 h (quantitative yield) [7143]. SM was obtained by acylation of 4-methoxyphenyl propionate with propionyl chloride in nitromethane in the presence of stannic chloride at 20° for 48 h (56%).

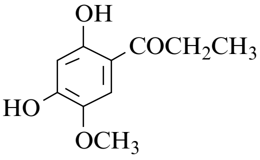
m.p. 68° [7143];
^{1}H NMR (Sadtler: standard n° 30292M), IR (Sadtler: standard n° 57337) [7143], UV [7143].

Propionate $C_{13}H_{16}O_4$ mol.wt. 236.27 [6351].

^{1}H NMR (Sadtler: standard n° 35282M), IR (Sadtler: standard n° 62650K).

1-(2,4-Dihydroxy-5-methoxyphenyl)-1-propanone

[79744-64-2] $C_{10}H_{12}O_4$ mol.wt. 196.20



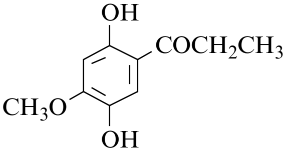
Syntheses

– Preparations using various methods [6870],
  - by Friedel–Crafts acylation of 4-methoxyresorcinol with propionyl chloride in the presence of aluminium chloride;
  - by Fries rearrangement of 4-methoxyresorcinol propionate with aluminium chloride;
  - by reaction of propionitrile with 4-methoxyresorcinol (Hoesch reaction).

^{13}C NMR [6870].

1-(2,5-Dihydroxy-4-methoxyphenyl)-1-propanone

[3839-58-5] $C_{10}H_{12}O_4$ mol.wt. 196.20



Synthesis

– Preparation by Fries rearrangement of 2-methoxyhydroquinone dipropionate (m.p. 59.5–60°) with aluminium chloride in nitrobenzene at r.t. for 64 h (82%) [7254].

m.p. 112–114° [7254].

Dibenzyl ether $C_{24}H_{24}O_4$ mol.wt. 376.45.

– Obtained by reaction of benzyl chloride with 2,5-dihydroxy-4-methoxypropiophenone in the presence of potash in ethanol at 20° for 4 h (85%) [7254].

m.p. 119–120° [7254].

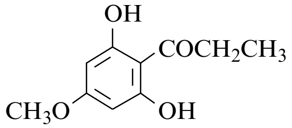
Diacetate $C_{14}H_{16}O_6$ mol.wt. 280.63.

- Obtained by reaction of acetic anhydride with 2,5-dihydroxy-4-methoxypropiophenone in the presence of pyridine at 20° for 48 h (90%) [7254].

m.p. 104° [7254].

1-(2,6-Dihydroxy-4-methoxyphenyl)-1-propanone *(Desaspidinol P)*

[69480-04-2] $C_{10}H_{12}O_4$ mol.wt. 196.20



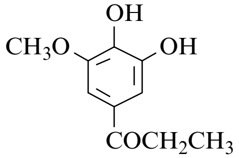
Syntheses

- Preparation according to: [7255,7256].
- Also refer to: [6895] (compound **15**) and [7257] (compound **17**).

m.p. 118–120° [7257];
MS [7257]; GLC [6895]; TLC [6895,7257]; paper chromatography [6895].

1-(3,4-Dihydroxy-5-methoxyphenyl)-1-propanone

[3934-94-9] $C_{10}H_{12}O_4$ mol.wt. 196.20



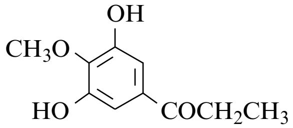
Synthesis

- Refer to: [7258].

Note: Redox potential [7258].

1-(3,5-Dihydroxy-4-methoxyphenyl)-1-propanone

[148204-59-5] $C_{10}H_{12}O_4$ mol.wt. 196.20



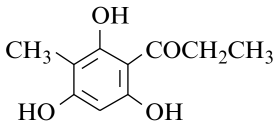
From natural sources

- In liquid wastes from eucalyptus wood and kraft lignin charring [7259].

GC [7259], GC-MS [7259].

1-(2,4,6-Trihydroxy-3-methylphenyl)-1-propanone

[57765-50-1] $C_{10}H_{12}O_4$ mol.wt. 196.20



Syntheses

- Obtained by Friedel–Crafts acylation of 2-methyl-phloroglucinol with propionyl chloride in the presence of aluminium chloride in nitrobenzene [7260].
- Also refer to: [6890] (compound **23**), [6895] (compound **8**) and [7261] (compound **5**).

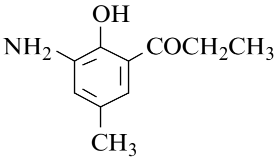
Isolation from natural sources

- From *Dryopteris caucasia* (A. Br.; Fraser-Jenkins, Corley) [7261].
- From *Dryopteris* of Japan (*Dryopteris bissetiana, Dryopteris lacera)* [7262].

m.p. 205° [6890,7260];
TLC [6890,6895,7262]; GLC [6895]; paper chromatography [6895,7262].

1-(3-Amino-2-hydroxy-5-methylphenyl)-1-propanone

[70978-23-3] $C_{10}H_{13}NO_2$ mol.wt. 179.22



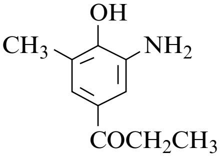
Syntheses

- Preparation by reduction of 2-hydroxy-5-methyl-3-nitropropiophenone in ethanol using 5% Pd/C as catalyst at atmospheric pressure and 25° [6926,6993].

m.p. 40° [6993].

1-(3-Amino-4-hydroxy-5-methylphenyl)-1-propanone

[141771-82-6] $C_{10}H_{13}NO_2$ mol.wt. 179.22

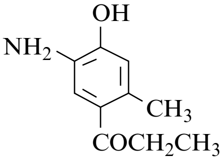


Synthesis

- Obtained by reduction of 4-hydroxy-3-methyl-5-nitropropiophenone with Raney nickel in ethanol (99%) [6995].

1-(5-Amino-4-hydroxy-2-methylphenyl)-1-propanone

$C_{10}H_{13}NO_2$ mol.wt. 179.22



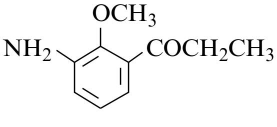
Synthesis

- Preparation by reduction of the nitro group of 4-hydroxy-2-methyl-5-nitropropiophenone with sodium hydrosulfite in boiling alkaline solution [6994].

m.p. 135° [6994].

1-(3-Amino-2-methoxyphenyl)-1-propanone

[103030-81-5] $C_{10}H_{13}NO_2$ mol.wt. 179.22



Synthesis

- Preparation from its hydrochloride [6533].

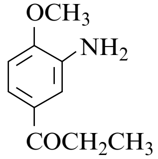
Hydrochloride $C_{10}H_{13}NO_2$, HCl mol.wt. 215.68.

- Preparation by reduction of 2-methoxy-3-nitropropiophenone with iron powder in the presence of concentrated hydrochloric acid in refluxing ethanol for 1 h (63%) [6533].

m.p. 154–155° (d) [6533]; UV [6533].

1-(3-Amino-4-methoxyphenyl)-1-propanone

[103028-92-8] $C_{10}H_{13}NO_2$ mol.wt. 179.22



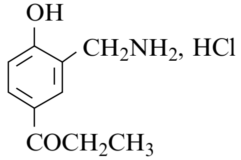
Syntheses

- Obtained from the 4-methoxy-3-nitropropiophenone by hydrogenation in the presence of Pd in benzene/ethyl acetate [7263] or by selective reduction with tin in aqueous hydrochloric acid [7002,7263].

m.p. 107–109° [7002,7263].

1-[3-(Aminomethyl)-4-hydroxyphenyl]-1-propanone *(Hydrochloride)*

[109314-49-0] $C_{10}H_{13}NO_2$, HCl mol.wt. 203.58

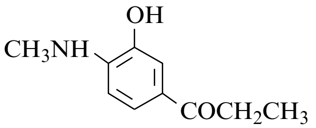


Synthesis

- Refer to: [6476].

1-[3-Hydroxy-4-(methylamino)phenyl]-1-propanone

[54903-58-1] $C_{10}H_{13}NO_2$ mol.wt. 179.22

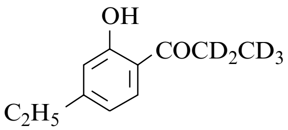


Syntheses

- Preparation from 3-methyl-6-propionylbenzoxazolinone by alkaline hydrolysis with boiling 10% aqueous sodium hydroxide solution for 4 h (90–100%) [6920,7264].

m.p. 149–150° [6920], 145–149° [7264].

1-(4-Ethyl-2-hydroxyphenyl)-1-propanone-*2,2,3,3,3-d*$_5$



$C_{11}H_9D_5O_2$ mol.wt. 183.26

Synthesis

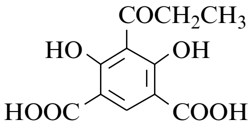
- Refer to: [6350].

ion (1^-), radical ion (1^-) [72051-81-1]; ESR spectrum [6350].

4,6-Dihydroxy-5-(1-oxopropyl)-1,3-benzenedicarboxylic acid

4,6-Dihydroxy-5-propionylisophthalic acid

$C_{11}H_{10}O_7$ mol.wt. 254.20

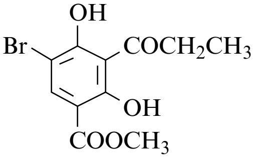


Synthesis

- Obtained by hydrolysis of its dimethyl ester with 4% potassium hydroxide in refluxing methanol [6809].

5-Bromo-2,4-dihydroxy-3-(1-oxopropyl)benzoic acid methyl ester

[99853-35-7] $C_{11}H_{11}BrO_5$ mol.wt. 303.11



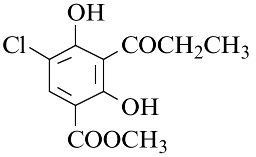
Syntheses

- Obtained by Fries rearrangement of methyl 5-bromo-2,4-di-propionoxybenzoate (m.p. 122°) with aluminium chloride (3.3 mol) at 125–130° for 1 h (32%) [6450]. The above reaction, when carried out using nitrobenzene at r.t. for 24 h or at 100° for 1 h gave the same keto ester.
- Also obtained by acylation of methyl 5-bromo-β-resorcylate with propionic anhydride under the conditions of the Friedel–Crafts reaction [6450].

m.p. 120° [6450].

5-Chloro-2,4-dihydroxy-3-(1-oxopropyl)benzoic acid methyl ester

[99854-30-5] $C_{11}H_{11}ClO_5$ mol.wt. 258.66



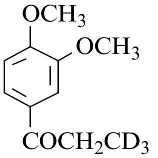
Syntheses

- Obtained by Fries rearrangement of methyl 5-chloro-2,4-dipropionoxybenzoate (m.p. 114°) with aluminium chloride at 125–130° for 1 h (37%) [6450].
- Also obtained by Friedel–Crafts acylation of methyl 5-chloro-2,4-dihydroxybenzoate with propionic anhydride in the presence of aluminium chloride [6450].
- Preparation by esterification of the corresponding keto acid [6450].

m.p. 107° [6450].

1-(3,4-Dimethoxyphenyl)-1-propanone-*3,3,3*-d_3

[63386-37-8] $C_{11}H_{11}D_3O_3$ mol.wt. 197.25



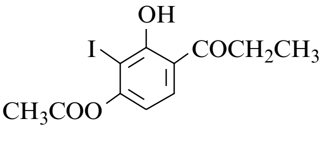
Synthesis

- Obtained by trideuteriomethylation of the lithio derivative of 3,4-dimethoxyacetophenone (41%) [7265].

m.p. 57–59° [7265]; ^{1}H NMR [7265].

1-[4-(Acetyloxy)-2-hydroxy-3-iodophenyl]-1-propanone

[130737-50-7] $C_{11}H_{11}IO_4$ mol.wt. 334.11

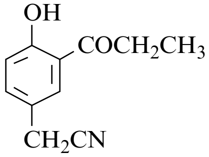


Syntheses

- Refer to: [7113,7266].

4-Hydroxy-3-(1-oxopropyl)phenylacetonitrile

[99842-69-0] $C_{11}H_{11}NO_2$ mol.wt. 189.21



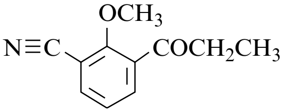
Synthesis

– Preparation by slowly adding a solution of 5-chloromethyl-2-hydroxypropiophenone in benzene to an aqueous solution of potassium cyanide at r.t. for 1 h, then keeping at 30–35° for 2–3 h (68%) [6942].

b.p.$_8$ 180–185° [6942]; m.p. 55–57° [6942].

2-Methoxy-3-(1-oxopropyl)benzonitrile

[99842-70-3] $C_{11}H_{11}NO_2$ mol.wt. 189.21



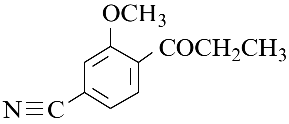
Synthesis

– Preparation by diazotization of 3-amino-2-methoxy-propiophenone hydrochloride, followed by treatment of the diazonium salt obtained with cuprous cyanide (74%) [6533].

m.p. 87–88° [6533]; UV [6533].

3-Methoxy-4-(1-oxopropyl)benzonitrile

[14004-66-1] $C_{11}H_{11}NO_2$ mol.wt. 189.21



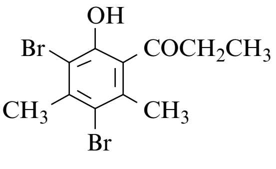
Synthesis

– Preparation by diazotization of 4-amino-2-methoxy-propiophenone hydrochloride (SM), followed by treatment of the diazonium salt obtained with cuprous cyanide (41%) [6927]. SM was prepared by hydrolysis of 4-acetamido-2-methoxypropiophenone with boiling 2% hydrochloric acid for 1 h.

m.p. 90–91° [6927].

1-(3,5-Dibromo-2-hydroxy-4,6-dimethylphenyl)-1-propanone

[5384-16-7] $C_{11}H_{12}Br_2O_2$ mol.wt. 336.02



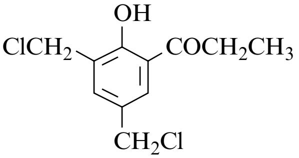
Syntheses

– Preparation by bromination of 2′-hydroxy-4′,6′-dimethyl-propiophenone, according to [6352], (good yield) [6356,6981].

m.p. 104° [6356,6981];
IR (Sadtler: standard n° 38088), UV [6356,6359]; TLC [6353,6358].

1-[3,5-Bis(chloromethyl)-2-hydroxyphenyl]-1-propanone

[99860-74-9] $C_{11}H_{12}Cl_2O_2$ mol.wt. 247.12



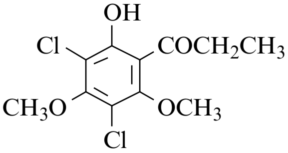
Synthesis

– Obtained by reaction of paraformaldehyde with o-hydroxy-propiophenone in the presence of hydrochloric acid at 60–70° for 7 h (15%) [6986].

b.p.$_2$ 161–164° [6986]; m.p. 73–74° [6986].

1-(3,5-Dichloro-2-hydroxy-4,6-dimethoxyphenyl)-1-propanone

[66021-78-1] $C_{11}H_{12}Cl_2O_4$ mol.wt. 279.12

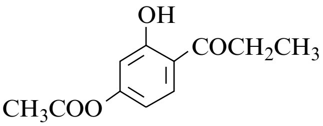


Synthesis

– Preparation by chlorination of 2-hydroxy-4,6-dimethoxy-propiophenone [6410].

1-[4-(Acetyloxy)-2-hydroxyphenyl]-1-propanone

[91143-73-6] $C_{11}H_{12}O_4$ mol.wt. 208.21



Syntheses

– Preparation by reaction of acetic anhydride with 2,4-di-hydroxypropiophenone (respropiophenone) in the presence of sodium acetate for 24 h at r.t. (80%) [6731].

– Also obtained by treatment of respropiophenone in a 9:1 (vol/vol) mixture of cyclohexane/tert-amyl alcohol with vinyl acetate (5 equiv) in the presence of *Pseudomonas cepacia* lipase adsorbed on Celite for 24 h at 40° (20%) [6761].

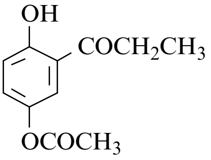
– Also refer to: [7267].

m.p. 80–81° [6731];

^{1}H NMR [6761], ^{13}C NMR [6761], IR [6761].

1-[5-(Acetyloxy)-2-hydroxyphenyl]-1-propanone

[148730-78-3] $C_{11}H_{12}O_4$ mol.wt. 208.21



Syntheses

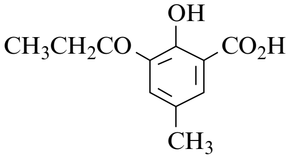
– Obtained by treatment of 2,5-dihydroxypropiophenone in a 9:1 (vol/vol) mixture of cyclohexane/tert-amyl alcohol with vinyl acetate (5 equiv) in the presence of *Pseudomonas cepacia* lipase adsorbed on Celite for 24 h at 40° (93%) [6761].

– Also obtained (by-product) by treatment of hydroquinone dipropionate with boron trifluoride complex (BF_3-OAc) at reflux for 1 h (9%) [6785].

^{1}H NMR [6761], ^{13}C NMR [6761], IR [6761].

2-Hydroxy-5-methyl-3-(1-oxopropyl)benzoic acid

[1760-86-7] $C_{11}H_{12}O_4$ mol.wt. 208.21



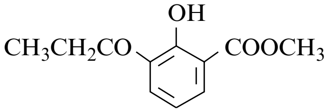
Syntheses

- Obtained by Fries rearrangement of 2-propionyloxy-5-methylbenzoic acid [6926], (m.p. 104°) with aluminium chloride (3.3 mol) at 170° for 3 h (30%) [7268].
- Also obtained by Friedel–Crafts acylation of 2-hydroxy-5-methylbenzoic acid with propionyl chloride in the presence of aluminium chloride (3 mol) in nitrobenzene, first at r.t. overnight, then heating on a water bath for 3 h (15%) [7268].

m.p. 110° [7268].

2-Hydroxy-3-(1-oxopropyl)benzoic acid methyl ester

[88466-30-2] $C_{11}H_{12}O_4$ mol.wt. 208.21

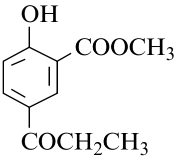


Syntheses

- Refer to: [6936,7269].

2-Hydroxy-5-(1-oxopropyl)benzoic acid methyl ester
Methyl 5-propionylsalicylate

[77526-99-9] $C_{11}H_{12}O_4$ mol.wt. 208.21



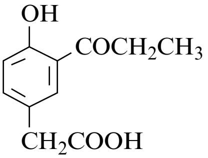
Syntheses

- Obtained by Fries rearrangement of methyl 2-(propionyloxy)benzoate (SM) with aluminium chloride in boiling carbon disulfide for 2 h [6938]. SM was prepared by treating the oil of *wintergreen* (methyl salicylate) with propionyl chloride at reflux for 2 h (90%) [6938].
- Preparation by Friedel–Crafts acylation of methyl salicylate with propionyl chloride using aluminium chloride as catalyst in carbon disulfide (96%) [6940].
- Also obtained by Fries rearrangement of methyl propionylsalicylate [7005].
- Also obtained by Fries rearrangement of 2-(propionyloxy)benzoic acid, according to [6961], followed by esterification (30%) [7270].
- Also refer to: [7271].

m.p. 64–65° [6938], 60–61° [6940], 59–60° [7270].

4-Hydroxy-3-(1-oxopropyl)phenylacetic acid

[115048-28-7] $C_{11}H_{12}O_4$ mol.wt. 208.21

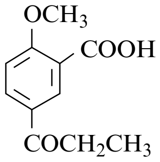


Synthesis

- Preparation by hydrolysis of 4-hydroxy-3-(1-oxopropyl) phenylacetonitrile with dilute sulfuric acid in boiling acetic acid for 2 h (82%) [6942].

m.p. 145–146° [6942].

2-Methoxy-5-(1-oxopropyl)benzoic acid



$C_{11}H_{12}O_4$ mol.wt. 208.21

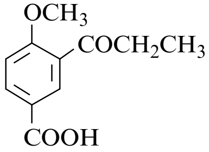
Syntheses

- Obtained by treatment of its methyl ester with sodium hydroxide [6937].
- Also refer to: [7272].

m.p. 127–128° [6937].

4-Methoxy-3-(1-oxopropyl)benzoic acid

[91143-39-4] $C_{11}H_{12}O_4$ mol.wt. 208.21



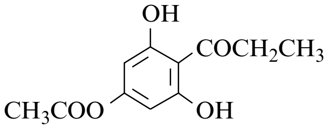
Syntheses

- Preparation by treatment of methyl 4-methoxy-3-propionyl-benzoate with potassium hydroxide in refluxing methanol for 30 min, then acidification with 6 N sulfuric acid (84%) [6943].
- Also refer to: [6941].

m.p. 209–211° [6943], 194° [6941]. One of the reported melting points is obviously wrong.

1-[4-(Acetyloxy)-2,6-dihydroxyphenyl]-1-propanone

[27178-00-3] $C_{11}H_{12}O_5$ mol.wt. 224.21



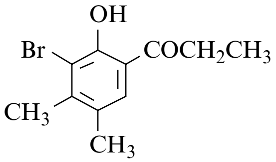
Synthesis

- Obtained by reaction of acetic anhydride with phloro-propiophenone in 2 N sodium hydroxide at 0° for 5 min [6629].

m.p. 130–131° [6629]; IR [6629]; TLC [6629].

1-(3-Bromo-2-hydroxy-4,5-dimethylphenyl)-1-propanone

[7376-15-0] $C_{11}H_{13}BrO_2$ mol.wt. 257.13



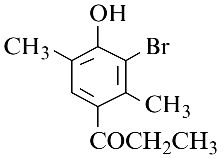
Syntheses

- Preparation by bromination of 2-hydroxy-4,5-dimethyl-propiophenone, according to the procedure [6354], (good yield) [6356,6981].

m.p. 124–125° [6356,6981];
IR (Sadtler: standard n° 38086) [6356,6981], UV [6356,6359];
TLC [6353,6358].

1-(3-Bromo-4-hydroxy-2,5-dimethylphenyl)-1-propanone

[5384-08-7] $C_{11}H_{13}BrO_2$ mol.wt. 257.13



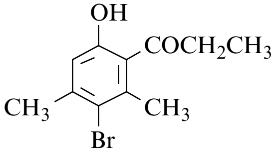
Syntheses

- Preparation by bromination of 4-hydroxy-2,5-dimethyl-propiophenone, according to the procedure [6354], (good yield) [6356,6981].

m.p. 77° [6356,6981];
^{1}H NMR (Sadtler: standard n° 8919M), IR (Sadtler: standard n° 38085) [6356, 6981],
UV [6356,6357]; TLC [6353,6358].

1-(3-Bromo-6-hydroxy-2,4-dimethylphenyl)-1-propanone

[5384-19-0] $C_{11}H_{13}BrO_2$ mol.wt. 257.13



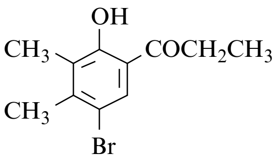
Syntheses

- Preparation by Fries rearrangement of 4-bromo-3,5-di-methylphenyl propionate (m.p. 30.5°) with aluminium chloride at 100° for 2 h (79%) [6356,6981].
- Also obtained by partial bromination of 2-hydroxy-4,6-di-methylpropiophenone, according to the procedure [6352], (51%) [6356,6981].
- Also obtained by partial debromination of 3′,5′-dibromo-2′-hydroxy-4′,6′-dimethylpropiophenone with copper powder in benzoic acid at 200° for 15 min (24%) [6356,6981].

m.p. 131° [6356,6981];
^{1}H NMR (Sadtler: standard n° 9091M), IR (Sadtler: standard n° 38087),
UV [6356,6359]; TLC [6353,6358].

1-(5-Bromo-2-hydroxy-3,4-dimethylphenyl)-1-propanone

[5384-03-2] $C_{11}H_{13}BrO_2$ mol.wt. 257.13



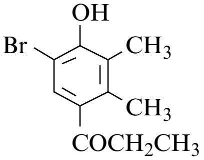
Syntheses

- Preparation by bromination of 2-hydroxy-3, 4-dimethyl-propiophenone, according to the procedure [6354], (good yield) [6356,6981].

m.p. 45° [6356,6981];
^{1}H NMR (Sadtler: standard n° 8921M), IR (Sadtler: standard n° 38084) [6356, 6981], UV [6356,6359]; TLC [6353,6358].

1-(5-Bromo-4-hydroxy-2,3-dimethylphenyl)-1-propanone

[5570-71-8] $C_{11}H_{13}BrO_2$ mol.wt. 257.13



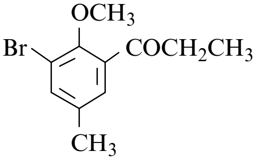
Syntheses

- Preparation by bromination of 4-hydroxy-2,3-dimethyl-propiophenone, according to the procedure [6354], (good yield) [6356,6981].

m.p. 69–70° [6356,6981];
^{1}H NMR (Sadtler: standard n° 9090M), IR (Sadtler: standard n° 38083) [6356, 6981], UV [6356,6357]; TLC [6353,6358].

1-(3-Bromo-2-methoxy-5-methylphenyl)-1-propanone

[106942-90-9] $C_{11}H_{13}BrO_2$ mol.wt. 257.13



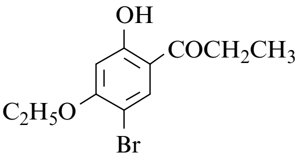
Synthesis

- Preparation by reaction of methyl iodide with 3-bromo-2-hydroxy-5-methylpropiophenone in the presence of potassium carbonate in acetone (93%) [6946].

b.p.$_{9.5}$ 95–100° [6945,6946];
^{1}H NMR [6945], ^{13}C NMR [6945], IR [6945].

1-(5-Bromo-4-ethoxy-2-hydroxyphenyl)-1-propanone

$C_{11}H_{13}BrO_3$ mol.wt. 273.13



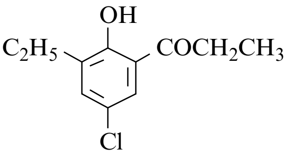
Synthesis

- Refer to: [7273].

Oxime [344367-93-7] $C_{11}H_{14}BrNO_3$ mol.wt. 288.14[7273].

1-(5-Chloro-3-ethyl-2-hydroxyphenyl)-1-propanone

[53347-07-2] $C_{11}H_{13}ClO_2$ mol.wt. 212.68



Synthesis

- Preparation by Fries rearrangement of 4-chloro-2-ethyl-phenyl propionate (b.p.$_{13}$ 137–140.5°) with aluminium chloride at 120° for 1 h (82%) [7274].

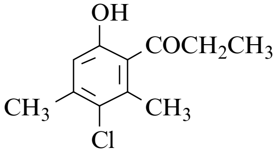
m.p. 44–45° [7274].

USE: Fungicide [7275].

BIOLOGICAL ACTIVITY: Bactericide [7274].

1-(3-Chloro-6-hydroxy-2,4-dimethylphenyl)-1-propanone

[105041-56-3] $C_{11}H_{13}ClO_2$ mol.wt. 212.68



Syntheses

- Preparation by Fries rearrangement of 4-chloro-3,5-di-methylphenyl propionate (b.p.$_3$ 123°) with aluminium chloride in carbon disulfide for 2 h at 80°, then at 110° for 2 h after solvent elimination [7276], (76%) [7277].
- Also obtained (by-product) by reaction of propionic anhydride with 4-chloro-3,5-dimethylanisole or 4-chloro-3,5-dimethylphenetole in the presence of aluminium chloride in refluxing carbon disulfide [7278].

white crystalline solid [7278], yellowish white [7277]; m.p. 106° [7277].

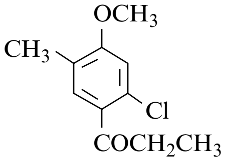
Ethyl ether $C_{13}H_{17}ClO_2$ mol.wt. 240.73.

- Obtained by reaction of propionic anhydride with 4-chloro-3,5-dimethylphenetole in the presence of aluminium chloride in refluxing carbon disulfide (62%) [7278].

b.p.$_7$ 155–156° [7278]; m.p. 53–54° [7278].

1-(2-Chloro-4-methoxy-5-methylphenyl)-1-propanone

[488106-61-2] $C_{11}H_{13}ClO_2$ mol.wt. 212.68

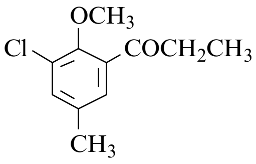


Synthesis

- Refer to: [7279].

1-(3-Chloro-2-methoxy-5-methylphenyl)-1-propanone

$C_{11}H_{13}ClO_2$ mol.wt. 212.68



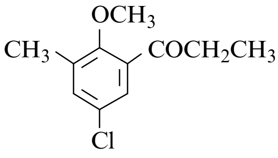
Synthesis

- Obtained by treatment of 3-chloro-2-hydroxy-5-methyl-propiophenone with methyl iodide in the presence of sodium ethoxide in refluxing ethanol for 3 h [6962].

oil [6962]; b.p.$_8$ 140° [6962].

1-(5-Chloro-2-methoxy-3-methylphenyl)-1-propanone

[107076-03-9] $C_{11}H_{13}ClO_2$ mol.wt. 212.68



Syntheses

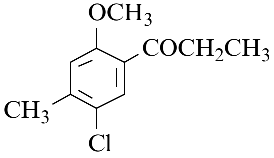
- Preparation from 5-chloro-2-hydroxy-3-methyl-propiophenone,

- by reaction with methyl iodide in the presence of sodium methoxide [6477] or ethoxide [6962];
- by reaction with dimethyl sulfate in the presence of sodium hydroxide in refluxing ethanol for 2 h (74%) [6966].

colourless oil [6966];
b.p.$_8$ 139° [6477,6962]; ^{1}H NMR [6966], IR [6966], MS [6966].

1-(5-Chloro-2-methoxy-4-methylphenyl)-1-propanone

[107076-02-8] $C_{11}H_{13}ClO_2$ mol.wt. 212.68



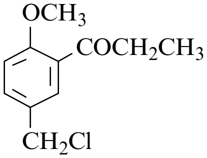
Synthesis

– Obtained by reaction of methyl iodide with 5-chloro-2-hydroxy-4-methylpropiophenone in the presence of sodium ethoxide in ethanol [6488].

m.p. 74° [6488].

1-[5-(Chloromethyl)-2-methoxyphenyl]-1-propanone

[100126-81-6] $C_{11}H_{13}ClO_2$ mol.wt. 212.68



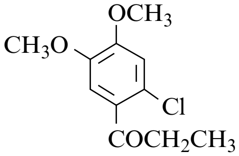
Synthesis

– Obtained by chloromethylation of 2-methoxypropiophenone (54%) [6986].

m.p. 107–108° [6986].

1-(2-Chloro-4,5-dimethoxyphenyl)-1-propanone

[149743-47-5] $C_{11}H_{13}ClO_3$ mol.wt. 228.68

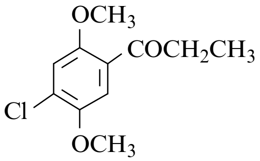


Syntheses

– Refer to: [6503,7280].

1-(4-Chloro-2,5-dimethoxyphenyl)-1-propanone

[13720-54-2] $C_{11}H_{13}ClO_3$ mol.wt. 228.68



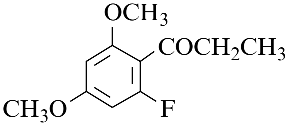
Synthesis

– Obtained by reaction of propionyl chloride with-2-chloro-1,4-dimethoxybenzene in the presence of aluminium chloride in carbon disulfide (50%) [7281].

m.p. 80–81° [7281].

1-(2-Fluoro-4,6-dimethoxyphenyl)-1-propanone

[864866-61-5] $C_{11}H_{13}FO_3$ mol.wt. 212.22

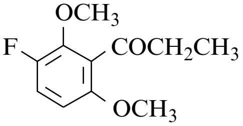


Synthesis

– Refer to: [6529].

1-(3-Fluoro-2,6-dimethoxyphenyl)-1-propanone

[119257-50-0] $C_{11}H_{13}FO_3$ mol.wt. 212.22

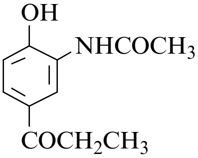


Synthesis

– Obtained by fluorination of 2,6-dimethoxypropiophenone [6817].

2-Hydroxy-5-(1-oxopropyl)acetanilide

[130521-17-4] $C_{11}H_{13}NO_3$ mol.wt. 207.23

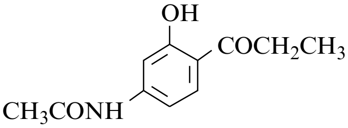


Synthesis

– Obtained by reaction of propionyl chloride with 2-acetyl-aminophenol in DMF in the presence of aluminium chloride at 85° for 5 h [6913].

3-Hydroxy-4-(1-oxopropyl)acetanilide

[66611-86-7] $C_{11}H_{13}NO_3$ mol.wt. 207.23



Syntheses

– Preparation by Fries rearrangement with aluminium chloride of 3-acetamidophenyl propionate [6351,7282], at 180° for 4 h (52%) [6918].

– Also obtained by Friedel–Crafts acylation of m-acetaminoanisole with propionyl chloride in the presence of aluminium chloride in refluxing ethylene dichloride for 2 h (40%) [6918].

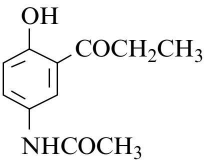
– Also refer to: [6429,6919,6927].

m.p. 161° [6351], 154° [6918];

^{1}H NMR (Sadtler: standard n° 28219M), IR (Sadtler: standard n° 55291).

4-Hydroxy-3-(1-oxopropyl)acetanilide

[99855-34-2] $C_{11}H_{13}NO_3$ mol.wt. 207.23



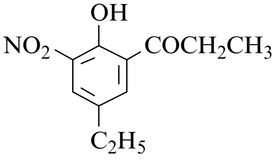
Syntheses

– Obtained by Fries rearrangement of 4-(propionyloxy)-acetanilide [7282], (m.p. 128°) with aluminium chloride, first at 110–120°, then at 160° for 3 h [6429].

– Also refer to: [7283,7284].

1-(5-Ethyl-2-hydroxy-3-nitrophenyl)-1-propanone

[70978-47-1] $C_{11}H_{13}NO_4$ mol.wt. 223.23



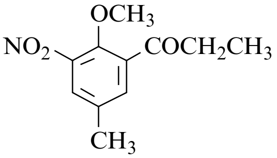
Syntheses

- Preparation by nitration of 5-ethyl-2-hydroxypropiophenone at −20° (25%) [6926], with concentrated nitric acid (d = 1.42) in concentrated sulfuric acid between −15° and 5° [6993].

m.p. 86.5–87.5° [6993], 86–87° [6926].

1-(2-Methoxy-5-methyl-3-nitrophenyl)-1-propanone

$C_{11}H_{13}NO_4$ mol.wt. 223.23



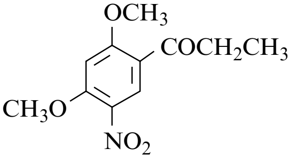
Synthesis

- Obtained by reaction of nitric acid with 2-methoxy-5-methylpropiophenone in acetic anhydride for 45 min (52%) [7285].

yellow oil [7285]; ^{1}H NMR [7285], MS [7285].

1-(2,4-Dimethoxy-5-nitrophenyl)-1-propanone

$C_{11}H_{13}NO_5$ mol.wt. 239.23



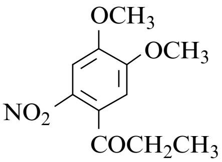
Synthesis

- Preparation by reaction of dimethyl sulfate with 2,4-di-hydroxy-5-nitropropiophenone in the presence of 20% sodium hydroxide (75%) [6555].

m.p. 155° [6555].

1-(4,5-Dimethoxy-2-nitrophenyl)-1-propanone

[91134-62-2] $C_{11}H_{13}NO_5$ mol.wt. 239.23



Syntheses

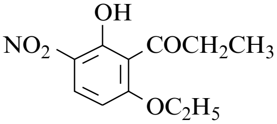
- Preparation by nitration of propioveratrone,
  - with fuming nitric acid in sulfuric acid (60%) [7001];
  - with nitric acid in acetic acid (65%) [7286].
- Also obtained by oxidation of 1-(4,5-dimethoxy-2-nitrophenyl)propyl nitrate (m.p. 99–100°) with potassium permanganate [7006].

- Also refer to: [7287,7288].

m.p. 132–133° [7001,7286], 130–131° [7006];
^{1}H NMR [7286], IR [7286].

USE: As intermediate for photoreactive protecting reagents [7288].
BIOLOGICAL ACTIVITY: Hypolipidemic [7287].

1-(6-Ethoxy-2-hydroxy-3-nitrophenyl)-1-propanone



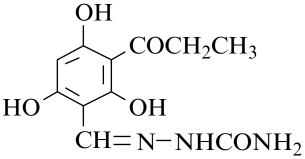
$C_{11}H_{13}NO_5$ mol.wt. 239.23

Syntheses

- Obtained by Friedel–Crafts reaction of propionic anhydride with 5-ethoxy-2-nitrophenol in the presence of aluminium chloride on heating in nitrobenzene [6407]. To see (*58C*) [6559].

2-[[2,4,6-Trihydroxy-3-(1-oxopropyl)phenyl]methylene]hydrazinecarboxamide

[96573-45-4] $C_{11}H_{13}N_3O_5$ mol.wt. 267.24



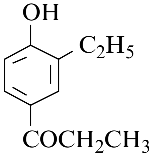
Synthesis

- Refer to: [7007] (compound **37**).

m.p. 193–195° (d) [7007];
^{1}H NMR [7007], IR [7007], MS [7007].

1-(3-Ethyl-4-hydroxyphenyl)-1-propanone

[540495-26-9] $C_{11}H_{14}O_2$ mol.wt. 178.23

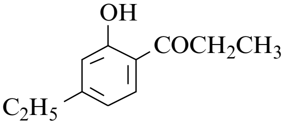


Synthesis

- Refer to: [7289].

1-(4-Ethyl-2-hydroxyphenyl)-1-propanone

$C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

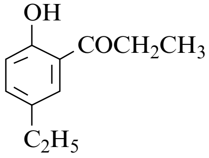
- Preparation by Fries rearrangement of 3-ethylphenyl propionate (b.p.$_{3.5}$ 97–98°) [7024] with aluminium chloride in nitrobenzene at 25° for 6 h (92%) or without solvent at 130° for 2 h (89%) [7290] or at 140–150° [7024].

Note: Ion (1^-), radical ion (1^-) [72051-80-0], ESR spectrum [6350].

b.p.$_4$ 116–120° [7024], b.p.$_5$ 146° [7290].

1-(5-Ethyl-2-hydroxyphenyl)-1-propanone

[63909-10-4] $C_{11}H_{14}O_2$ mol.wt. 178.23



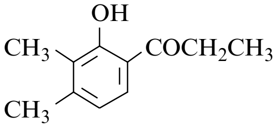
Syntheses

- Preparation by Fries rearrangement of 4-ethylphenyl propionate with aluminium chloride at 100° for 2 h (70%) [7291] or at 170° for 1–2 h (80–96%) [6492].
- Preparation by Friedel–Crafts acylation of 4-ethylphenol,
  - with propionic anhydride in the presence of magnesium perchlorate at 170° for 90 min (82%) [6491];
  - with propionyl chloride in the presence of aluminium chloride in refluxing methylene chloride (59%) [6993].
- Also refer to: [6926,7048].

Hydrazone [70136-40-2] $C_{11}H_{16}N_2O$ mol.wt. 192.26 [6646].

1-(2-Hydroxy-3,4-dimethylphenyl)-1-propanone

[5384-01-0] $C_{11}H_{14}O_2$ mol.wt. 178.23



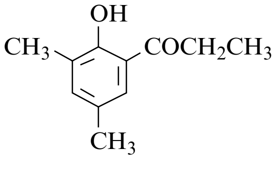
Syntheses

- Preparation by Fries rearrangement of 2,3-dimethylphenyl propionate,
  - with titanium tetrachloride at 50° for 3 h or 100° for 2 h (82–84%) [6356,6576,6981];
  - with aluminium chloride at 165° for 2 h (85%) [6356,6981].
- Also obtained by isomerization of 2′,3′-dimethyl-4′-hydroxypropiophenone by treatment with aluminium chloride [6482], for 1 h at 180–200° (59–71%) [6356,6468].
- Also refer to: [7292].

m.p. 44–45° [6356,6981];
^{1}H NMR (Sadtler: standard n° 8381M), IR (Sadtler: standard n° 37037) [6356,6981],
UV [6356,6359,6457]; TLC [6353,6356,6358].

1-(2-Hydroxy-3,5-dimethylphenyl)-1-propanone

[5570-72-9] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

- Preparation by Fries rearrangement of 2,4-dimethylphenyl propionate,

- with aluminium chloride, without solvent, at 100° for 7 h (63%) [6356,6576,6981], 130–140° for 5 h (90%) [7293], 165° for 1 h (74%) [6356,6981] or in nitromethane at 20° for 7 h (72%) [6569];
- with titanium tetrachloride at 100° for 7 h (83–86%) [6356,6576,6981].

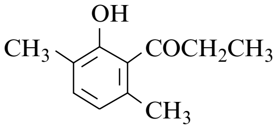
– Also obtained by Fries rearrangement of 2,5-dimethylphenyl propionate with aluminium chloride at 100° for 7 h (60%) [6356,6981] or 165° for 1 h (92%) [6981].
– Also obtained by isomerization of 2,5-dimethyl-4-hydroxypropiophenone by heating with aluminium chloride [6482], at 180–200° for 1 h (57%) [6356,6468].
– Also obtained by isomerization of 4-hydroxy-2,6-dimethylpropiophenone with aluminium chloride (3 mol) for 1 h at 180–200° (10%) [6482].
– Also obtained by reaction of sec-butyllithium (1.1 equiv) with 2-bromo-4,6-dimethylphenyl propionate in tetrahydrofuran/ethyl ether/hexane at −95° for 30 min and −78° for 30 min, then hydrolysis with saturated ammonium chloride (31%) (Metal-promoted Fries rearrangement) [6590].
– Also refer to: [7294].

m.p. 54° [6356,6981], 52–53° [7293];
^{1}H NMR (Sadtler: standard n° 8382M) [6590], ^{13}C NMR [6590],
IR (Sadtler: standard n° 37038) [6356,6590,6981], UV [6356,6359,6457]; TLC [6353,6358].

Hydrazone [70136-44-6] $C_{11}H_{16}N_2O$ mol.wt. 192.26 [6646].

1-(2-Hydroxy-3,6-dimethylphenyl)-1-propanone

[51233-75-1] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

– Preparation by Fries rearrangement of 2,5-dimethylphenyl propionate,

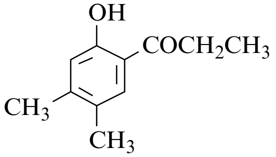
- with titanium tetrachloride at 120° for 1 h (76–79%) [7031];
- with aluminium chloride in nitromethane at r.t. for 21 days (11%) [6569].

– Also refer to: [7292].

m.p. 43° [6569,7031];
^{1}H NMR (Sadtler: standard n° 21708M) [6569,7031], IR [6569,7031],
UV [6569,7031], MS [7031].

1-(2-Hydroxy-4,5-dimethylphenyl)-1-propanone

[5384-13-4] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

– Preparation by Fries rearrangement of 3,4-dimethylphenyl propionate with aluminium chloride,

  - without solvent between 120° and 150° (quantitative yield) [7295], at 165° for 1 h (93%) [6356,6981], at 100° for 2 h (90%) [6356], at 110° (85%) [7296] and in the presence of sodium chloride [7297];
  - in nitromethane at 20° for 7 days (89%) [6569].

– Preparation by Fries rearrangement of 3,4-dimethylphenyl propionate with titanium tetrachloride,

  - without solvent at 100° for 1–2 h (91%) [6356,6981];
  - in nitromethane at 20° (63%) [6569] for 100 h (99%) [7031].

– Preparation by Friedel–Crafts acylation of 3,4-xylenol with propionic acid in the presence of boron trifluoride etherate (quantitative yield) [7298].

– Also obtained by isomerization,

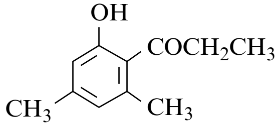
  - of 2-hydroxy-4,6-dimethylpropiophenone (1 mol) with aluminium chloride (3 mol) [6482] at 140–180° some hours (quantitative yield) [6468,7295] and in the presence of sodium chloride [7297];
  - of 2-hydroxy-3,5-dimethylpropiophenone (1 mol) with aluminium chloride (1.5 mol) at 180–200° for 1 h (20%) [6468].

– Also refer to: [7292,7294].

b.p.$_7$ 101° [7296], b.p.$_{10}$ 140° [6569,7031]. One of the reported boiling points is obviously wrong.
m.p. 61° [6569,7031], 60–61° [6356,6981], 60° [7295,7297];
^{1}H NMR (Sadtler: standard n° 8384M), IR (Sadtler: standard n° 37041) [6356,6981,7031]; UV [6356,6359,6399,6457,7031], MS [7031];
TLC [6353,6358].

1-(2-Hydroxy-4,6-dimethylphenyl)-1-propanone

[5384-54-3] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

– Preparation by Fries rearrangement of 3,5-dimethylphenyl propionate (m-5-xylyl propionate, b.p.$_{56}$ 167°) [7299],

  - in the presence of aluminium chloride at 120–150° (quantitative yield) [7295], at 130° for 2 h (95%) [7290], at 120° for 1 h (98%) [7031], at 100° for 5 h (83%) [7299] or at 50° for 3 h (61%) [6356,6576];
  - in the presence of aluminium chloride in nitrobenzene at 25° for 6 h (96%) [7290];
  - in the presence of an aluminium chloride and sodium chloride mixture at 85° for 5 h [7297];
  - in the presence of titanium tetrachloride at 120° for 1 h (98%) [7031] or at 50° for 3 h (79%) [6356,6576,6981].

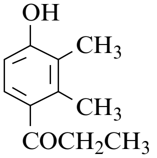
– Also obtained by reaction of propionic acid with 3,5-xylenol in the presence of boron trifluoride at 70° for 2 h (90%) [6583] or boron trifluoride etherate at 97–100° for 30 min (15%) [7027].

- Also obtained by reaction of propionic anhydride,
  - with 3,5-dimethylphenol in the presence of aluminium chloride in refluxing ethylene dichloride (79%) [7300];
  - with 3,5-dimethylanisole in the presence of aluminium chloride in refluxing carbon disulfide (small amount) [7278].
- Also obtained by alkaline degradation of 2,3,5,7-tetramethylchromone [6427].
- Also obtained by reaction of 2-bromo-4,6-dimethylphenyl propionate at low temperature (−78 to −95°) with sec-butyllithium to give, after hydrolysis, the titled ketone (metal-promoted Fries rearrangement) (yields: 31% by GC; 17% isolated) [6590].
- Also refer to: [7294,7301].

b.p.$_{2}$ 150° [7290], b.p.$_{15}$ 151_151.5° [6583].
m.p. 78° [6583,7278,7295], [6356,6427,6981,7031], 76° [7027,7299], 75° [7297].
^{1}H NMR (Sadtler: standard n° 8385M) [6590,7031], ^{13}C NMR [6590],
IR (Sadtler: standard n° 37042) [6356,6590,6981,7027,7031],
UV [6356,6359,6457,7027,7031], MS [7031];
TLC [6353,6356,6358]; GC [6590].

1-(4-Hydroxy-2,3-dimethylphenyl)-1-propanone

[5355-81-7] $C_{11}H_{14}O_2$ mol.wt. 178.23



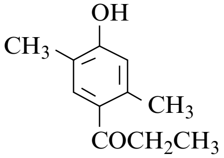
Syntheses

- Obtained by Fries rearrangement of 2,3-dimethylphenyl propionate with aluminium chloride in nitrobenzene at 50° for 3 h (10%) [6356,6576,6981].

m.p. 140° [6356,6981];
^{1}H NMR (Sadtler: standard n° 9088M), IR (Sadtler: standard n°38071) [6356,6981],
UV [6356,6357,6457]; TLC [6353,6358].

1-(4-Hydroxy-2,5-dimethylphenyl)-1-propanone

[5384-06-5] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

- Preparation by Fries rearrangement of 2,5-dimethylphenyl propionate with aluminium chloride,
  - in nitrobenzene at r.t. for 24 h (70%) [7302] or 44 h (41%) [6981];
  - in nitromethane at r.t. for 21 days (61%) [6569]. The same result (61%) was obtained by using titanium tetrachloride as catalyst at r.t. for 7 days;
  - in nitromethane at 20° for 24 h, but using different quantities of aluminium chloride by mole of ester:

$AlCl_3$ (mol)	Yield (%)
1	9
2	74
3	82

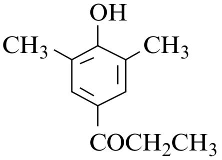
- without solvent at 100° for 7 h (36%) [6356,6981].

– Also refer to: [6468,6482,7294].

m.p. 128° [7302], 127° [6356,6569,6981];
^{1}H NMR (Sadtler: standard n° 8379M), IR (Sadtler: standard n°37039) [6356,6981],
UV [6356,6357,6457]; TLC [6353,6358].

1-(4-Hydroxy-3,5-dimethylphenyl)-1-propanone

[5384-09-8] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

– Preparation by Fries rearrangement of 2,6-dimethylphenyl propionate with aluminium chloride,

- without solvent at 50° for 10 h (56%) [6356,6981], at 130–140° [7303], (59%) [7304] or at 165° for 1 h [7305], (59%) [6981];
- in nitrobenzene at r.t. for 24 h (70%) [7294] or in nitromethane at 20° for 7 days (69%) [6569].

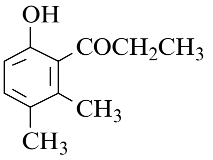
– Preparation by Fries rearrangement of 2,6-dimethylphenyl propionate with titanium tetrachloride at 100° for 2 h (20%) [6981].
– Preparation by oxidation of (3,5-dimethyl-4-hydroxyphenyl)ethylcarbinol with DDQ in dioxane at r.t. for 16 h (80%) [7306].
– Also refer to: [6405,6406,6519,7307,7308].

m.p. 108° [6356,6981], 106–108° [7303], 106–107° [7294], 106–106.5° [7304];
^{1}H NMR (Sadtler: standard n° 8383M), IR (Sadtler: standard n° 37040) [6356,6981],
UV [6356,6357,6457]; TLC [6353,6358].

Oxime [75408-97-8] $C_{11}H_{15}NO_2$ mol.wt. 193.26 [6384].

1-(6-Hydroxy-2,3-dimethylphenyl)-1-propanone

[121194-66-9] $C_{11}H_{14}O_2$ mol.wt. 178.23



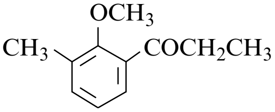
Synthesis

– Preparation by treatment of 3-tert-butyl-2-hydroxy-5,6-dimethylpropiophenone with aluminium chloride in nitromethane at 20° for 24 h (quantitative yield) [7031].

m.p. 70° [7031];
^{1}H NMR (Sadtler: standard n° 52710M) [7031],
IR (Sadtler: standard n° 79767K) [7031], UV [7031], MS [7031].

1-(2-Methoxy-3-methylphenyl)-1-propanone

[68597-45-5] $C_{11}H_{14}O_2$ mol.wt. 178.23



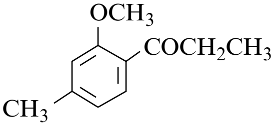
Syntheses

- Preparation by adding a solution of 5-chloro-2-methoxy-3-methylpropiophenone and ammonium formate in ethanol to a suspension of 10% Pd/C in aqueous 2.5 N potassium hydroxide and heating at 60° for 20 min (82%) [6966].
- Also obtained by reaction of methyl iodide with 2-methoxy-3-methylbenzoyl chloride in the presence of zinc in an ethyl acetate/toluene mixture (70%) [7309].
- Also obtained by reaction of ethylmagnesium bromide with 2-methoxy-3-methylbenzonitrile (69%) [6973].
- Also refer to: [7292].

colourless oil [6966]; b.p.$_{0.3}$ 86° [6973], b.p.$_{12}$ 122° [7309];
^{1}H NMR [6966], IR [6966], MS [6966].

1-(2-Methoxy-4-methylphenyl)-1-propanone

[36871-54-2] $C_{11}H_{14}O_2$ mol.wt. 178.23



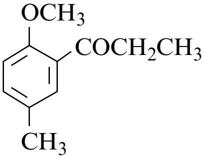
Syntheses

- Obtained by reaction of methyl iodide with 2-methoxy-4-methylbenzoyl chloride in the presence of zinc in an ethyl acetate/toluene mixture (68%) [7309].
- Obtained by reaction of dimethyl sulfate with 2-hydroxy-4-methylpropiophenone in the presence of sodium hydroxide in methanol [6980,6981].
- Also obtained by reaction of propionyl chloride with 3-methylanisole in the presence of aluminium chloride in carbon disulfide at r.t. for 24 h (27%) [7310].

b.p.$_{15}$ 142–144° [7310], b.p.$_{14}$ 147° [7309]; IR [6980].

1-(2-Methoxy-5-methylphenyl)-1-propanone

[82620-73-3] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

- Preparation by reaction of propionyl chloride with 4-methylanisole in the presence of aluminium chloride in carbon disulfide (97%) [7311].
- Also obtained by reaction of methyl iodide with 2-methoxy-5-methylbenzoyl chloride in the presence of zinc in an ethyl acetate/toluene mixture (60%) [7309].
- Also refer to: [7033,7312,7313].

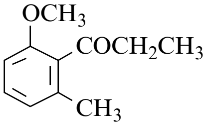
b.p.$_{10}$ 128–130° [7311], b.p.$_{13}$ 142–143° [7309], b.p.$_{16–17}$ 144.8–146.4° [7033];
^{1}H NMR [7311], IR [7311], UV [7033].

2,4-Dinitrophenylhydrazone [82623-49-2] $C_{17}H_{18}N_4O_5$ mol.wt. 358.39

(m.p. 112–112.5°) [7311].

1-(2-Methoxy-6-methylphenyl)-1-propanone

[137937-37-2] $C_{11}H_{14}O_2$ mol.wt. 178.23



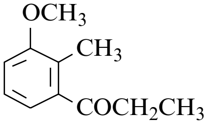
Syntheses

- Preparation by reaction of dimethyl sulfate with 2-hydroxy-6-methylpropiophenone (92%) [7011].
- Preparation by reaction of 3-methoxy-o-tolunitrile (m.p. 64°) [7314] with ethylmagnesium iodide in ethyl ether at reflux for 6 h (68%) [7058].

b.p.$_{11}$ 124–125° [7011], b.p.$_6$ 137° [7058];
m.p. 8° [7058];
^{1}H NMR (Sadtler: standard n° 57892M) [7011],
IR (Sadtler: standard n° 84940K) [7011], UV [7011], MS [7011].

1-(3-Methoxy-2-methylphenyl)-1-propanone

[104216-40-2] $C_{11}H_{14}O_2$ mol.wt. 178.23



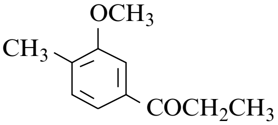
Syntheses

- Preparation by reaction of 3-methoxy-2-methylbenzoyl chloride with diethylcadmium according to the procedure [7315] for 3 h at 40–50° (50%) [7316].

oil [7316]; b.p.$_{0.1}$ 95–105° [7316].

1-(3-Methoxy-4-methylphenyl)-1-propanone

[18158-58-2] $C_{11}H_{14}O_2$ mol.wt. 178.23



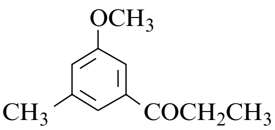
Syntheses

- Obtained by reaction of dimethyl sulfate with 3-hydroxy-4-methylpropiophenone (70%) [7064] in the presence of aqueous 2 N sodium hydroxide at 40–45° (60%) [7061].

yellow oil [7064]; b.p.$_5$ 134–136° [7061], b.p.$_{12}$ 147° [7064];
^{1}H NMR [7061].

1-(3-Methoxy-5-methylphenyl)-1-propanone

[29578-84-5] $C_{11}H_{14}O_2$ mol.wt. 178.23

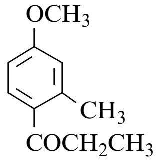


Synthesis

- Refer to: [7317].

1-(4-Methoxy-2-methylphenyl)-1-propanone

[53773-76-5] $C_{11}H_{14}O_2$ mol.wt. 178.23



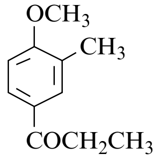
Syntheses

- Preparation by reaction of propionyl chloride with 3-methylanisole in the presence of aluminium chloride in methylene chloride for 3 h at 0° [6976], in carbon disulfide at r.t. for 24 h (52%) [7310] or by Friedel–Crafts acylation using samarium triiodide [7318].
- Also refer to: [6672,7292,7312].

m.p. 43° [7310].

1-(4-Methoxy-3-methylphenyl)-1-propanone

[76805-57-7] $C_{11}H_{14}O_2$ mol.wt. 178.23

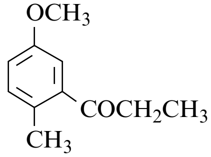


Syntheses

- Refer to: [6519,7312,7319,7320].

1-(5-Methoxy-2-methylphenyl)-1-propanone

[29578-81-2] $C_{11}H_{14}O_2$ mol.wt. 178.23



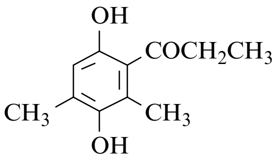
Syntheses

- Obtained (method P) by reaction of ethylmagnesium bromide with 5-methoxy-2-methylbenzoyl chloride in THF at −78° (73%) [6979].
- Also refer to: [7321].

oil [6979]; b.p.$_{18}$ 146–147° [7321].

1-(3,6-Dihydroxy-2,4-dimethylphenyl)-1-propanone

[210104-11-3] $C_{11}H_{14}O_3$ mol.wt. 194.23

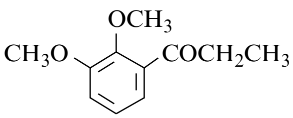


Synthesis

- Preparation by reaction of 2,6-dimethylbenzoquinone (2.5 equiv) with 2-oxobutanoic acid in aqueous acetonitrile or an acetonitrile/methylene chloride mixture (38%) [6788].

1-(2,3-Dimethoxyphenyl)-1-propanone

[76049-04-2] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

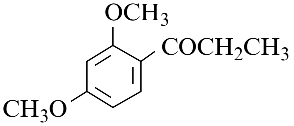
- Obtained by reaction of ethylmagnesium bromide with 2,3-dimethoxybenzonitrile [6724, 7130,7322].

- Preparation by reaction of dimethyl sulfate with 2-hydroxy-3-methoxypropiophenone in aqueous alkaline solution [7130].
- Also obtained by a Jones oxidation of 1-(2,3-dimethoxyphenyl)-1-propanol in acetone with sodium dichromate in dilute sulfuric acid for 3 h at 20° [6726].
- Also obtained by reaction of 2,3-dimethoxybenzoyl chloride with diethylcadmium in ethyl ether [7322].
- Also refer to: [6725,7129,7131,7323].

colourless oil [6724];
b.p.$_{0.2}$ 97–98° [7130], b.p.$_{0.7}$ 99–101° [7322], b.p.$_{0.2-0.3}$ 100–102° [6725],
b.p.$_{2}$ 114° [6724];
^{1}H NMR [6726], ^{13}C NMR [6726], IR [6726].

1-(2,4-Dimethoxyphenyl)-1-propanone

[831-00-5] $C_{11}H_{14}O_3$ mol.wt. 194.23



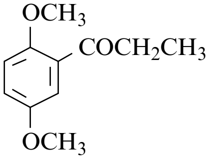
Syntheses

- Preparation by reaction of dimethyl sulfate with respropiophenone in methanol,
  - in the presence of aqueous sodium hydroxide solution (89%) [6389];
  - in the presence of potassium hydroxide, according to the procedure [7324], (68%) [6555].
- Also obtained by reaction of methyl iodide with respropiophenone in butanone in the presence of potassium carbonate [7325].
- Also obtained by Friedel–Crafts acylation of 1,3-dimethoxybenzene with propionyl chloride [6796] using stannic chloride in carbon disulfide (80%) [7111] or 10% mol. samarium triiodide in acetonitrile [7318].
- Also obtained by reaction of propionic acid with 1,3-dimethoxybenzene in the presence of PPA [6738], (quantitative yield) [7326], (74%) [7327].
- Also obtained by decarboxylation of 2,4-dimethoxy-6-carboxypropiophenone with Cu dust [7325].
- Also refer to: [6530,6796,7097,7328–7333].

b.p.$_{2}$ 143.5° [7327], b.p.$_{18}$ 174–176° [6796], b.p.$_{20}$ 180° [6389];
m.p. 83° [6389], 78° [6738], 75° [7325], 74–75° [7326], 72° [6796], 67° [6555], 66–67° [7111]. Some melting points are obviously wrong.
^{1}H NMR [7111,7318], IR [7111,7318,7326],
UV [7111,7326,7333], MS [7111]; Luminescence spectroscopy [7333].

1-(2,5-Dimethoxyphenyl)-1-propanone

[5803-30-5] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

- Obtained by acylation of hydroquinone dimethyl ether,

- with propionyl chloride [6796,7147,7149,7334] in the presence of aluminium chloride in petroleum ether (34%) [7335], in carbon disulfide [7336], (64%) [7281] or using 10% mol. samarium triiodide in acetonitrile (60%) [7318];
- with propionic anhydride in the presence of aluminium chloride in nitromethane [7337];
- with propionic acid in the presence of PPA at 70° for 2 h (95%) [7338];
- in the presence of zeolite catalyst [7339].

– Also obtained by oxidation of 1-(2,5-dimethoxyphenyl)-1-propanol with PDC in methylene chloride for 2 days at r.t. [7340].

– Also refer to: [6407,6713,7014,7341–7345].

Isolation from natural sources

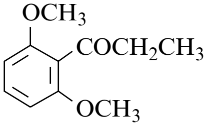
– From leaf and root of *Asarum forbesii* Maxim (Aristolochiaceae) [7346].

b.p.$_{0.3}$ 102–103° [7342], b.p.$_{4}$ 130–137° [7147], b.p.$_{13}$ 160° [6796],
b.p.$_{12}$ 160–162° [7341], b.p.$_{19}$ 165–172° [7337], b.p.$_{13}$ 167–169° [7335];
m.p. 8° [7338];
^{1}H NMR (Sadtler: standard n° 24405M) [7318,7336],
IR (Sadtler: standard n° 52517) [7318,7336], UV [7336],
MS [7336,7347]; GC-MS [7346].

BIOLOGICAL ACTIVITY: Antiinflammatory [6713].

1-(2,6-Dimethoxyphenyl)-1-propanone

[3840-02-6] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

– Preparation by reaction of dimethyl sulfate,

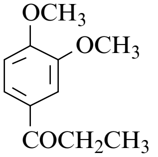
- with 2-hydroxy-6-methoxypropiophenone in the presence of aqueous sodium hydroxide (quantitative yield) [6801];
- with 2,6-dihydroxypropiophenone in the presence of alkali (70%) [6801] or 2 N sodium hydroxide at r.t. for 2 h (38%) [6800].

– Also refer to: [7348].

b.p.$_{9}$ 148–150° [6801];
m.p. 45° [6800], 44–45° [6801], 42–43° [7348].

1-(3,4-Dimethoxyphenyl)-1-propanone *(Propioveratrone)*

[1835-04-7] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

– Preparation by reaction of dimethyl sulfate with propionylcatechol in alkaline solution (43%) [7349].

– Preparation by reaction of propionyl chloride with veratrole [6796,7350],

  - in the presence of aluminium chloride in nitrobenzene for 24 h at r.t. (85%) [7351] or 1 h at −5° (36%) [6854], in refluxing benzene [7352], (84%) [7353], (65%) [7354], in chloroform for 1 h at 0–5° under nitrogen (79%) [7355], in carbon disulfide (61%) [7354], in ethylene dichloride for 1 h at 35° (54%) [7356] or in boiling petroleum ether for 30 h (28%) [7335];
  - in the presence of zinc chloride in boiling carbon disulfide (36%) [7357];
  - over cation-exchanged clays [7358] or using zeolite-H-beta catalysts [7359].

– Preparation by reaction of propionic acid with veratrole,

  - in the presence of PPA at 60° for 2.5 h (98%) [7360], at 100° for 90 min (75%) [7361] or at 70–80° for 4 h (55%) [7362];
  - in the presence of zinc chloride (12–15%) [7363].

– Preparation by reaction of propionic anhydride with veratrolein the presence of iodine for 3 h at reflux (46%) [6721].
– Also obtained by oxidation of 1-(3,4-dimethoxyphenyl)propane with DDQ in wet dioxane/silica gel under sonication (78%) [7093].
– Also obtained from (3,4-dimethoxyphenyl) (dimethylaminoethyl) ketone by hydrogenation in the presence of Pd-$BaSO_4$ catalyst in tetralin at 130° (65%) [7364].
– Also obtained by reaction of sodium methoxide with 1,2-dibromo-1-(3,4-dimethoxyphenyl)-propane [7100,7241,7335].
– Also refer to: [6591,6828,7167,7365,7366].

Isolation from natural sources

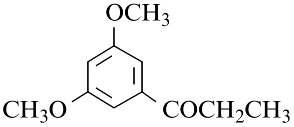
– From the leaves of *Hedyosmum* spp. from Bolivia (Chlorantaceae) [7367].
– From the aerial parts of *Pteronia camphorata* (Compositae) [7368].
– From *Asarum max*. [7369].

b.p.$_{0.2}$ 137° [7351], b.p.$_{0.8}$ 140° [7354], b.p.$_{2}$ 153–156° [6854],
b.p.$_{5}$ 157–160° [7361], b.p.$_{3}$ 158–160° [7353], b.p.$_{20}$ 188° [7354];
m.p. 62–63° [7349,7364], 62° [7100], 61° [7356], 60° [7335],
59–60° [7093,7351,7353], 58.5–59.5° [7355],
58–59° [6796,7241,7351,7362,7370,7371],
58° [7357], 57.5° [7354,7361], 56–58° [6721], 55–56° [6854];
^{1}H NMR (Sadtler: standard n° 24409M) [7093,7368], ^{13}C NMR [7093],
IR (Sadtler: standard n° 52522) [7368], UV [7213], MS [7368];
CG-MS [7372]; GC [7368]; TLC [7368].

BIOLOGICAL ACTIVITY: Antiinflammatory [7350].

1-(3,5-Dimethoxyphenyl)-1-propanone

[41497-31-8] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

– Preparation by reaction of ethylmagnesium bromide,

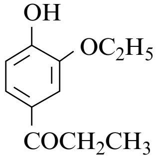
- with 3,5-dimethoxybenzamide (m.p. 145–146°) (80–85%) [6856,7373];
- with 3,5-dimethoxybenzonitrile (m.p. 86–87°) [7373].

- Also obtained by decarboxylation of 3,5-dimethoxy-2-carboxypropiophenone with Cu dust [7325] or by heating its ethyl ether with 25% sulfuric acid for 13 h [7374].
- Preparation by following the method [6856] starting from benzoic acid (good yield) [7349].
- Preparation (method O) by reaction of diethylcadmium with 3,5-dimethoxybenzoyl chloride (71%) [6979].
- Obtained by reaction of methyl iodide with 3,5-dimethoxybenzoyl chloride in the presence of zinc in an ethyl acetate/toluene mixture (19%) [7309].
- Also refer to: [7375].

b.p.$_{0.08}$ 98–100° [7373], b.p.$_{11}$ 162–163° [6856], b.p.$_{17}$ 168–170° [7309], b.p.$_{15}$ 170–172° [7374];
m.p. 34–35° [7325,7374], 33.5° [7349], 32.5° [6856], 27° [6979].

1-(3-Ethoxy-4-hydroxyphenyl)-1-propanone

[159186-06-8] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

- Obtained by reaction of ethyl bromide with 3,4-dihydroxypropiophenone in the presence of potassium hydroxide in refluxing ethanol (38%) [6851].
- Also refer to: [7376,7377].

BIOLOGICAL ACTIVITY: Antiasthmatic [7377].

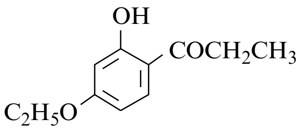
Ethyl ether $C_{13}H_{18}O_3$ mol.wt. 222.28.

- Preparation by reaction of propionyl chloride with o-diethoxybenzene in the presence of aluminium chloride in nitrobenzene at −5° for 2 h (73%) [6851].
- Also obtained by reaction of excess ethyl bromide with 3,4-dihydroxypropiophenone in the presence of potassium hydroxide in refluxing ethanol for 9 h (54%) [6851].

b.p.$_{32}$ 181–184° [6851]; m.p. 38–39° [6851].

1-(4-Ethoxy-2-hydroxyphenyl)-1-propanone

[63411-90-5] $C_{11}H_{14}O_3$ mol.wt. 194.23



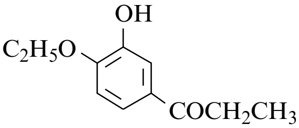
Syntheses

- Preparation by reaction of an ethyl halide (unspecified) with respropiophenone in the presence of potassium carbonate in refluxing acetone for 5 h (51%) [6380].
- Also obtained by reaction of ethyl iodide with respropiophenone in the presence of potassium hydroxide in ethanol [7140,7378].

Notes: Polymer with 1,2-ethanediol [161450-86-8] [7379]; polymer with 1,3-propanediol [163706-93-2] [7076]; polymer with 1,4-butanediol [160308-44-1] [7380].

m.p. 54° [7140], 52–53° [6380], 49° [7378];
^{1}H NMR [6380], IR [6380]; TLC [6380].

1-(4-Ethoxy-3-hydroxyphenyl)-1-propanone

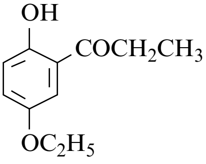


$C_{11}H_{14}O_3$ mol.wt. 194.23

Synthesis

- Obtained (by-product) by reaction of ethyl bromide with 3,4-dihydroxypropiophenone in boiling 25% ethanolic potassium hydroxide solution for 9 h (38%) [6851].

1-(5-Ethoxy-2-hydroxyphenyl)-1-propanone



$C_{11}H_{14}O_3$ mol.wt. 194.23

Synthesis

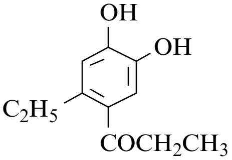
- Obtained (by-product) by Friedel–Crafts acylation of hydroquinone diethyl ether with propionyl chloride or propionic anhydride in the presence of aluminium chloride, first below 5° and short reflux times [7149].

N.B.: The amount of dealkylation could, however, be held below 20% under mild conditions. When more drastic conditions were employed, for example with longer reflux times, up to about 40% of the titled ketone could be obtained [7149].

m.p. 82° [7149].

1-(2-Ethyl-4,5-dihydroxyphenyl)-1-propanone

[267008-04-8]



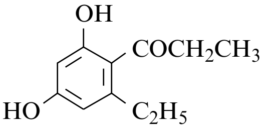
$C_{11}H_{14}O_3$ mol.wt. 194.23

Synthesis

- Preparation by demethylation of 4-propionyl-5-ethylveratrole (b.p.$_2$ 95–101°) with boron tribromide in methylene chloride, first at 0°, then at r.t. for 12 h (95–97%) [7381].

^{1}H NMR [7381].

1-(2-Ethyl-4,6-dihydroxyphenyl)-1-propanone



$C_{11}H_{14}O_3$ mol.wt. 194.23

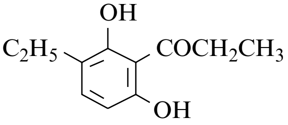
Synthesis

- Obtained by reaction of propionitrile with 5-ethylresorcinol (Hoesch reaction) (19%) [7382].

m.p. 142° [7382].

1-(3-Ethyl-2,6-dihydroxyphenyl)-1-propanone

[116867-95-9] $C_{11}H_{14}O_3$ mol.wt. 194.23



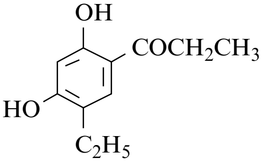
Syntheses

- Obtained from 5-ethyl-2,4-dihydroxy-3-propionylbenzoic acid in refluxing dilute acetic acid in the presence of a few drops of concentrated hydrochloric acid (37%) [6450].
- Also obtained by hydrolysis of 8-propionyl-7-hydroxy-6-ethyl-4-methylcoumarin in refluxing 10% sodium hydroxide for 2 h [6450].

m.p. 102° [6450].

1-(5-Ethyl-2,4-dihydroxyphenyl)-1-propanone

[158153-04-9] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

- Preparation by Fries rearrangement of 4-ethylresorcinol dipropionate in the presence of aluminium chloride (2 equiv) without solvent at 60–70° for 3–4 h or in nitrobenzene at 110° for 5 h [7383].
- Preparation by reaction of propionitrile with 4-ethyl-resorcinol (Hoesch reaction) (76%) [7115].
- Also refer to: [7384].

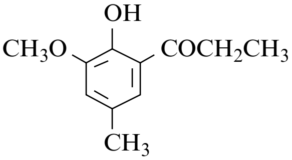
m.p. 69–70° [7115], 67.5° [7383].

Oxime [114113-06-3] $C_{11}H_{15}NO_3$ mol.wt. 209.22.

- For determination of cobalt and copper by gravimetry [7385].

1-(2-Hydroxy-3-methoxy-5-methylphenyl)-1-propanone

[91970-96-6] $C_{11}H_{14}O_3$ mol.wt. 194.23

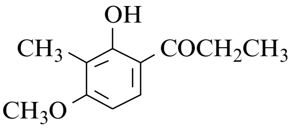


Syntheses

- Preparation by partial demethylation of 2,3-dimethoxy-5-methylpropiophenone with aluminium chloride in nitromethane, first at 0°, then at 20° for 5 days (88%) [7285].
- Also obtained by partial methylation of 2,3-dihydroxy-5-methylpropiophenone with dimethyl sulfate in the presence of potassium carbonate in acetone (39%) [7110].

m.p. 69–70° [7110], 67–68° [7285];
^1H NMR (Sadtler: standard n° 49338M) [7285],
IR (Sadtler: standard n° 76411K) [7285], UV [7285].

1-(2-Hydroxy-4-methoxy-3-methylphenyl)-1-propanone



$C_{11}H_{14}O_3$ mol.wt. 194.23

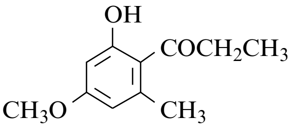
Syntheses

- Obtained by reaction of methyl iodide (5 mol) with respropiophenone in methanol in the presence of potassium hydroxide, first at 0° and at r.t. overnight, then at reflux for 6 h (22%) [6769].
- Also obtained by alkaline hydrolysis of 7-methoxy-2,3,8-trimethylchromone [6769].

m.p. 78–79° [6769].

1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1-propanone

[64030-63-3]



$C_{11}H_{14}O_3$ mol.wt. 194.23

Syntheses

- Preparation by partial methylation of 2,4-dihydroxy-6-methylpropiophenone with dimethyl sulfate,
  - in the presence of excess 0.5 N sodium hydroxide [6744];
  - in the presence of potassium carbonate in refluxing acetone [7120], (65%) [7121].
- Also obtained by reaction of propionitrile with orcinol monomethyl ether (Hoesch reaction) [6744].
- Also refer to: [7386].

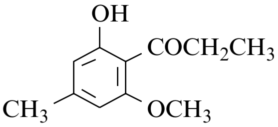
Isolation from natural sources

- From *Juniperus sabina* leaves (Cupressaceae) [7387].
- Obtained by alkaline degradation of *Coumarsabin* with potassium hydroxide in refluxing methanol for 2 h (40%) [7388]. *Coumarsabin* (3,5-dimethyl-4,7-dimethoxycoumarin) (m.p. 86–87°) was isolated from the leaves of *Juniperus sabina* (Cupressaceae).

m.p. 98–100° [7387], 84–85° [7121], 83–84° [7120], 82–83° [7388], 73.5° [6744];

^{1}H NMR [7121,7387,7388], ^{13}C NMR [7387], IR [7387,7388], UV [7387,7388].

1-(2-Hydroxy-6-methoxy-4-methylphenyl)-1-propanone



$C_{11}H_{14}O_3$ mol.wt. 194.23

Syntheses

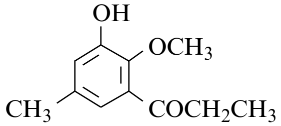
- Obtained by partial methylation of 2,6-dihydroxy-4-methyl-propiophenone with dimethyl sulfate in the presence of 0.5 N sodium hydroxide (40%) [6744].

- Also obtained by reaction of propionic anhydride with orcinol monomethyl ether in the presence of concentrated sulfuric acid at 130° [6744].

m.p. 75° [6744].

1-(3-Hydroxy-2-methoxy-5-methylphenyl)-1-propanone

[108439-91-4] $C_{11}H_{14}O_3$ mol.wt. 194.23



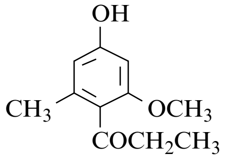
Synthesis

- Obtained by treatment of 2-methoxy-5-methyl-3-nitro-propiophenone (yellow oil) with stannous chloride dihydrate, then sodium nitrite, according to the Woodward procedure [7389], (23%) [7285].

m.p. 31° [7285]; ^1H NMR [7285], IR [7285], UV [7285].

1-(4-Hydroxy-2-methoxy-6-methylphenyl)-1-propanone

$C_{11}H_{14}O_3$ mol.wt. 194.23



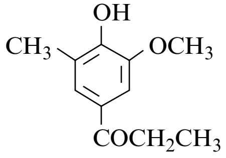
Syntheses

- Obtained by partial methylation of 2,4-dihydroxy-6-methylpropiophenone with dimethyl sulfate in the presence of 0.5 N sodium hydroxide [6744].
- Also obtained by reaction of ethyl cyanide with orcinol monomethyl ether (Hoesch reaction) [6744].

m.p. 114° [6744].

1-(4-Hydroxy-3-methoxy-5-methylphenyl)-1-propanone

[91970-97-7] $C_{11}H_{14}O_3$ mol.wt. 194.23



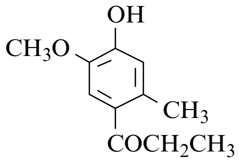
Synthesis

- Obtained by Fries rearrangement of 2-methoxy-6-methyl-phenyl propionate with aluminium chloride in refluxing carbon disulfide for 3 h (55%) [7390].

m.p. 106–108° [7390].

1-(4-Hydroxy-5-methoxy-2-methylphenyl)-1-propanone

[104216-16-2] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

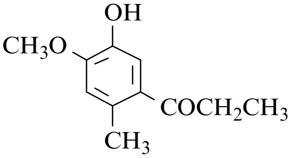
- Preparation by Fries rearrangement of isocreosol propionate with aluminium chloride in nitromethane, first at 0°, then at 30° for 24 h (97%) [7285].
- Also obtained by reaction of propionyl chloride with isocreosol in the presence of aluminium chloride in nitrobenzene, first at 30–35°, then at r.t. overnight (25%) [7391].

m.p. 87° [7285], 85.5° [7391];
^{1}H NMR (Sadtler: standard n° 49334M) [7285],
IR (Sadtler: standard n° 76407K) [7285], UV [7285]; pK_a [7285].

Benzoate [109469-57-0] $C_{18}H_{18}O_4$ mol.wt. 298.34 (m.p. 69°) [7391].

1-(5-Hydroxy-4-methoxy-2-methylphenyl)-1-propanone

[91970-98-8] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

- Preparation by reaction of propionyl chloride with creosol in the presence of aluminium chloride in methylene chloride, first at −20°, then at 20° for 2 h (95%) [7040].
- Preparation by reaction of propionic acid with creosol in the presence of boron trifluoride, first at 0°, then at 25° for 2 days (80%) [7110].
- Also obtained by Fries rearrangement of creosol propionate with aluminium chloride in methylene chloride, first at −20°, then at 20° for 8 h (59%) [7285].

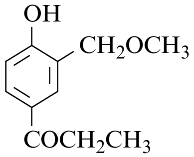
m.p. 98° [7285], 97–99° [7110];
^{1}H NMR (Sadtler: standard n° 49333M) [7285],
IR (Sadtler: standard n° 76406K) [7285], UV [7285]; pK_a [7285].

Propionate [108439-90-3] $C_{14}H_{18}O_4$ mol.wt. 250.29 (m.p. 46°) [7285].

^{1}H NMR (Sadtler: standard n° 49331M) [7285], IR (Sadtler: standard n° 76404K) [7285], UV [7285].

1-[4-Hydroxy-3-(methoxymethyl)phenyl]-1-propanone

[136715-21-4] $C_{11}H_{14}O_3$ mol.wt. 194.23



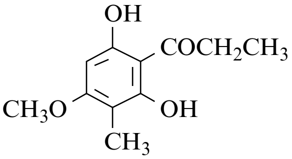
Synthesis

- Preparation [7392] (Czech patent).

USE: Pharmaceutical intermediate [7392].

1-(2,6-Dihydroxy-4-methoxy-3-methylphenyl)-1-propanone *(Aspidinol-P)*

[55765-53-4] $C_{11}H_{14}O_4$ mol.wt. 210.23



Syntheses

- Refer to: [6890] (compound **15**), [6895] (compound **23**) and [7393] (compound **31-P**).
- Also refer to: [7394].

N.B.: On the EI mass spectra drawing, the formula of the "titled ketone" was displayed (Fig. 9, page 136) [7394]. Actually, the concerned ketone was the upper homologous, i.e. the 2,6-dihydroxy-4-methoxy-3-methylbutyrophenone, called *Aspidinol* (compound **7**). On the drawing of the displayed formula of compound **7**, a methylene group is missing in the lateral chain [7394].

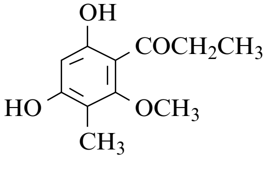
Isolation from natural sources

- From *Dryopteris* of Japan [7262].

m.p. 190–192° [6890];
UV [7394], MS [7262,7393,7394];
GLC [6895]; TLC [6890,6895,7262]; HPLC [7394];
paper chromatography [6895,7262].

1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)-1-propanone

[39026-68-1] $C_{11}H_{14}O_4$ mol.wt. 210.23



Isolation from natural sources

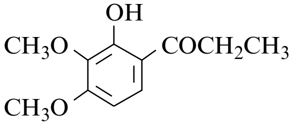
- Preparation by acid hydrolysis of its glycoside (SM1) ($C_{20}H_{30}O_9$, m.p. 179–180°, [39027-10-6]), itself obtained by treatment of (SM2) ($C_{36}H_{40}O_{12}$, m.p. 270°, [39036-20-9]) with barium hydroxide in boiling aqueous dioxane. SM2 is the hexamethyl ether of *poriolide*. The *poriolide* (major compound) ($C_{29}H_{26}O_{12}$, m.p. 265°, [39262-30-1]) was isolated from the leaves of *Leucothoe Keiskei Miq.* (Ericaceae) [7395,7396].

N.B.: *Isoporiolide* ($C_{29}H_{26}O_{12}$, m.p. 293–295°, [39262-31-2]) is an isomer of *poriolide*. *Isoporiolide* hexamethyl ether (SM3) ($C_{36}H_{40}O_{12}$, [39084-13-4]), decomposed under the same conditions using barium hydroxide, yields the phenol glycoside SM1 [7395,7396].

m.p. 132–134° [7395,7396];
^{1}H NMR [7395,7396], IR [7395,7396], UV [7395,7396].

1-(2-Hydroxy-3,4-dimethoxyphenyl)-1-propanone

[61948-26-3] $C_{11}H_{14}O_4$ mol.wt. 210.23

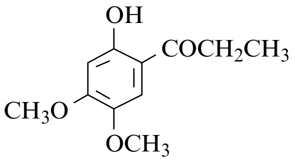


Syntheses

- Preparation by reaction of propionyl chloride with pyrogallol trimethyl ether in the presence of aluminium chloride in ethyl ether at 0° [7133], (56%) [7132] or at r.t. for 24 h (92%) [7397].
- Preparation by reaction of methyl iodide with 2,3,4-tri-hydroxypropiophenone in the presence of potassium carbonate in acetone, first at r.t. for 20 h, then at reflux for 8 h (94%) [6863].
- Also refer to: [6757,6758,7398] (compound **1f**).

m.p. 94–96° [6863], 93–94° [7132], 91–92° [7397];
^{1}H NMR [6863,7132,7397], IR [6863].

1-(2-Hydroxy-4,5-dimethoxyphenyl)-1-propanone



$C_{11}H_{14}O_4$ mol.wt. 210.23

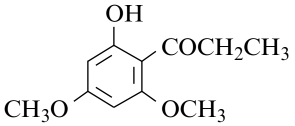
Syntheses

- Obtained by condensation of propionitrile with 3,4-di-methoxyphenol (Hoesch reaction) (16%) [7399].
- Also obtained (by-product) by reaction of propionyl chloride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride in carbon disulfide [7400], (29%) [7401].
- Also refer to: [7133,7402,7403].

m.p. 125° [7399], 124–126° [7401,7402], 124–125° [7400].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-1-propanone

[2215-82-9]



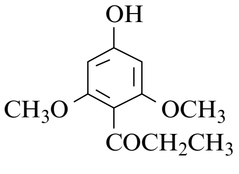
$C_{11}H_{14}O_4$ mol.wt. 210.23

Syntheses

- Preparation by reaction of propionyl chloride with phloroglucinol trimethyl ether in the presence of aluminium chloride in ethyl ether at 0° for 40 h (64%) [7404] or 48 h (71%) [7405].
- Also obtained by reaction of propionitrile with phloroglucinol dimethyl ether (Hoesch reaction) (67%) [7406], (38%) [6873,7407].
- Also obtained by reaction of dimethyl sulfate with phloropropiophenone in acetone in the presence of potassium carbonate [6629].
- Also obtained (by-product) by irradiation of 3,5-dimethoxy-2-propionylphenyl methacrylate in acetonitrile under argon atmosphere with high-pressure Hg lamp at r.t. (25%) [7408].
- Also refer to: [6757,6758,7138,7386,7409] (compound **1d**).

m.p. 113–114° [6629], 113° [7406], 111° [6873,7405], 110–111° [7404]; TLC [6629].

1-(4-Hydroxy-2,6-dimethoxyphenyl)-1-propanone



$C_{11}H_{14}O_4$ mol.wt. 210.23

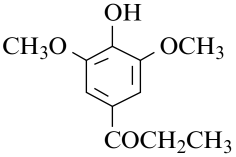
Syntheses

- Obtained by saponification of 4-(benzoyloxy)-2, 6-di-methoxypropiophenone (m.p. 103°) with 15% methanolic potassium hydroxide at r.t. for 2 h [6873].
- Also obtained (by-product) by reaction of propionitrile with phloroglucinol dimethyl ether (Hoesch reaction) [6873].
- Also refer to: [6743].

m.p. 180° [6743,6873].

1-(4-Hydroxy-3,5-dimethoxyphenyl)-1-propanone *(Propiosyringone)*

[5650-43-1] $C_{11}H_{14}O_4$ mol.wt. 210.23



Syntheses

- Preparation by oxidation of (3,5-dimethoxy-4-hydroxy-phenyl)ethylcarbinol also named 1-(4-hydroxy-3,5-di-methoxyphenyl)-1-propanol,
  - with DDQ in dioxane at r.t. for 16 h (70%) [7306];
  - using silver oxide in sodium hydroxide as described by [7169], (40%) [7170].
- Also obtained by partial demethylation of 3,4,5-trimethoxypropiophenone,
  - with hydrobromic acid at 40° or hydrochloric acid at 100° (20–30%) [6591];
  - in concentrated sulfuric acid solution [6973,7410], at 45–47° for 12 h (67%) [7411];
  - in the course of the reaction of ethylmagnesium bromide with 3,4,5-trimethoxybenzonitrile (14%) [7349].
- Also obtained by Fries rearrangement of 2,6-dimethoxyphenyl propionate (b.p.$_{0.5}$ 125–127°) with aluminium chloride in nitrobenzene [7411,7412], (30%) [7413] or at 80° for 30–60 min [7414], according to the procedure [6620], (11%) [6700,7161,7411].
- Preparation by reaction of sodium methoxide with 3-bromo or 3-iodo-4-hydroxy-5-methoxy-propiophenone in the presence of anhydrous cupric chloride (trace) in DMF at 100° (64%) [6959].

Isolation from natural sources

- Preparation from *Aspen poplar wood* [7415].
- From the aerial parts of *Baccharis magellanica* (Compositae) [7416].
- Identification in Spanish oak heartwood of *Quercus robur*, *Quercus petraea*, *Quercus pyrenaica* and *Quercus faginea* [7210].
- Identification in organic aerosols emitted from the combustion of biomass indigenous to South Asia [7417].
- In biodegradation of oak (Quercus alba) wood during growth of the shiitake mushroom (Lentinula edodes) [7418].
- One of aromatic degradation products in the steam hydrolysis residue of birchwood *(Betula pubescens)* [7419].
- Identification from volatile components of hardwood sawdust smoke [7420].
- Identification, in hydrogenolysis products of *Fraxinus mandshurica* [7421].
- Determination in black kraft liquor by GLC [7422].
- Of raw cane sugar flavour [7202].
- A flavour component of whiskey [7423].

- Its β-D-glucopyranoside [367502-03-2] was isolated from *Kokuto*, non-centrifuged Cane Sugar (*Saccharum officinarum*) [7247] or from the aerial parts of *Conyza blinii* [7424].
- Also refer to: [7092,7180,7188,7425–7430].

m.p. (monohydrate) 99–100° [6591];
(anhydrous) 113–115° [7431], 111.5–112.5° [6696],
109–110° [7161,7411,7413], 109° [7349],
108.5–109.5° [6959], 108–109° [7170], 107–109° [7415],
107° [6591], 106.5–108° [7414];
^{1}H NMR [7416], IR [6700,7161,7416,7420],
UV [6696,7161,7212,7213], MS [7416,7420];
GC-MS [7210,7220,7221,7423,7432];
chromatography [6700]; paper chromatography [7224,7419];
GC [7420]; GLC [7419]; HPLC [7423]; ionization potential [7232].
USE: Antioxidizing agent [7414].

Benzyl ether $C_{18}H_{20}O_4$ mol.wt. 300.35 (m.p. 71–72°) [7412].

Ethyl ether [184963-79-9] $C_{13}H_{18}O_4$ mol.wt. 238.28 [7433,7434].

Propyl ether [184963-86-8] $C_{14}H_{20}O_4$ mol.wt. 252.31 [7433,7434].

Isobutyl ether [184963-80-2] $C_{15}H_{22}O_4$ mol.wt. 266.34 [7434].

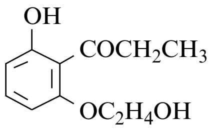
Acetate [52536-84-2] $C_{13}H_{16}O_5$ mol.wt. 252.27.

- Refer to: [6591,7419,7435].

m.p. 118° [7419], 110–112° [6591], 105° [7435], 95° [7419];
MS [7251,7372]; GC-MS [7251,7372].

1-[2-Hydroxy-6-(2-hydroxyethoxy)phenyl]-1-propanone

[3361-73-7] $C_{11}H_{14}O_4$ mol.wt. 210.23



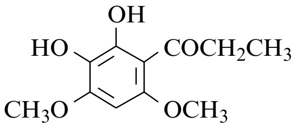
Syntheses

- Obtained by treatment of 2,6-dihydroxypropiophenone with epoxyethane [6802].
- Also refer to: [6805].

m.p. 91–92° [6802].

1-(2,3-Dihydroxy-4,6-dimethoxyphenyl)-1-propanone

[94190-89-3] $C_{11}H_{14}O_5$ mol.wt. 226.23



Syntheses

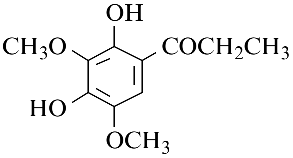
- Preparation by Fries rearrangement of 1,2-dihydroxy-3,5-dimethoxybenzene dipropionate with aluminium chloride in methylene chloride at r.t. for 3 h (94%) [7436,7437].

- Also obtained by hydrolysis of 1-[2-hydroxy-4,6-di-methoxy-3-(propionyloxy) phenyl]-1-propanone with 6 N hydrochloric acid in refluxing methanol for 30 min (79%) [7438].
- Also refer to: [7439].

m.p. 128° [7436,7437], 127° [7438];
^{1}H NMR [7438], ^{13}C NMR [7438], MS [7438]; TLC [7438].

1-(2,4-Dihydroxy-3,5-dimethoxyphenyl)-1-propanone

[99964-98-4] $C_{11}H_{14}O_5$ mol.wt. 226.23

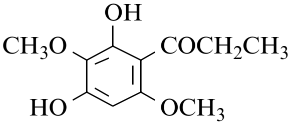


Isolation from natural sources

- From *Leucanthemopsis pallida* subsp. *flaveola* (Compositae) (compound **1**) [7440].

Colourless solid [7440]; m.p. 122–123° [7440];
^{1}H NMR [7440], ^{13}C NMR [7440], IR [7440],
UV [7440].

1-(2,4-Dihydroxy-3,6-dimethoxyphenyl)-1-propanone



$C_{11}H_{14}O_5$ mol.wt. 226.23

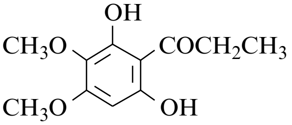
Synthesis

- Preparation by reaction of propionitrile with 2,5-dimethoxy-resorcinol (Hoesch condensation) (63%) [7441].

m.p. 126–127° [7441].

1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-propanone

[134081-93-9] $C_{11}H_{14}O_5$ mol.wt. 226.23

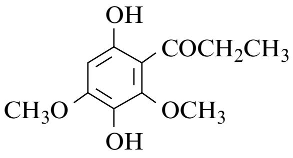


Synthesis

- Preparation by hydrolysis of 6-hydroxy-3, 4-dimethoxy-2-(tosyloxy)propiophenone (m.p. 120.5–121°) with potassium carbonate in refluxing methanol for 1–3 h (82%) [7442].

m.p. 142–143° [7442]; ^{1}H NMR [7442].

1-(3,6-Dihydroxy-2,4-dimethoxyphenyl)-1-propanone



$C_{11}H_{14}O_5$ mol.wt. 226.23

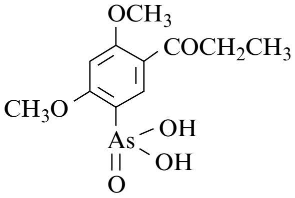
Synthesis

- Obtained by Elbs persulfate oxidation of 2-hydroxy-4,6-di-methoxypropiophenone (31%) [7405].

m.p. 120–121° [7405].

[2,4-Dimethoxy-5-(1-oxopropyl)phenyl]arsonic acid

2,4-Dimethoxy-5-arsonopropiophenone



$C_{11}H_{15}AsO_6$ mol.wt. 318.16.

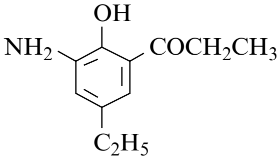
Syntheses

- Obtained from 5-amino-2,4-dimethoxypropiophenone through diazo reaction and coupling with sodium nitrite (61%) [6555].
- Also obtained by direct arsonation of respropiophenone with arsenic acid for 75 h at 100°, followed by methylation of the intermediate compound (11%) [6555].

m.p. 243° [6555].

1-(3-Amino-5-ethyl-2-hydroxyphenyl)-1-propanone

[70978-24-4]



$C_{11}H_{15}NO_2$ mol.wt. 193.25

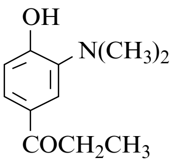
Syntheses

- Preparation by hydrogenation of 5-ethyl-2-hydroxy-3-nitropropiophenone in ethanol using 5% Pd/C as catalyst at atmospheric pressure and 25° [6926,6993].

m.p. 30° [6993].

1-[3-(Dimethylamino)-4-hydroxyphenyl]-1-propanone

[141771-86-0]



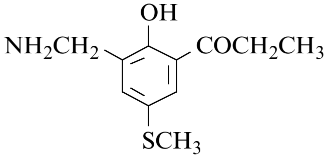
$C_{11}H_{15}NO_2$ mol.wt. 193.25

Synthesis

- Obtained from methylation of 3-amino-4-hydroxy-propiophenone by methyl iodide and triethylamine in methanol [6995].

1-[3-(Aminomethyl)-2-hydroxy-5-(methylthio)phenyl]-1-propanone

[75061-07-3] $C_{11}H_{15}NO_2S$ mol.wt. 225.31



Synthesis

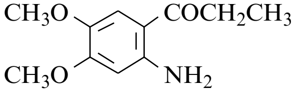
– Refer to: [7106].

Hydrochloride [75060-88-7] $C_{11}H_{15}NO_2S$, HCl mol.wt. 261.77.

BIOLOGICAL ACTIVITY: Antiinflammatory [7106].

1-(2-Amino-4,5-dimethoxyphenyl)-1-propanone

[4765-46-2] $C_{11}H_{15}NO_3$ mol.wt. 209.25



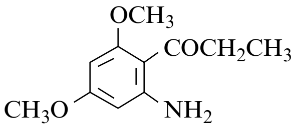
Synthesis

- Preparation by successively adding propionitrile and aluminium chloride to a solution of boron trichloride and 3,4-dimethoxyaniline in benzene under nitrogen and refluxing the mixture obtained for 8 h (60%) [7009].
- Preparation by reduction of 4,5-dimethoxy-2-nitropropiophenone with iron and acetic acid (68%) [7286].

m.p. 128–129° [7009];
^{1}H NMR [7286], IR [7286], MS [7009].

1-(2-Amino-4,6-dimethoxyphenyl)-1-propanone

[124623-19-4] $C_{11}H_{15}NO_3$ mol.wt. 209.25



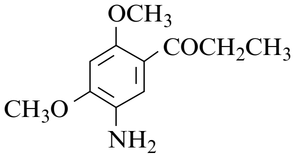
Synthesis

- Preparation by successively adding propionitrile and aluminium chloride to a solution of boron trichloride and 3,5-dimethoxyaniline in benzene under nitrogen and refluxing the mixture obtained for 8 h (92%) [7009].

m.p. 63–66° [7009]; MS [7009].

1-(5-Amino-2,4-dimethoxyphenyl)-1-propanone

$C_{11}H_{15}NO_3$ mol.wt. 209.25



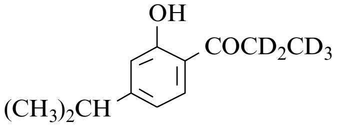
Synthesis

- Preparation from the corresponding nitro compound by catalytic reduction in acetone solution using Raney nickel. Then, after filtration of the catalyst and half elimination of solvent by vacuum distillation, the hydrochloride was prepared by passing dry hydrogen chloride into the remaining solution [6555].

m.p. 107° [6555].

Hydrochloride $C_{11}H_{15}NO_3,HCl$ mol.wt. 245.71 (m.p. > 300°) [6555].

1-[2-Hydroxy-4-(1-methylethyl)phenyl]-1-propanone-*2,2,3,3,3-d_5*



$C_{12}H_{11}D_5O_2$ mol.wt. 197.29

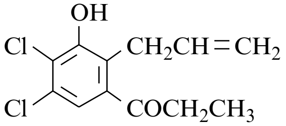
Synthesis

- Refer to: [6350].

Ion (1^-), radical ion (1^-) [72051-85-5], ESR spectrum [6350].

1-[4,5-Dichloro-3-hydroxy-2-(2-propenyl)phenyl]-1-propanone

[113730-43-1]



$C_{12}H_{12}Cl_2O_2$ mol.wt. 259.13

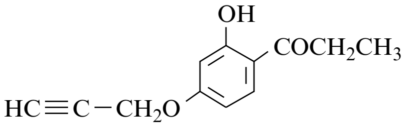
Synthesis

- Obtained by heating 2-allyl-4,5-dichloro-3-hydroxy-propiophenone (m.p. 50°) at 235° for 8 min (Claisen rearrangement) (59%) [6386].

m.p. 80° [6386]; ^{1}H NMR [6386].

1-[2-Hydroxy-4-(2-propynyloxy)phenyl]-1-propanone

[457628-03-4]



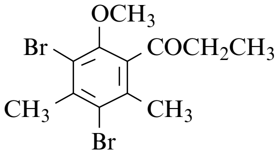
$C_{12}H_{12}O_3$ mol.wt. 204.23

Syntheses

- Refer to: [6524,6526].

1-(3,5-Dibromo-2-methoxy-4,6-dimethylphenyl)-1-propanone

[5384-17-8]



$C_{12}H_{14}Br_2O_2$ mol.wt. 350.05

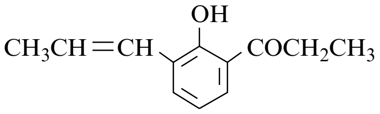
Synthesis

- Preparation by reaction of dimethyl sulfate with 3,5-di-bromo-4,6-dimethyl-2-hydroxypropiophenone in methanol in the presence of aqueous sodium hydroxide [6981].

m.p. 70° [6981].

1-[2-Hydroxy-3-(1-propenyl)phenyl]-1-propanone

[35888-89-2]



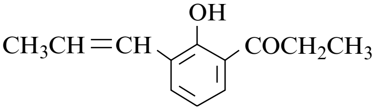
$C_{12}H_{14}O_2$ mol.wt. 190.24

Syntheses

- Preparation by potassium hydroxide-catalyzed isomerization of 3-allyl-2-hydroxypropiophenone [6933] in refluxing diethylene glycol (55%) [6935].
- Also refer to: [6934,7443,7444] (Japanese patents) and [6936,7445,7446].

1-[2-Hydroxy-3-(1-propenyl)phenyl]-1-propanone *(E)*

[187276-37-5]



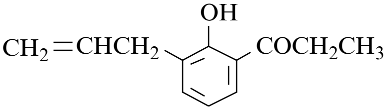
$C_{12}H_{14}O_2$ mol.wt. 190.24

Synthesis

- Refer to: [7269].

1-[2-Hydroxy-3-(2-propenyl)phenyl]-1-propanone

[35888-91-6]



$C_{12}H_{14}O_2$ mol.wt. 190.24

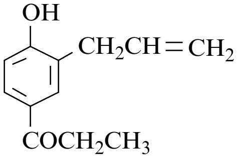
Syntheses

- Obtained by Claisen rearrangement of 2-(allyloxy)-propiophenone (b.p.$_{5.5}$ 112–113°) without solvent at 250–260° (79%) [7447].
- Also refer to: [6933–6935,7443,7446].

b.p.$_5$ 100–102° [7447].

1-[4-Hydroxy-3-(2-propenyl)phenyl]-1-propanone

[91496-09-2]



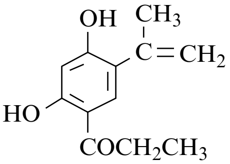
$C_{12}H_{14}O_2$ mol.wt. 190.24

Syntheses

- Obtained by Claisen rearrangement of 4-(allyloxy)-propiophenone (b.p.$_{4.5}$ 140–142°) [7447],
  - in boiling dimethylaniline [6667], for 5–6 h [7448];
  - without solvent at 200–210° for 12 h [6667] or at 260–270° (80%) [7447].
- Also refer to: [7449].

b.p.$_{21}$ 203–204° [7448]; m.p. 83° [6667,7448], 65° [7447].
One of the reported melting points is obviously wrong.

1-[2,4-Dihydroxy-5-(1-methylethenyl)phenyl]-1-propanone



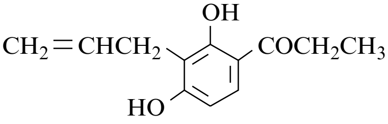
$C_{12}H_{14}O_3$ mol.wt. 206.24

Synthesis

- Obtained from 2,4-dihydroxy-5-propionyl-β-cinnamic acid on heating above its melting point [6800].

1-[2,4-Dihydroxy-3-(2-propenyl)phenyl]-1-propanone

[74815-88-6]



$C_{12}H_{14}O_3$ mol.wt. 206.24

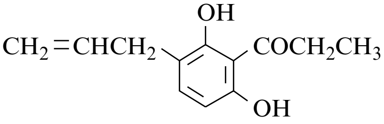
Syntheses

- Preparation by Claisen rearrangement of 4-(allyl-oxy)-2-hydroxypropiophenone,
  - at 210–215° for 1.5 h (90%) [7450] or at 205° for 2 h (85%) [7451];
  - in refluxing o-dichlorobenzene for 20–24 h (75–80%) [6766].
- Also refer to: [7452,7453].

m.p. 124° [7451]; ^{1}H NMR [7450], MS [7450].

1-[2,6-Dihydroxy-3-(2-propenyl)phenyl]-1-propanone

[17488-75-4]



$C_{12}H_{14}O_3$ mol.wt. 206.24

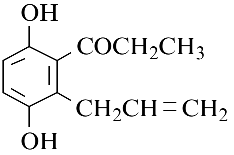
Synthesis

- Obtained by heating 6-allyl-8-propionyl-β-methyl-umbelliferone (m.p. 136–137.5°) in sodium hydroxide solution containing sodium hydrosulfite [7153].

m.p. 57–59° [7153].

1-[3,6-Dihydroxy-2-(2-propenyl)phenyl]-1-propanone

[28885-62-3]



$C_{12}H_{14}O_3$ mol.wt. 206.24

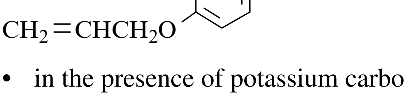
Syntheses

- Obtained by thermal Claisen rearrangement of 5-(allyloxy)-2-hydroxypropiophenone [7454], at 180–225° for 1 h under nitrogen (65%) [6787].

m.p. 90–91° [7454], 90–90.5° [6787];
^{1}H NMR [6787], MS [6787].

1-[2-Hydroxy-4-(2-propenyloxy)phenyl]-1-propanone

[106627-40-1]



$C_{12}H_{14}O_3$ mol.wt. 206.24

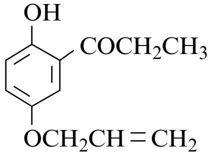
Syntheses

- Preparation by reaction of allyl bromide with respropiophenone,
  - in the presence of potassium carbonate in refluxing acetone for 7 h [7451] or in refluxing methyl ethyl ketone for 3 days (55%) [7450];
  - in the presence of cesium carbonate in DMF at 20° (95–98%) [6766].

b.p.$_7$ 156–162° [7450], b.p.$_{14}$ 174° [7451]; MS [7450].

1-[2-Hydroxy-5-(2-propenyloxy)phenyl]-1-propanone

[29026-02-6] $C_{12}H_{14}O_3$ mol.wt. 206.24



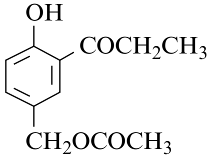
Syntheses

– Obtained by allylation of 2,5-dihydroxypropiophenone [7454], with allyl bromide in the presence of potassium carbonate in refluxing acetone for 8 h (65%) [6787].

b.p.$_{10}$ 163–165° [6787]; m.p. 50–51° [6787,7454]; ^{1}H NMR [6787], MS [6787].

1-[5-(Acetoxymethyl)-2-hydroxyphenyl]-1-propanone

[108540-33-6] $C_{12}H_{14}O_4$ mol.wt. 222.24



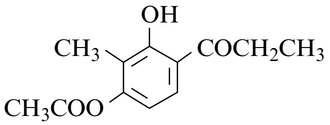
Synthesis

– Preparation by reaction of sodium acetate with 5-chloromethyl-2-hydroxypropiophenone in acetic acid at 125–130° for 2 h (92%) [6986].

m.p. 55–57° [6986].

1-[4-(Acetyloxy)-2-hydroxy-3-methylphenyl]-1-propanone

[130737-47-2] $C_{12}H_{14}O_4$ mol.wt. 222.24

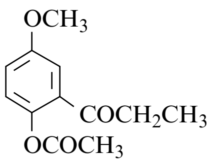


Syntheses

– Preparation by partial acetylation of 2, 4-dihydroxy-3-methylpropiophenone [7113] with acetyl chloride in the presence of boron trifluoride etherate [7114].
– Also refer to: [7455].

1-[2-(Acetyloxy)-5-methoxyphenyl]-1-propanone

$C_{12}H_{14}O_4$ mol.wt. 222.24



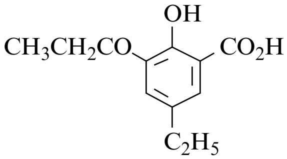
Synthesis

– Preparation by acetylation of 2-hydroxy-5-methoxypropiophenone [7149].

m.p. 54° [7149].

5-Ethyl-2-hydroxy-3-(1-oxopropyl)benzoic acid

[67127-81-5] $C_{12}H_{14}O_4$ mol.wt. 222.24



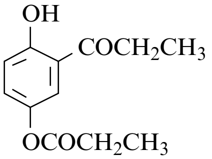
Synthesis

- Obtained by Fries rearrangement of 5-ethyl-2-propionoxy-benzoic acid according to [7268], (45%) [6926].

m.p. 135–136° [6926].

1-[2-Hydroxy-5-(1-oxopropoxy)phenyl]-1-propanone

[459124-92-6] $C_{12}H_{14}O_4$ mol.wt. 222.24



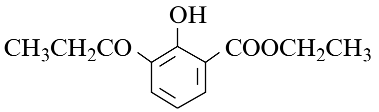
Syntheses

- Obtained by Fries rearrangement of 1,4-dipropionyloxy-benzene with aluminium chloride (20%) [6745], at 142° for 30 min [6783,6798] or at 120° for 4 h (43%) [6733].
- Also obtained by heating 2-propionylhydroquinone with propionic anhydride [6745].

m.p. 71–73° [6783,6798], 71° [6733], 53° [6745].
One of the reported melting points is obviously wrong.
^1H NMR [6783,6798], IR [6733,6783,6798], UV [6733].

2-Hydroxy-3-(1-oxopropyl)benzoic acid ethyl ester

[35888-93-8] $C_{12}H_{14}O_4$ mol.wt. 222.24

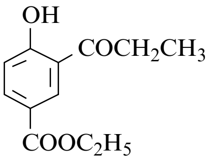


Syntheses

- Obtained by esterification of the corresponding acid [6935].
- Also refer to: [6933,6936,7269].

4-Hydroxy-3-(1-oxopropyl)benzoic acid ethyl ester

[100117-91-7] $C_{12}H_{14}O_4$ mol.wt. 222.24

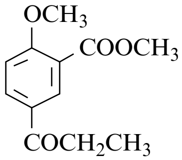


Syntheses

- Obtained by reaction of propionyl chloride with ethyl p-hydroxybenzoate in the presence of aluminium chloride in tetrachloroethane at 120° for 3–4 h [6941].
- Also refer to: [6942].

m.p. 73–75° [6942], 65° [6941].
One of the reported melting points is obviously wrong.

2-Methoxy-5-(1-oxopropyl)benzoic acid methyl ester



$C_{12}H_{14}O_4$ mol.wt. 222.24

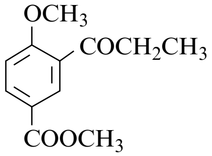
Syntheses

- Preparation by methylation of 5-propionylsalicylic acid with dimethyl sulfate in 25% sodium hydroxide at 80° (62%) [6937,7005].
- Also obtained by refluxing 1 h methyl 5-propionylsalicylate in a methanolic solution of sodium methoxide with dimethyl sulfate (74%) [6937,7005].

m.p. 83.5–84.5° [6937,7005].

4-Methoxy-3-(1-oxopropyl)benzoic acid methyl ester

[91497-18-6] $C_{12}H_{14}O_4$ mol.wt. 222.24



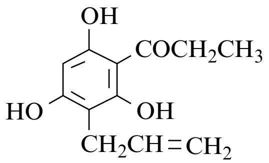
Synthesis

- Obtained by reaction of 4-hydroxy-3-propionylbenzoic acid with potassium carbonate in refluxing acetone for 15 min, then treatment in situ of the potassium salt formed with methyl iodide at reflux for 20 h (67%) [6943].

m.p. 74–75° [6943].

1-[2,4,6-Trihydroxy-3-(2-propenyl)phenyl]-1-propanone

[85602-23-9] $C_{12}H_{14}O_4$ mol.wt. 222.24



Syntheses

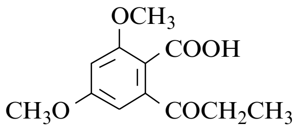
- Preparation by alkenylation of phloropropiophenone with allyl chloride in aqueous ethyl ether in the presence of sodium carbonate and cuprous chloride at r.t. for 3 h (90%) [7456].
- Also refer to: [7457].

m.p. 157–159° [7456]; ^{13}C NMR [7456,7458], IR [7456], MS [7456].

USE: Fungicide [7457].

BIOORGANIC ACTIVITY: Bactericide [7457].

2,4-Dimethoxy-6-(1-oxopropyl)benzoic acid



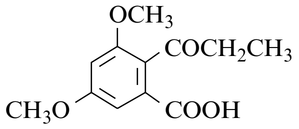
$C_{12}H_{14}O_5$ mol.wt. 238.24

Synthesis

- Obtained by heating 3,5-dimethoxyphthalic anhydride, propionic acid and sodium propionate at 170–180° for 1.5 h [7325].

m.p. 158° [7325].

3,5-Dimethoxy-2-(1-oxopropyl)benzoic acid



$C_{12}H_{14}O_5$ mol.wt. 238.24

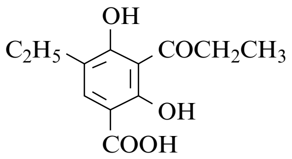
Synthesis

- Obtained by heating 3,5-dimethoxyphthalic anhydride, propionic acid and sodium propionate at 170–180° for 1.5 h [7325].

m.p. 160° [7325].

5-Ethyl-2,4-dihydroxy-3-(1-oxopropyl)benzoic acid

[105339-17-1]



$C_{12}H_{14}O_5$ mol.wt. 238.24

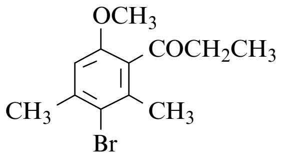
Syntheses

- Obtained by Fries rearrangement of methyl 2,4-dipropio-noxy-5-ethylbenzoate (m.p. 89°) with aluminium chloride at 110–120° for 1 h (26%) [6450].
- Also obtained by Friedel–Crafts acylation of methyl 2,4-di-hydroxy-5-ethylbenzoate with propionic anhydride in the presence of aluminium chloride in nitrobenzene, first at 25–30° for 20 h, then at 105° for 4 h [6450].
- Also obtained by treatment of methyl 5-ethyl-2,4-dihydroxy-3-propionylbenzoate with 10% sodium hydroxide solution at r.t. for 24 h [6450].

m.p. 183° [6450].

1-(3-Bromo-6-methoxy-2,4-dimethylphenyl)-1-propanone

[36871-61-1]



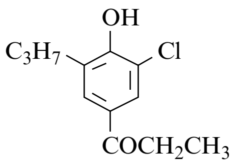
$C_{12}H_{15}BrO_2$ mol.wt. 271.15

Syntheses

- Preparation by reaction of dimethyl sulfate with 3-bromo-6-hydroxy-2,4-dimethylpropiophenone [6351].
- Preparation by reaction of bromine with 2-methoxy-4,6-dimethylpropiophenone in dilute acetic acid at r.t. for 2 h [6980].

^{1}H NMR [6980].

1-(3-Chloro-4-hydroxy-5-propylphenyl)-1-propanone



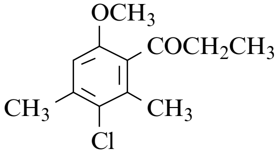
$C_{12}H_{15}ClO_2$ mol.wt. 226.70

Synthesis

- Preparation by heating a suspension of sodium propionate and 2-chloro-6-propylphenol in neat triflic acid [6402].

1-(3-Chloro-6-methoxy-2,4-dimethylphenyl)-1-propanone

[107076-12-0] $C_{12}H_{15}ClO_2$ mol.wt. 226.70



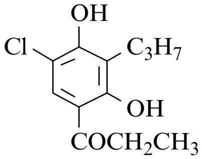
Synthesis

- Obtained by reaction of propionic anhydride with 4-chloro-3,5-dimethylanisole in the presence of aluminium chloride in refluxing carbon disulfide (55%) [7278].

m.p. 66.5–67.5° [7278].

1-(5-Chloro-2,4-dihydroxy-3-propylphenyl)-1-propanone

[612812-31-4] $C_{12}H_{15}ClO_3$ mol.wt. 242.70

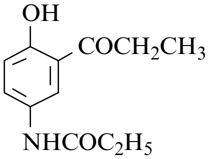


Synthesis

- Preparation by heating a mixture of 4-chloro-2-propylresorcinol, sodium propionate and triflic acid [7459].

4-Hydroxy-3-(1-oxopropyl)propionanilide

[91641-62-2] $C_{12}H_{15}NO_3$ mol.wt. 221.26



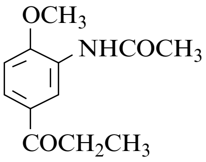
Syntheses

- Obtained by Fries rearrangement of p-aminophenol dipropionate (m.p. 165°) with aluminium chloride at 140° for 1 h (60%) [7460].
- Also refer to: [7283].

m.p. 125° [7460].

2-Methoxy-5-(1-oxopropyl)acetanilide

$C_{12}H_{15}NO_3$ mol.wt. 221.26



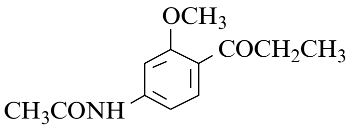
Synthesis

- Obtained by reaction of acetic anhydride with 3-amino-4-methoxypropiophenone at r.t. for 12 h [7002].

m.p. 134–138° [7002].

3-Methoxy-4-(1-oxopropyl)acetanilide

$C_{12}H_{15}NO_3$ mol.wt. 221.26

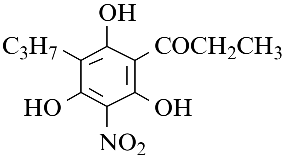


Synthesis

- Refer to: [6927].

1-(2,4,6-Trihydroxy-3-nitro-5-propylphenyl)-1-propanone

[119691-98-4] $C_{12}H_{15}NO_6$ mol.wt. 269.25



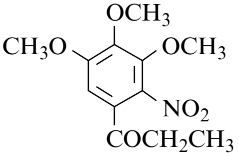
Synthesis

- Obtained by reaction of fuming nitric acid with 3′-propyl-2′,4′,6′-trihydroxypropiophenone in acetic acid at 60° (30–40%) [6561].

m.p. 100–102° [6561];
^{1}H NMR [6561], IR [6561], MS [6561].

1-(3,4,5-Trimethoxy-2-nitrophenyl)-1-propanone

$C_{12}H_{15}NO_6$ mol.wt. 269.25



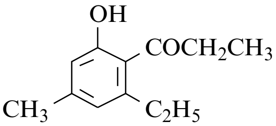
Synthesis

- Obtained from ethyl α-(nitrotrimethylgallyl)propionate by boiling a short time with dilute sulfuric acid [7461].

heavy brown oil [7461].

1-(2-Ethyl-6-hydroxy-4-methylphenyl)-1-propanone

$C_{12}H_{16}O_2$ mol.wt. 192.26



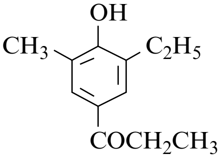
Syntheses

- Preparation by Fries rearrangement of 3-ethyl-5-methyl-phenyl propionate (b.p.$_{28}$ 142°) with aluminium chloride,
  - without solvent at 130° for 2 h (78%) [6480];
  - in nitrobenzene at 25° for 6 h (80%) [6480].
- To see (*12i*) [6407].

b.p.$_3$ 240° [6480].

1-(3-Ethyl-4-hydroxy-5-methylphenyl)-1-propanone

$C_{12}H_{16}O_2$ mol.wt. 192.26

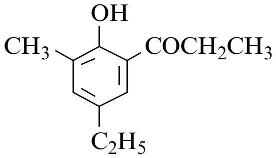


Synthesis

- Obtained by Fries rearrangement of 2-methyl-6-ethylphenyl propionate with aluminium chloride at 120° (73%) [7304].

m.p. 101–102° [7304].

1-(5-Ethyl-2-hydroxy-3-methylphenyl)-1-propanone



$C_{12}H_{16}O_2$ mol.wt. 192.26

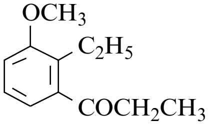
Synthesis

- Obtained by Fries rearrangement of 2-methyl-4-ethylphenyl propionate with aluminium chloride at 110° (71%) [7304].

b.p.$_{14}$ 152–154° [7304].

1-(2-Ethyl-3-methoxyphenyl)-1-propanone

[17055-42-4] $C_{12}H_{16}O_2$ mol.wt. 192.26



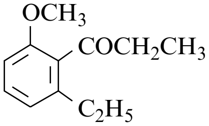
Syntheses

- Preparation by reaction of 2-ethyl-3-methoxybenzonitrile with ethylmagnesium bromide (60%) [7130] or ethyl-magnesium iodide (69%) [7129].
- Also refer to: [7462].

b.p.$_{0.2}$ 82–84° [7130], b.p.$_2$ 142° [7129].

1-(2-Ethyl-6-methoxyphenyl)-1-propanone

[38463-02-4] $C_{12}H_{16}O_2$ mol.wt. 192.26



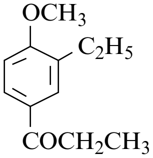
Synthesis

- Obtained from o-bromoanisole by treatment with sodium amide and diethyl ketone in THF at 65° for 6 h (30%) [7463].

^{1}H NMR [7463], IR [7463].

1-(3-Ethyl-4-methoxyphenyl)-1-propanone

[2129-07-9] $C_{12}H_{16}O_2$ mol.wt. 192.26



Syntheses

- Preparation by Friedel–Crafts acylation of 2-ethylanisole with propionyl chloride in the presence of aluminium chloride in carbon disulfide [7350], (81%) [7464].

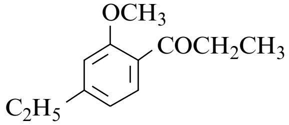
b.p.$_3$ 145–147° [7464].

BIOLOGICAL ACTIVITY: Antiinflammatory [7350].

Semicarbazone $C_{13}H_{19}N_3O_2$ mol.wt. 249.31 (m.p. 175°) [7464].

2,4-Dinitrophenylhydrazone $C_{18}H_{20}N_4O_5$ mol.wt. 372.38 (m.p. 155°) [7464].

1-(4-Ethyl-2-methoxyphenyl)-1-propanone



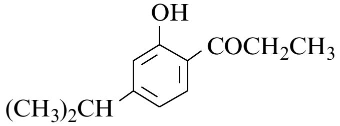
$C_{12}H_{16}O_2$ mol.wt. 192.26

Synthesis

- Preparation by reaction of dimethyl sulfate with 4-ethyl-2-hydroxypropiophenone in the presence of 10% aqueous sodium hydroxide at r.t. (87%) [7290].

b.p.$_{29}$ 215° [7290].

1-[2-Hydroxy-4-(1-methylethyl)phenyl]-1-propanone



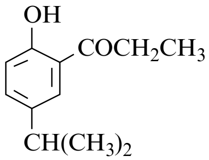
$C_{12}H_{16}O_2$ mol.wt. 192.26

Synthesis

- Refer to: [6350].

N.B.: Ion (1⁻), radical ion (1⁻) [72051-84-4], ESR spectrum [6350].

1-[2-Hydroxy-5-(1-methylethyl)phenyl]-1-propanone



$C_{12}H_{16}O_2$ mol.wt. 192.26

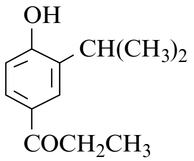
Synthesis

- Refer to: [6646].

Hydrazone [70136-41-3] $C_{12}H_{18}N_2O$ mol.wt. 206.29 [6646].

1-[4-Hydroxy-3-(1-methylethyl)phenyl]-1-propanone

[1807-41-6] $C_{12}H_{16}O_2$ mol.wt. 192.26

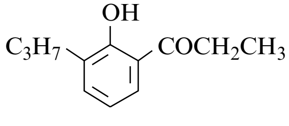


Syntheses

- Preparation by Fries rearrangement of o-isopropylphenyl propionate with aluminium chloride in nitrobenzene at r.t. for 3 days, then at 50–60° for 3–4 h (64%) [7465].
- Refer to: [7289].

m.p. 123° [7465].

1-(2-Hydroxy-3-propylphenyl)-1-propanone



$C_{12}H_{16}O_2$ mol.wt. 192.26

Syntheses

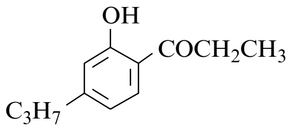
- Obtained (poor yield) by Fries rearrangement of 2-propyl-phenyl propionate (b.p.$_{11}$ 120°)

[7466] with aluminium chloride at 130–135° for 30 min (15%) [7466] or at 120° according to [6961], (87%) [7467].

N.B.: The compound obtained by [7467] is in reality the *para* isomer, 4-hydroxy-3-propyl-propiophenone.

b.p.$_{13}$ 142° [7466], b.p.$_3$ 164° [7467]. Boiling points incoherents.
m.p. 78–79° [7467], 43° [7466].
One of the reported melting points is obviously wrong.

1-(2-Hydroxy-4-propylphenyl)-1-propanone



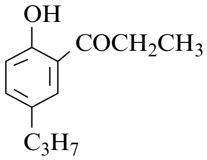
$C_{12}H_{16}O_2$ mol.wt. 192.26

Synthesis

- Preparation by reaction of propionic acid with m-propyl-phenol in the presence of zinc chloride for 5 h at 180° (Nencki reaction) (38%) [7468].

b.p.$_{0.7}$ 90° [7468], b.p.$_{13}$ 124–125° [7468].

1-(2-Hydroxy-5-propylphenyl)-1-propanone



$C_{12}H_{16}O_2$ mol.wt. 192.26

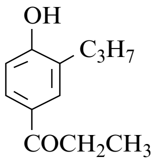
Syntheses

- Preparation by Fries rearrangement of p-propylphenyl propionate (b.p. 254–256°) [6575] with aluminium chloride for 2 h at 100° and the reaction terminated at 120° (83%) [6575] or at 120° according to [6961], (61%) [7467].

b.p.$_3$ 115–118° [7467], b.p. 270° [6575].

1-(4-Hydroxy-3-propylphenyl)-1-propanone

[194792-41-1] $C_{12}H_{16}O_2$ mol.wt. 192.26



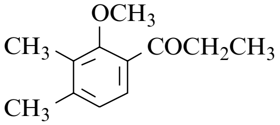
Syntheses

- Preparation by Fries rearrangement of 2-propylphenyl propionate (b.p.$_{1.3-1.4}$ 84–86°) [7469], (b.p.$_{11}$ 120°) [7466], (b.p. 245°) [6575] with aluminium chloride at 120° according to [6961] (87%) [7467], for 2 h at 100° and the reaction terminated at 120° (70%) [6575], for 30 min at 130–135° (63%) [7466] or for 3 h at 140–150° (51%) [7469].
- Also obtained by reaction of propionyl chloride with 2-propylphenol in the presence of aluminium chloride in nitrobenzene at r.t. overnight (Behn's method) [7470].
- Also refer to: [7289,7452,7471].

b.p.$_3$ 164° [7467];
m.p. 80–81° [7470], 78–79° [7467], 77–78° [7469], 77° [6575], 75° [7466];
^{1}H NMR [7469], ^{13}C NMR [7469].

1-(2-Methoxy-3,4-dimethylphenyl)-1-propanone

[107075-91-2] $C_{12}H_{16}O_2$ mol.wt. 192.26

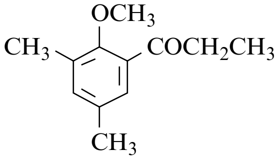


Synthesis

– Refer to: [7292].

1-(2-Methoxy-3,5-dimethylphenyl)-1-propanone

[5384-04-3] $C_{12}H_{16}O_2$ mol.wt. 192.26



Syntheses

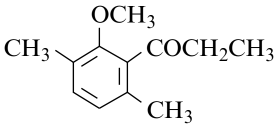
– Preparation by reaction of dimethyl sulfate with 3,5-di-methyl-2-hydroxypropiophenone in methanol in the presence of aqueous sodium hydroxide [6981].

– Also refer to: [7292].

b.p.$_{13}$ 154° [6981];
m.p. −17° [6981]; $n_D^{20} = 1.523$ [6981].

1-(2-Methoxy-3,6-dimethylphenyl)-1-propanone

[107075-92-3] $C_{12}H_{16}O_2$ mol.wt. 192.26

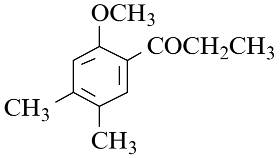


Synthesis

– Refer to: [7292].

1-(2-Methoxy-4,5-dimethylphenyl)-1-propanone

[36871-58-6] $C_{12}H_{16}O_2$ mol.wt. 192.26



Syntheses

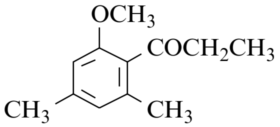
– Preparation by reaction of dimethyl sulfate with 2-hydroxy-4,5-dimethylpropiophenone in methanolic sodium hydroxide [6980].

– Also refer to: [7292].

b.p.$_{25}$ 190° [7472]; m.p. 69° [7472].

1-(2-Methoxy-4,6-dimethylphenyl)-1-propanone

[5384-14-5] $C_{12}H_{16}O_2$ mol.wt. 192.26



Syntheses

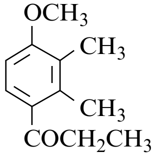
– Preparation by reaction of propionic anhydride with 3,5-di-methylanisole in the presence of aluminium chloride in refluxing carbon disulfide (75%) [7278].

- Also obtained by reaction of dimethyl sulfate with 2-hydroxy-4,6-dimethylpropiophenone in the presence of aqueous or dilute methanolic sodium hydroxide [7278].
- Also refer to: [6980,7292].

b.p.$_2$ 120–122° [7278], b.p.$_{13}$ 143° [6981], b.p.$_{36}$ 175° [7290];
m.p. 28–29° [6981]; IR [6981].

1-(4-Methoxy-2,3-dimethylphenyl)-1-propanone

[90852-26-9] $C_{12}H_{16}O_2$ mol.wt. 192.26

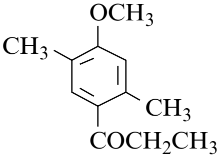


Synthesis

- Refer to: [7473] (Japanese patent).

1-(4-Methoxy-2,5-dimethylphenyl)-1-propanone

[36871-56-4] $C_{12}H_{16}O_2$ mol.wt. 192.26



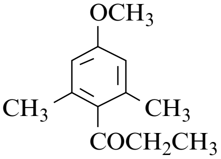
Syntheses

- Preparation by reaction of dimethyl sulfate with 4-hydroxy-2,5-dimethylpropiophenone in methanolic sodium hydroxide solution [6980].
- Also refer to: [7292].

IR [6980].

1-(4-Methoxy-2,6-dimethylphenyl)-1-propanone

[107075-90-1] $C_{12}H_{16}O_2$ mol.wt. 192.26



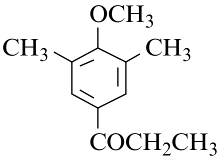
Syntheses

- Refer to: [7292,7472].

b.p.$_{15}$ 154° [7472]; m.p. 76° [7472].

1-(4-Methoxy-3,5-dimethylphenyl)-1-propanone

[5384-11-2] $C_{12}H_{16}O_2$ mol.wt. 192.26



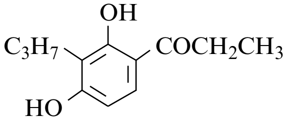
Syntheses

- Preparation by reaction of dimethyl sulfate with 3, 5-di-methyl-4-hydroxypropiophenone in methanol in the presence of 20% potassium hydroxide (90%) [6981].
- Also refer to: [7292,7474].

b.p.$_{12}$ 154–156° [6981]; m.p. 27° [6981].

1-(2,4-Dihydroxy-3-propylphenyl)-1-propanone

[79558-49-9]



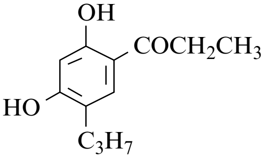
$C_{12}H_{16}O_3$ mol.wt. 208.26

Syntheses

- Preparation by reaction of propionitrile with 2-propyl-resorcinol (Hoesch reaction) (80%) [7451].
- Also obtained by reaction of hydrogen with 3-allyl-respropiophenone,
  - in the presence of $PdCl_2$ in ethanol for 1 h [7451];
  - in the presence of 5% Pd/C in MTBE (tert-butyl methyl ether) (quantitative yield) [6766];
  - in the presence of Raney nickel at r.t. for 4.5 h at 60 psi of hydrogen (18%) [7450].
- Preparation by reaction of propionic acid with 2-propylresorcinol in the presence of hydrobromic acid [7475].
- Also obtained by heating a mixture of 2-propylresorcinol, sodium propionate and triflic acid (TfOH) [7459].
- Also refer to: [7452,7476–7483].

m.p. 109–110° [7451]; MS [7450].

1-(2,4-Dihydroxy-5-propylphenyl)-1-propanone



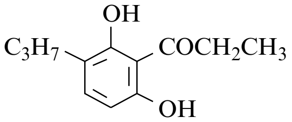
$C_{12}H_{16}O_3$ mol.wt. 208.26

Syntheses

- Preparation by Fries rearrangement of 4-propylresorcinol dipropionate in the presence of aluminium chloride (2 equiv) without solvent at 60–70° for 3–4 h or in nitrobenzene at 110° for 5 h [7383].
- Preparation by reaction of propionitrile with 4-propyl-resorcinol (Hoesch reaction) (80%) [7115].

b.p.$_{9.5}$ 190–195° [7383]; m.p. 82° [7115,7484], 73–74° [7383].

1-(2,6-Dihydroxy-3-propylphenyl)-1-propanone



$C_{12}H_{16}O_3$ mol.wt. 208.26

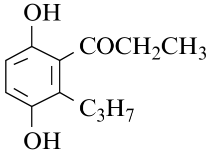
Synthesis

- Obtained by catalytic reduction of 2,6-dipropionylresorcinol with Pd in acetic acid [7383].

m.p. 92° [7383].

1-(3,6-Dihydroxy-2-propylphenyl)-1-propanone

[28885-63-4] $C_{12}H_{16}O_3$ mol.wt. 208.26



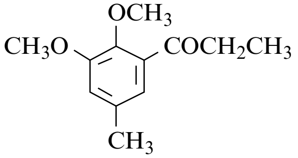
Synthesis

– Obtained by catalytic hydrogenation of 2-allyl-3,6-di-hydroxypropiophenone [7454] over Pd/C in ethanol with hydrogen at 1 atmosphere for 3.5 h (89%) [6787].

m.p. 114–114.5° [6787], 114° [7454]; ^{1}H NMR [6787], MS [6787].

1-(2,3-Dimethoxy-5-methylphenyl)-1-propanone

[108439-93-6] $C_{12}H_{16}O_3$ mol.wt. 208.26



Syntheses

– Preparation by reaction of 2,3-dimethoxy-5-methylbenzoyl chloride with zinc ethyl iodide (prepared according to [7309] in toluene at r.t. for 2 h (70%) [7110]).

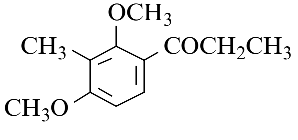
– Obtained by reaction of 2,3-dimethoxy-5-methylbenzonitrile and ethylmagnesium bromide [7370]. **N.B.**: Analysis showed that the oil had a composition corresponding to a mixture of about equal parts of 2,3-dimethoxy-5-methylpropiophenone and 2-ethyl-3-methoxy-5-methylpropiophenone. This oil by distillation in vacuo gave no apparent separation [7370].

– Also refer to: [6351,7285].

oil [7370];
b.p.$_1$ 100–102° [7370], b.p.$_{0.2}$ 103–105° [6351], b.p.$_{0.2-0.3}$ 104–105° [7110];
^{1}H NMR (Sadtler: standard n° 49339M), IR (Sadtler: standard n° 76412K).

1-(2,4-Dimethoxy-3-methylphenyl)-1-propanone

[77942-13-3] $C_{12}H_{16}O_3$ mol.wt. 208.26



Syntheses

– Preparation by methylation of 2,4-dihydroxy-3-methyl-propiophenone [6530] with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 48 h (86%) [7111].

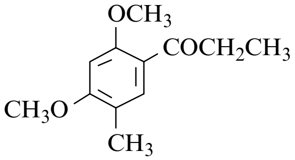
– This dimethyl ether can be obtained by reaction of propionyl chloride with 2,6-dimethoxytoluene, according to the procedure used to prepare the acetyl derivative (92–99%) [7485,7486].

– Also refer to: [7112].

m.p. 40–41° [7111]; ^{1}H NMR [7111], IR [7111], UV [7111], MS [7111].

1-(2,4-Dimethoxy-5-methylphenyl)-1-propanone

[77942-24-6] $C_{12}H_{16}O_3$ mol.wt. 208.26



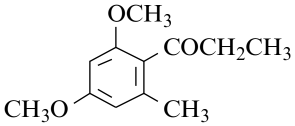
Synthesis

- Preparation by reaction of propionyl chloride with 1,3-dimethoxy-4-methylbenzene in the presence of stannic chloride in carbon disulfide (80%) [7111].

m.p. 72.5–73.5° [7111]; ^{1}H NMR [7111], IR [7111], UV [7111], MS [7111].

1-(2,4-Dimethoxy-6-methylphenyl)-1-propanone

$C_{12}H_{16}O_3$ mol.wt. 208.26



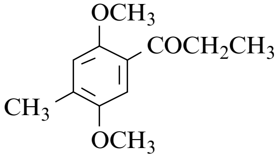
Synthesis

- Obtained by reaction of dimethyl sulfate with 2,4-di-hydroxy-6-methylpropiophenone in the presence of aqueous sodium hydroxide [6744].

m.p. 42–43° [6744].

1-(2,5-Dimethoxy-4-methylphenyl)-1-propanone

[13720-53-1] $C_{12}H_{16}O_3$ mol.wt. 208.26



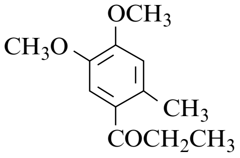
Syntheses

- Preparation by reaction of propionyl chloride with 2,5-dimethoxytoluene in the presence of aluminium chloride in carbon disulfide (75%) [7281].
- Also refer to: [7487].

m.p. 76–77° [7281].

1-(4,5-Dimethoxy-2-methylphenyl)-1-propanone

[3307-02-6] $C_{12}H_{16}O_3$ mol.wt. 208.26



Synthesis

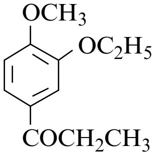
- Preparation by reaction of propionyl chloride with homoveratrole (3,4-dimethoxytoluene) in carbon disulfide in the presence of aluminium chloride (82%) [7488].

b.p.$_5$ 165° [7488]; m.p. 55° [6351], 40° [7488]. One of the reported melting points is obviously wrong.

^{1}H NMR (Sadtler: standard n° 49336M), IR (Sadtler: standard n° 76409K).

1-(3-Ethoxy-4-methoxyphenyl)-1-propanone

[833-53-4] $C_{12}H_{16}O_3$ mol.wt. 208.26



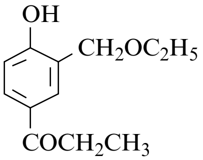
Syntheses

- Obtained by ethylation of 3-hydroxy-4-methoxy-propiophenone formed from guaiacol propionate by Fries reaction [6850].
- Also prepared by oxidation of the secondary alcohol obtained from 3-ethoxy-4-methoxybenzaldehyde and ethylmagnesium iodide [6850].
- Also obtained by hydration of 3-ethoxy-4-methoxy-1-propynylbenzene [7489].

m.p. 66–67° [6850], 65–65.7° [7489].

1-[3-(Ethoxymethyl)-4-hydroxyphenyl]-1-propanone

[136715-22-5] $C_{12}H_{16}O_3$ mol.wt. 208.26

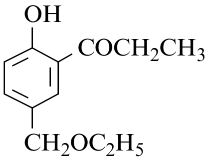


Synthesis

- Preparation [7392] (Czech patent).
- USE: Pharmaceutical intermediate [7392].

1-[5-(Ethoxymethyl)-2-hydroxyphenyl]-1-propanone

[100257-32-7] $C_{12}H_{16}O_3$ mol.wt. 208.26



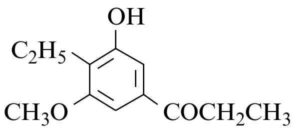
Synthesis

- Obtained by reaction of 95% ethanol with 5-chloromethyl-2-hydroxypropiophenone in the presence of iron and concentrated hydrochloric acid at r.t. for 2 h (56%) [6986].

b.p.$_4$ 145–147° [6986]; m.p. 31.5–34° [6986].

1-(4-Ethyl-3-hydroxy-5-methoxyphenyl)-1-propanone

$C_{12}H_{16}O_3$ mol.wt. 208.26

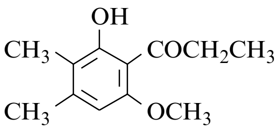


Synthesis

- Refer to: [7434].

1-(2-Hydroxy-6-methoxy-3,4-dimethylphenyl)-1-propanone

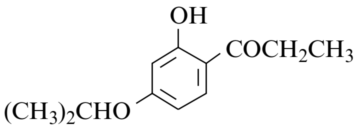
[185207-93-6] $C_{12}H_{16}O_3$ mol.wt. 208.26



Syntheses

- Refer to: [7490,7491].

1-[2-Hydroxy-4-(1-methylethoxy)phenyl]-1-propanone



$C_{12}H_{16}O_3$ mol.wt. 208.26

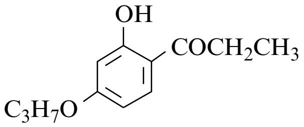
Synthesis

- Refer to: [7492].

Oxime [214398-51-3] $C_{12}H_{17}NO_3$ mol.wt. 223.27.

USE: Spectrophotometric reagent for iron [7492].

1-(2-Hydroxy-4-propoxyphenyl)-1-propanone



$C_{12}H_{16}O_3$ mol.wt. 208.26

Synthesis

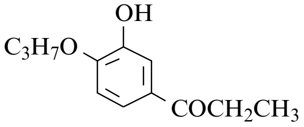
- Refer to: [7493].

Oxime [77697-21-3] $C_{12}H_{17}NO_3$ mol.wt. 223.27.

USE: For spectrophotometric determination of various metals: copper [7493,7494], molybdenum [7495] or vanadium [7496].

1-(3-Hydroxy-4-propoxyphenyl)-1-propanone

[54560-82-6] $C_{12}H_{16}O_3$ mol.wt. 208.26



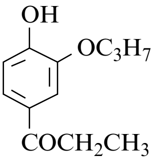
Synthesis

- Obtained (by-product) by reaction of 3,4-methylenedioxy-benzonitrile and ethyl magnesium bromide in toluene for 30 min (4%) [7497].

m.p. 81.5–82.5° [7497]; ^{1}H NMR [7497], IR [7497], UV [7497], MS [7497]; TLC [7497];

1-(4-Hydroxy-3-propoxyphenyl)-1-propanone

[54560-83-7] $C_{12}H_{16}O_3$ mol.wt. 208.26



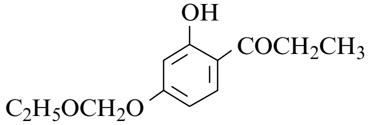
Synthesis

- Obtained (by-product) by reaction of 3,4-methylenedioxy-benzonitrile and ethyl magnesium bromide in toluene for 30 min (3%) [7497].

m.p. 76.5–77° [7497]; ^{1}H NMR [7497], IR [7497], UV [7497], MS [7497]; TLC [7497].

1-[4-(Ethoxymethoxy)-2-hydroxyphenyl]-1-propanone

[124300-17-0] $C_{12}H_{16}O_4$ mol.wt. 224.26



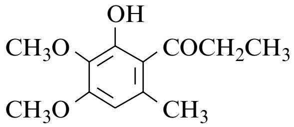
Syntheses

- Obtained by reaction of ethoxymethyl chloride with respropiophenone in the presence of potassium carbonate in acetone at 60° for 3 h (37%) [6757].
- Also obtained by treatment of 4-ethoxymethoxy-2-hydroxy-α-(hydroxymethyl) propiophenone (pale yellow oil) with 4% aqueous sodium carbonate in refluxing ethanol for 1.5 h (36%) [6757].

pale yellow oil [6757]; ^{1}H NMR [6757], IR [6757], UV [6757]; TLC [6757].

1-(2-Hydroxy-3,4-dimethoxy-6-methylphenyl)-1-propanone

[81421-70-7] $C_{12}H_{16}O_4$ mol.wt. 224.26



Syntheses

- Obtained by Friedel–Crafts acylation of 3,4, 5-trimethoxy-toluene with propionyl chloride in the presence of aluminium chloride,
  - in carbon tetrachloride at r.t. for 1.3 h (30%) [7388];
  - in ethyl ether, first at 10° for 1 h, then at r.t. overnight (68%) [7120].

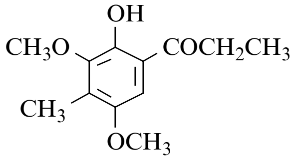
Isolation from natural sources

- From *Juniperus sabina* (Cupressaceae) [7387].
- Obtained by alkaline degradation of *8-Methoxycoumarsabin* with potassium hydroxide in refluxing methanol for 2 h (41%) [7388]. *8-Methoxycoumarsabin* (3,5-dimethyl-4,7,8-tri-methoxycoumarin) (m.p. 125–126°) was isolated from the leaves of *Juniperus sabina* (Cupressaceae).

m.p. 104–106° [7120,7387], 103–104° [7388]; ^{1}H NMR [7120,7387,7388], ^{13}C NMR [7387], IR [7387,7388],
UV [7387,7388].

1-(2-Hydroxy-3,5-dimethoxy-4-methylphenyl)-1-propanone

[383187-35-7] $C_{12}H_{16}O_4$ mol.wt. 224.26



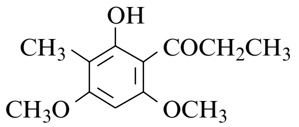
Syntheses

- Preparation by Fries rearrangement of 2,4-dimethoxy-3-methylphenyl propionate in the presence of boron trifluoride dimethyl etherate for 3 h at 90° (74–76%) [7498,7499].

m.p. 78–79° [7499]; ^{1}H NMR [7499], ^{13}C NMR [7499], IR [7499], MS [7499], HRMS [7499].

1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-1-propanone

[69480-07-5] $C_{12}H_{16}O_4$ mol.wt. 224.26



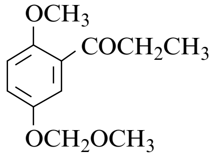
Syntheses

– Refer to: [6895,7500].

m.p. 133° [6895];
GLC [6895], TLC [6895], paper chromatography [6895].

1-[2-Methoxy-5-(methoxymethoxy)phenyl]-1-propanone

[268234-16-8] $C_{12}H_{16}O_4$ mol.wt. 224.26

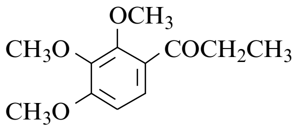


Synthesis

– Refer to: [7501].

1-(2,3,4-Trimethoxyphenyl)-1-propanone

[18060-58-7] $C_{12}H_{16}O_4$ mol.wt. 224.26



Syntheses

– Preparation by reaction of propionic anhydride with pyrogallol trimethyl ether in the presence of PPA at 45–60° for 3 h (82%) [7502].

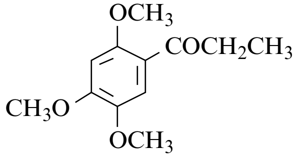
– Obtained by reaction of ethylmagnesium bromide with 2,3,4-trimethoxybenzonitrile (25%) [6973].

– Also refer to: [7343,7433,7434,7503].

b.p.$_{0.2}$ 118–119° [6973], b.p.$_4$ 131–138° [7502];
^{1}H NMR [7502], IR [7502], MS [7503].

1-(2,4,5-Trimethoxyphenyl)-1-propanone *(Isoacoramone)*

[3904-18-5] $C_{12}H_{16}O_4$ mol.wt. 224.26



Syntheses

– Preparation from 1,2,4-trimethoxybenzene by reaction,

- with propionic anhydride catalyzed either by iodine or aluminium chloride [7504], (95%) [7505];
- with propionyl chloride in the presence of aluminium chloride in methylene chloride at 10° for 1 h (80%) [7506] or in carbon disulfide (67%) [7254,7507], (60%) [7281,7401].

- Also obtained by treatment of 2-hydroxy-4,5-dimethoxypropiophenone with dimethyl sulfate in the presence of sodium hydroxide [7401].
- Also obtained by reaction of dimethyl sulfate with 2,5-dihydroxy-4-methoxypropiophenone in the presence of alkali (61%) [7254].
- Also obtained by oxidation of 1-(3,4,5-trimethoxyphenyl)propane with DDQ in wet dioxane/silica gel under sonication (64%) [7093].
- Also obtained by heating dibromoasarone with zinc powder in the presence of potassium hydroxide at reflux some hours [7508].
- Also refer to: [7509].

Isolation from natural sources

- From the root of *Asarum maximum* [7369].
- From *Acorus tatarinowii* (Araceae) [7510].
- From *Piper marginatum* [7511].
- Also refer to: [7280,7433,7434].

b.p.$_{13}$ 186° [7508];
m.p. 108.5–109.5° [7254], 108–110° [7506], 108–109° [7093,7281], 106–108° [7401,7507,7508], 105–106° [7505]; ^{1}H NMR [7093, 7505,7510,7512], ^{13}C NMR [7093,7510],
IR [7505,7510], MS [7505].

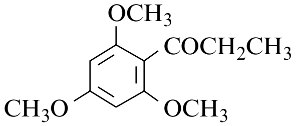
BIOLOGICAL ACTIVITY: Hypolipaemic effect [7504]; toxicity [7504].

Oxime $C_{12}H_{17}NO_4$ mol.wt. 239.27 (m.p. 106–108°) [7507].

Semicarbazone $C_{13}H_{19}N_3O_4$ mol.wt. 281.31 (m.p. 165–167°) [7507].

1-(2,4,6-Trimethoxyphenyl)-1-propanone

[834-94-6] $C_{12}H_{16}O_4$ mol.wt. 224.26



Syntheses

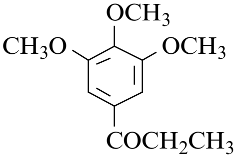
- Obtained by reaction of propionic acid with phloropropiophenone trimethyl ether in the presence of PPA as a condensing agent [7326,7513], (55%) [7514].
- Also obtained by reaction of propionitrile with phloroglucinol trimethyl ether [7515].
- Also obtained from *trans*-2,4,6-trimethoxy-1-propenylbenzene by a Wacker oxidation [7097].
- Also obtained (by-product) by reaction of propionyl chloride with phloroglucinol trimethyl ether in the presence of aluminium chloride in ethyl ether [7405].
- Refer to: [7516–7518].

m.p. 136–137° [7405], 89° [7514], 85° [7515], 84.5–85° [7326], 82–83° [7516]. One of the reported melting points is obviously wrong.
IR [7326].

USE: 3D QSAR study of hypolipidemic asarones by comparative molecular surface analysis [7519].

1-(3,4,5-Trimethoxyphenyl)-1-propanone

[5658-50-4] $C_{12}H_{16}O_4$ mol.wt. 224.26



Syntheses

- Obtained by reaction of 5% ethanolic potassium hydroxide with 2-(3,4,5-trimethoxybenzoyl)-2-methylacetic acid ethyl ester (m.p. 40°) at 60° for 1 h (76%) [7520], (73%) [7521].
- Also obtained by oxidation of ethyl-(3,4,5-trimethoxy-phenyl)carbinol with DDQ in dioxane at 20° for 14 h (89%) [7522].
- Also obtained by reaction of ethylmagnesium bromide with 3,4,5-trimethoxybenzonitrile (56%) [7349] or with 3,4,5-trimethoxybenzamide [6591] or 3,4,5-trimethoxybenzaldehyde (83%) [7523] or still with 3,4,5-trimethoxybenzoyl chloride (87%) [7524].
- Also obtained by reaction of diethylcadmium with 3,4,5-trimethoxybenzoyl chloride [7525].
- Also obtained by reaction of ethyllithium with 3,4,5-trimethoxy-N,N-dimethylbenzamide (51%) [7526].
- Also obtained by bubbling ozone through the warm solution of 3-(3,4,5-trimethoxyphenyl)-pentene(2) in 70% acetic acid for 4 h (45%) [7527], (60%) [7528].
- Also obtained by oxidation of 1-(3,4,5-trimethoxyphenyl)propane with DDQ in wet dioxane/silica gel under sonication (65%) [7093,7512].
- Also obtained by oxidation of 1-(3,4,5-trimethoxyphenyl)-1-propanol with Jones reagent [7529,7530].
- Also refer to: [6828,7340,7343,7411,7531–7536], (74–84%) [7537], (66%) [7538,7539].

Isolation from natural sources

- From *Virola surinamensis* (Myristicaceae) [6523].

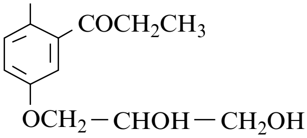
b.p.$_{11}$ 177–178° [7521], b.p.$_{10}$ 181–182° [7525], b.p.$_{40}$ 210° [7527];
m.p. 53.5° [7527], 53–56° [7538], 53–55.5° [7526], 53–54° [7524],
53° [6591,7520], 52–53° [7093,7512,7523],
52° [7349], 51–53° [7529], 51.5–52.5° [7525], 51–52° [7521];
^{1}H NMR [6523,7093,7512,7523,7524,7529],
^{13}C NMR [6523,7093], IR [6523], UV [7213], MS [6523,7529].

USE: Fungicide [7539]; antioxidizing agent [6846].

1-[2-Hydroxy-5-(2,3-dihydroxypropoxy)phenyl]-1-propanone

3-(4-Hydroxy-3-propionylphenoxy)-1,2-propanediol

[956-23-0] $C_{12}H_{16}O_5$ mol.wt. 240.26



Syntheses

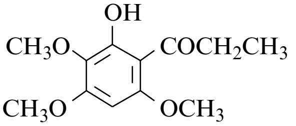
- Preparation by reaction of 3-chloro-1,2-propanediol with 2,5-dihydroxypropiophenone in the presence of sodium ethoxide in ethanol [6465].

- Also refer to: [7540], (83%).

b.p.$_{0.06}$ 165–173° [7540]; m.p. 79.5–81° [6465]; ^{1}H NMR [6465,6472].

1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-propanone

[51379-76-1] $C_{12}H_{16}O_5$ mol.wt. 240.26



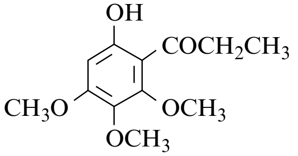
Syntheses

- Preparation by reaction of propionyl chloride with 1,2,3,5-tetramethoxybenzene (m.p. 47°) [7541] in the presence of aluminum chloride in ethyl ether first at 0°, then at r.t. [7542], (56%) [7405], (55%) [7543] or in carbon disulfide at r.t. for 3-4h (33%) [7541]
- Preparation by reaction of propionic anhydride with 1,2,3,5-tetramethoxybenzene in the presence of aluminium chloride [7544] in ethyl ether, first 5 h at 0°, then at r.t. overnight (66%) [7545].
- Preparation by partial demethylation of 2,3,4,6-tetramethoxypropiophenone with aluminium chloride in acetonitrile at 50° for 1–2 h (82%) [7442].
- Also refer to: [7546,7547].

m.p. 132° [7545], 129–130° [7405], 128–129.5° [7543], 128–130° [7442], 124–126° [7541]; ^{1}H NMR [7442,7543], IR [7543]; TLC [7543].

1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-propanone

[52099-20-4] $C_{12}H_{16}O_5$ mol.wt. 240.26



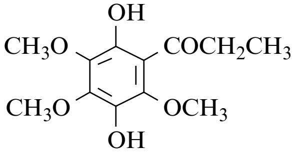
Syntheses

- Preparation by hydrogenolysis of 6-(benzyloxy)-2,3,4-tri-methoxypropiophenone over 10% Pd/C in ethyl acetate-methanol (1:1) (89%) [7442].
- Also obtained by partial methylation of 3,6-dihydroxy-2,4-dimethoxypropiophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing benzene for 12 h [7542], (43%) [7405].
- Also refer to: [7546].

oil [7405]; m.p. 42.5–43.5° [7442]; ^{1}H NMR [7442].

1-(2,5-Dihydroxy-3,4,6-trimethoxyphenyl)-1-propanone

[119232-80-3] $C_{12}H_{16}O_6$ mol.wt. 256.26

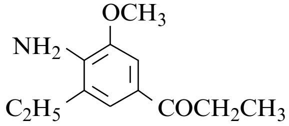


Synthesis

- Preparation by Elbs persulfate oxidation of 2-hydroxy-3,4,6-trimethoxypropiophenone [7548], according to the method [7549].

1-(4-Amino-3-ethyl-5-methoxyphenyl)-1-propanone

[403854-11-5]



$C_{12}H_{17}NO_2$ mol.wt. 207.27

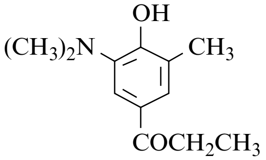
Isolation from natural sources

- From *paradica* and *maghraby banana psedudostem* [7550].

Note: Banana plant juice and its pulping liquor as anti-corrosive materials [7550].

1-[3-(Dimethylamino)-4-hydroxy-5-methylphenyl]-1-propanone

[141771-83-7]

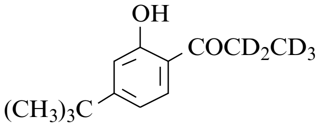


$C_{12}H_{17}NO_2$ mol.wt. 207.27

Synthesis

- Obtained from methylation of 3-amino-4-hydroxy-5-methylpropiophenone by methyl iodide and triethylamine in methanol (64%) [6995].

1-[4-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-propanone-*2,2,3,3,3-d_5*

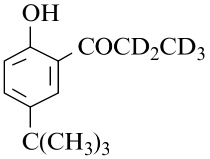


$C_{13}H_{13}D_5O_2$ mol.wt. 211.32

Synthesis

- Refer to: [6350].
  Ion (1^-), radical ion (1^-) [72051-89-9], ESR spectrum [6350].

1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-propanone-*2,2,3,3,3-d_5*



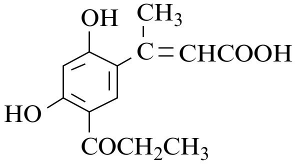
$C_{13}H_{13}D_5O_2$ mol.wt. 211.32

Synthesis

- Refer to: [6350].
  Ion (1^-), radical ion (1^-) [69858-32-8], ESR spectrum [6350]; high-resolution ESR spectrum [7551].

3-[2,4-Dihydroxy-5-(1-oxopropyl)phenyl]-3-methyl-2-propenoic acid *(E)*

2,4-Dihydroxy-5-propionyl-β-methylcinnamic acid



$C_{13}H_{14}O_5$ mol.wt. 250.24

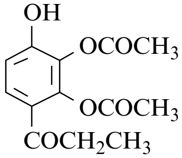
Syntheses

- Obtained (poor yield) by saponification of the residual mixture (m.p. 165–185°) obtained in Fries rearrangement of 4-methylumbelliferone propionate (m.p. 150°), after 8-propionyl and 6-propionyl derivatives separation (4%) [6800].
- Also obtained by hydrolysis of 6-propionyl-4-methyl-umbelliferone (m.p. 228°) with sodium hydroxide [6800].

m.p. 164–166° [6800].

1-[2,3-Bis(acetyloxy)-4-hydroxyphenyl]-1-propanone

[161583-88-6] $C_{13}H_{14}O_6$ mol.wt. 266.25



Syntheses

- Obtained by enzymatic deacetylation of 2,3,4-triacetoxy-propiophenone (m.p. 75–76°) with two lipases in various solvents [7552] (see table below).
- Also refer to: [7553].

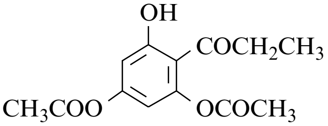
Solvent	With PPL[a] (yield %)	With CCL[b] (yield %)
THF	70	50
Acetone	20	20
DIPE	65	65

N.B.: [a]PPL = porcine pancreas lipase [b]CCL = candida cylindracea lipase

m.p. 68–70° [7552]; ^{1}H NMR [7552], IR [7552], UV [7552], MS [7552]; TLC [7552].

1-[2,4-Bis(acetyloxy)-6-hydroxyphenyl]-1-propanone

[22665-89-0] $C_{13}H_{14}O_6$ mol.wt. 266.25



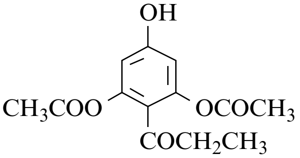
Synthesis

- Obtained (poor yield) by reaction of acetic anhydride with phloropropiophenone in the presence of pyridine at 95° for 2 h (7%) [6629].

m.p. 69–70° [6629];
IR [6629,7554]; TLC [6629].

1-[2,6-Bis(acetyloxy)-4-hydroxyphenyl]-1-propanone

[145747-21-3] $C_{13}H_{14}O_6$ mol.wt. 266.25



Syntheses

- Obtained by enzymatic deacetylation of 2,4,6-triacetoxy-propiophenone (m.p. 56–57°) [6565] with two lipases in various solvents [7552] (see table below).
- Also obtained by regioselective deacetylation of 2,4,6-tri-acetoxypropiophenone with PPL in THF/n-butanol at 42–45° (60%) [7555].
- Also refer to: [7553].

Solvent	With PPL[a] (yield %)	With CCL[b] (yield %)
THF	60	35
Acetone	35	30
DIPE	50	20

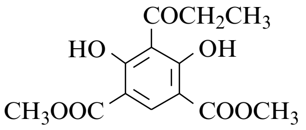
N.B.: [a]PPL = porcine pancreas lipase [b]CCL = candida cylindracea lipase

m.p. 110–114° [7552]; ^{1}H NMR [7552], MS [7552]; TLC [7552].

4,6-Dihydroxy-5-(1-oxopropyl)-1,3-benzenedicarboxylic acid dimethyl ester

4,6-Dihydroxy-5-propionylisophthalic acid dimethyl ester

[13936-94-2]



$C_{13}H_{14}O_7$ mol.wt. 282.25

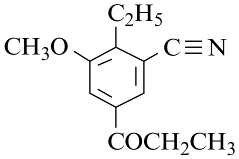
Synthesis

- Obtained by reaction of propionyl chloride with dimethyl resorcinol-4,6-dicarboxylate in the presence of aluminium chloride in nitrobenzene for 3 h, then at r.t. overnight (75%) [6809].

m.p. 112–113° [6809].

2-Ethyl-3-methoxy-5-(1-oxopropyl)benzonitrile

[105838-23-1]



$C_{13}H_{15}NO_2$ mol.wt. 217.20

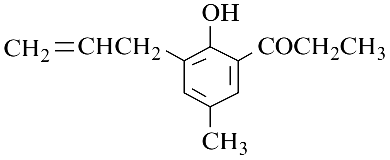
Synthesis

- Preparation by reaction of ethylmagnesium bromide with 4,5-dimethoxyisophthalonitrile (75%) [6973].

m.p. 110° [6973].

1-[2-Hydroxy-5-methyl-3-(2-propenyl)phenyl]-1-propanone

[108293-74-9]



$C_{13}H_{16}O_2$ mol.wt. 204.27

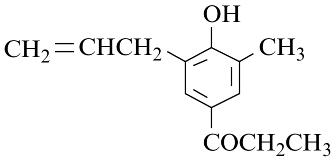
Synthesis

- Obtained by thermal Claisen rearrangement of 2-(allyloxy)-5-methylpropiophenone [7447] at 190° for 20 h in an argon atmosphere (80%) [7556].

m.p. 40–41° [7556]; ^{1}H NMR [7556], MS [7556].

1-[4-Hydroxy-3-methyl-5-(2-propenyl)phenyl]-1-propanone

[5551-29-1]



$C_{13}H_{16}O_2$ mol.wt. 204.27

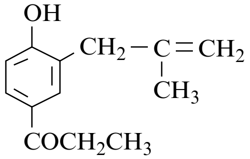
Synthesis

- Preparation by thermal Claisen rearrangement of 4-(allyloxy)-3-methylpropiophenone (b.p.$_{25}$ 185°) in refluxing N,N-diethylaniline for 7 h (95%) [7557].

b.p.$_{25}$ 215° [7557]; m.p. 74° [7557].

1-[4-Hydroxy-3-(2-methyl-2-propenyl)phenyl]-1-propanone

[98017-40-4] $C_{13}H_{16}O_2$ mol.wt. 204.27



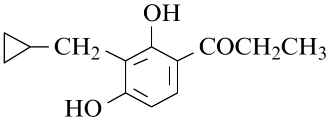
Synthesis

– Obtained by thermal Claisen rearrangement of 4-(β-methallyloxy)propiophenone (m.p. 39°) in boiling dimethylaniline for 5–6 h (70–75%) [7448].

b.p.$_{15}$ 197–198° [7448]; m.p. 75° [7448].

1-[3-(Cyclopropylmethyl)-2,4-dihydroxyphenyl]-1-propanone

[194792-34-2] $C_{13}H_{16}O_3$ mol.wt. 220.27

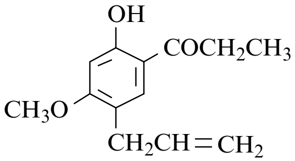


Synthesis

– Refer to: [7452].

1-[2-Hydroxy-4-methoxy-5-(2-propenyl)phenyl]-1-propanone

[869562-75-4] $C_{13}H_{16}O_3$ mol.wt. 220.27

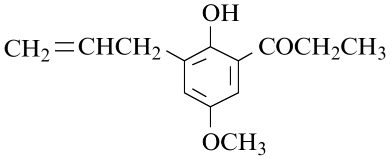


Synthesis

– Refer to: [7558].

1-[2-Hydroxy-5-methoxy-3-(2-propenyl)phenyl]-1-propanone

[102569-09-5] $C_{13}H_{16}O_3$ mol.wt. 220.27

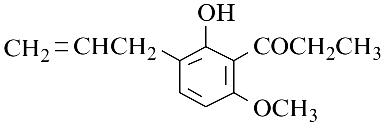


Synthesis

– Refer to: [7559].

1-[2-Hydroxy-6-methoxy-3-(2-propenyl)phenyl]-1-propanone

[17488-54-9] $C_{13}H_{16}O_3$ mol.wt. 220.27



Syntheses

– Preparation by partial methylation of 3-allyl-2,6-di-hydroxypropiophenone with dimethyl sulfate (60%) [7153].

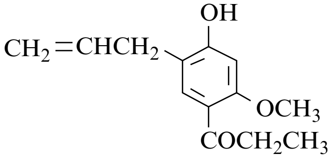
– Also obtained by isomerization of 2-(allyloxy)-6-methoxypropiophenone by distillation at a high pressure and long storage or by heating in a sealed tube for 24 h at 215–220° (Claisen rearrangement) (30%) [7153].

b.p.$_2$ 126–130° [7153];
m.p. 99–101° [7153]; n_D^{20} = 1.5524 [7153].

1-[4-Hydroxy-2-methoxy-5-(2-propenyl)phenyl]-1-propanone

[152719-59-0]

$C_{13}H_{16}O_3$ mol.wt. 220.27



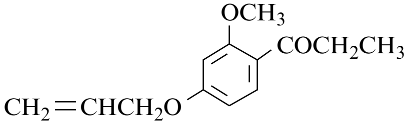
Syntheses

- Preparation by thermal Claisen rearrangement of 2-methoxy-4-(allyloxy)propiophenone (2-O-methyl-4-O-allylrespropiophenone) by heating at 200° for 2 h (80%) [7451].
- Also refer to: [7449].

m.p. 132–133° [7451].

1-[2-Methoxy-4-(2-propenyloxy)phenyl]-1-propanone

$C_{13}H_{16}O_3$ mol.wt. 220.27



Synthesis

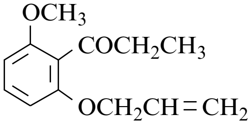
- Obtained by reaction of dimethyl sulfate with 4-(allyloxy)-2-hydroxypropiophenone in the presence of 20% potassium hydroxide in acetone [7451].

m.p. 31° [7451].

1-[2-Methoxy-6-(2-propenyloxy)phenyl]-1-propanone

[17488-72-1]

$C_{13}H_{16}O_3$ mol.wt. 220.27



Synthesis

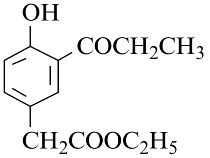
- Preparation by reaction of allyl bromide with 2-hydroxy-6-methoxypropiophenone in acetone in the presence of potassium carbonate (76%) [7153].

b.p.$_1$ 142–144° [7153].

4-Hydroxy-3-(1-oxopropyl)phenylacetic acid ethyl ester

[108994-28-1]

$C_{13}H_{16}O_4$ mol.wt. 236.27



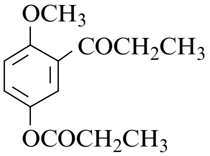
Synthesis

- Preparation by reaction of ethanol with 4-hydroxy-3-(1-oxopropyl)phenylacetonitrile in the presence of hydrogen chloride, first at 0° for 2 h, then at reflux for 2 h (91%) [6942].

m.p. 35–37° [6942].

1-[2-Methoxy-5-(1-oxopropoxy)phenyl]-1-propanone

[80427-25-4] $C_{13}H_{16}O_4$ mol.wt. 236.27



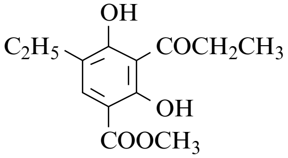
Synthesis

- Obtained by reaction of propionyl chloride with 4-(propionyloxy)anisole in the presence of stannic chloride in nitromethane at 20° for 48 h (56%) [7143].

m.p. 115° [7143]; IR [7143], UV [7143].

5-Ethyl-2,4-dihydroxy-3-(1-oxopropyl)benzoic acid methyl ester

[106214-15-7] $C_{13}H_{16}O_5$ mol.wt. 252.27



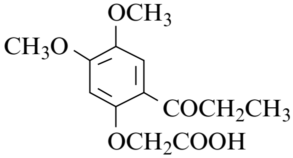
Syntheses

- Obtained by Fries rearrangement of methyl 2,4-di-propionoxy-5-ethylbenzoate (m.p. 89°) with aluminium chloride at 110–120° for 1 h (33%) [6450].
- Also obtained by Friedel–Crafts acylation of methyl 2,4-di-hydroxy-5-ethylbenzoate with propionic anhydride in the presence of aluminium chloride in nitrobenzene, first at 25–30° for 20 h, then at 105° for 4 h [6450].

m.p. 91° [6450].

2-[4,5-Dimethoxy-2-(1-oxopropyl)phenoxy]acetic acid

$C_{13}H_{16}O_6$ mol.wt. 268.27



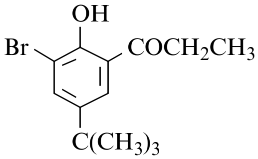
Synthesis

- Obtained by treatment of ethyl α-(2-propionyl-4,5-dimethoxyphenoxy)acetate with 10% aqueous sodium hydroxide on a steam bath, then at r.t. overnight [7400].

m.p. 138–140° [7400].

1-[3-Bromo-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-propanone

[22362-64-7] $C_{13}H_{17}BrO_2$ mol.wt. 285.18



Syntheses

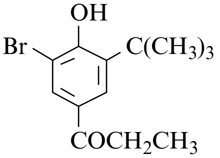
- Preparation by adding a solution of 5-tert-butyl-2-hydroxy-propiophenone in an acetic acid and hydrochloric acid (d = 1.19) mixture to an aqueous solution of 0.2 N potassium bromate and potassium bromide, then stirring 24 h after the addition of ketone (97%) [7560,7561].

- Also obtained by Fries rearrangement of 2-bromo-4-tert-butylphenyl propionate (b.p.$_3$ 128°) [7562] with aluminium chloride without solvent at 110° for 2h (68%) [7562] or at 20° for 8 days (24%) [7560,7561].

b.p.$_3$ 165° [7562]; m.p. 125° [7560,7561];
^{1}H NMR (Sadtler: standard n° 16616M), IR (Sadtler: standard n° 44742) [7560,7561],
UV [6359,7560,7561]; TLC [6353].

1-[3-Bromo-5-(1,1-dimethylethyl)-4-hydroxyphenyl]-1-propanone

[20683-42-5] $C_{13}H_{17}BrO_2$ mol.wt. 285.18



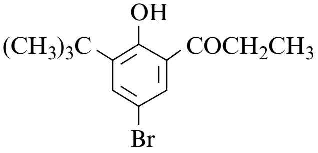
Syntheses

- Preparation by reaction of potassium bromate and potassium bromide on the 3-tert-butyl-4-hydroxypropiophenone according to the procedure [6352], (97%) [7560,7561].

m.p. 70° [7560,7561];
^{1}H NMR (Sadtler: standard n° 16615M),
IR (Sadtler: standard n° 44736) [7560,7561],
UV [6357,7560,7561]; TLC [6353].

1-[5-Bromo-3-(1,1-dimethylethyl)-2-hydroyphenyl]-1-propanone

[35154-19-9] $C_{13}H_{17}BrO_2$ mol.wt. 285.18



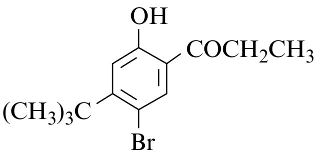
Syntheses

- Preparation by reaction of potassium bromate and potassium bromide on the 3-tert-butyl-2-hydroxy-propiophenone according to the procedure [6352], (93%) [7560,7561].

m.p. 96° [7560,7561];
^{1}H NMR (Sadtler: standard n° 16613M), IR (Sadtler: standard n° 44734) [7560,7561],
UV [7560,7561]; TLC [6353].

1-[5-Bromo-4-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-propanone

[35154-28-0] $C_{13}H_{17}BrO_2$ mol.wt. 285.18



Syntheses

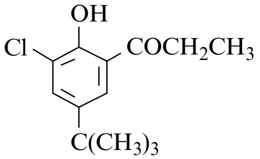
- Preparation by reaction of potassium bromate and potassium bromide on the 4-tert-butyl-2-hydroxy-propiophenone according to the procedure [6352], (82%) [7560,7561].

– Also obtained by Fries rearrangement of 4-bromo-3-tert-butylphenyl propionate [6351].

m.p. 47° [7560,7561]; ^{1}H NMR (Sadtler: standard n° 18462M) [7560,7561], IR (Sadtler: standard n° 44739) [7560,7561], UV [7560,7561]; TLC [6353].

1-[3-Chloro-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-propanone

[51233-86-4] $C_{13}H_{17}ClO_2$ mol.wt. 240.73



Syntheses

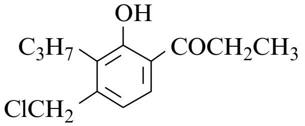
– Preparation by Fries rearrangement of 2-chloro-4-tert-butylphenyl propionate [6351] (b.p.$_{10}$ 129°) [7563],

- with aluminium chloride (1.5 mol) at 110° (80%) [7563];
- with titanium tetrachloride (1.5 mol) at 140° for 15 min (28%) [6466].

b.p.$_{10}$ 142° [7563]; m.p. 113° [6351,6466];
^{1}H NMR (Sadtler: standard n° 20028M), IR (Sadtler: standard n° 47037) [6466],
UV [6466].

1-[4-(Chloromethyl)-2-hydroxy-3-propylphenyl]-1-propanone

[107223-72-3] $C_{13}H_{17}ClO_2$ mol.wt. 240.73



Syntheses

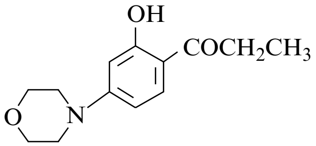
– Obtained by reaction of ethyl chloroformate with 1-[4-[(dimethylamino)methyl]-2-hydroxy-3-propylphenyl]-1-propanone in toluene, first in an ice-water bath for 2 h, then at r.t. for 16 h (75%) [7564].

– Also refer to: [7565].

m.p. 41–44° [7564].

1-[2-Hydroxy-4-(4-morpholinyl)phenyl]-1-propanone

[404009-42-3] $C_{13}H_{17}NO_3$ mol.wt. 235.28

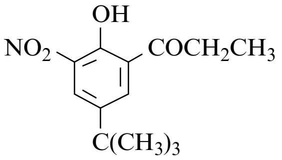


Synthesis

– Refer to: [7566].

1-[5-(1,1-Dimethylethyl)-2-hydroxy-3-nitrophenyl]-1-propanone

[85052-32-0] $C_{13}H_{17}NO_4$ mol.wt. 251.28

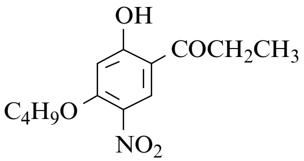


Synthesis

– Refer to: [7567].

1-(4-Butoxy-2-hydroxy-5-nitrophenyl)-1-propanone

[182184-03-8] $C_{13}H_{17}NO_5$ mol.wt. 267.28



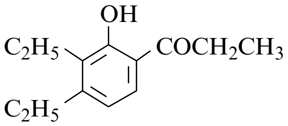
Syntheses

– Refer to: [7568,7569].

Note: Complex with Fe (III) [7569].

1-(3,4-Diethyl-2-hydroxyphenyl)-1-propanone

[936642-85-2] $C_{13}H_{18}O_2$ mol.wt. 206.28



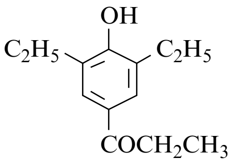
Synthesis

– Obtained by reaction of 2,3-diethylcyclobutenone with ethyl vinyl ketone in the presence of a catalytic amount of $[RhCl(C_2H_4)_2]_2$ and P(*cyclo*-$C_6H_{11})_3$ in toluene at 130° for 12 h under an argon atmosphere (73%) [7570].

Yellow liquid [7570]; b.p.$_{0.1}$ 120–130° [7570];
^{1}H NMR [7570], ^{13}C NMR [7570], IR [7570], MS [7570]; GLC [7570].

1-(3,5-Diethyl-4-hydroxyphenyl)-1-propanone

[104008-46-0] $C_{13}H_{18}O_2$ mol.wt. 206.28

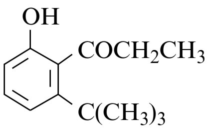


Synthesis

– Refer to: [7308] (Japanese patent).

1-[2-(1,1-Dimethylethyl)-6-hydroxyphenyl]-1-propanone

[86690-30-4] $C_{13}H_{18}O_2$ mol.wt. 206.28

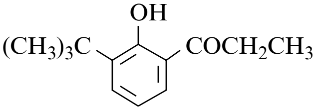


Synthesis

– Obtained from 2-allyl-3-tert-butylphenol by treatment with perbenzoic acid in ethyl ether, first at 0°, then between 0° and 25° for 24 h (68%) [6589].

1-[3-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-propanone

[25441-52-5] $C_{13}H_{18}O_2$ mol.wt. 206.28



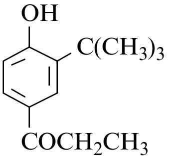
Syntheses

- Obtained by Fries rearrangement of 2-tert-butylphenyl propionate,
  - with titanium tetrachloride in nitromethane or nitrobenzene at 20° for 24 h (15–17%) [7561] or without solvent at 100° for 2 h (21%) [7560];
  - with niobium pentachloride in nitromethane at 20° for 6 h (26%) [7561];
  - with tantalum pentachloride in nitromethane at 20° for 2 h (15%) [7561].
- Also refer to: [6586,7571,7572].

b.p.$_{13}$ 143° [7560,7561]; m.p. 36° [7560,7561];
^{1}H NMR (Sadtler: standard n° 18459M), IR (Sadtler: standard n° 44733) [7560,7561],
UV [7560,7561]; TLC [6353,7560,7561].

1-[3-(1,1-Dimethylethyl)-4-hydroxyphenyl]-1-propanone

[20683-32-3] $C_{13}H_{18}O_2$ mol.wt. 206.28



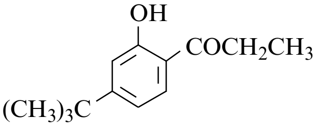
Syntheses

- Preparation by Fries rearrangement of 2-tert-butylphenyl propionate (1 mol) with titanium tetrachloride (2 mol) in nitromethane at 20° for 24 h (72–81%) [6586,7560,7561].
- Also refer to: [7571,7572].

m.p. 159° [7560,7561];
^{1}H NMR (Sadtler: standard n° 16614M), IR (Sadtler: standard n° 44735) [7560,7561],
UV [6357,7560,7561]; TLC [6353].

1-[4-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-propanone

[22362-62-5] $C_{13}H_{18}O_2$ mol.wt. 206.28



Syntheses

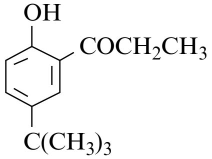
- Preparation by Fries rearrangement of 3-tert-butylphenyl propionate in nitromethane at 20° for 24 h,
  - with titanium tetrachloride (91%) [7560,7561];
  - with aluminium chloride (66%) [7560,7561].

N.B.: Ion (1$^-$), radical ion (1$^-$) [72051-88-8], ESR spectrum [6350].

b.p.$_{11}$ 149–150° [7560,7561]; m.p. 48–49° [7560,7561];
^{1}H NMR (Sadtler: standard n° 18461M), IR (Sadtler: standard n° 44738) [7560,7561],
UV [6359,7560,7561]; TLC [6353].

1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-propanone

[22362-63-6] $C_{13}H_{18}O_2$ mol.wt. 206.28



Syntheses

- Obtained by Fries rearrangement of various propionic esters and experimental conditions [7561],

  - from 4-tert-butylphenyl propionate *(classic reaction)*,

- in nitrobenzene at 50° for 5–6 h with titanium tetrachloride (93%) [7106] or aluminium chloride (50%) [7560],
- in nitromethane at 20° for 192 h with titanium tetrachloride (78%) [7560], stannic chloride (70%) [6570,7560], aluminium chloride (60%) [6466] and ferric chloride (45%) [7560];
- without solvent in the presence of aluminium chloride at 120° for 1 h and at 140° or 180° for 15 min (41–43%) [6466,7573];

  - from 2-tert-butylphenyl propionate *(reaction with intermolecular migration of tert-butyl group)*. Cf. [7561,7571,7572,7574–7577];

- in nitromethane with aluminium chloride at 20° for 72 h (92%) [6570] or 168–192 h (45–60%) [6570,6586,7560],
- in nitromethane with other catalysts at 20° for 168–192 h: Gallium chloride (73–74%) [6570], zirconium chloride (63–71%) [6570,6586,7560] and ferric chloride or stannic chloride (49–53%) [6570,6586,7560];

  - from di-tert-butyl or tri-tert-butylphenyl propionates in nitromethane at 20° using various catalysts *(reactions with migration of an o-tert-butyl group and sometimes elimination of these)*.

Esters	Catalysts	Reaction times (h)	Yield (%)	Reference
2,4-Di-tert-butylphenyl propionate	$SnCl_4$	144	59	[6570,7712]
	$AlCl_3$	168	54	[6570]
2,6-Di-tert-butylphenyl propionate[a]	$AlCl_3$	240	50	[7712]
2,4,6-Tri-tert-butylphenyl propionate[b]	$SbCl_5$	2	16	[7712]

[a] Cf. Fries rearrangement of 2-tert-butylphenyl propionate.

[b] formation of a very important tar.

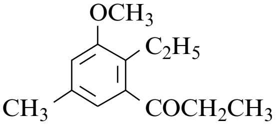
- Also obtained by Fries rearrangement of phenyl propionate with aluminium chloride in nitromethane in the presence of tert-butyl chloride at 20° for 24 h (63%) [6570]. *(There are simultaneously acylation and alkylation of the aromatic ring).*
- Also obtained by Friedel–Crafts acylation of p-tert-butylphenol with propionyl chloride in nitromethane in the presence of aluminium chloride at 20° for 120 h (66%) [6466].

- Also obtained by demethylation of 5-tert-butyl-2-methoxypropiophenone in the presence of a 47% hydrobromic acid/57% hydriodic acid mixture in refluxing acetic acid (70%) [7106].
- Also obtained by alkylation of o-hydroxypropiophenone with tert-butyl chloride in nitromethane in the presence of aluminium chloride at 20° for 40 h (79%) [6570].
- Also obtained from 3,5-di-tert-butyl-2-hydroxypropiophenone after total elimination of the tert-butyl group in $3'$ position by action of aluminium chloride in nitromethane at 20° for 72 h (98%) [6570].

b.p.$_6$ 119–122° [7106], b.p.$_{12}$ 145–146° [7560], b.p.$_{26}$ 165° [7573];
m.p. 31° [7560];
^{1}H NMR (Sadtler: standard n° 18464M), IR (Sadtler: standard n° 44741) [7560]; UV [6359,7560]; high-resolution ESR spectrum [7551]; TLC [6353].

Note: Ion (1^-), radical ion (1^-) [69858-31-7], ESR spectrum [6350].

1-(2-Ethyl-3-methoxy-5-methylphenyl)-1-propanone



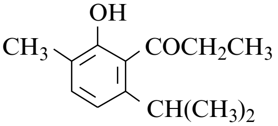
$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

- Obtained by reaction of 2,3-dimethoxy-5-methylbenzonitrile and ethylmagnesium bromide [7370]. **N.B.**: Analysis showed that the oil had a composition corresponding to a mixture of about equal parts of 2,3-dimethoxy-5-methyl-propiophenone and 2-ethyl-3-methoxy-5-methylpropiophenone. This oil by distillation in vacuo gave no apparent separation [7370].

oil [7370]; b.p.$_1$ 100–102° [7370].

1-[2-Hydroxy-3-methyl-6-(1-methylethyl)phenyl]-1-propanone



$C_{13}H_{18}O_2$ mol.wt. 206.28

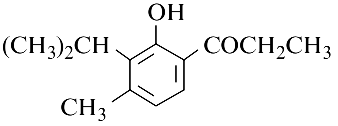
Synthesis

- Obtained by Fries rearrangement of carvacryl propionate (b.p.$_3$ 121°) with aluminium chloride at 120° (71%) [7578], Cf. (*11i*) [6407].

b.p.$_6$ 164° [7578].

1-[2-Hydroxy-4-methyl-3-(1-methylethyl)phenyl]-1-propanone

[129375-02-6]



$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

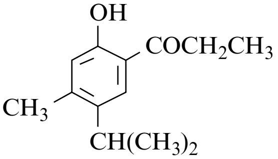
- Preparation by Fries rearrangement of 3-methyl-2-iso-propylphenyl propionate with aluminium chloride in nitromethane at 20° for 170 h (98%) [7026].

b.p.$_{11}$ 143–144° [7026];

m.p. 47–48° [7026];
^{1}H NMR (Sadtler: standard n° 52702M), [7026],
IR (Sadtler: standard n° 79759K) [7026], UV [7026], MS [7026].

1-[2-Hydroxy-4-methyl-5-(1-methylethyl)phenyl]-1-propanone

[121194-62-5] $C_{13}H_{18}O_2$ mol.wt. 206.28



Syntheses

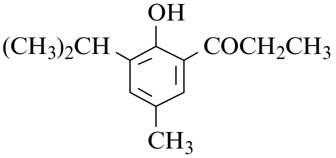
- Preparation by Fries rearrangement of 3-methyl-4-isopropyl-phenyl propionate with titanium tetrachloride or aluminium chloride in nitromethane at r.t. for 100 h (97%) [7031].
- Also obtained by Fries rearrangement of various esters with aluminium chloride (1.4 equiv) without solvent at 100° for 2 h [7026]:

Esters	Yields (%)
3-Methyl-4-isopropylphenyl propionate	44
3-Methyl-2-isopropylphenyl propionate	17
5-Methyl-2-isopropylphenyl propionate	14

b.p.$_{17}$ 161–162° [7031];
m.p. 35° [7031];
^{1}H NMR (Sadtler: standard n° 52704M) [7031], IR (Sadtler: standard n° 79761K) [7031],
UV [7031], MS [7031].

1-[2-Hydroxy-5-methyl-3-(1-methylethyl)phenyl]-1-propanone

[129375-04-8] $C_{13}H_{18}O_2$ mol.wt. 206.28



Syntheses

- Preparation by Fries rearrangement of 2-isopropyl-4-methylphenyl propionate with titanium tetrachloride at 100° for 2 h (95%) [7026].
- Also obtained by treatment of 2-hydroxy-3-isopropyl-6-methylpropiophenone with aluminium chloride (1.4 equiv) without solvent at 100° for 2 h (28%) [7026].
- Also obtained (by-product) by Fries rearrangement of two esters with aluminium chloride (1.4 equiv) without solvent at 100° for 2 h [7026]:

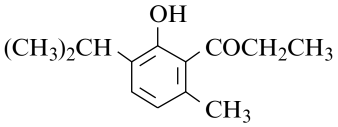
Esters	Ketone (%)
2-Isopropyl-5-methylphenyl propionate	16
4-Isopropyl-3-methylphenyl propionate	8

- Also refer to: [7031].

b.p.$_{15}$ 148–149° [7026];
m.p. 28–29° [7026];
^{1}H NMR (Sadtler: standard n° 52741M) [7026],
IR (Sadtler: standard n° 79800K) [7026], UV [7026], MS [7026].

1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-propanone

[121194-61-4] $C_{13}H_{18}O_2$ mol.wt. 206.28



Syntheses

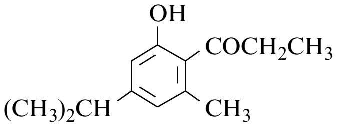
- Preparation by Fries rearrangement of thymyl propionate (b.p.$_{19}$ 180°) [7579] with aluminium chloride [7580], at 120° (83%) [7579] Cf. (*13i*) [6407] or titanium tetrachloride at 120° for 1 h (79%) [7031].
- Also obtained by Friedel–Crafts acylation of thymol with propionic acid in the presence of aluminium chloride at reflux for 12 h (82%) [7581].

N.B.: Two reported boiling points (117–118°/11 mbar [7581] and 161°/6 mbar [7579]) are obviously erroneous, since they correspond to a mixture of products, not containing the expected compound.

b.p.$_{11}$ 117–118° [7580,7581], b.p.$_{14}$ 156–157° [7031], b.p.$_6$ 161° [7579];
m.p. 39° [7031]; ^{1}H NMR (Sadtler: standard n° 52706M) [7031],
IR (Sadtler: standard n° 79763K) [7031], UV [7031], MS [7031].

1-[2-Hydroxy-6-methyl-4-(1-methylethyl)phenyl]-1-propanone

[121194-63-6] $C_{13}H_{18}O_2$ mol.wt. 206.28



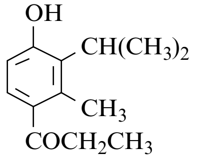
Syntheses

- Preparation by Fries rearrangement of 3-isopropyl-5-methylphenyl propionate,
  - with titanium tetrachloride at 120° for 1 h (94%) [7031];
  - with aluminium chloride at 120° for 1 h (94%) [7031] or at 100° for 2 h (76%) [7026].
- Also obtained by Fries rearrangement of 2-isopropyl-3-methylphenyl propionate with aluminium chloride (1.4 equiv) at 100° for 2 h (53%) [7026].

b.p.$_{14}$ 164° [7031]; m.p. 23° [7031];
^{1}H NMR (Sadtler: standard n° 52705M) [7031], IR (Sadtler: standard n° 79762K) [7031],
UV [7031], MS [7031].

1-[4-Hydroxy-2-methyl-3-(1-methylethyl)phenyl]-1-propanone

[129375-03-7] $C_{13}H_{18}O_2$ mol.wt. 206.28



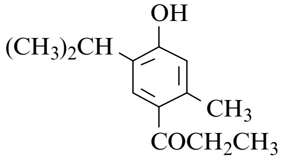
Synthesis

- Obtained (poor yield) by Fries rearrangement of 3-methyl-2-isopropylphenyl propionate with aluminium chloride in nitromethane at 20° for 170 h (1%) [7026].

m.p. 91° [7026];
^{1}H NMR (Sadtler: standard n° 52740M) [7026],
IR (Sadtler: standard n° 79799K) [7026], UV [7026], MS [7026].

1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-propanone

[37847-36-2] $C_{13}H_{18}O_2$ mol.wt. 206.28



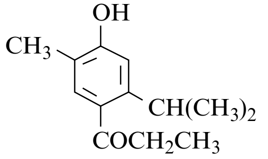
Syntheses

- Preparation by Fries rearrangement of 2-isopropyl-5-methylphenyl propionate (thymyl propionate) (b.p.$_{13}$ 133.5–134.5°) [7302] with aluminium chloride,
  - in nitrobenzene, first in an ice bath, then at r.t. for 24 h [7582], (90%) [6961], (85%) [7302];
  - without solvent at 100° for 2 h (35%) [7026].
- Also obtained by demethylation of 4-methoxy-2-methyl-5-isopropylpropiophenone (b.p.$_{15}$ 169–171°) with boiling pyridinium chloride for 37 min (60%) [7583].
- Also obtained by reaction of propionyl chloride with thymol in nitrobenzene in the presence of aluminium chloride [7470].
- Also obtained (by-product) by Fries rearrangement of 3-methyl-4-isopropylphenyl propionate with aluminium chloride (1.4 equiv) without solvent at 100° for 2 h (17%) [7026].
- Also refer to: [7031,7584].

b.p.$_{13}$ 195–200° [7583], b.p.$_{13}$ 196–200° [7585], b.p.$_{15}$ 200° [7470];
m.p. 113° [6351,7302], 112° [6961,7470], 111° [7583,7585];
^{1}H NMR (Sadtler: standard n° 52707M), IR (Sadtler: standard n° 79764K).

1-[4-Hydroxy-5-methyl-2-(1-methylethyl)phenyl]-1-propanone

$C_{13}H_{18}O_2$ mol.wt. 206.28



Syntheses

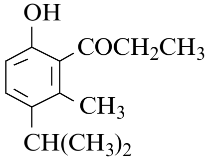
- Preparation by Fries rearrangement of 2-methyl-5-isopropylphenyl propionate (carvacryl propionate) (b.p.$_{12.5}$ 135–136.5°) with aluminium chloride in nitrobenzene, first in an ice bath, then at r.t. or at 30° for 24 h (86–87%) [6961,7302].

- Also obtained by reaction of propionyl chloride with carvacrol in the presence of aluminium chloride in nitrobenzene, first at <50°, then at r.t. for 24 h (quantitative yield) [7586] or simply at r.t. for 24 h (12%) [7587].
- Also refer to: [7067].

b.p.$_{15}$ 201° [7586];
m.p. 110° [7302,7586], 76° [6961,7587]. One of the reported melting points is obviously wrong.

1-[6-Hydroxy-2-methyl-3-(1-methylethyl)phenyl]-1-propanone

[121194-67-0] $C_{13}H_{18}O_2$ mol.wt. 206.28



Synthesis

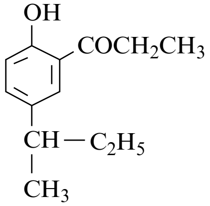
- Preparation by treatment of 3-tert-butyl-2-hydroxy-6-methyl-5-isopropylpropiophenone with aluminium chloride in nitromethane at 20° for 24 h (99%) [7031].

N.B.: The structure of this ketone previously reported in the literature [7588] (150°/15 mbar) is wrong. The procedure described by these authors leads to 2-hydroxy-4-methyl-5-isopropylpropiophenone instead of the titled ketone.

m.p. 70° [7031];
^{1}H NMR (Sadtler: standard n° 52703M) [7031], IR (Sadtler: standard n° 79760K) [7031],
UV [7031], MS [7031].

1-[2-Hydroxy-5-(1-methylpropyl)phenyl]-1-propanone

[131867-27-1] $C_{13}H_{18}O_2$ mol.wt. 206.28



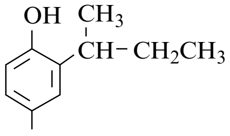
Synthesis

- Preparation by reaction of propionic acid with p-sec-butylphenol in the presence of boron trifluoride (71%) [6509].

b.p.$_6$ 134–137° [6509];
n_D^{25} = 1.5251 [6509].

1-[4-Hydroxy-3-(1-methylpropyl)phenyl]-1-propanone

[16648-74-1] $C_{13}H_{18}O_2$ mol.wt. 206.28



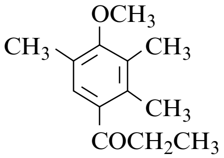
Synthesis

- Preparation by Fries rearrangement of 2-sec-butylphenyl propionate with aluminium chloride in nitrobenzene at 50° for 24 h [7589].

m.p. 110–112° [7589].
USE: Fungicide [7590].

1-(4-Methoxy-2,3,5-trimethylphenyl)-1-propanone

[107076-00-6] $C_{13}H_{18}O_2$ mol.wt. 206.28

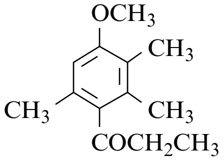


Synthesis

- Refer to: [7292].

1-(4-Methoxy-2,3,6-trimethylphenyl)-1-propanone

[107076-01-7] $C_{13}H_{18}O_2$ mol.wt. 206.28



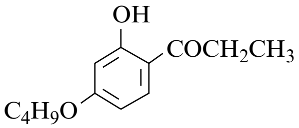
Syntheses

- Preparation from 2,3,5-trimethylanisole and propionyl chloride [7591] according to the procedures [7592,7593].
- Also refer to: [7292].

m.p. 56–57° [7591]; ^{1}H NMR [7591], IR [7591], MS [7591].

1-(4-Butoxy-2-hydroxyphenyl)-1-propanone

[178693-81-7] $C_{13}H_{18}O_3$ mol.wt. 222.28



Syntheses

- Refer to: [7568,7569,7594,7595].

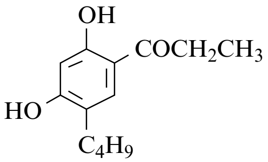
USE: As indicator for determination of Fe (III) [7569,7595].

Oxime [161140-14-3] $C_{13}H_{19}NO_3$ mol.wt. 237.30.

USE: Analytical reagent for Ni (II) [7594,7596–7598], Cu (II) [7594,7599] and Mo (VI) [7600].

1-(5-Butyl-2,4-dihydroxyphenyl)-1-propanone

$C_{13}H_{18}O_3$ mol.wt. 222.28



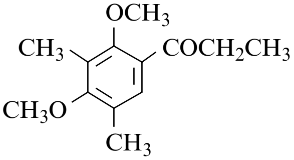
Synthesis

- Preparation by reaction of propionitrile with 4-butyl-resorcinol (Hoesch reaction) (79%) [7115].

m.p. 62–63° [7115].

1-(2,4-Dimethoxy-3,5-dimethylphenyl)-1-propanone

[20935-63-1] $C_{13}H_{18}O_3$ mol.wt. 222.28



Syntheses

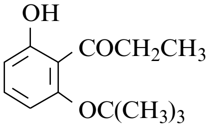
– Preparation by reaction of propionyl chloride with 1,3-dimethoxy-2,4-dimethylbenzene in the presence of,

- stannic chloride in carbon disulfide (87%) [7111];
- aluminium chloride in carbon disulfide (80%) [7601].

oil [7111]; b.p.$_{0.5}$ 120° [7111], b.p.$_{12}$ 155–158° [7601];
^{1}H NMR [7111], IR [7111,7601], UV [7111,7601],
MS [7111].

1-[2-(1,1-Dimethylethoxy)-6-hydroxyphenyl]-1-propanone

[868731-80-0] $C_{13}H_{18}O_3$ mol.wt. 222.28

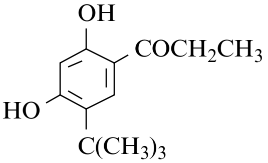


Synthesis

– Refer to: [6816].

1-[5-(1,1-Dimethylethyl)-2,4-dihydroxyphenyl]-1-propanone

[95102-26-4] $C_{13}H_{18}O_3$ mol.wt. 222.28



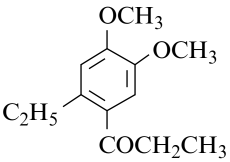
Syntheses

– Preparation from various methods [7602,7603] (Japanese patents),

- by Fries rearrangement of 4-tert-butylresorcinol propionate with aluminium chloride in nitrobenzene;
- by reaction of propionitrile with 4-tert-butylresorcinol (Hoesch reaction);
- by alkylation of respropiophenone with tert-butyl chloride.

1-(2-Ethyl-4,5-dimethoxyphenyl)-1-propanone

[92156-94-0] $C_{13}H_{18}O_3$ mol.wt. 222.28



Syntheses

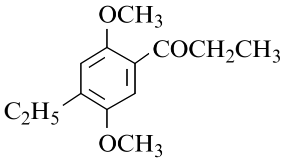
– Preparation by reaction of 1,2-dimethoxy-4-ethylbenzene,

- with propionyl chloride in the presence of aluminium chloride in methylene chloride, first between −5° and 0°, then at r.t. for 4 h (78%) [7604];
- with propionic anhydride in the presence of iodine (66%) [7381].

b.p.$_2$ 95–101° [7381]; m.p. 43–44° [7604]; ^{1}H NMR [7381,7604], IR [7381,7604].

1-(4-Ethyl-2,5-dimethoxyphenyl)-1-propanone

[153756-50-4] $C_{13}H_{18}O_3$ mol.wt. 222.28

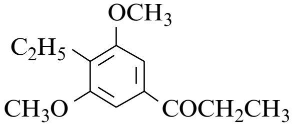


Synthesis

– Obtained by acylation of 2-ethyl-1,4-dimethoxybenzene with propionic acid in the presence of trifluoroacetic anhydride [7605].

1-(4-Ethyl-3,5-dimethoxyphenyl)-1-propanone

[184963-81-3] $C_{13}H_{18}O_3$ mol.wt. 222.28



Syntheses

– Obtained by reaction of ethylmagnesium bromide with 3,4,5-trimethoxybenzonitrile (20%) [6973].

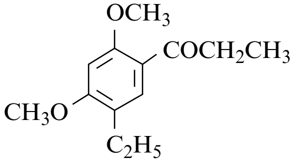
– Also refer to: [7433,7434].

m.p. 72° [6973].

Oxime $C_{13}H_{19}NO_3$ mol.wt. 237.3 (m.p. 94–95°) [6973].

1-(5-Ethyl-2,4-dimethoxyphenyl)-1-propanone

[158153-03-8] $C_{13}H_{18}O_3$ mol.wt. 222.28

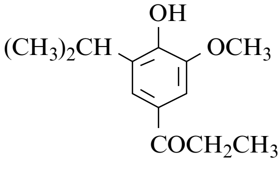


Synthesis

– Refer to: [7384].

1-[4-Hydroxy-3-methoxy-5-(1-methylethyl)phenyl]-1-propanone

[83569-68-0] $C_{13}H_{18}O_3$ mol.wt. 222.28



Syntheses

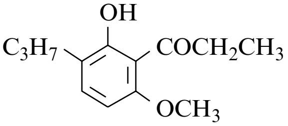
– Preparation by reaction of propionic anhydride with 1,2-dimethoxy-3-isopropylbenzene (b.p.$_3$ 80–84°) in the presence of aluminium chloride in methylene chloride under nitrogen, first at 0° then at r.t. for 4 h (83%) [7606].

- Preparation by reaction of propionic acid with 6-isopropyl-guaiacol in the presence of boron trifluoride for 90 min at 70° (60%) [7607].
- Preparation by reaction of dimethyl sulfate with 3,4-dihydroxy-5-isopropylpropiophenone in the presence of potassium carbonate in acetone at r.t. for 2 h (95%) [7606].

m.p. 87.5–88° [7606], 87–88° [7607]; ^{1}H NMR [7606], IR [7606].

1-(2-Hydroxy-6-methoxy-3-propylphenyl)-1-propanone

[17488-55-0] $C_{13}H_{18}O_3$ mol.wt. 222.28



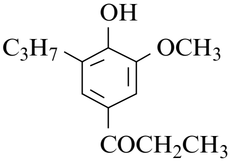
Synthesis

- Preparation by hydrogenation of 3-allyl-2-hydroxy-6-methoxypropiophenone over Pd/C (80%) [7153].

m.p. 77–78° [7153].

1-(4-Hydroxy-3-methoxy-5-propylphenyl)-1-propanone

$C_{13}H_{18}O_3$ mol.wt. 222.28



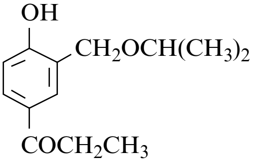
Synthesis

- Preparation by reaction of propionic acid with 6-propyl-guaiacol in the presence of boron trifluoride for 90 min at 70° (70%) [7607].

m.p. 85–86° [7607].

1-[4-Hydroxy-3-[(1-methylethoxy)methyl]phenyl]-1-propanone

[136715-23-6] $C_{13}H_{18}O_3$ mol.wt. 222.28

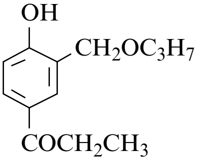


Synthesis

- Refer to: [7392].

1-[4-Hydroxy-3-(propoxymethyl)phenyl]-1-propanone

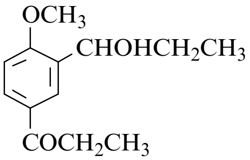
[114477-32-6] $C_{13}H_{18}O_3$ mol.wt. 222.28



Syntheses

- Obtained by reaction of 1-propanol with 3-(chloromethyl)-4-hydroxypropiophenone in the presence of sodium hydrogenocarbonate [6982].
- Also refer to: [7392,7608–7610].

1-[3-(1-Hydroxypropyl)-4-methoxyphenyl]-1-propanone



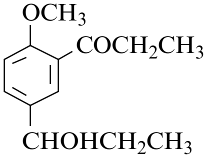
$C_{13}H_{18}O_3$ mol.wt. 222.28

Syntheses

- Obtained by treatment of 2,4-dipropionylanisole with sodium borohydride in refluxing mixture of nitromethane/water (8v/1v) for 2 h (31%) [6733,7611].

m.p. 80° [6733,7611];
^{1}H NMR [6733,7611], IR [6733,7611].

1-[5-(1-Hydroxypropyl)-2-methoxyphenyl]-1-propanone



$C_{13}H_{18}O_3$ mol.wt. 222.28

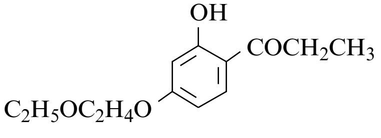
Syntheses

- Obtained by treatment of 2,4-dipropionylanisole with sodium borohydride in refluxing mixture of nitromethane/water (8v/1v) for 2 h (22%) [6733,7611].

colourless oil [6733,7611];
^{1}H NMR [6733,7611], IR [6733,7611].

1-[4-(2-Ethoxyethoxy)-2-hydroxyphenyl]-1-propanone

[63411-93-8]



$C_{13}H_{18}O_4$ mol.wt. 238.28

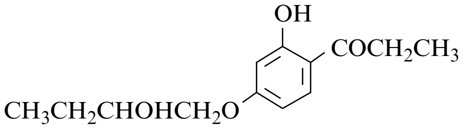
Synthesis

- Preparation by reaction of a 2-ethoxyethyl halide (unspecified) with res-propiophenone in the presence of potassium carbonate in refluxing acetone for 5 h (74%) [6380].

m.p. 45–46° [6380] (purified by vacuum sublimation);
^{1}H NMR [6380], IR [6380]; TLC [6380].

1-[2-Hydroxy-4-(2-hydroxybutoxy)phenyl]-1-propanone

[149454-58-0]



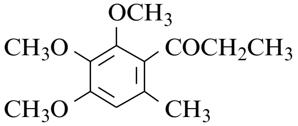
$C_{13}H_{18}O_4$ mol.wt. 238.28

Synthesis

- Obtained by reaction of res-propiophenone with 1,2-butylene oxide in the presence of ethanolic sodium hydroxide [7612].

1-(2,3,4-Trimethoxy-6-methylphenyl)-1-propanone

[81421-71-8] $C_{13}H_{18}O_4$ mol.wt. 238.28

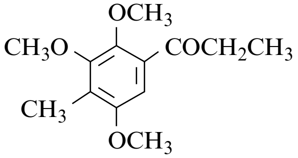


Synthesis

– Obtained by reaction of propionyl chloride with 3,4,5-tri-methoxytoluene (m.p. 35–36°) in the presence of aluminium chloride in carbon tetrachloride at r.t. (55%) (impure ketone) [7388].

1-(2,3,5-Trimethoxy-4-methylphenyl)-1-propanone

[383187-40-4] $C_{13}H_{18}O_4$ mol.wt. 238.28

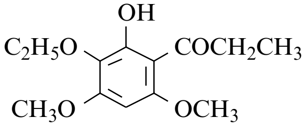


Synthesis

– Preparation by reaction of dimethyl sulfate with 2-hydroxy-3,5-dimethoxy-4-methylpropiophenone in acetone for 40 h at r.t. (99%) [7498].

1-(3-Ethoxy-2-hydroxy-4,6-dimethoxyphenyl)-1-propanone

[89880-49-9] $C_{13}H_{18}O_5$ mol.wt. 254.28



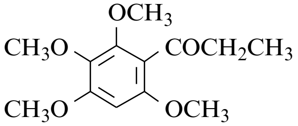
Synthesis

– Obtained (by-product) by reaction of propionyl chloride with 1,2,3,5-tetramethoxybenzene in the presence of aluminium chloride in ethyl ether at r.t. for 20 h (16%) [7543].

m.p. 100.5–101.5° [7543]; ^{1}H NMR [7543], IR [7543].

1-(2,3,4,6-Tetramethoxyphenyl)-1-propanone

[89880-48-8] $C_{13}H_{18}O_5$ mol.wt. 254.28



Syntheses

– Obtained by reaction of propionyl chloride with 1,2,3,5-tetramethoxybenzene in the presence of aluminium chloride in carbon disulfide [7541] or in ethyl ether (45%) [7543].

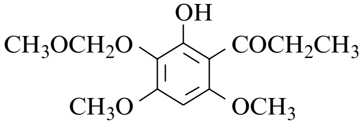
– Also obtained by reaction of dimethyl sulfate with 2-hydroxy-3,4,6-trimethoxypropiophenone in the presence of potassium carbonate in refluxing acetone [7541], (94%) [7543].

– Also refer to: [7442].

m.p. 55–56° [7541], 50–52° [7543]; ^{1}H NMR [7543], IR [7543].

1-[2-Hydroxy-4,6-dimethoxy-3-(methoxymethoxy)phenyl]-1-propanone

[276690-11-0] $C_{13}H_{18}O_6$ mol.wt. 270.28

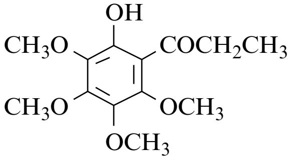


Synthesis

- Refer to: [7439].

1-(2-Hydroxy-3,4,5,6-tetramethoxyphenyl)-1-propanone

[117970-66-8] $C_{13}H_{18}O_6$ mol.wt. 270.28

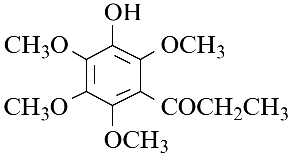


Syntheses

- Preparation by partial methylation of 2,5-dihydroxy-3,4,6-trimethoxypropiophenone with dimethyl sulfate in the presence of potassium carbonate in benzene (50%) [7548].
- Also obtained by reaction of propionyl chloride with pentamethoxybenzene (m.p. 58–59°) [7549] in the presence of aluminium chloride,
  - in ethyl ether at r.t. for 16 h [7548], according to the method [7549];
  - in methylene chloride at r.t. (55%) [7116].

1-(3-Hydroxy-2,4,5,6-tetramethoxyphenyl)-1-propanone

[50816-73-4] $C_{13}H_{18}O_6$ mol.wt. 270.28



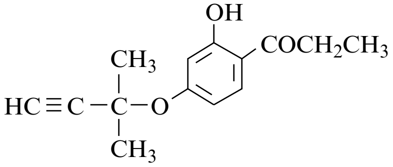
Synthesis

- Obtained by hydrogenation of patagonaldehyde (SM) (m.p. 108–109°) at atmospheric pressure over Pt in ethanol containing a few drops of perchloric acid for 1 h (25%) [7613]. SM was isolated from the heartwood of *Patagonula americana* L. (Boraginaceae).

oil [7613]; ^{1}H NMR [7613], IR [7613], MS [7613]; TLC [7613].

1-[4-[(1,1-Dimethyl-2-propynyl)oxy]-2-hydroxyphenyl]-1-propanone

[118609-36-2] $C_{14}H_{16}O_3$ mol.wt. 232.28



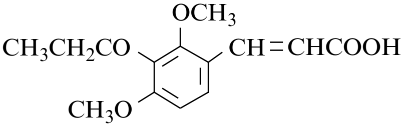
Synthesis

- Refer to: [7614].

3-[2,4-Dimethoxy-3-(1-oxopropyl)phenyl]-2-propenoic acid

2,4-Dimethoxy-3-propionylcinnamic acid

[100884-41-1]



$C_{14}H_{16}O_5$ mol.wt. 264.28

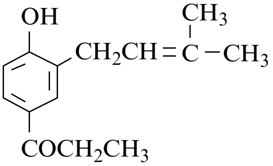
Synthesis

- Obtained (one-pot) by saponification of 8-propionylumbelliferone (m.p. 168°) and treatment in situ with dimethyl sulfate in boiling 3 N potassium hydroxide of the residual mixture [6806].

m.p. 134° [6806].

1-[4-Hydroxy-3-(3-methyl-2-butenyl)phenyl]-1-propanone

[561046-07-9]



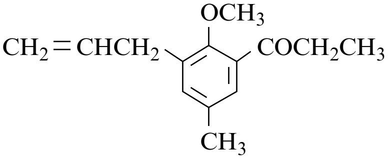
$C_{14}H_{18}O_2$ mol.wt. 218.30

Synthesis

- Refer to: [7615] (compound **84**).

1-[2-Methoxy-5-methyl-3-(2-propenyl)phenyl]-1-propanone

[108293-76-1]



$C_{14}H_{18}O_2$ mol.wt. 218.30

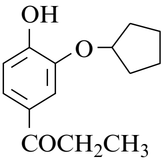
Synthesis

- Preparation by reaction of dimethyl sulfate with 3-allyl-2-hydroxy-5-methylpropiophenone in THF in the presence of an aqueous solution of potassium hydroxide at reflux for 3 h (96%) [7556].

b.p.$_{0.1}$ 88–90° [7556]; ^{1}H NMR [7556], MS [7556].

1-[3-(Cyclopentyloxy)-4-hydroxyphenyl]-1-propanone

[137053-39-5]



$C_{14}H_{18}O_3$ mol.wt. 234.30

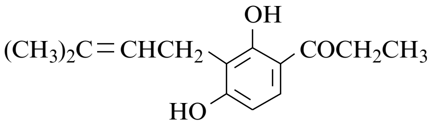
Synthesis

- Preparation [7616] (Czech patent).

USE: Pharmaceutical intermediate [7616].

1-[2,4-Dihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-propanone

[97304-06-8]



$C_{14}H_{18}O_3$ mol.wt. 234.30

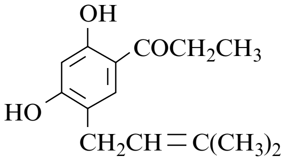
Synthesis

- Obtained by reaction of isopentenyl bromide with respropiophenone in an aqueous 8% potassium hydroxide solution at r.t. for 5 h (17%) [6777].

USE: Fungicide [6777].
m.p. 118° [6777];
^{1}H NMR [6777], IR [6777], UV [6777], MS [6777].

1-[2,4-Dihydroxy-5-(3-methyl-2-butenyl)phenyl]-1-propanone

[97304-11-5]



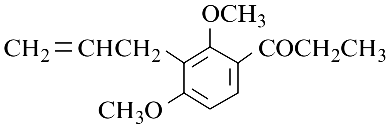
$C_{14}H_{18}O_3$ mol.wt. 234.30

Synthesis

- Obtained by reaction of isopentenyl bromide with respropiophenone in an aqueous 8% potassium hydroxide solution at r.t. for 5 h (12%) [6777].

USE: Fungicide [6777].
m.p. 53° [6777];
^{1}H NMR [6777], IR [6777], UV [6777], MS [6777].

1-[2,4-Dimethoxy-3-(2-propenyl)phenyl]-1-propanone



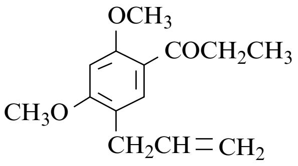
$C_{14}H_{18}O_3$ mol.wt. 234.30

Synthesis

- Preparation by reaction of dimethyl sulfate in acetone with 3-allylrespropiophenone in the presence of 20% potassium hydroxide solution [7451].

b.p.$_{18}$ 180° [7451];
n_D^{15} = 1.540 [7451].

1-[2,4-Dimethoxy-5-(2-propenyl)phenyl]-1-propanone



$C_{14}H_{18}O_3$ mol.wt. 234.30

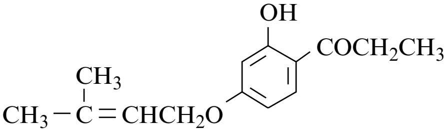
Synthesis

- Preparation by reaction of dimethyl sulfate with 5-allyl-4-hydroxy-2-methoxypropiophenone in the presence of 10% aqueous sodium hydroxide [7451].

m.p. 67° [7451].

1-[2-Hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-1-propanone

[87061-00-5]



$C_{14}H_{18}O_3$ mol.wt. 234.30

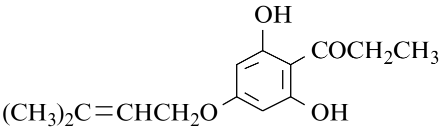
Syntheses

- Refer to: [6748,7617–7619] (Japanese patents).

1-[2,6-Dihydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-1-propanone

2,4,6-Trihydroxypropiophenone-4-O-3,3′-dimethylallyl ether

[70219-80-6]



$C_{14}H_{18}O_4$ mol.wt. 250.29

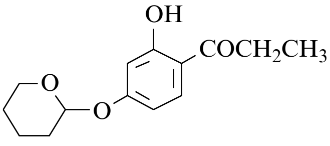
Isolation from natural sources

- From *Leontonyx squarrosus* (compound **4**) [7620].

  colourless oil [7620]; IR [7620], MS [7620].

1-[2-Hydroxy-4-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]-1-propanone

[270084-45-2]



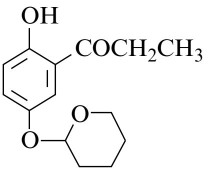
$C_{14}H_{18}O_4$ mol.wt. 250.29

Syntheses

- Preparation by reaction of THP with res-propiophenone [7621], according to the procedure [7622].
- Also refer to: [7623] (compound **7-OTHP**).

1-[2-Hydroxy-5-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]-1-propanone

[54437-06-8]



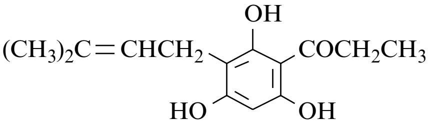
$C_{14}H_{18}O_4$ mol.wt. 250.29

Synthesis

- Refer to: [6795].

1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-propanone

[69916-07-0]



$C_{14}H_{18}O_4$ mol.wt. 250.29

Syntheses

- Obtained by reaction of prenyl bromide with phloropropiophenone in a benzene/ethyl ether solution in the presence of methanolic sodium methoxide, first at 5°, then at r.t. for 6 h (21%) [6880].

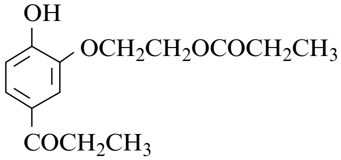
– Also refer to: [6901,7457,7624,7625].

m.p. 170° [6880];
^{1}H NMR [6880,7458], ^{13}C NMR [7458], IR [6880], UV [6880], MS [6880].

USE: Fungicide [6880,7457,7624,7625].
BIOLOGICAL ACTIVITY: Antimicrobial [6901]; bactericide [7457].

1-[4-Hydroxy-3-[2-(1-oxopropoxy)ethoxy]phenyl]-1-propanone

[63437-97-8] $C_{14}H_{18}O_5$ mol.wt. 266.29

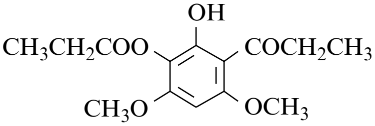


Synthesis

– Refer to: [7626].

1-[2-Hydroxy-4,6-dimethoxy-3-(1-oxopropoxy)phenyl]-1-propanone

[94190-88-2] $C_{14}H_{18}O_6$ mol.wt. 282.29



Syntheses

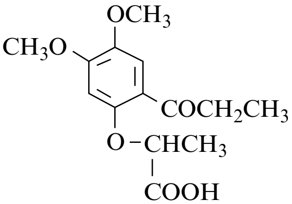
– Obtained by treatment of 1-(2-hydroxy-4,6-di-methoxy-3-propionylphenyl)-1-propanone with a mixture of m-chloroperbenzoic acid and trifluoroacetic acid in refluxing methylene chloride for 2 h (31%) [7438].

– Also refer to: [7439].

m.p. 120° [7438];
^{1}H NMR [7438], ^{13}C NMR [7438]; TLC [7438].

2-[4,5-Dimethoxy-2-(1-oxopropyl)phenoxy]propanoic acid

$C_{14}H_{18}O_6$, 0.5 H_2O mol.wt. 291.30



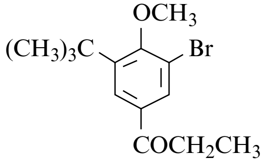
Synthesis

– Obtained by treatment of ethyl α-(2-propionyl-4,5-dimethoxyphenoxy)propionate with 10% aqueous sodium hydroxide on a steam bath, then at r.t. overnight (91%) [7400].

m.p. 100–104° [7400].

1-[3-Bromo-5-(1,1-dimethylethyl)-4-methoxyphenyl]-1-propanone

[35154-10-0] $C_{14}H_{19}BrO_2$ mol.wt. 299.22



Synthesis

- Obtained by reaction of dimethyl sulfate with 3-bromo-5-tert-butyl-4-hydroxypropiophenone (81%) [7560].

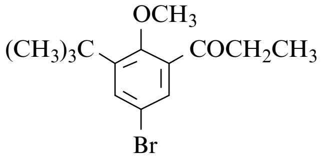
colourless oil [7560]; b.p.$_{15}$ 172° [7560];
IR [7560], UV [7560].

2,4-Dinitrophenylhydrazone $C_{20}H_{23}BrN_4O_5$ mol.wt. 479.33

[35154-12-2] *(E)* ^{1}H NMR [7627], UV [7627] and
[35154-11-1] *(Z)* ^{1}H NMR [7627], UV [7627].
syn (m.p. 160°) (red crystals) [7560,7627] and
anti (m.p. 188°) (yellow crystals) [7560,7627].

1-[5-Bromo-3-(1,1-dimethylethyl)-2-methoxyphenyl]-1-propanone

[35154-21-3] $C_{14}H_{19}BrO_2$ mol.wt. 299.22



Synthesis

- Obtained by reaction of dimethyl sulfate with 5-bromo-3-tert-butyl-2-hydroxypropiophenone (60%) [7560].

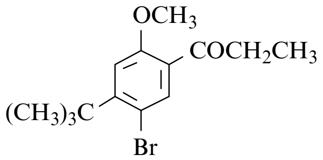
colourless oil [7560]; b.p.$_{18}$ 190° [7560];
IR [7560], UV [7560].

2,4-Dinitrophenylhydrazone [35154-22-4] $C_{20}H_{23}BrN_4O_5$ mol.wt. 479.33

(m.p. 181°) [7560].

1-[5-Bromo-4-(1,1-dimethylethyl)-2-methoxyphenyl]-1-propanone

[35155-00-1] $C_{14}H_{19}BrO_2$ mol.wt. 299.22



Synthesis

- Preparation by reaction of dimethyl sulfate with 5-bromo-4-tert-butyl-2-hydroxypropiophenone (99%) [7560].

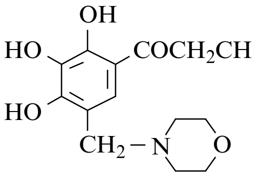
m.p. 98° [7560];
^{1}H NMR (Sadtler: standard 18463M), IR (Sadtler: standard 44740) [7560],
UV [7560].

2,4-Dinitrophenylhydrazone [35155-01-2] $C_{20}H_{23}BrN_4O_5$ mol.wt. 479.33

(m.p. 155–156°) [7560].

1-[2,3,4-Trihydroxy-5-(4-morpholinomethyl)phenyl]-1-propanone

[73044-15-2] $C_{14}H_{19}NO_5$ mol.wt. 281.31



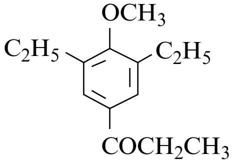
Synthesis

– Preparation by reaction of equimolar amounts of 2,3,4-tri-hydroxypropiophenone, morpholine and formaldehyde in alcoholic solution (Mannich reaction) [7628].

USE: Antioxidizing agent and antiradiation [7628].

1-(3,5-Diethyl-4-methoxyphenyl)-1-propanone

[104008-43-7] $C_{14}H_{20}O_2$ mol.wt. 220.31

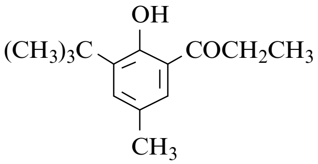


Synthesis

– Refer to: [7308] (Japanese patent).

1-[3-(1,1-Dimethylethyl)-2-hydroxy-5-methylphenyl]-1-propanone

[133903-09-0] $C_{14}H_{20}O_2$ mol.wt. 220.31



Syntheses

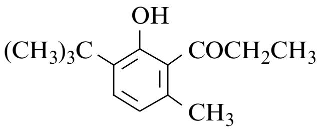
– The reaction of [AlMe(dbmp)$_2$] (Hdbmp = 2, 6-di-tert-butyl-4-methylphenol) with O:C(Cl) Et leads to acylation of one of the dbmp ligands and affords [AlMe(dbmp)(bhmpp)] (SM). (Hbhmpp = 3-tert-butyl-2-hydroxy-5-methyl-propiophenone). Hydrolysis of SM yields uncomplexed Hbhmpp, the titled ketone [7629].

– Also refer to: [7630].

^{1}H NMR [7629], ^{13}C NMR [7629], IR [7629]; X-ray crystallography [7629].

1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methylphenyl]-1-propanone

[137937-38-3] $C_{14}H_{20}O_2$ mol.wt. 220.31



Syntheses

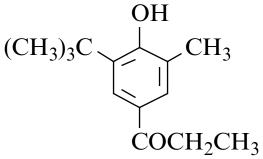
– Preparation by Fries rearrangement of 2-tert-butyl-5-methylphenyl propionate (b.p.$_{19}$ 152°) [7631],

- with titanium tetrachloride in chlorobenzene at 100° for 2 h (55%) [7011] or in ethylene dichloride at 20° for 7 days (16%) [7025];
- with aluminium chloride at 110° for 2 h (80%) or in nitrobenzene at 25° for 6 h (82%) [7631].

b.p.$_8$ 108° [7631]; m.p. 70° [7011], 68° [7025];
^{1}H NMR (Sadtler: standard n° 57887M) [7011],
IR (Sadtler: standard n° 84935K) [7011,7025], UV [7011,7025], MS [7011],
Absolute X-ray crystal structure data [7011,7632].

1-[3-(1,1-Dimethylethyl)-4-hydroxy-5-methylphenyl]-1-propanone

[52069-29-1] $C_{14}H_{20}O_2$ mol.wt. 220.31

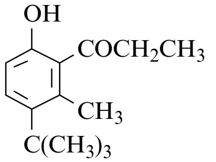


Syntheses
- Preparation [7308].
- Also refer to: [7633,7634] (Japanese patent).

BIOLOGICAL ACTIVITY: Lipoxygenase inhibitor [7308].

1-[3-(1,1-Dimethylethyl)-6-hydroxy-2-methylphenyl]-1-propanone

[51451-25-3] $C_{14}H_{20}O_2$ mol.wt. 220.31



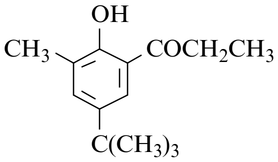
Synthesis
- Described as being obtained by Fries rearrangement of 2-tert-butyl-5-methylphenyl propionate with titanium tetrachloride in ethylene dichloride at 20° for 7 days (16%) (compound **3e**) [7025].

m.p. 68° [7025];
^{1}H NMR [7025], IR (Sadtler: standard n° 47035) [7025], UV [7025].

N.B.: After examination of ^{1}H NMR spectra, the title ketone was formulated as 1-[3-(1,1-dimethyl-ethyl)-6-hydroxy-2-methylphenyl]-1-propanone [7025]. This contradicted some results with respect to the mobility of the tert-butyl group [6586,7560,7571]. So, a structural analysis from X-ray data has been realized [7632] and has led to the formula 1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methylphenyl]-1-propanone (compound **5**) [7011], (compound **2e**) in the paper [7025].

1-[5-(1,1-Dimethylethyl)-2-hydroxy-3-methylphenyl]-1-propanone

[51233-85-3] $C_{14}H_{20}O_2$ mol.wt. 220.31



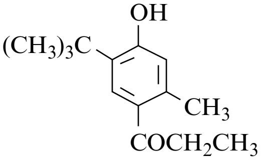
Syntheses
- Preparation by Fries rearrangement of 4-tert-butyl-2-methylphenyl propionate in the presence of aluminium chloride in nitromethane at 20° for 7 days (83%) [6466].

- Preparation by Friedel–Crafts alkylation of 2-hydroxy-3-methylpropiophenone with tert-butyl chloride in nitromethane in the presence of aluminium chloride at 20° for 24 h (78%) [6466].

m.p. M67° [6466];
^{1}H NMR (Sadtler: standard n° 20029M), IR (Sadtler: standard n° 47038) [6466], UV [6466].

1-[5-(1,1-Dimethylethyl)-4-hydroxy-2-methylphenyl]-1-propanone

[51451-24-2] $C_{14}H_{20}O_2$ mol.wt. 220.31



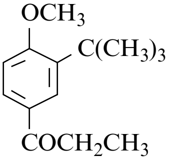
Synthesis

- Preparation by Fries rearrangement of 2-tert-butyl-5-methyl-phenyl propionate with titanium tetrachloride in nitromethane at r.t. for 24 h (31%) [7025].

m.p. 133° [7025];
^{1}H NMR (Sadtler: standard n° 20027M) [7025],
IR (Sadtler: standard n° 47036) [7025], UV [7025].

1-[3-(1,1-Dimethylethyl)-4-methoxyphenyl]-1-propanone

[35154-05-3] $C_{14}H_{20}O_2$ mol.wt. 220.31



Synthesis

- Preparation by reaction of dimethyl sulfate with 3-tert-butyl-4-hydroxypropiophenone (quantitative yield) [7560].

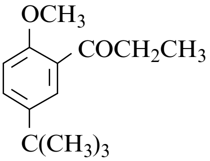
b.p.$_{20}$ 167° [7560]; m.p. 66–67° [7560];
^{1}H NMR (Sadtler: standard 18460M),
IR (Sadtler: standard 44737) [7560], UV [7560].

2,4-Dinitrophenylhydrazones $C_{20}H_{24}N_4O_5$ mol.wt. 400.48

[35154-07-5] *(E)* ^{1}H NMR [7627] and [35154-06-4] *(Z)* ^{1}H NMR [7627];
syn (m.p. 162°) (red crystals) [7560,7627]; anti (m.p. 178°) (yellow crystals) [7560,7627].

1-[5-(1,1-Dimethylethyl)-2-methoxyphenyl]-1-propanone

[35155-04-5] $C_{14}H_{20}O_2$ mol.wt. 220.31

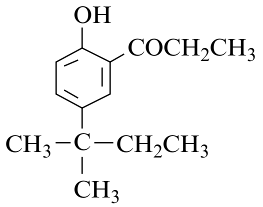


Synthesis

- Preparation by reaction of dimethyl sulfate with 5-tert-butyl-2-hydroxypropiophenone [7560].

b.p.$_{15}$ 166–167° [7560]; m.p. 46° [7560];
IR [7560], UV [7560].

1-[2-Hydroxy-5-(1,1-dimethylpropyl)phenyl]-1-propanone



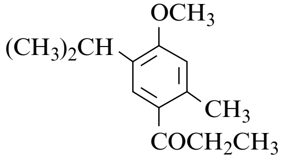
$C_{14}H_{20}O_2$ mol.wt. 220.31

Syntheses

- Obtained by Fries rearrangement of p-tert-amylphenol propionate (b.p.$_{20}$ 118°) with aluminium chloride at 120° for 1 h (45%) [7573], Cf. (*10i*) [6407].
- Also obtained by reaction of propionic acid with p-tert-amyl-phenol in the presence of boron trifluoride at 70–80° for 2 h [6363].

yellow amber-coloured oil [6363];
b.p.$_{30}$ 110° [7573], b.p.$_{24}$ 206–209° [6363]. One of the reported boiling points is obviously wrong. $n_D^{24} = 1.5320$ [6363].

1-[4-Methoxy-2-methyl-5-(1-methylethyl)phenyl]-1-propanone



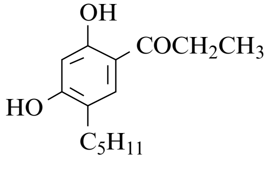
$C_{14}H_{20}O_2$ mol.wt. 220.31

Syntheses

- Preparation by reaction of dimethyl sulfate with 4-hydroxy-2-methyl-5-isopropylpropiophenone in the presence of aqueous sodium hydroxide (76%) [7582].
- Also obtained by reaction of propionyl chloride with 2-iso-propyl-5-methylanisole in the presence of aluminium chloride in carbon disulfide [7583].
- Also refer to: [7319].

b.p.$_{15}$ 169–171° [7583], b.p.$_{20}$ 169–171° [7582].

1-(2,4-Dihydroxy-5-pentylphenyl)-1-propanone



$C_{14}H_{20}O_3$ mol.wt. 236.31

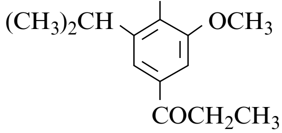
Syntheses

- Preparation by reaction of propionitrile with 4-amyl-resorcinol (Hoesch reaction) [7484], (83%) [7115].

b.p.$_{6}$ 192–195° [7484];
m.p. 52–53° [7115,7484].

1-[3,4-Dimethoxy-5-(1-methylethyl)phenyl]-1-propanone

[83569-69-1]



$C_{14}H_{20}O_3$ mol.wt. 236.31

Syntheses

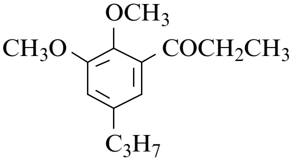
- Preparation by reaction of propionyl chloride with 1,2-dimethoxy-3-isopropylbenzene,

- in the presence of ferric chloride in refluxing carbon disulfide (56%) [7635];
- in the presence of aluminium chloride in methylene chloride at r.t. (71%) [7635].

– Also obtained by reaction of dimethyl sulfate with 4-hydroxy-5-methoxy-3-isopropyl-propiophenone in the presence of potassium carbonate in acetone at r.t. for 2 h (95%) [7606].

b.p.$_{0.04}$ 113–116° [7635], b.p.$_{0.5}$ 130–132° [7606];
n_D^{20} = 1.5257 [7635], n_D^{24} = 1.531 [7606];
^{1}H NMR [7606], IR [7606,7635].

1-(2,3-Dimethoxy-5-propylphenyl)-1-propanone



$C_{14}H_{20}O_3$ mol.wt. 236.31

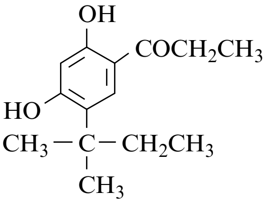
Synthesis

– Obtained by reaction of ethylmagnesium bromide with 2,3-dimethoxy-5-propylbenzonitrile (15%) [6973].

b.p.$_{0.5}$ 121–124° [6973].

1-[5-(1,1-Dimethylpropyl)-2,4-dihydroxyphenyl]-1-propanone

[95102-28-6]



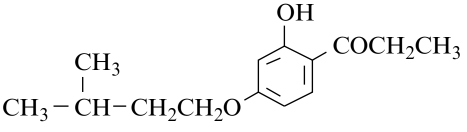
$C_{14}H_{20}O_3$ mol.wt. 236.31

Syntheses

– Refer to: [7602,7603] (Japanese patents).

1-[2-Hydroxy-4-(3-methylbutoxy)phenyl]-1-propanone

[37456-35-2]



$C_{14}H_{20}O_3$ mol.wt. 236.31

Syntheses

– Obtained by reaction of isoamyl bromide with respropiophenone in the presence of potassium carbonate in refluxing acetone for 3 days (45%) [7636].

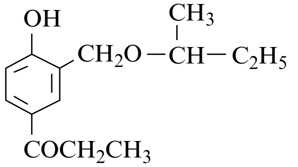
– Also refer to: [7637,7638].

N.B.: The name of this ketone, called 2-(3-methylbutoxy)-4-hydroxypropiophenone (7b) was erroneous [7636] (page 82).

m.p. 43–44° [7636];
^{1}H NMR [7636], MS [7636]; TLC [7636].

1-[4-Hydroxy-3-[(1-methylpropoxy)methyl]phenyl]-1-propanone

[136715-26-9] $C_{14}H_{20}O_3$ mol.wt. 236.31

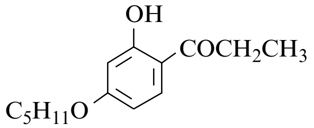


Synthesis

- Preparation by reaction of 4-hydroxy-3-(chloromethyl)-propiophenone with 2-butanol in the presence of sodium bicarbonate at 30–40° [7392].

1-[2-Hydroxy-4-(pentyloxy)phenyl]-1-propanone

[63411-91-6] $C_{14}H_{20}O_3$ mol.wt. 236.31



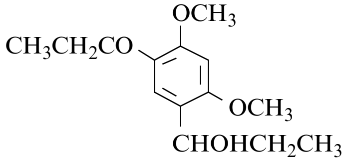
Synthesis

- Preparation by reaction of a pentyl halide (unspecified) with respropiophenone in the presence of potassium carbonate in refluxing acetone for 5 h (64%) [6380].

viscous oil, purified by preparative TLC [6380];
^1H NMR [6380], IR [6380]; TLC [6380].

1-[2,4-Dimethoxy-5-(1-hydroxypropyl)phenyl]-1-propanone

$C_{14}H_{20}O_4$ mol.wt. 252.31



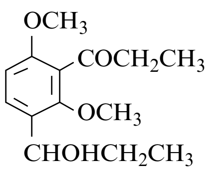
Syntheses

- Obtained by treatment of 4,6-dipropionyl-1,3-dimethoxy-benzene with sodium borohydride in a mixture of nitromethane/water (8v/1v) for 24 h at 20° (48%) [6733,7611].

m.p. 134° [6733,7611];
^1H NMR [6733,7611], IR [6733,7611].

1-[2,6-Dimethoxy-3-(1-hydroxypropyl)phenyl]-1-propanone

$C_{14}H_{20}O_4$ mol.wt. 252.31



Syntheses

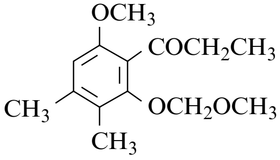
- Obtained by treatment of 2,4-dipropionyl-1,3-dimethoxy-benzene with sodium borohydride in a mixture of nitromethane/water (8v/1v) for 24 h at 20° (88%) [6733,7611].

m.p. 58° [6733,7611];
^1H NMR [6733,7611], IR [6733,7611].

2,4-Dinitrophenylhydrazone $C_{20}H_{24}N_4O_7$ mol.wt. 432.48 (m.p. 257°) [6733];
IR [6733], UV [6733].

1-[6-Methoxy-2-(methoxymethoxy)-3,4-dimethylphenyl]-1-propanone

[185207-91-4] $C_{14}H_{20}O_4$ mol.wt. 252.31

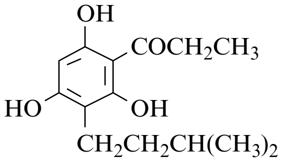


Syntheses

- Refer to: [7490,7491] (Japanese patents).

1-[2,4,6-Trihydroxy-3-(3-methylbutyl)phenyl]-1-propanone

[96756-27-3] $C_{14}H_{20}O_4$ mol.wt. 252.31



Syntheses

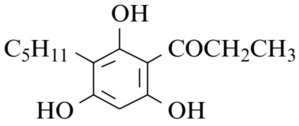
- Preparation by hydrogenation of 2,4,6-trihydroxy-3-iso-pentenylpropiophenone in the presence of PtO_2 at r.t. for 1 h (86%) [6880].
- Also obtained by reaction of propionic acid with C-isoamyl-phloroglucinol in the presence of boron trifluoride at 28–30° for 24 h (63%) [7639].

USE: Fungicide [6880].

m.p. 175° (table 1) and 180° (table 2) [7639], 173° [6880];
^1H NMR [6880], IR [6880], UV [6880], MS [6880].

1-(2,4,6-Trihydroxy-3-pentylphenyl)-1-propanone

[66711-60-2] $C_{14}H_{20}O_4$ mol.wt. 252.31



Syntheses

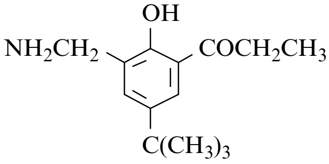
- Obtained by Friedel–Crafts acylation of 2,4,6-trihydroxy-pentylbenzene with propionyl chloride in a carbon disulfide/nitrobenzene solution in the presence of aluminium chloride, first at r.t., then at 30–35° for 6 h (58%) [6880].
- Also refer to: [7640].

USE: Fungicide [7640].

m.p. 166° [6880]; ^1H NMR [6880], IR [6880], UV [6880], MS [6880].

1-[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-propanone

[75060-92-3] $C_{14}H_{21}NO_2$ mol.wt. 235.33



Syntheses

- Refer to: [7106,7641].
- Also refer to: [7642–7653].

USE: Antioxidizing agent [7654].
BIOLOGICAL ACTIVITY: Antiinflammatory [7654–7659]; antiarteriosclerotic agent [7641]; atherosclerosis [7660]; heart infarction inhibition [7661]; antihypertensive [7662]; antiallergic [7663].

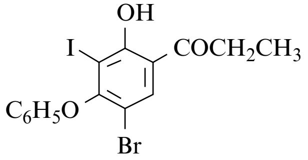
Hydrochloride *(ONO-3144)* [75060-66-1] $C_{14}H_{21}NO_2$, HCl mol. wt. 271.79.

- For syntheses, refer to: [7106,7641,7648,7662].

Note: Ion (1^-) [118464-88-3] [7654].
BIOLOGICAL ACTIVITY: Antiinflammatory [7106,7654]; antiarteriosclerotic agent [7641].

1-(5-Bromo-2-hydroxy-3-iodo-4-phenoxyphenyl)-1-propanone

[245407-13-0] $C_{15}H_{12}BrIO_3$ mol.wt. 447.07



Syntheses

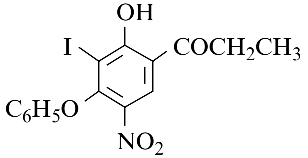
- Obtained by oxidation of 5-bromo-2,4-dihydroxy-propiophenone [6368] with phenyliodonium diacetate (PIDA) under three conditions (compound **4h**) [6452]:

(a) (*basic*): in the presence of potassium hydroxide in methanol at 0° and stirring overnight (38%).
(b) (*neutral*): in refluxing methanol (10%).
(c) (*acidic*): in refluxing acetic acid (20%).

m.p. 146–147° [6452]; ^{1}H NMR [6452], IR [6452], MS [6452]; TLC [6452].

1-(2-Hydroxy-3-iodo-5-nitro-4-phenoxyphenyl)-1-propanone

[245407-11-8] $C_{15}H_{12}INO_5$ mol.wt. 413.17



Syntheses

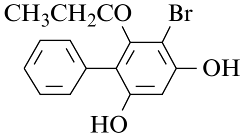
- Obtained by oxidation of 2,4-dihydroxy-5-nitro-propiophenone [6555] with phenyliodonium diacetate (PIDA) under three conditions (compound **4g**) [6452]:

(a) (*basic*): in the presence of potassium hydroxide in methanol at 0° and stirring overnight (48%).
(b) (*neutral*): in refluxing methanol (27%).
(c) (*acidic*): in refluxing acetic acid (41%).

m.p. 190–192° [6452]; ^{1}H NMR [6452], IR [6452], MS [6452]; TLC [6452].

1-(3-Bromo-4,6-dihydroxy[1,1′-biphenyl]-2-yl)-1-propanone

[860152-45-0] $C_{15}H_{13}BrO_3$ mol.wt. 321.17

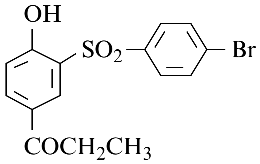


Synthesis

- Refer to: [7664].

1-[3-[(4-Bromophenyl)sulfonyl]-4-hydroxyphenyl]-1-propanone

[67474-16-2] $C_{15}H_{13}BrO_4S$ mol.wt. 369.24



Synthesis

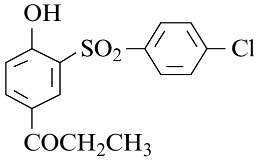
- Preparation by treatment of 4-propionylphenyl p-bromophenylsulfonate (1 mol) with aluminium chloride (3 mol) at 140° for 3–4 h (84–100%) [7665].

m.p. 105–106° [7665].

BIOLOGICAL ACTIVITY: Bactericide (no data) [7665].

1-[3-[(4-Chlorophenyl)sulfonyl]-4-hydroxyphenyl]-1-propanone

[67474-15-1] $C_{15}H_{13}ClO_4S$ mol.wt. 324.78



Synthesis

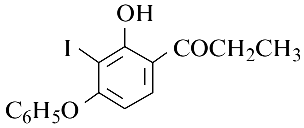
- Preparation by treatment of 4-propionylphenyl p-chlorophenylsulfonate (1 mol) with aluminium chloride (3 mol) at 140° for 3–4 h (84–100%) [7665].

m.p. 68–70° [7665].

BIOLOGICAL ACTIVITY: Bactericide (no data) [7665].

1-(2-Hydroxy-3-iodo-4-phenoxyphenyl)-1-propanone

[245407-09-4] $C_{15}H_{13}IO_3$ mol.wt. 368.17



Syntheses

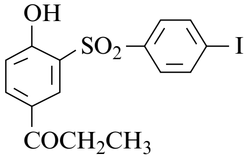
- Obtained by oxidation of respropiophenone [6734] with phenyliodonium diacetate (PIDA) under three conditions (compound **4e**) [6452]:

(a) (*basic*): in the presence of potassium hydroxide in methanol at 0° and stirring overnight (68%).
(b) (*neutral*): in refluxing methanol (40%).
(c) (*acidic*): in refluxing acetic acid (55%).

m.p. 136–138° [6452]; ^{1}H NMR [6452], IR [6452]; TLC [6452].

1-[4-Hydroxy-3-[(4-iodophenyl)sulfonyl]phenyl]-1-propanone

[67474-17-3] $C_{15}H_{13}IO_4S$ mol.wt. 416.24



Synthesis

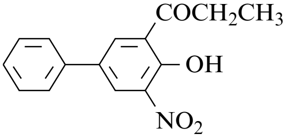
- Preparation by treatment of 4-propionylphenyl p-iodophenylsulfonate (1 mol) with aluminium chloride (3 mol) in nitrobenzene at 100–110° (84–100%) [7665].

BIOLOGICAL ACTIVITY: Bactericide (no data) [7665].

m.p. 135° [7665].

1-(4-Hydroxy-5-nitro[1,1′-biphenyl]-3-yl)-1-propanone

[92554-09-1] $C_{15}H_{13}NO_4$ mol.wt. 271.27



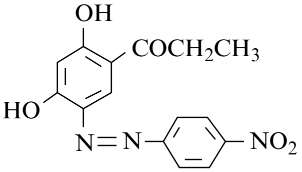
Synthesis

– Obtained by reaction of concentrated nitric acid with 2-hydroxy-5-phenylpropiophenone in concentrated sulfuric acid between –5° and 0° [7666].

m.p. 55–60° [7666].

1-[2,4-Dihydroxy-5-[(4-nitrophenyl)azo]phenyl]-1-propanone

[65412-16-0] $C_{15}H_{13}N_3O_5$ mol.wt. 315.29



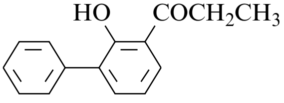
Synthesis

– Refer to: [7667].

USE: Dye for polypropylene fibres [7667].

1-(2-Hydroxy[1,1′-biphenyl]-3-yl)-1-propanone

$C_{15}H_{14}O_2$ mol.wt. 226.27



Syntheses

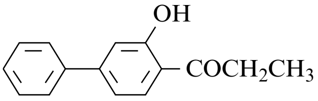
– Obtained (by-product) by Fries rearrangement of o-xenyl propionate,

- with aluminium chloride at 160° for 30–45 min (8%) [7668], (4%) [7669];
- with titanium tetrachloride in nitromethane at 20° for 24 h (10%) [7561].

b.p.$_{3.5}$ 183–185° [7668]; n_D^{20} = 1.6145 [7561]; IR [7561], UV [7561].

1-(3-Hydroxy[1,1′-biphenyl]-4-yl)-1-propanone

[108478-15-5] $C_{15}H_{14}O_2$ mol.wt. 226.27



Syntheses

– Preparation by Fries rearrangement of 3-hydroxybiphenyl propionate (b.p.$_2$ 160–165°) [7668,7670] with aluminium chloride (71–74%) [7668,7671].

– Preparation by Friedel–Crafts acylation of 3-methoxy-biphenyl with propionyl chloride in the presence of aluminium chloride, first in refluxing methylene chloride for 11 h, then at r.t. for 11 h (71%) [7671].

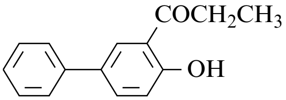
– Also obtained by ether cleavage of 3-methoxy-4-propionylbiphenyl with aluminium chloride in refluxing methylene chloride overnight (53%) [7671].

- Also obtained (poor yield) by heating diethyl propionylmalonate and 2-phenyl-3-yn-2-ol together for 1.5 h, the final temperature was 230° (7%) [7672].

m.p. 113–113.5° [7672], 110.5–111° [7671], 109° [7668,7670];
IR [7672], UV [7672].

1-(4-Hydroxy[1,1′-biphenyl]-3-yl)-1-propanone

2-Hydroxy-5-phenylpropiophenone



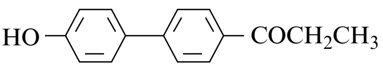
$C_{15}H_{14}O_2$ mol.wt. 226.27

Syntheses

- Refer to: [7666,7673].

USE: Plasticizer for vinyl chloride-vinylene chloride polymers [7673].

1-(4′-Hydroxy[1,1′-biphenyl]-4-yl)-1-propanone



$C_{15}H_{14}O_2$ mol.wt. 226.27

Syntheses

- Obtained by Fries rearrangement of 4-(propionyloxy)biphenyl (m.p. 93°) with aluminium chloride in nitrobenzene, first at 20° for 12 h, then at 60° for 1 h (38%) [7674].
- Also obtained by treatment of 4-methoxy-4′-propionylbiphenyl with 48% hydrobromic acid in boiling acetic acid for 7–8 h (70%) [7675].

m.p. 179° [7674], 174° [7675].

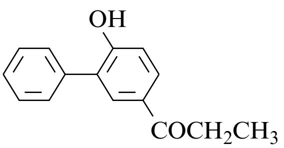
Benzoate $C_{22}H_{18}O_3$ mol.wt. 330.38.

- Preparation by reaction of benzoyl chloride with 4-hydroxy-4′-propionylbiphenyl in the presence of pyridine (82%) [7675].

m.p. 167–168° [7675].

1-(6-Hydroxy[1,1′-biphenyl]-3-yl)-1-propanone

[95102-29-7]



$C_{15}H_{14}O_2$ mol.wt. 226.27

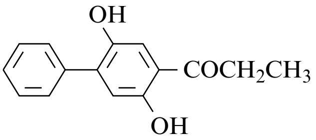
Syntheses

- Preparation by Fries rearrangement of o-xenyl propionate (2-hydroxydiphenyl propionate),
  - with aluminium chloride [7603], at 120° for 5 h [7676] or 160° for 30–45 min [7668], (46%) [7669];
  - with titanium tetrachloride in nitromethane at 20° for 24 h (22%) [7561].

- Also obtained by direct condensation of o-hydroxydiphenyl with propionic acid in the presence of boron trifluoride for 2–3 h between 65° and 85° [6819].
- Also obtained by treatment of 5-propionyl-2-methoxydiphenyl (m.p. 92°) with boiling pyridinium chloride [7677].
- Also refer to: [7602,7670].

m.p. 154° [6819], 151–152° [7668], 150° [7561], 148–149° [7677], 148° [7676], 147.5–148° [7669];
IR [7561], UV [7561].

1-(2,5-Dihydroxy[1,1′-biphenyl]-4-yl)-1-propanone



$C_{15}H_{14}O_3$ mol.wt. 242.27

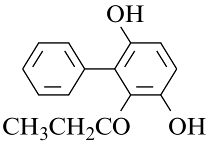
Synthesis

- Obtained (by-product) by Friedel–Crafts acylation of 2,5-dimethoxydiphenyl (b.p.$_4$ 147–149°) with propionyl chloride or propionic anhydride in the presence of aluminium chloride (15%) [7668].

b.p.$_6$ 220–230° [7668]; m.p. 138–139° [7668].

1-(3,6-Dihydroxy[1,1′-biphenyl]-2-yl)-1-propanone

6-Propionyl-2,5-dihydroxydiphenyl



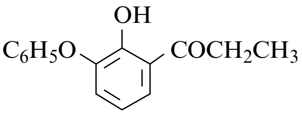
$C_{15}H_{14}O_3$ mol.wt. 242.27

Synthesis

- Obtained by direct propionylation of 2,5-dimethoxydiphenyl, but total demethylation took place at the same time (15%) [7668].

1-(2-Hydroxy-3-phenoxyphenyl)-1-propanone

[307000-30-2]



$C_{15}H_{14}O_3$ mol.wt. 242.27

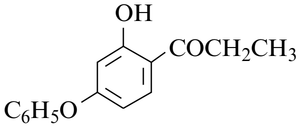
Synthesis

- Refer to: [7678].

USE: For inhibiting development of body odours in cosmetic compositions [7679–7683].

1-(2-Hydroxy-4-phenoxyphenyl)-1-propanone

[479580-94-4] $C_{15}H_{14}O_3$ mol.wt. 242.27



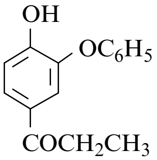
Synthesis

– Refer to: [7678].

USE: For inhibiting development of body odours in cosmetic compositions [7679–7683].

1-(4-Hydroxy-3-phenoxyphenyl)-1-propanone

[307000-51-7] $C_{15}H_{14}O_3$ mol.wt. 242.27

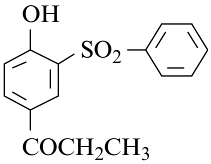


Synthesis

– Refer to: [7678].

1-[4-Hydroxy-3-(phenylsulfonyl)phenyl]-1-propanone

[67474-13-9] $C_{15}H_{14}O_4S$ mol.wt. 290.34



Synthesis

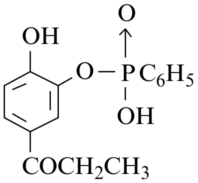
– Preparation by treatment of 4-propionylphenyl benzenesulfonate (1 mol) with aluminium chloride (3 mol) at 140° for 3–4 h (84–100%) [7665].

BIOLOGICAL ACTIVITY: Bactericide (no data) [7665].
m.p. 64° [7665].

1-(3,4-Dihydroxyphenyl)-1-propanone 3-phenylphosphonate

2-Hydroxy-5-propionylphenyl hydrogen phenylphosphonate

$C_{15}H_{15}O_5P$ mol.wt. 306.25

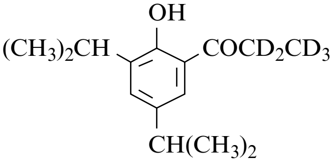


Synthesis

– Obtained by reaction of phenylphosphonic dichloride with 3,4-dihydroxypropiophenone in boiling bromobenzene for 6 h under nitrogen (83%) [6822].

m.p. 163–164.5° [6822]; IR [6822]; pK$_a$ [6822].

1-[2-Hydroxy-3,5-bis(1-methylethyl)phenyl]-1-propanone-*2,2,3,3,3-d*$_5$



$C_{15}H_{17}D_5O_2$ mol.wt. 239.37

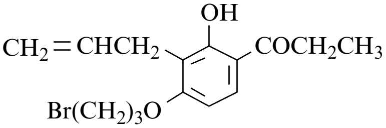
Synthesis

- Refer to: [6350].

Ion (1$^-$), radical ion (1$^-$) [57209-24-2], ESR spectrum [6350].

1-[4-(3-Bromopropoxy)-2-hydroxy-3-(2-propenyl)phenyl]-1-propanone

[194792-40-0]



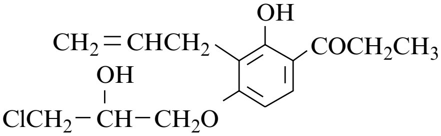
$C_{15}H_{19}BrO_3$ mol.wt. 327.22

Synthesis

- Refer to: [7452].

1-[4-(3-Chloro-2-hydroxypropoxy)-2-hydroxy-3-(2-propenyl)phenyl]-1-propanone

[63360-15-6]



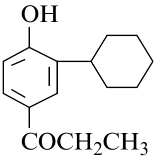
$C_{15}H_{19}ClO_4$ mol.wt. 298.77

Syntheses

- Refer to: [7112,7453,7482,7684].

1-(3-Cyclohexyl-4-hydroxyphenyl)-1-propanone

[95185-71-0]



$C_{15}H_{20}O_2$ mol.wt. 232.32

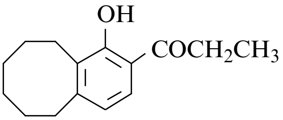
Synthesis

- Preparation by Fries rearrangement of 2-cyclohexylphenyl propionate [7603].

USE: As colour developer [7603].

1-(5,6,7,8,9,10-Hexahydro-1-hydroxy-2-benzocyclooctenyl)-1-propanone

[936642-86-3]



$C_{15}H_{20}O_2$ mol.wt. 232.32

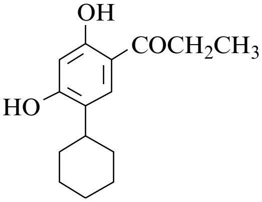
Synthesis

- Obtained by reaction of bicyclo[6.2.0]dec-8(1)-en-9-one with ethyl vinyl ketone in the presence of a catalytic amount of $[RhCl(C_2H_4)_2]_2$ and P(*cyclo*-$C_6H_{11})_3$ in toluene at 130° for 12 h under an argon atmosphere (43%) [7570].

Colourless liquid [7570]; b.p.$_{0.1}$ 120–130° [7570];
^{1}H NMR [7570], ^{13}C NMR [7570], IR [7570], MS [7570]; GLC [7570].

1-(5-Cyclohexyl-2,4-dihydroxyphenyl)-1-propanone

[159977-37-4] $C_{15}H_{20}O_3$ mol.wt. 248.32



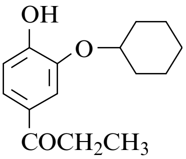
Synthesis

- Preparation by reaction of propionic acid with 4-cyclohexyl-resorcinol in the presence of boron trifluoride etherate at 125° for 15 min (80%) [6737].

m.p. 72–73° [6737]; IR [6737], UV [6737].

1-[3-(Cyclohexyloxy)-4-hydroxyphenyl]-1-propanone

[137053-40-8] $C_{15}H_{20}O_3$ mol.wt. 248.32



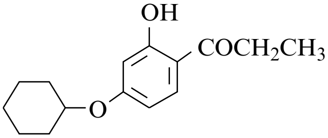
Synthesis

- Preparation [7616] (Czech patent).

USE: As pharmaceutical intermediate [7616].

1-[4-(Cyclohexyloxy)-2-hydroxyphenyl]-1-propanone

[101002-35-1] $C_{15}H_{20}O_3$ mol.wt. 248.32



Syntheses

- Obtained by Fries rearrangement of 3-(cyclohexyloxy)-phenyl propionate [7685].
- Also obtained by reaction of cyclohexyl bromide with respropiophenone in the presence of potassium carbonate in refluxing acetone [7237].

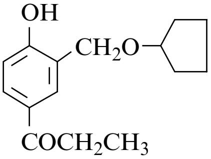
m.p. 80–81° [7237].

Oxime [101002-22-6] $C_{15}H_{21}NO_3$ mol.wt. 263.34 [7686] (m.p. 65–69°) [7237,7687].

- As lipoxygenase inhibitor [7685]; antiallergic and antiinflammatory agent [7688].

1-[3-[(Cyclopentyloxy)methyl]-4-hydroxyphenyl]-1-propanone

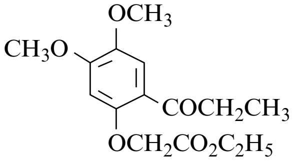
[154783-68-3] $C_{15}H_{20}O_3$ mol.wt. 248.32



Synthesis

- Refer to: [7689] (Czech patent).

2-[4,5-Dimethoxy-2-(1-oxopropyl)phenoxy]acetic acid ethyl ester



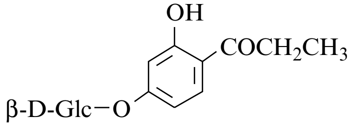
$C_{15}H_{20}O_6$ mol.wt. 296.32

Synthesis

- Obtained by reaction of ethyl bromoacetate with 2-hydroxy-4,5-dimethoxypropiophenone in the presence of potassium hydroxide in refluxing acetone (96%) [7400].

m.p. 112–114° [7400].

1-[2-Hydroxy-4-(β-D-glucopyranosyloxy)phenyl]-1-propanone



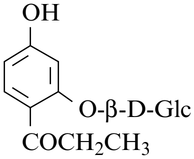
$C_{15}H_{20}O_8$ mol.wt. 328.32

Synthesis

- Preparation by treatment of its tetraacetate (m.p. 129–130°) with boiling methanolic 0.2 M sodium methoxide for 3 min (75%) [6731].

m.p. (monohydrate) 144–145° [6731]; $(\alpha)_D^{25} = -51.4°$ (c = 2 in DMF) [6731];
(anhydrous) 79–181° [6731]; $(\alpha)_D^{20} = -53.2°$ (c = 2 in DMF) [6731];
paper chromatography [6731].

1-[4-Hydroxy-2-(β-D-glucopyranosyloxy)phenyl]-1-propanone *(Trihydrate)*



$C_{15}H_{20}O_8$, 3 H_2O mol.wt. 382.36

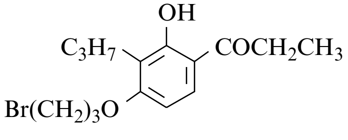
Synthesis

- Obtained by treatment of its pentaacetate (m.p. 110–112°) with boiling methanolic 0.2 M sodium methoxide for 3 min (50%) [6731].

m.p. 69–70° [6731]; paper chromatography [6731];
$(\alpha)_D^{30} = -74.5°$ (c = 1 in water) [6731].

1-[4-(3-Bromopropoxy)-2-hydroxy-3-propyl]-1-propanone

[194608-83-8]



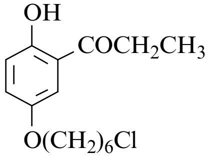
$C_{15}H_{21}BrO_3$ mol.wt. 329.23

Syntheses

- Preparation by reaction of 1,3-dibromopropane with 2,4-dihydroxy-3-propylpropiophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone [7452,7478].
- Also refer to: [7480,7483].

1-[5-[(6-Chlorohexyl)oxy]-2-hydroxyphenyl]-1-propanone

[140439-50-5] $C_{15}H_{21}ClO_3$ mol.wt. 284.78



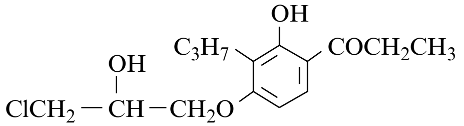
Synthesis

– Preparation by reaction of 1-bromo-6-chlorohexane with 2,5-dihydroxypropiophenone in the presence of potassium carbonate in refluxing 2-butanone for 16 h (83%) [6789].

yellow solid [6789]; ^1H NMR [6789].

1-[4-(3-Chloro-2-hydroxypropoxy)-2-hydroxy-3-propylphenyl]-1-propanone

[69076-30-8] $C_{15}H_{21}ClO_4$ mol.wt. 300.78



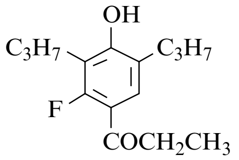
Synthesis

– Preparation by reaction of epichlorohydrin with 2,4-dihydroxy-3-propylpropiophenone, according to the method [7690] (quantitative yield) [7482].

oily solid [7482].

1-(2-Fluoro-4-hydroxy-3,5-dipropylphenyl)-1-propanone

[449779-85-5] $C_{15}H_{21}FO_2$ mol.wt. 252.33

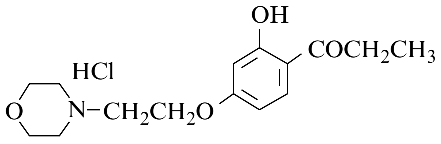


Synthesis

– Refer to: [7479].

1-[2-Hydroxy-4-(2-morpholinoethoxy)phenyl]-1-propanone *(Hydrochloride)*

[20800-11-7] $C_{15}H_{21}NO_4$, HCl mol.wt. 315.80



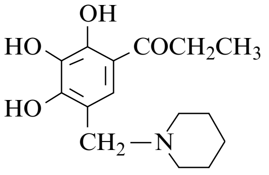
Synthesis

– Obtained by adding a solution of o-hydroxy-propiophenone and sodium ethoxide in ethanol to morpholine hydrochloride and the mixture refluxed 3 h [7691].

m.p. 193–195° [7691]; LD_{50} [7691].

1-[2,3,4-Trihydroxy-5-(1-piperidinylmethyl)phenyl]-1-propanone

[73044-16-3] $C_{15}H_{21}NO_4$ mol.wt. 279.34



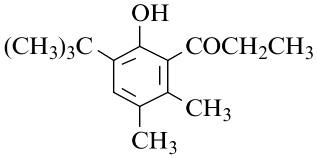
Synthesis

- Preparation by reaction of equimolar amounts of 2,3,4-trihydroxypropiophenone, piperidine and formaldehyde in alcoholic solution (Mannich reaction) [7628].

USE: Antioxidizing agent and antiradiation [7628].

1-[3-(1,1-Dimethylethyl)-2-hydroxy-5,6-dimethylphenyl]-1-propanone

[121194-64-7] $C_{15}H_{22}O_2$ mol.wt. 234.34



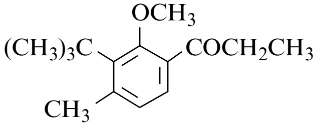
Synthesis

- Preparation by Fries rearrangement of 2-tert-butyl-4,5-di-methylphenyl propionate with titanium tetrachloride in chlorobenzene at 100° for 2 h (52%) [7031].

m.p. 56° [7031];
^1H NMR (Sadtler: standard n° 52743M) [7031],
IR (Sadtler: standard n° 79802K) [7031], UV [7031], MS [7031].

1-[3-(1,1-Dimethylethyl)-2-methoxy-4-methylphenyl]-1-propanone

$C_{15}H_{22}O_2$ mol.wt. 234.34



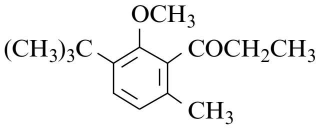
Synthesis

- Refer to: [7472].

b.p.$_{15}$ 180° [7472]; m.p. 72° [7472].

1-[3-(1,1-Dimethylethyl)-2-methoxy-6-methylphenyl]-1-propanone

$C_{15}H_{22}O_2$ mol.wt. 234.34



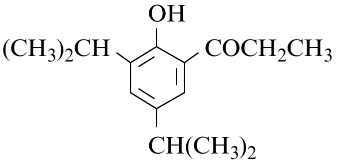
Synthesis

- Preparation by reaction of dimethyl sulfate with 3-tert-butyl-2-hydroxy-6-methylpropiophenone [6351].

^1H NMR (Sadtler: standard n° 57888M), IR (Sadtler: standard n° 84936K).

1-[2-Hydroxy-3,5-bis(1-methylethyl)phenyl]-1-propanone

[57139-00-1] $C_{15}H_{22}O_2$ mol.wt. 234.34



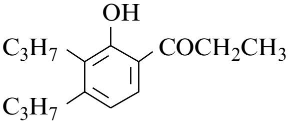
Synthesis

- Refer to: [6350].

Note: Ion (1^-), radical ion (1^-) [57139-00-1], ESR spectrum [6350].

1-(2-Hydroxy-3,4-dipropylphenyl)-1-propanone

[936642-84-1] $C_{15}H_{22}O_2$ mol.wt. 234.34



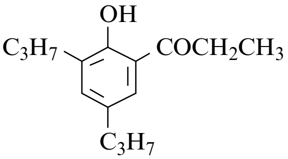
Synthesis

- Obtained by reaction of 2,3-dipropylcyclobutenone with ethyl vinyl ketone in the presence of a catalytic amount of $[RhCl(C_2H_4)_2]_2$ and P(*cyclo*-$C_6H_{11})_3$ in toluene at 130° for 12 h under an argon atmosphere (75%) [7570].

White solid [7570]; b.p.$_1$ 120–130° [7570];
^{1}H NMR [7570], ^{13}C NMR [7570], IR [7570], MS [7570]; GLC [7570].

1-(2-Hydroxy-3,5-dipropylphenyl)-1-propanone

[92729-83-4] $C_{15}H_{22}O_2$ mol.wt. 234.34



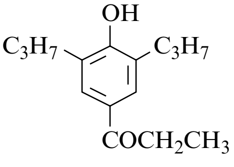
Syntheses

- Preparation by Fries rearrangement of 2,4-dipropylphenyl propionate (b.p.$_{15}$ 154°) [7466], (b.p. 277–279°) [6575] with aluminium chloride for 30 min at 130–135° (75%) [7466] or 2 h at 100° and the reaction terminated at 120° (62%) [6575].

amber-coloured oil [7466]; b.p.$_9$ 154–155° [7466], b.p. 295–298° [6575];
n_D^{26} = 1.5217 [7466].

1-(4-Hydroxy-3,5-dipropylphenyl)-1-propanone

[449779-75-3] $C_{15}H_{22}O_2$ mol.wt. 234.34



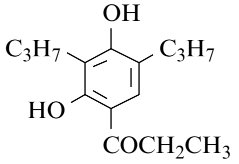
Synthesis

- Preparation by heating a suspension of sodium propionate and 2,6-dipropylphenol in neat triflic acid at 50° for 1 h (85%) [6402].

white solid [6402]; ^{1}H NMR [6402], MS [6402].

1-(2,4-Dihydroxy-3,5-dipropylphenyl)-1-propanone

[449779-73-1] $C_{15}H_{22}O_3$ mol.wt. 250.34

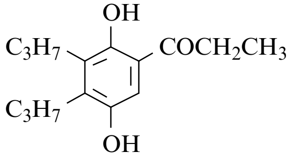


Syntheses

- Preparation by heating a mixture of 1,3-dihydroxy-2,4-di-propylbenzene, sodium propionate and triflic acid (TfOH) (75%) [7459].
- Also refer to: [7479].

1-(2,5-Dihydroxy-3,4-dipropylphenyl)-1-propanone

[873222-90-3] $C_{15}H_{22}O_3$ mol.wt. 250.34



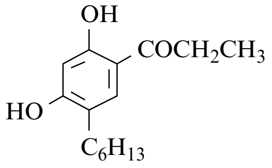
Synthesis

- Obtained by catalytic cocyclization of 4-octyne, ethyl vinyl ketone and carbon monoxide (20 atmospheres) in the presence of $[Cp^*RuCl_2]_2$ in DMF at 140° for 20 h (54%) [7692].

yellow solid [7692]; m.p. 116–117° [7692];
^1H NMR [7692], ^{13}C NMR [7692], IR [7692], MS [7692]; TLC [7692].

1-(2,4-Dihydroxy-5-hexylphenyl)-1-propanone

$C_{15}H_{22}O_3$ mol.wt. 250.34



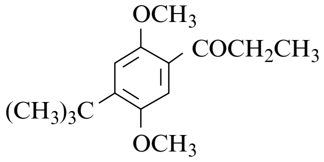
Synthesis

- Preparation by reaction of propionitrile with 4-hexyl-resorcinol (Hoesch reaction) (67%) [7115].

m.p. 50–51° [7115,7484].

1-[4-(1,1-Dimethylethyl)-2,5-dimethoxyphenyl]-1-propanone

[120350-19-8] $C_{15}H_{22}O_3$ mol.wt. 250.34



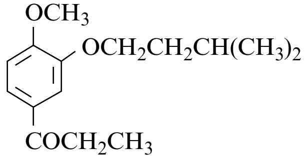
Synthesis

- Preparation by reaction of propionyl chloride with 2-tert-butylhydroquinone dimethyl ether in the presence of aluminium chloride in carbon disulfide [7336].

^1H NMR [7336], IR [7336], UV [7336], MS [7336].

1-[4-Methoxy-3-(3-methylbutoxy)phenyl]-1-propanone

$C_{15}H_{22}O_3$ mol.wt. 250.34



Synthesis

- Obtained by treatment of 2-isopentyloxy-1-methoxy-4-(1-propynyl)benzene with aqueous-ethanolic sulfuric acid [7693].

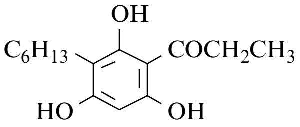
m.p. 64.5–65° [7693].

4-Nitrophenylhydrazone $C_{21}H_{27}N_3O_4$ mol.wt. 385.46 (m.p. 106–108°)[7693].

2,4-Dinitrophenylhydrazone $C_{21}H_{26}N_4O_6$ mol.wt. 430.46 (m.p. 136–137°)[7693].

1-(3-Hexyl-2,4,6-trihydroxyphenyl)-1-propanone

[66711-55-5] $C_{15}H_{22}O_4$ mol.wt. 266.34



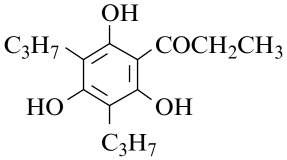
Synthesis

– Preparation, refer to: [7640].

USE: Fungicide [7640].

1-(2,4,6-Trihydroxy-3,5-dipropylphenyl)-1-propanone

[54556-10-4] $C_{15}H_{22}O_4$ mol.wt. 266.34

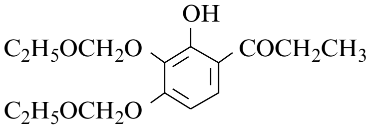


Synthesis

– This ketone was synthetic or obtained from hops [7694].

1-[3,4-Bis(ethoxymethoxy)-2-hydroxyphenyl]-1-propanone

[124300-26-1] $C_{15}H_{22}O_6$ mol.wt. 298.34



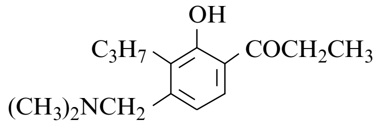
Synthesis

– Obtained by treatment of 3,4-di(ethoxymethoxy)-2-hydroxy-α-hydroxymethylpropiophenone (pale yellow oil) with 4% ethanolic sodium carbonate at reflux for 1 h (31%) [6757].

pale yellow oil [6757]; ^{1}H NMR [6757]; TLC [6757].

1-[4-[(Dimethylamino)methyl]-2-hydroxy-3-propylphenyl]-1-propanone

[107223-71-2] $C_{15}H_{23}NO_2$ mol.wt. 249.35



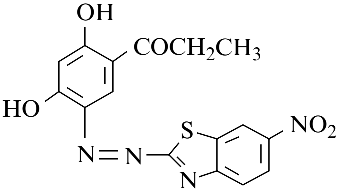
Synthesis

– Obtained by reaction of propionyl chloride with 3-[(dimethylamino)methyl]-2-propylphenyl propionate hydrochloride (m.p. 178–180°) in the presence of aluminium chloride at 165° for 2 h (86%) [7564].

b.p.$_{0.2}$ 105–112° [7564].

1-[2,4-Dihydroxy-5-[(6-nitro-2-benzothiazolyl)azo]phenyl]-1-propanone

[65412-18-2] $C_{16}H_{12}N_4O_5S$ mol.wt. 372.36



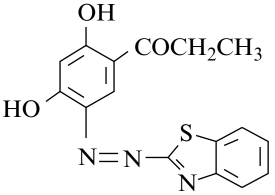
Synthesis

– Refer to: [7667].

USE: Dye for polypropylene fibres [7667].

1-[5-(2-Benzothiazolylazo)-2,4-dihydroxyphenyl]-1-propanone

[65412-17-1] $C_{16}H_{13}N_3O_3S$ mol.wt. 327.36



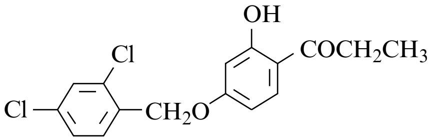
Synthesis

– Refer to: [7667].

USE: Dye for polypropylene fibres [7667].

1-[4-[(2,4-Dichlorophenyl)methoxy]-2-hydroxyphenyl]-1-propanone

[63411-96-1] $C_{16}H_{14}Cl_2O_3$ mol.wt. 325.19



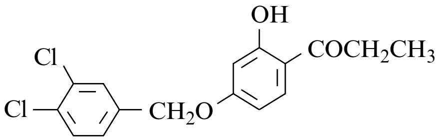
Synthesis

– Obtained by reaction of a 2,4-dichlorobenzyl halide (unspecified) with respropiophenone in the presence of potassium carbonate in refluxing acetone for 5 h (43%) [6380].

m.p. 109–110° [6380]; ^{1}H NMR [6380], IR [6380]; TLC [6380].

1-[4-[(3,4-Dichlorophenyl)methoxy]-2-hydroxyphenyl]-1-propanone

[63411-95-0] $C_{16}H_{14}Cl_2O_3$ mol.wt. 325.19

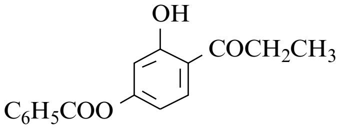


Synthesis

– Obtained by reaction of a 3,4-dichlorobenzyl halide (unspecified) with respropiophenone in the presence of potassium carbonate in refluxing acetone for 5 h (55%) [6380].

m.p. 126–127° [6380]; ^{1}H NMR [6380], IR [6380]; TLC [6380].

1-[4-(Benzoyloxy)-2-hydroxyphenyl]-1-propanone



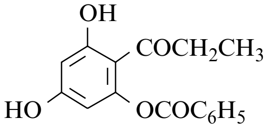
$C_{16}H_{14}O_4$ mol.wt. 270.28

Synthesis

- Obtained (by-product) by action of potassium hydroxide with 2,4-di(benzoyloxy) propiophenone in pyridine at 50° for 5 min [6762].

b.p.$_{0.4}$ 210–220° [6762]; m.p. 80° [6762].

1-[2-(Benzoyloxy)-4,6-dihydroxyphenyl]-1-propanone



$C_{16}H_{14}O_5$ mol.wt. 286.28

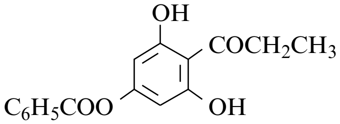
Syntheses

- Obtained by reaction of benzoyl chloride with phloropropiophenone in the presence of 1.5% aqueous potassium hydroxide (8%) [6873].
- Also refer to: [6743].

m.p. 191–192° [6873].

N.B.: A mixture of the two monobenzoates (see below its isomer) melts at about 167°.

1-[4-(Benzoyloxy)-2,6-dihydroxyphenyl]-1-propanone



$C_{16}H_{14}O_5$ mol.wt. 286.28

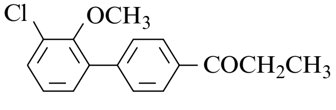
Syntheses

- Obtained by reaction of benzoyl chloride with phloropropiophenone in the presence of 1.5% aqueous potassium hydroxide (19%) [6873].
- Also refer to: [6743].

m.p. 193° [6743,6873].

N.B.: A mixture of the two monobenzoates (see above its isomer) melts at about 167°.

1-(3′-Chloro-2′-methoxy[1,1′-biphenyl]-4-yl)-1-propanone



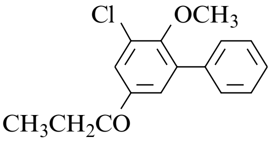
$C_{16}H_{15}ClO_2$ mol.wt. 274.75

Synthesis

- Obtained (by-product) by reaction of propionyl chloride with 3-chloro-2-methoxybiphenyl in the presence of aluminium chloride, in mixture with its 3-propionyl isomer (major product). Total yield (73%) [7695].

b.p.$_{20}$ 230–235° [7695].

1-(5-Chloro-6-methoxy[1,1′-biphenyl]-3-yl)-1-propanone



$C_{16}H_{15}ClO_2$ mol.wt. 274.75

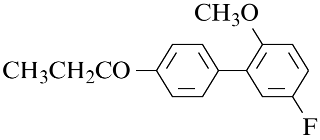
Synthesis

- Obtained (major product) by reaction of propionyl chloride with 3-chloro-2-methoxybiphenyl in the presence of aluminium chloride, in mixture with its 4′-propionyl isomer (minor product). Total yield (73%) [7695].

b.p.$_{20}$ 230–235° [7695].

1-(5′-Fluoro-2′-methoxy[1,1′-biphenyl]-4-yl)-1-propanone

[864287-73-0]



$C_{16}H_{15}FO_2$ mol.wt. 258.29

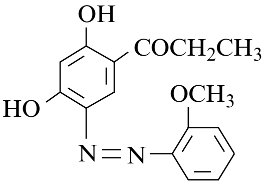
Synthesis

- Refer to: [7696].

light-yellow crystals [7696];
^{1}H NMR [7696], ^{13}C NMR [7696], MS [7696].

1-[2,4-Dihydroxy-5-[(2-methoxyphenyl)azo]phenyl]-1-propanone

[65412-10-4]



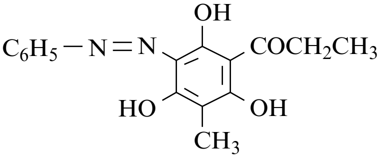
$C_{16}H_{16}N_2O_4$ mol.wt. 300.31

Synthesis

- Refer to: [7667].

USE: Dye for polypropylene fibres [7667].

1-[2,4,6-Trihydroxy-3-methyl-5-(phenylazo)phenyl]-1-propanone *(E)*



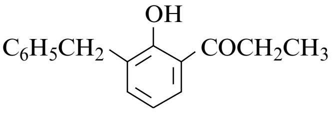
$C_{16}H_{16}N_2O_4$ mol.wt. 300.31

Syntheses

- Preparation by reaction of 1,3-diphenyltriazene (diazoaminobenzene) with 2,4,6-trihydroxy-3-methylpropiophenone [7260,7697].

m.p. 211° [7260,7697].

1-[2-Hydroxy-3-(phenylmethyl)phenyl]-1-propanone



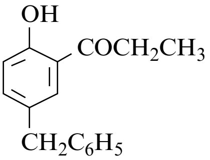
$C_{16}H_{16}O_2$ mol.wt. 240.30

Synthesis

- Obtained by Fries rearrangement of 2-benzylphenyl propionate with aluminium chloride [6351].

m.p. 53° [6351];
^{1}H NMR (Sadtler: standard n° 29909M).

1-[2-Hydroxy-5-(phenylmethyl)phenyl]-1-propanone



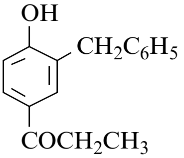
$C_{16}H_{16}O_2$ mol.wt. 240.30

Syntheses

- Obtained by alkylation of 2-hydroxypropiophenone with benzyl chloride in the presence of zinc chloride, at reflux (21%) [7698].
- Also obtained by Fries rearrangement of 4-benzylphenyl propionate (b.p.$_{20}$ 205°) with aluminium chloride [7699].

b.p.$_{0.6}$ 148–150° [7698]; $n_D^{24.5}$ = 1.5601 [7698]; m.p. 75–76° [7699].

1-[4-Hydroxy-3-(phenylmethyl)phenyl]-1-propanone



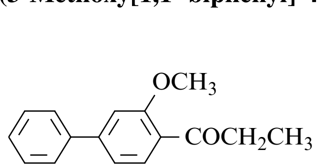
$C_{16}H_{16}O_2$ mol.wt. 240.30

Syntheses

- Obtained by Fries rearrangement of 2-benzylphenyl propionate (b.p.$_{25}$ 203°) [7699] with aluminium chloride [6351,7699].
- Also obtained by alkylation of 4-hydroxypropiophenone with benzyl chloride in the presence of zinc chloride, at reflux (18%) [7698].

b.p.$_1$ 216–220° [7698]; m.p. 157° [6351,7698], 153° [7699];
^{1}H NMR (Sadtler: standard n° 28217M), IR (Sadtler: standard n° 55289).

1-(3-Methoxy[1,1′-biphenyl]-4-yl)-1-propanone



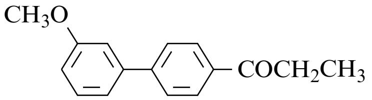
$C_{16}H_{16}O_2$ mol.wt. 240.30

Syntheses

- Preparation by reaction of propionyl chloride with 3-methoxybiphenyl [7671].
- Also obtained by reaction of ethylmagnesium bromide with 3-methoxybiphenyl-4-carbonitrile [7671].

m.p. 68.5–69° [7671].

1-(3′-Methoxy[1,1′-biphenyl]-4-yl)-1-propanone



$C_{16}H_{16}O_2$ mol.wt. 240.30

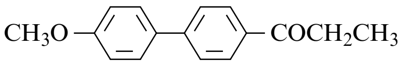
Synthesis

– Preparation by reaction of propionyl chloride with 3-methoxybiphenyl in the presence of aluminium chloride in refluxing methylene chloride for 11 h (92%) [7671].

m.p. 87–88° [7671].

1-(4′-Methoxy[1,1′-biphenyl]-4-yl)-1-propanone

[56116-76-8]



$C_{16}H_{16}O_2$ mol.wt. 240.30

Syntheses

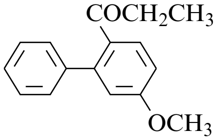
– Obtained by reaction of propionyl chloride with 4-methoxybiphenyl in the presence of aluminium chloride,

- in carbon disulfide (30%) [7675];
- in nitrobenzene, first at 0°, then at r.t. overnight (26%) [7674].

– Also refer to: [7700–7702].

m.p. 149° [7674].

1-(5-Methoxy[1,1′-biphenyl]-2-yl)-1-propanone



$C_{16}H_{16}O_2$ mol.wt. 240.30

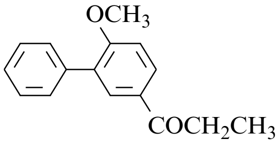
Syntheses

– Preparation by reaction of propionic anhydride with 3-methoxybiphenyl in the presence of aluminium chloride in carbon disulfide [7668,7670].

– Also obtained by reaction of ethylmagnesium bromide with 5-methoxybiphenyl-2-carbonitrile [7671].

b.p.$_{2.5}$ 171.5–173° [7671]; m.p. 72° [7668,7670].

1-(6-Methoxy[1,1′-biphenyl]-3-yl)-1-propanone



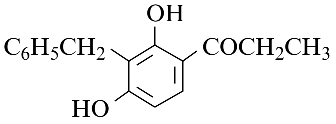
$C_{16}H_{16}O_2$ mol.wt. 240.30

Syntheses

– Obtained by reaction of propionyl chloride [7677] or propionic anhydride [7668,7670] with 2-methoxybiphenyl in the presence of aluminium chloride.

m.p. 93–94° [7668,7670], 92° [7677].

1-[2,4-Dihydroxy-3-(phenylmethyl)phenyl]-1-propanone



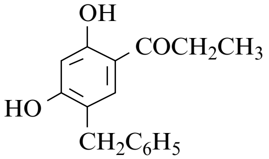
$C_{16}H_{16}O_3$ mol.wt. 256.30

Syntheses

- Obtained by O-debenzylation of 2-hydroxy-3-benzyl-4-benzyloxypropiophenone with concentrated hydrochloric acid in refluxing acetic acid for 2 h (27%) [7703].
- Also obtained by reaction of propionitrile with 2-benzylresorcinol (Hoesch reaction) (12%) [7703].

m.p. 157–158° [7703].

1-[2,4-Dihydroxy-5-(phenylmethyl)phenyl]-1-propanone



$C_{16}H_{16}O_3$ mol.wt. 256.30

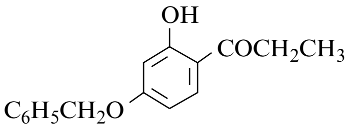
Synthesis

- Obtained by Fries rearrangement of 2,4-dihydroxy-diphenylmethane dipropionate (b.p.$_{16}$ 240°) with aluminium chloride [7699].

m.p. 92° [7699].

1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1-propanone

[63411-94-9]



$C_{16}H_{16}O_3$ mol.wt. 256.30

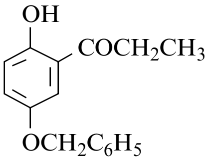
Syntheses

- Preparation by partial benzylation of respropiophenone with benzyl halide (unspecified) [6380] or with benzyl chloride [7135],
  - in the presence of potassium carbonate in refluxing acetone for 2 h (75%) [7135], for 5 h (45%) [6380] or for 8 h (47%) [7703];
  - in the presence of methanolic potassium hydroxide, first at r.t. overnight, then at reflux for 5 h [7703].

m.p. 113–114° [6380,7703], 112–114° [7135];
^{1}H NMR [6380], IR [6380]; TLC [6380].

1-[2-Hydroxy-5-(phenylmethoxy)phenyl]-1-propanone

[54437-05-7]



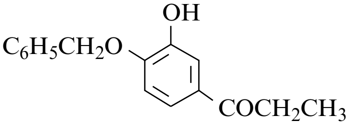
$C_{16}H_{16}O_3$ mol.wt. 256.30

Synthesis

- Refer to: [6795].

1-[3-Hydroxy-4-(phenylmethoxy)phenyl]-1-propanone

[178375-14-9] $C_{16}H_{16}O_3$ mol.wt. 256.30

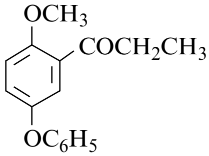


Syntheses

- Refer to: [6405,6406].

1-(2-Methoxy-5-phenoxyphenyl)-1-propanone

[502924-47-2] $C_{16}H_{16}O_3$ mol.wt. 256.30

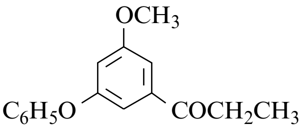


Synthesis

- Refer to: [6958].

1-(3-Methoxy-5-phenoxyphenyl)-1-propanone

[502924-51-8] $C_{16}H_{16}O_3$ mol.wt. 256.30

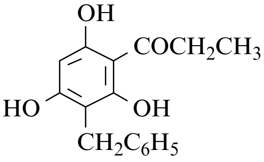


Synthesis

- Refer to: [6958].

1-[2,4,6-Trihydroxy-3-(phenylmethyl)phenyl]-1-propanone

$C_{16}H_{16}O_4$ mol.wt. 272.30



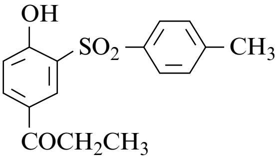
Synthesis

- Refer to: [7458].

^{13}C NMR [7458].

1-[4-Hydroxy-3-[(4-methylphenyl)sulfonyl]phenyl]-1-propanone

[67474-14-0] $C_{16}H_{16}O_4S$ mol.wt. 304.37



Synthesis

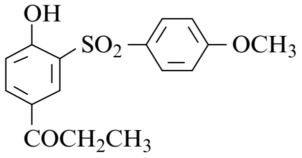
- Preparation by treatment of 4-propionylphenyl p-toluenesulfonate (1 mol) with aluminium chloride (3 mol) at 140° for 3–4 h (84–100%) [7665].

m.p. 84° [7665].

BIOLOGICAL ACTIVITY: Bactericide (no data) [7665].

1-[4-Hydroxy-3-[(4-methoxyphenyl)sulfonyl]phenyl]-1-propanone

[67474-18-4]



$C_{16}H_{16}O_5S$ mol.wt. 320.37.

Synthesis

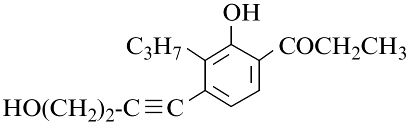
- Preparation by treatment of 4-propionylphenyl p-methoxyphenylsulfonate (1 mol) with aluminium chloride (3 mol) at 140° for 3–4 h (84–100%) [7665].

m.p. 64–65° [7665].

BIOLOGICAL ACTIVITY: Bactericide (no data) [7665].

1-[2-Hydroxy-4-(4-hydroxy-1-butynyl)-3-propylphenyl]-1-propanone

[194792-37-5]



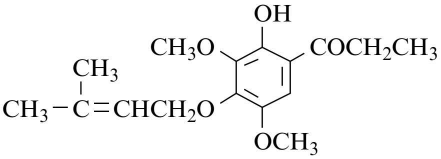
$C_{16}H_{20}O_3$ mol.wt. 260.33

Synthesis

- Refer to: [7452].

1-[2-Hydroxy-3,5-dimethoxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-1-propanone

[99965-00-1]



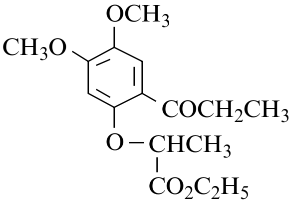
$C_{16}H_{22}O_5$ mol.wt. 294.35

Isolation from natural sources

- From *Leucanthemopsis pallida* subsp. *flaveola* (Compositae) (compound **2**) [7440].

^{1}H NMR [7440], ^{13}C NMR [7440], IR [7440], UV [7440].

2-[4,5-Dimethoxy-2-(1-oxopropyl)phenoxy]propanoic acid ethyl ester



$C_{16}H_{22}O_6$ mol.wt. 310.35

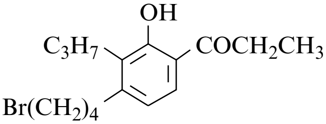
Synthesis

- Obtained by reaction of ethyl α-bromopropionate with 2-hydroxy-4,5-dimethoxypropiophenone in the presence of potassium hydroxide in refluxing acetone for 6–10 h (84%) [7400].

m.p. 62–63° [7400].

1-[4-(4-Bromobutyl)-2-hydroxy-3-propylphenyl]-1-propanone

[194792-39-7] $C_{16}H_{23}BrO_2$ mol.wt. 327.26

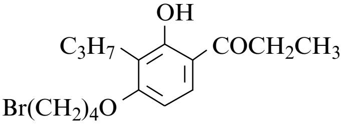


Synthesis

– Refer to: [7452].

1-[4-(4-Bromobutoxy)-2-hydroxy-3-propylphenyl]-1-propanone

[194793-05-0] $C_{16}H_{23}BrO_3$ mol.wt. 343.26

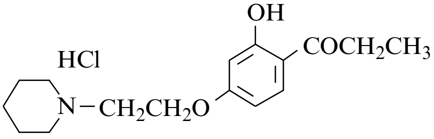


Synthesis

– Refer to: [7452].

1-[2-Hydroxy-4-(2-piperidinoethoxy)phenyl]-1-propanone *(Hydrochloride)*

[20800-22-0] $C_{16}H_{23}NO_3$, HCl mol.wt. 313.82



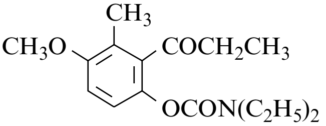
Synthesis

– Obtained by adding a solution of o-hydroxy-propiophenone and sodium ethoxide in ethanol to piperidine hydrochloride and the mixture refluxed 3 h [7691].

m.p. 181–183° [7691]; LD_{50} [7691].

4-Methoxy-3-methyl-2-(1-oxopropyl)phenyl diethylcarbamate

$C_{16}H_{23}NO_4$ mol.wt. 293.16



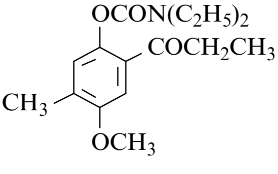
Synthesis

– Obtained by lithiation-zinc transmetallation of 4-methoxy-3-methylphenyl diethylcarbamate followed by cross coupling with propionyl chloride (6%) [7500].

^{1}H NMR [7500].

4-Methoxy-5-methyl-2-(1-oxopropyl)phenyl diethylcarbamate

$C_{16}H_{23}NO_4$ mol.wt. 293.16



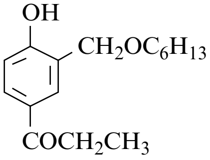
Synthesis

– Obtained by lithiation-zinc transmetallation of 4-methoxy-3-methylphenyl diethylcarbamate followed by cross coupling with propionyl chloride (76%) [7500].

m.p. 67–68° [7500];
^{1}H NMR [7500], ^{13}C NMR [7500], IR [7500], MS [7500].

1-[3-[(Hexyloxy)methyl]-4-hydroxyphenyl]-1-propanone

[114477-31-5] $C_{16}H_{24}O_3$ mol.wt. 264.36



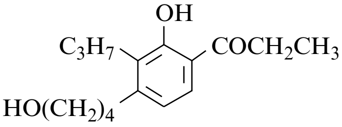
Syntheses

- Obtained by reaction of 4-hydroxy-3-chloromethylpropiophenone with 1-hexanol in the presence of sodium bicarbonate [7392].
- Also refer to: [7610].

USE: Pharmaceutical intermediate [7392].

1-[2-Hydroxy-4-(4-hydroxybutyl)-3-propylphenyl]-1-propanone

[194792-38-6] $C_{16}H_{24}O_3$ mol.wt. 264.36

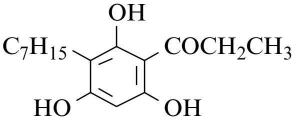


Synthesis

- Refer to: [7452].

1-(3-Heptyl-2,4,6-trihydroxyphenyl)-1-propanone

[74477-94-4] $C_{16}H_{24}O_4$ mol.wt. 280.36



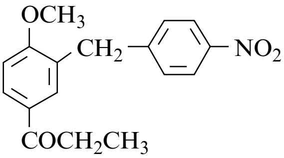
Synthesis

- Preparation [7625] (Japanese patent).

USE: Fungicide [7625].

1-[4-Methoxy-3-[(4-nitrophenyl)methyl]phenyl]-1-propanone

[188984-62-5] $C_{17}H_{17}NO_4$ mol.wt. 299.33

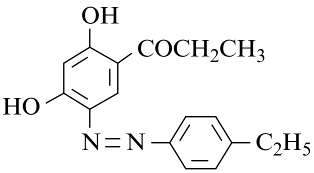


Synthesis

- Refer to: [7704].

1-[5-[(4-Ethylphenyl)azo]-2,4-dihydroxyphenyl]-1-propanone

[65412-09-1] $C_{17}H_{18}N_2O_3$ mol.wt. 298.34

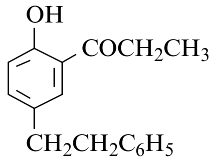


Synthesis

- Refer to: [7667].

USE: Dye for polypropylene fibres [7667].

1-[2-Hydroxy-5-(phenylethyl)phenyl]-1-propanone



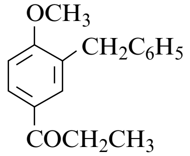
$C_{17}H_{18}O_2$ mol.wt. 254.33

Synthesis

- Preparation by Fries rearrangement of 4-(phenylethyl) phenyl propionate (b.p.$_{18}$ 220°; m.p. 38–39°) with aluminium chloride (70%) [7699].

b.p.$_{18}$ 220° [7699]; m.p. 47–48° [7699].

1-[4-Methoxy-3-(phenylmethyl)phenyl]-1-propanone



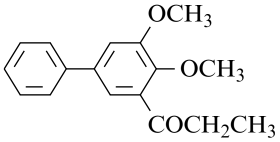
$C_{17}H_{18}O_2$ mol.wt. 254.33

Syntheses

- Obtained by reaction of dimethyl sulfate with 3-benzyl-4-hydroxypropiophenone in the presence of aqueous sodium hydroxide solution [7699].
- Also refer to: [7698].

b.p.$_{0.5}$ 187–188° [7698]; m.p. 120° [7698], 119° [7699].

1-(4,5-Dimethoxy[1,1′-biphenyl]-3-yl)-1-propanone



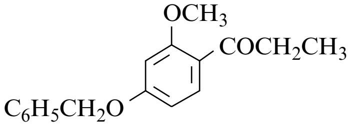
$C_{17}H_{18}O_3$ mol.wt. 270.33

Synthesis

- Obtained by reaction of propionic anhydride with 3,4-dimethoxydiphenyl in the presence of aluminium chloride in carbon disulfide (10%) [7668].

b.p.$_{3}$ 228–230° [7668]; m.p. 113° [7668].

1-[2-Methoxy-4-(phenylmethoxy)phenyl]-1-propanone



$C_{17}H_{18}O_3$ mol.wt. 270.33

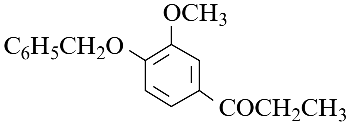
Synthesis

- Preparation by reaction of methyl iodide with 4-(benzyloxy)-2-hydroxypropiophenone in the presence of potassium carbonate in boiling acetone [7135].

m.p. 59–60° [7135].

1-[3-Methoxy-4-(phenylmethoxy)phenyl]-1-propanone

[1835-15-0]



$C_{17}H_{18}O_3$ mol.wt. 270.33

Syntheses

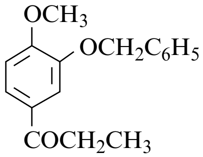
- Obtained by reaction of sodium methoxide with 1-benzyloxy-4-(1,2-dibromopropyl)-2-methoxybenzene [7245].

- Also obtained by reaction of ethylmagnesium bromide with o-benzylvanillin (85%) [7705].
- Also obtained by reaction of benzyl chloride with 4-hydroxy-3-methoxypropiophenone in the presence of potassium carbonate and sodium iodide in refluxing dilute ethanol for 5 h (92%) [7163].
- Also refer to: [6406,7244].

m.p. 99–101° [7705], 98–101° [7163], 93° [7245], 91–93° [7194]; ^{1}H NMR [7705]; Polarographic half-wave potential [7706].

1-[4-Methoxy-3-(phenylmethoxy)phenyl]-1-propanone

[83674-89-9] $C_{17}H_{18}O_3$ mol.wt. 270.33

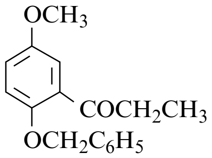


Syntheses

- Refer to: [7707,7708].

1-[5-Methoxy-2-(phenylmethoxy)phenyl]-1-propanone

$C_{17}H_{18}O_3$ mol.wt. 270.33



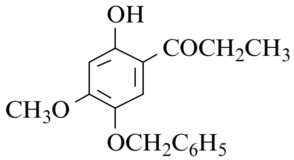
Synthesis

- Obtained by reaction of benzyl chloride with 2-hydroxy-5-methoxypropiophenone in the presence of potassium hydroxide in methanol [7149].

b.p.$_1$ 194–196° [7149]; m.p. 47° [7149].

1-[2-Hydroxy-4-methoxy-5-(phenylmethoxy)phenyl]-1-propanone

[3904-19-6] $C_{17}H_{18}O_4$ mol.wt. 286.33



Synthesis

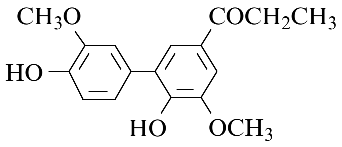
- Obtained by reaction of benzyl chloride with 2,5-dihydroxy-4-methoxypropiophenone in the presence of sodium ethoxide in refluxing ethanol for 12 h (88%) [7254].

m.p. 99° [7254].

1-(4′,6-Dihydroxy-3′,5-dimethoxy[1,1′-biphenyl]-3-yl)-1-propanone

4′-Hydroxy-3′-(4-hydroxy-3-methoxyphenyl)-5′-methoxypropiophenone

[18593-00-5] $C_{17}H_{18}O_5$ mol.wt. 302.33



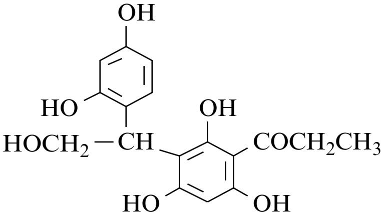
Synthesis

- Obtained by saponification of 3,3′-dimethoxy-4-hydroxy-2′-propionoxy-5′-propionylbiphenyl [7161].

^{1}H NMR [7161].

1-[3-[1-(2,4-Dihydroxyphenyl)-2-hydroxyethyl]-2,4,6-trihydroxyphenyl]-1-propanone

[128347-39-7]

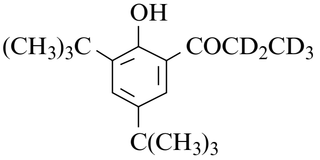


$C_{17}H_{18}O_7$ mol.wt. 334.33

Syntheses

- Refer to: [7709,7710] (Japanese patents).

1-[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]-1-propanone-*2,2,3,3,3-d*$_5$



$C_{17}H_{21}D_5O_2$ mol.wt. 267.42

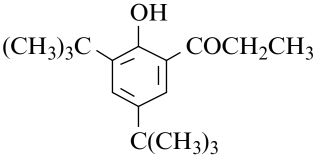
Synthesis

- Refer to: [7711].

Radical ion (1^-) [65282-39-5], ESR spectrum [7711].
Ion (1^-), radical ion (1^-) [72051-73-1], ESR spectrum [6350].

1-[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]-1-propanone

[40662-81-5]



$C_{17}H_{26}O_2$ mol.wt. 262.39

Syntheses

- Preparation by Fries rearrangement of 2,4-di-tert-butyl-phenyl propionate in nitromethane,
  - with titanium tetrachloride at 20° for a week (69%) [7712];
  - with stannic chloride at 20° for 24 h (50%) [6570,7712].
- Also obtained by Fries rearrangement of 2,6-di-tert-butyl-phenyl propionate with titanium tetrachloride in nitromethane at 20° for a week (17%) [7712]. There is a tert-butyl group migration on the ring.
- Also obtained by Fries rearrangement of 2,4,6-tri-tert-butylphenyl propionate in nitromethane,
  - with titanium tetrachloride at 20° for 8 days (25%) [7712];
  - with antimony pentachloride at 20° for 2 h (38%) [7712].

  In these above two reactions, there are tert-butyl group eliminations.
- Also obtained (by-product) by Fries rearrangement of 2-tert-butylphenyl propionate in the presence of aluminium bromide, antimony pentachloride, antimony pentafluoride or boron trifluoride in nitromethane at 20° for 24 h (5–8%) [7576].

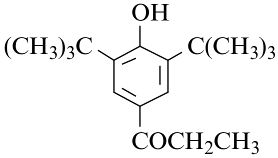
b.p.$_{18-20}$ 155–156° [7712]; m.p. 68° [7712];

^{1}H NMR (Sadtler: standard n° 16617M), IR (Sadtler: standard n° 44743) [7712], UV [7712].

Notes: Radical ion (1^-) [72051-72-0], ESR spectrum [6350]; radical ion (1^-) [65282-38-4], ESR spectrum [7711].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-propanone

[14035-34-8] $C_{17}H_{26}O_2$ mol.wt. 262.39



Syntheses

- Preparation by Friedel–Crafts acylation of 2, 6-di-tert-butylphenol with propionyl chloride in the presence of aluminium chloride,
  - at 0° for 45 min [7713] according to the procedure [7714];
  - at −10° for 1–13 min (92%) [7715];
  - at 20° for 20 min, followed by hydrolysis of the formed keto ester (85%, m.p. 46.5–47.5°) [7716].
- Preparation by Friedel–Crafts acylation of 2,6-di-tert-butylphenol with propionyl chloride in the presence of titanium tetrachloride in ethylene dichloride [7717].
- Preparation by Fries rearrangement of 2,6-di-tert-butylphenyl propionate with titanium tetrachloride in nitromethane at 20° for a week (66%) [7712].
- Also obtained by hydrolysis of 2,6-di-tert-butyl-4-ethylmethoxymethylenequinone (m.p. 98–101°) with hydrochloric acid (98%) [7718].
- Also obtained by oxidation of (3,5-di-tert-butyl-4-hydroxyphenyl)-ethylcarbinol with DDQ in dioxane at r.t. for 16 h (94%) [7306].
- Also obtained from 4-bromo-2,6-di-tert-butyl-4-(1-hydroxypropyl)-2,5-cyclohexadienone (m.p. 51°) [7719],
  - by treatment with 80% aqueous acetic acid or ethanol at 60° for 15 min (92%) [7719];
  - by treatment in petroleum ether at r.t. for 48 h (92%) [7719];
  - by treatment with pyridine in methanol at r.t. for 6 h (18%) [7720];
  - by treatment with 5% methanolic sulfuric acid at r.t. for 18 h in the presence of silver nitrate (19%) or without this one (9%) [7721].
- Also obtained from 2,6-di-tert-butyl-4-(1-methoxypropyl)phenol (m.p. 45°) by bromine oxidation on heating in 80% aqueous acetic acid at 60° for 15 min (64%) [7718].
- Also obtained by treatment of 4-bromo-2,6-di-tert-butyl-4-(1-bromopropyl)-2,5-cyclohexadienone (m.p. 75–77°) with a water-petroleum ether mixture (69%) [7719].
- Also obtained from 2,6-di-tert-butyl-4-(1-hydroxypropyl)phenol (m.p. 77°) by bromine oxidation on heating in 80% aqueous acetic acid (37%) or in petroleum ether with ice cooling (21%) [7719].
- Also obtained from 2,6-di-tert-butyl-4-ethylmethylenequinone (m.p. 37°) by bromine oxidation on heating in 80% aqueous acetic acid at 80° for 15 min (25%) [7719].

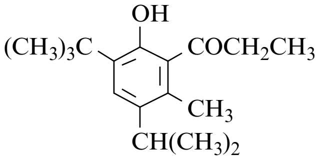
- Also obtained (trace) by oxidation of 2,6-di-tert-butyl-4-propylphenol with cumene hydroperoxide in the presence of cobalt phthalate in cumene under air bubbling at 80–100° for 100 h (<4%) [7722].
- Also refer to: [6485,7723–7730].

m.p. 137° [7712,7718,7719], 136–137° [7713,7716,7721], 136° [7722], 134–135° [7715];
^{1}H NMR (Sadtler: standard n° 18465M) [7731], IR (Sadtler: standard n° 44745) [7712],
UV [7712], MS [7732].

BIOLOGICAL ACTIVITY: Antiinflammatory [7717], anticholesteremic [7733].

1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methyl-5-(1-methylethyl)phenyl]-1-propanone

[121194-65-8] $C_{17}H_{26}O_2$ mol.wt. 262.39



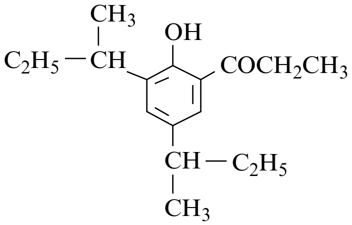
Synthesis

- Preparation by Fries rearrangement of 2-tert-butyl-5-methyl-4-isopropylphenyl propionate with titanium tetrachloride in chlorobenzene at 100° for 2 h (51%) [7031].

m.p. 73° [7031];
^{1}H NMR (Sadtler: standard n° 52744M) [7031], IR (Sadtler: standard n° 79803K) [7031], UV [7031], MS [7031].

1-[2-Hydroxy-3,5-di(1-methylpropyl)phenyl]-1-propanone

[107621-04-5] $C_{17}H_{26}O_2$ mol.wt. 262.39



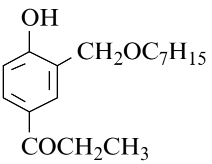
Synthesis

- Preparation by reaction of propionic acid with 2,4-di-sec-butylphenol (b.p.$_1$ 86–89°) in the presence of boron trifluoride (67%) [6509].

b.p.$_1$ 106–109° [6509];
n_D^{25} = 1.5153 [6509].

1-[3-(Heptyloxy)methyl-4-hydroxyphenyl]-1-propanone

[136715-27-0] $C_{17}H_{26}O_3$ mol.wt. 278.39



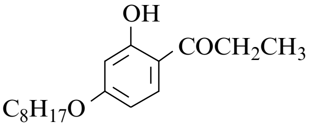
Synthesis

- Obtained by reaction of 4-hydroxy-3-chloromethylpropiophenone with 1-heptanol in the presence of sodium bicarbonate [7392].

USE: Pharmaceutical intermediate [7392].

1-[2-Hydroxy-4-(octyloxy)phenyl]-1-propanone

[63411-92-7]



$C_{17}H_{26}O_3$ mol.wt. 278.39

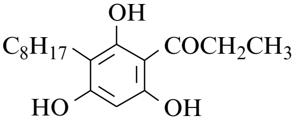
Synthesis

- Preparation by reaction of an octyl halide (unspecified) with respropiophenone in the presence of potassium carbonate in refluxing acetone for 5 h (60%) [6380].

viscous oil, purified by preparative TLC [6380];
^{1}H NMR [6380], IR [6380]; TLC [6380].

1-(2,4,6-Trihydroxy-3-octylphenyl)-1-propanone

[74477-95-5]



$C_{17}H_{26}O_4$ mol.wt. 294.39

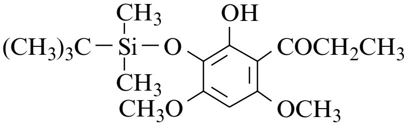
Synthesis

- Preparation [7625] (Japanese patent).

USE: Fungicide [7625].

1-[3-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-2-hydroxy-4,6-dimethoxyphenyl]-1-propanone

[293744-05-5]



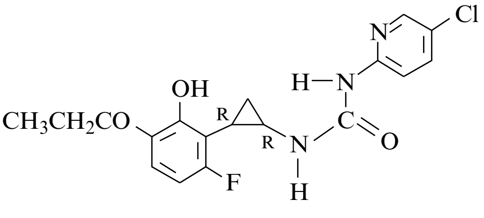
$C_{17}H_{28}O_5Si$ mol.wt. 340.49

Syntheses

- Preparation by reaction of tert-butyldimethylsilyl chloride with 2,3-dihydroxy-4,6-dimethoxy-propiophenone in the presence of imidazole in DMF (95%) [7436,7437].

N-(5-Chloro-2-pyridinyl)-N′-[(1*R*,2*R*)-2-[6-fluoro-2-hydroxy-3-(1-oxopropyl) phenyl]-cyclopropyl]-urea *(MSC 197)*

[247230-90-6]



$C_{18}H_{17}ClFN_3O_3$ mol.wt. 377.80

Syntheses

- Obtained by treatment of its methyl ether with boron trichloride in methylene chloride [6522].
- Also refer to: [6521].

m.p. 196.5–198.5° [6522];
$(\alpha)_D^{22} = -176.8°$ (CH_2Cl_2) [6522];
^{1}H NMR [6522].

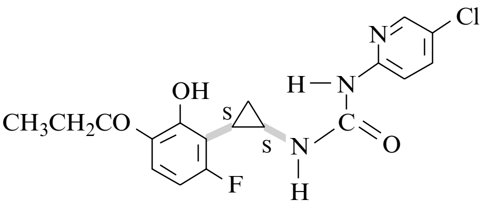
N-(5-Chloro-2-pyridinyl)-N′-[(1*S*,2*S*)-2-[6-fluoro-2-hydroxy-3-(1-oxopropyl)phenyl]-cyclopropyl]-urea *(MSC 198)*

[247230-91-7]

$C_{18}H_{17}ClFN_3O_3$ mol.wt. 377.80

Syntheses

– Refer to: [6521,6522,7734].



m.p. 196–197° [6522];
$(\alpha)_D^{22}$ = +176.8° (CH_2Cl_2) [6522];
^{1}H NMR [6522].

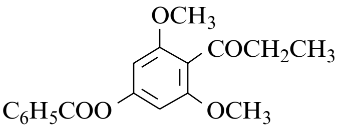
1-[4-(Benzoyloxy)-2,6-dimethoxyphenyl]-1-propanone

$C_{18}H_{18}O_5$ mol.wt. 314.34

Syntheses

– Obtained by reaction of 4-(benzoyloxy)-2,6-di-hydroxypropiophenone with methyl iodide in the presence of potassium carbonate in boiling acetone for 30 h [6873].

– Also refer to: [6743].



m.p. 103° [6873].

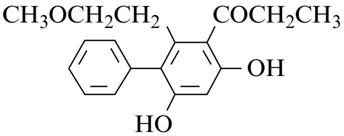
1-[4,6-Dihydroxy-2-(2-methoxyethyl)[1,1′-biphenyl]-3-yl]-1-propanone

[860152-82-5]

$C_{18}H_{20}O_4$ mol.wt. 300.35

Synthesis

– Refer to: [7664].



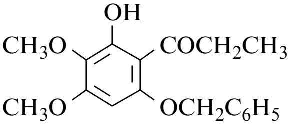
1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-1-propanone

[134082-01-2]

$C_{18}H_{20}O_5$ mol.wt. 316.35

Synthesis

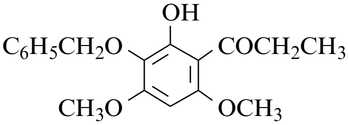
– Preparation by reaction of benzyl chloride with 2,6-di-hydroxy-3,4-dimethoxypropiophenone in N,N-dimethyl-formamide in the presence of potassium carbonate at 150–160° for 10–20 min, followed by treatment of the dibenzyl ether obtained by concentrated hydrochloric acid in acetic acid at r.t. for 2–3 h (85%) [7442].



m.p. 111.5–113° [7442]; ^{1}H NMR [7442].

1-[2-Hydroxy-4,6-dimethoxy-3-(phenylmethoxy)phenyl]-1-propanone

[293744-03-3] $C_{18}H_{20}O_5$ mol.wt. 316.35



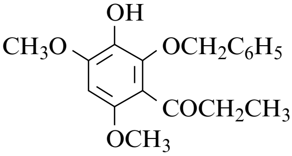
Syntheses

- Preparation by reaction of benzyl bromide with 2,3-dihydroxy-4,6-dimethoxypropiophenone in the presence of Hünig base in methylene chloride for 1 day (71%) [7436,7437].

m.p. 68° [7436,7437].

1-[3-Hydroxy-4,6-dimethoxy-2-(phenylmethoxy)phenyl]-1-propanone

[293744-04-4] $C_{18}H_{20}O_5$ mol.wt. 316.35



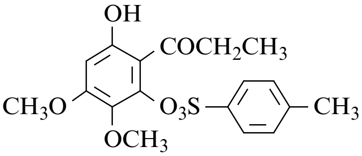
Syntheses

- Obtained (by-product) by reaction of benzyl bromide with 2,3-dihydroxy-4,6-dimethoxypropiophenone in the presence of Hünig base in methylene chloride for 1 day (9%) [7436,7437].

m.p. 132° [7436,7437].

1-[6-Hydroxy-3,4-dimethoxy-2-[[(4-methylphenyl)sulfonyl]oxy]phenyl]-1-propanone

[134081-85-9] $C_{18}H_{20}O_7S$ mol.wt. 380.42



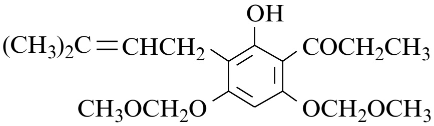
Synthesis

- Preparation by partial demethylation of 3,4,6-tri-methoxy-2-(tosyloxy)propiophenone (m.p. 121–123°) with aluminium bromide in acetonitrile at r.t. for 2–3 h (88%) [7442].

m.p. 120.5–121° [7442]; ^1H NMR [7442].

1-[2-Hydroxy-4,6-bis(methoxymethoxy)-3-(3-methyl-2-butenyl)phenyl]-1-propanone

[199166-83-1] $C_{18}H_{26}O_6$ mol.wt. 338.40



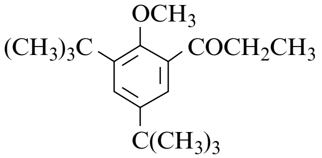
Synthesis

- Refer to: [7735].

USE: Preparation of flavones and naphthalenes as estrogens [7735].

1-[3,5-Bis(1,1-dimethylethyl)-2-methoxyphenyl]-1-propanone

[40662-83-7] $C_{18}H_{28}O_2$ mol.wt. 276.42



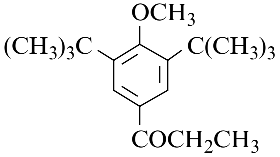
Synthesis

– Obtained by reaction of methyl iodide with the sodium salt of 3,5-di-tert-butyl-2-hydroxypropiophenone in THF [7712].

b.p.$_{15}$ 163° [7712]; m.p. 55° [7712];
^{1}H NMR (Sadtler: standard n° 16618M), IR (Sadtler: standard n° 44744) [7712], UV [7712].

1-[3,5-Bis(1,1-dimethylethyl)-4-methoxyphenyl]-1-propanone

[40662-88-2] $C_{18}H_{28}O_2$ mol.wt. 276.42



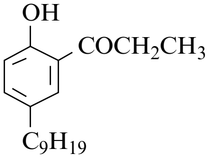
Synthesis

– Preparation by reaction of dimethyl sulfate with 3,5-di-tert-butyl-4-hydroxypropiophenone in the presence of sodium hydroxide in dilute methanol [7712].

yellow oil [7712]; $n_D^{20} = 1.5165$ [7712]; IR [7712], UV [7712].

1-(2-Hydroxy-5-nonylphenyl)-1-propanone

[908130-90-5] $C_{18}H_{28}O_2$ mol.wt. 276.42

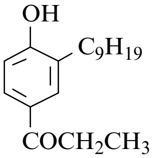


Synthesis

– Obtained by acylation of 4-nonylanisole in the presence of aluminium halide or boron halide [7736].

1-(4-Hydroxy-3-nonylphenyl)-1-propanone

[104557-28-0] $C_{18}H_{28}O_2$ mol.wt. 276.42

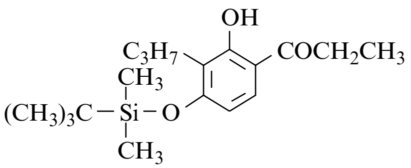


Synthesis

– Refer to: [7737] (Japanese patent).

1-[4-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-2-hydroxy-3-propylphenyl]-1-propanone

[194854-84-7] $C_{18}H_{30}O_3Si$ mol.wt. 322.52



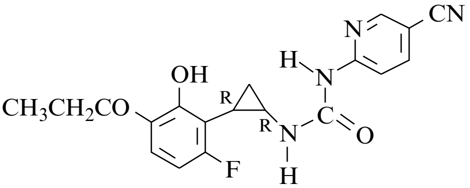
Syntheses

– Refer to: [7476,7477].

N-(5-Cyano-2-pyridinyl)-N′-[(1*R*,2*R*)-2-[6-fluoro-2-hydroxy-3-(1-oxopropyl) phenyl]-cyclopropyl]-urea

[231957-52-1]

$C_{19}H_{17}FN_4O_3$ mol.wt. 368.37



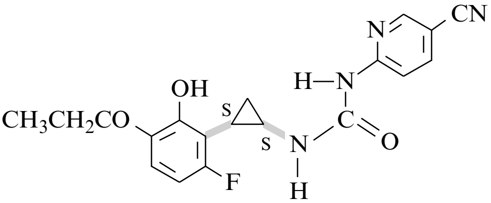
Synthesis

– Refer to: [6990].

N-(5-Cyano-2-pyridinyl)-N′-[(1*S*,2*S*)-2-[6-fluoro-2-hydroxy-3-(1-oxopropyl) phenyl]-cyclopropyl]-urea *(MIV 150)*

[231957-54-3]

$C_{19}H_{17}FN_4O_3$ mol.wt. 368.37



Syntheses

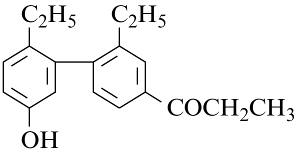
– Refer to: [6522,6990,7734, 7738].

^{1}H NMR [6522].

1-(2,2′-Diethyl-5′-hydroxy[1,1′-biphenyl]-4-yl)-1-propanone

[540495-32-7]

$C_{19}H_{22}O_2$ mol.wt. 282.32



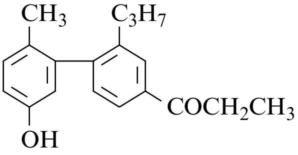
Synthesis

– Refer to: [7289].

1-(5′-Hydroxy-2′-methyl-2-propyl[1,1′-biphenyl]-4-yl)-1-propanone

[540495-19-0]

$C_{19}H_{22}O_2$ mol.wt. 282.32



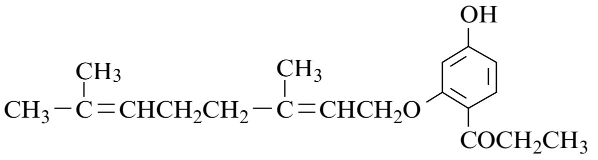
Synthesis

– Refer to: [7289].

1-[2-[(3,7-Dimethyl-2,6-octadienyl)oxy]-4-hydroxyphenyl]-1-propanone *(E)*

[87108-26-7]

$C_{19}H_{26}O_3$
mol.wt. 302.42



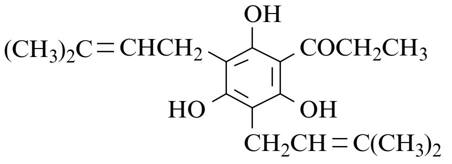
Synthesis

– Refer to: [7617] (Japanese patent).

1-[2,4,6-Trihydroxy-3,5-bis(3-methyl-2-butenyl)phenyl]-1-propanone

[50874-47-0]

$C_{19}H_{26}O_4$ mol.wt. 318.41



Syntheses

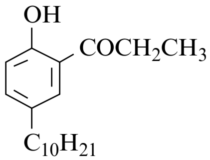
– Obtained by reaction of 1-bromo-3-methyl-2-butene with phloropropiophenone in the presence of the weakly basic resin DeAcidite H-IP (OH^- form) in boiling benzene for 16 h (12%) [7739]. The yield was similar to the one obtained by using 2-methyl-3-buten-2-ol and a Lewis acid but the isolation was simpler.

– Also refer to: [7740].

m.p. 83° [7739].

1-(5-Decyl-2-hydroxyphenyl)-1-propanone

[102020-39-3] $C_{19}H_{30}O_2$ mol.wt. 290.45



Synthesis

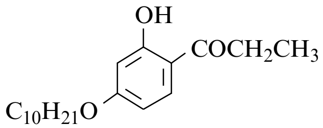
– Preparation by reaction of propionic acid with 4-decylphenol (m.p. 55–56.5°) in the presence of boron trifluoride (81%) [6509].

b.p.$_{1}$ 161–165° [6509]; n_D^{25} = 1.5072 [6509].

1-[4-(Decyloxy)-2-hydroxyphenyl]-1-propanone

[101002-30-6]

$C_{19}H_{30}O_3$ mol.wt. 306.45



Syntheses

– Obtained by Fries rearrangement of 3-(decyloxy)phenyl propionate [7685].

– Also obtained by reaction of n-decyl bromide with respropiophenone in the presence of potassium carbonate in refluxing acetone [7237].

– Also refer to: [7688].

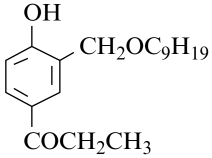
m.p. 30° [7237].

Oxime [101002-16-8] $C_{19}H_{31}NO_3$ mol.wt. 321.46 (m.p. 58–59°) [7237,7641,7686].

USE: Antiallergic and antiinflammatory agent [7688], lipoxygenase inhibitor [7685].

1-[4-Hydroxy-3-[(nonyloxy)methyl]phenyl]-1-propanone

[136715-28-1] $C_{19}H_{30}O_3$ mol.wt. 306.45



Synthesis

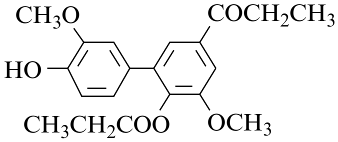
- Preparation [7392] (Czech patent).

USE: As pharmaceutical intermediate [7392].

1-(4′-Hydroxy-3′,5-dimethoxy-6-propionyloxy[1,1′-biphenyl]-3-yl)-1-propanone

4′-Hydroxy-3′-(4-hydroxy-3-methoxyphenyl)-5′-methoxypropiophenone 4′-propionate
3,3′-Dimethoxy-4-hydroxy-2′-propionoxy-5′-propionylbiphenyl

[18592-99-9] $C_{20}H_{22}O_6$ mol.wt. 358.39



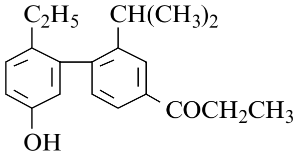
Synthesis

- Isolation from the dehydrogenation resin of propioguaiacone through preparative TLC (compound **12**) (6%) [7161].

^{1}H NMR [7161], IR [7161], UV [7161].

1-[2′-Ethyl-5′-hydroxy-2-(1-methylethyl)[1,1′-biphenyl]-4-yl]-1-propanone

[540495-43-0] $C_{20}H_{24}O_2$ mol.wt. 296.41

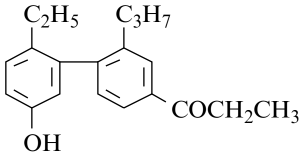


Synthesis

- Refer to: [7289].

1-(2′-Ethyl-5′-hydroxy-2-propyl[1,1′-biphenyl]-4-yl)-1-propanone

[540495-37-2] $C_{20}H_{24}O_2$ mol.wt. 296.41

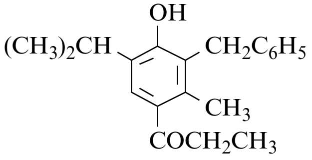


Syntheses

- Refer to: [7289,7741].

1-[4-Hydroxy-2-methyl-5-(1-methylethyl)-3-(phenylmethyl)phenyl]-1-propanone

[102168-44-5] $C_{20}H_{24}O_2$ mol.wt. 296.41



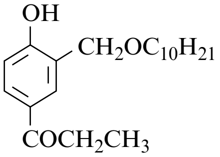
Synthesis

– Obtained (by-product) by benzylation of propionyl-4-thymol (m.p. 111°) with benzyl chloride in the presence of zinc chloride in refluxing chloroform for 22 h (4%) [7585].

b.p.$_{22}$ 244–246° [7585]; n_D^{22} = 1.5640 [7585].

1-[3-[(Decyloxy)methyl]-4-hydroxyphenyl]-1-propanone

[136715-29-2] $C_{20}H_{32}O_3$ mol.wt. 320.47



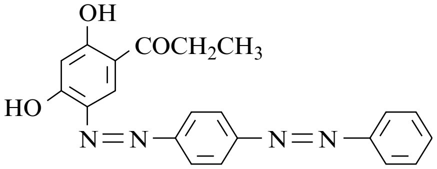
Synthesis

– Preparation [7392] (Czech patent).

USE: As pharmaceutical intermediate [7392].

1-[2,4-Dihydroxy-5-[[4-(phenylazo)phenyl]azo]phenyl]-1-propanone

[65561-66-2] $C_{21}H_{18}N_4O_3$ mol.wt. 374.40



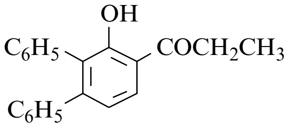
Synthesis

– Preparation, refer to: [7742,7743].

USE: For dyeing nickel-containing polypropylene fibres [7742,7743].

1-(3′-Hydroxy[1,1′:2′,1″-terphenyl]-4′-yl)-1-propanone

[777067-73-9] $C_{21}H_{18}O_2$ mol.wt. 302.37



Synthesis

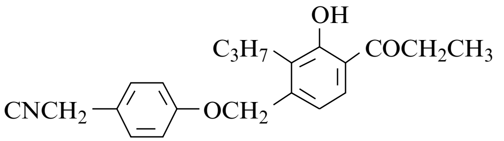
– Obtained by treatment of 2,3-dichloro-6-propionylphenol (3.2 mmol) in dioxane with cesium carbonate (5 mmol), phenylboronic acid (4.8 mmol), tris(dibenzylideneacetone)-dipalladium (0) (0.1 mmol) and a 20% solution of tricyclo-hexylphosphine (0.25 mmol) in toluene. The resulting mixture was heated at 80° for 24 h (48%) [6385].

m.p. 129–130° [6385]; ^{1}H NMR [6385].

Oxime $C_{21}H_{19}NO_2$ mol.wt. 317.39 (m.p. 220–221°) [6385].

4-[[3-Hydroxy-4-(1-oxopropyl)-2-propylphenyl]methoxy]benzeneacetonitrile

[107223-73-4]



$C_{21}H_{23}NO_3$ mol.wt. 337.42

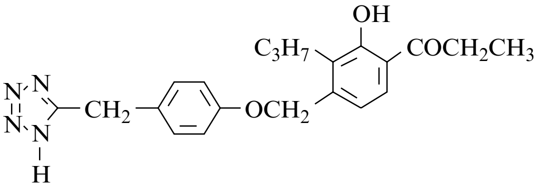
Synthesis

- Obtained by adding 1-[4-(chloromethyl)-2-hydroxy-3-propylphenyl]-1-propanone, then sodium iodide to a solution of 4-hydroxybenzeneacetonitrile and potassium tert-butoxide in DMF cooled with an ice-water bath, then stirring at r.t. for 24 h (51%) [7564].

m.p. 122–126° [7564].

1-[2-Hydroxy-3-propyl-4-[[4-(1*H*-tetrazol-5-ylmethyl)phenoxy]methyl] phenyl]- 1-propanone

[107223-61-0]



$C_{21}H_{24}N_4O_3$ mol.wt; 380.45

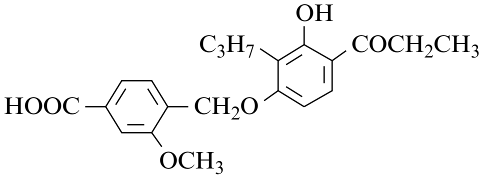
Synthesis

- Obtained by reaction of sodium azide with 4-[(4-propionyl-2-propyl-3-hydroxyphenyl)methoxy] benzene-acetonitrile in the presence of ammonium chloride in DMF at 105° for 20 h (32%) [7564].

m.p. 162–166° [7564].

4-[[3-Hydroxy-4-(1-oxopropyl)-2-propylphenoxy]methyl]-3-methoxybenzoic acid

[118683-25-3]



$C_{21}H_{24}O_6$ mol.wt. 372.42

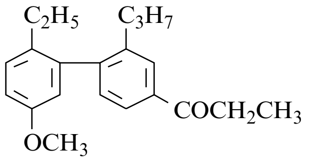
Synthesis

- Preparation from the substituted 2-propyl-resorcinol intermediate described in [7744], (compound **56**) [7481].

m.p. 217–218° [7481].

1-(2′-Ethyl-5′-methoxy-2-propylphenyl[1,1′-biphenyl]-4-yl)-1-propanone

[888009-25-4]

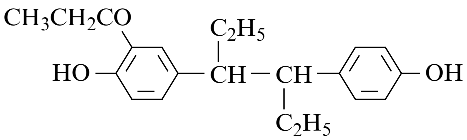


$C_{21}H_{26}O_2$ mol.wt. 310.44

Synthesis

- In a four-step process from 1-(4-hydroxy-3-propylphenyl)-1-propanone [7741].

1-[5-[(1*RS*,2*SR*)-1-Ethyl-2-(4-hydroxyphenyl)butyl]-2-hydroxyphenyl]-1-propanone *(3-Propionylhexestrol)*



$C_{21}H_{26}O_3$ mol.wt. 326.44

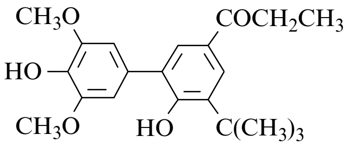
Synthesis

- Obtained by treatment of its dimethyl ether with refluxing pyridinium chloride for 20 min [7745].

m.p. 168° [7745].

1-[5-(1,1-Dimethylethyl)-4′,6-dihydroxy-3′,5′-dimethoxy[1,1′-biphenyl]-3-yl]-1-propanone

[75482-12-1]



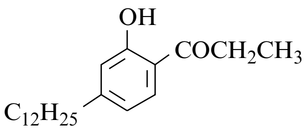
$C_{21}H_{26}O_5$ mol.wt. 358.43

Synthesis

- Obtained by oxidative coupling of 2,6-dimethoxy-phenol and 2,6-di-tert-butyl-4-propionylphenoxy radical (SM) in 1,2-dimethoxyethane under nitrogen at less than −10°, followed by treatment with boron trifluoride etherate (39%) [7409]. SM was prepared using a published procedure [7713].

m.p. 102° [7409]; ^{1}H NMR [7409], MS [7409].

1-(4-Dodecyl-2-hydroxyphenyl)-1-propanone



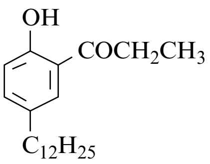
$C_{21}H_{34}O_2$ mol.wt. 318.50

Synthesis

- Refer to: [7685].

Oxime [101002-26-0] $C_{21}H_{35}NO_2$ mol.wt. 333.51 [7685].

1-(5-Dodecyl-2-hydroxyphenyl)-1-propanone



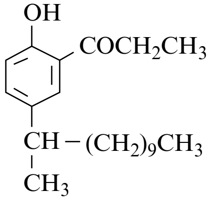
$C_{21}H_{34}O_2$ mol.wt. 318.50

Synthesis

- Refer to: [6646].

Hydrazone [70136-42-4] $C_{21}H_{36}N_2O$ mol.wt. 332.53 [6646].

1-(5-sec-Dodecyl-2-hydroxyphenyl)-1-propanone



$C_{21}H_{34}O_2$ mol.wt. 318.50

Synthesis

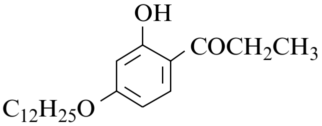
- Refer to: [7746].

Oxime [37769-66-7] $C_{21}H_{35}NO_2$ mol.wt. 333.51

- extg. agents, for copper from iron-contg. aq. solns. [7746].

1-[4-(Dodecyloxy)-2-hydroxyphenyl]-1-propanone

[143286-88-8]



$C_{21}H_{34}O_3$ mol.wt. 334.50

Synthesis

- Obtained by reaction of n-dodecyl bromide with respropiophenone in the presence of potassium carbonate in refluxing acetone [7237].

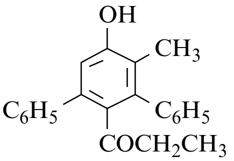
m.p. 41° [7237].

Oxime [99283-87-1] $C_{21}H_{35}NO_3$ mol.wt. 349.51 (m.p. 63–65°) [7747].

- Also refer to: [7685,7686].

1-[4-Hydroxy-3-methyl-2,6-di(phenyl)phenyl]-1-propanone

[14164-72-8]



$C_{22}H_{20}O_2$ mol.wt. 316.40

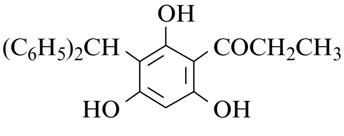
Synthesis

- Obtained (poor yield) by dehydrogenation of 3,5-diphenyl-2-methyl-4-propionylcyclohexan-1-ol (m.p. 140–141°) with Pd-C at 300–320° for 30 min under nitrogen (2%) [7748].

m.p. 220–221° [7748]; IR [7748].

1-[3-(Diphenylmethyl)-2,4,6-trihydroxyphenyl]-1-propanone

[105630-22-6]



$C_{22}H_{20}O_4$ mol.wt. 348.40

Synthesis

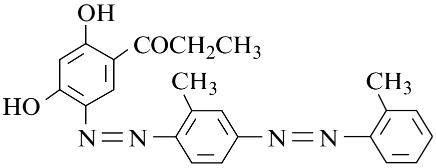
- Obtained by reaction of 2,4,6-trihydroxypropiophenone with diphenylcarbinol in dioxan in the presence of boron trifluoride etherate at 60–70° for 4 h (28%) [6886].

m.p. 155–156° [6886];

^{1}H NMR [6886], IR [6886], UV [6886]; TLC [6886].

1-[2,4-Dihydroxy-5-[[2-methyl-4-[(2-methylphenyl)azo]phenyl]azo]phenyl]-1-propanone

[65561-67-3] $C_{23}H_{22}N_4O_3$ mol.wt. 402.45



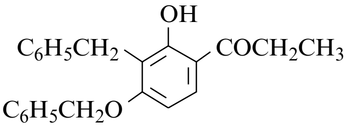
Syntheses

– Preparation, refer to: [7742,7743].

USE: Dye, for polypropylene fibres [7742,7743].

1-[2-Hydroxy-4-(phenylmethoxy)-3-(phenylmethyl)phenyl]-1-propanone

$C_{23}H_{22}O_3$ mol.wt. 346.43



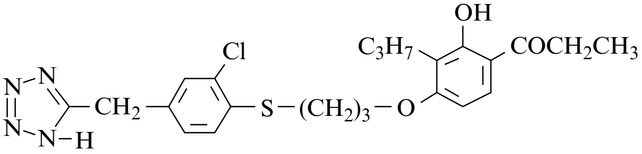
Synthesis

– Obtained by reaction of benzyl chloride with respropiophenone in the presence of methanolic potassium hydroxide, first at r.t. overnight, then at reflux for 5 h (12%) [7703].

m.p. 120° [7703].

1-[4-[3-[[2-Chloro-4-(1*H*-tetrazol-5-ylmethyl)phenyl]thio]propoxy]-2-hydroxy-3-propylphenyl]-1-propanone

[194791-95-2] $C_{23}H_{27}ClN_4O_3S$ mol.wt. 475.01



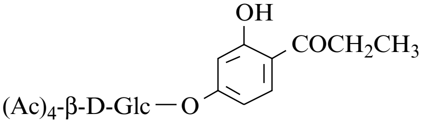
Synthesis

– Refer to: [7452].

Oxime [194791-97-4] $C_{23}H_{28}ClN_5O_3S$ mol.wt. 490.03 [7452].

1-[2-Hydroxy-4-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy]phenyl]-1-propanone

$C_{23}H_{28}O_{12}$ mol.wt. 496.47



Synthesis

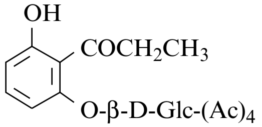
– Obtained by reaction of α-acetobromoglucose (α-ABG) with respropiophenone in the presence of silver oxide in quinoline for 2 h (50%) [6731].

m.p. 129–130° [6731];

$(\alpha)_D^{20}$ = –25.6° (c = 2 in chloroform) [6731].

1-[2-Hydroxy-6-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy]phenyl]-1-propanone

[190777-99-2] $C_{23}H_{28}O_{12}$ mol.wt. 496.47



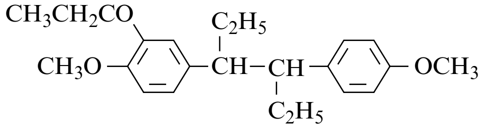
Syntheses

- Preparation by treatment of 2,6-dihydroxypropiophenone with cadmium carbonate in refluxing toluene for 1 h with removal of the generated water with a Dean-stark apparatus. Then, adding acetobromoglucose and the whole heated at reflux for 15 h (55%) [6813].
- Also refer to: [7749].

m.p. 175.5–177.5° [6813]; ^{1}H NMR [6813], IR [6813], FAB-MS [6813].
Na salt [6813].

1-[5-[(1*RS*,2*SR*)-1-Ethyl-2-(4-methoxyphenyl)butyl]-2-methoxyphenyl]-1-propanone

$C_{23}H_{30}O_3$ mol.wt. 354.49



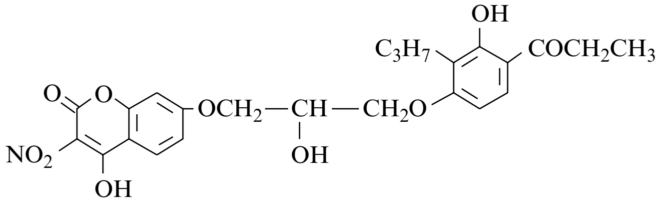
Synthesis

- Obtained by reaction of propionyl chloride with hexestrol dimethyl ether in the presence of aluminium chloride in nitrobenzene, first at 0°, then at r.t. for 3 h [7745].

b.p.$_{18}$ 280° [7745]; m.p. 96° [7745].

4-Hydroxy-7-[2-hydroxy-3-[3-hydroxy-4-(1-oxopropyl)-2-propylphenoxy]propoxy]-3-nitro-2*H*-1-benzopyran-2-one

[69076-17-1] $C_{24}H_{25}NO_{10}$ mol.wt. 447.58



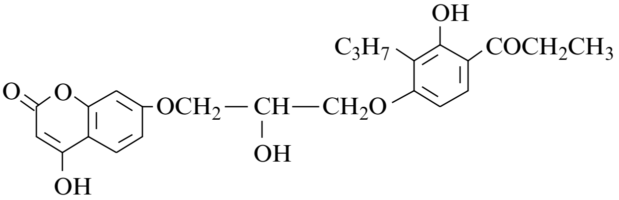
Syntheses

- Preparation by method H (compound **169**) (83%) [7482].
- Also refer to: [7112,7453].

m.p. 121° [7482].

4-Hydroxy-7-[2-hydroxy-3-[3-hydroxy-4-(1-oxopropyl)-2-propylphenoxy]propoxy]-2*H*-1-benzopyran-2-one

[69076-45-5] $C_{24}H_{26}O_8$ mol.wt. 442.47



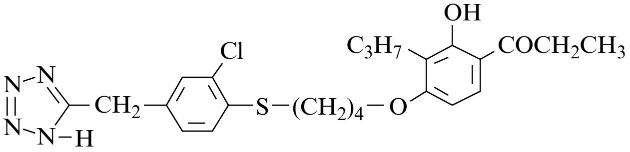
Syntheses

- Preparation by method G (compound **81**) (61%) [7482].
- Also refer to: [7112,7453].

m.p. 89° [7482].

1-[4-[4-[[2-Chloro-4-(1*H*-tetrazol-5-ylmethyl)phenyl]thio]butoxy]-2-hydroxy-3-propylphenyl]-1-propanone

[194791-98-5] $C_{24}H_{29}ClN_4O_3S$ mol.wt. 489.04



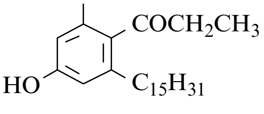
Synthesis

- Refer to: [7452].

Oxime [194791-99-6] $C_{24}H_{30}ClN_5O_3S$ mol.wt. 504.05 [7452].

1-(2,4-Dihydroxy-6-pentadecylphenyl)-1-propanone

[95818-32-9] $C_{24}H_{40}O_3$ mol.wt. 376.58



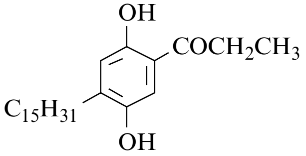
Synthesis

- Preparation by condensation of propionic acid with 5-pentadecylresorcinol (m.p. 95°) (SM) in xylene in the presence of BF_3-HF for 1 h at 50–60°, then at r.t. overnight [6820]. SM was prepared by catalytic hydrogenation of *cardol* 1,3-dihydroxy-5-(8,11-pentadecadienyl)benzene [7750], abundant material present in oil of cashew nuts *(Anacarde)* [7751].

m.p. 76° [6820].

1-(2,5-Dihydroxy-4-pentadecylphenyl)-1-propanone

[127498-04-8] $C_{24}H_{40}O_3$ mol.wt. 376.58

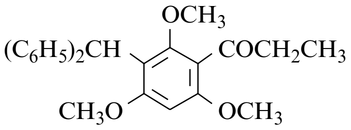


Synthesis

– Refer to: [7752] (Japanese patent).

1-[3-(Diphenylmethyl)-2,4,6-trimethoxyphenyl]-1-propanone

[105630-23-7] $C_{25}H_{26}O_4$ mol.wt. 390.48



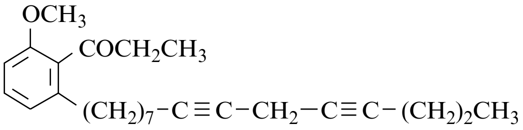
Synthesis

– Obtained by reaction of dimethyl sulfate with 3-diphenyl-methyl-2,4,6-trihydroxypropiophenone in the presence of potassium carbonate in refluxing acetone for 4 h [6886].

m.p. 156–157° [6886]; ^{1}H NMR [6886], UV [6886]; TLC [6886].

1-[2-Methoxy-6-(8,11-pentadecadiynyl)phenyl]-1-propanone

[188579-52-4] $C_{25}H_{34}O_2$ mol.wt. 366.54



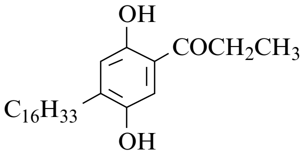
Synthesis

– Refer to: [7753].

1-(4-Hexadecyl-2,5-dihydroxyphenyl)-1-propanone

2-Hexadecyl-5-propionylhydroquinone

[65208-26-6] $C_{25}H_{42}O_3$ mol.wt. 390.61



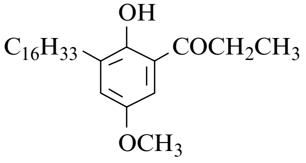
Synthesis

– Preparation by reaction of propionic acid with 2-hexadecyl-hydroquinone (m.p. 112.5–113.5°) in the presence of boron trifluoride (50%) [6509].

m.p. 93–94° [6509].

1-(3-Hexadecyl-2-hydroxy-5-methoxyphenyl)-1-propanone

[103048-60-8] $C_{26}H_{44}O_3$ mol.wt. 404.63



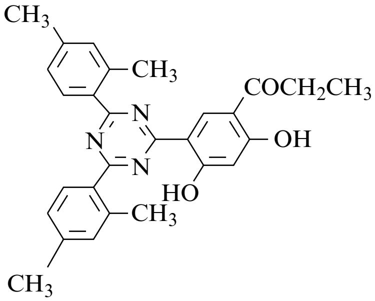
Synthesis

– Preparation by reaction of propionic acid with 2-hexadecyl-4-methoxyphenol in the presence of boron trifluoride (54%) [6509].

m.p. 65.4–66.5° [6509].

1-[5-[4,6-Bis(2,4-dimethylphenyl)-1,3,5-triazin-2-yl]-2,4-dihydroxyphenyl]-1-propanone

[176843-49-5]



$C_{28}H_{27}N_3O_3$ mol.wt. 453.54

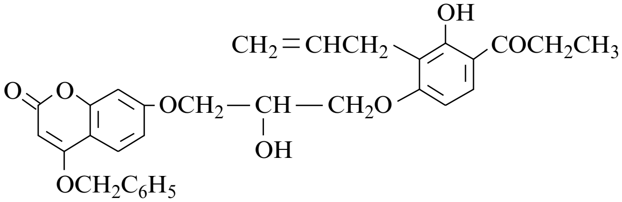
Syntheses

- Refer to: [7754,7755].

UV [7754,7755].

7-[2-Hydroxy-3-[3-hydroxy-4-(1-oxopropyl)-2-(2-propenyl)phenoxy]propoxy]-4-(phenylmethoxy)-2*H*-1-benzopyran-2-one

[63360-36-1] $C_{31}H_{30}O_8$ mol.wt. 530.57



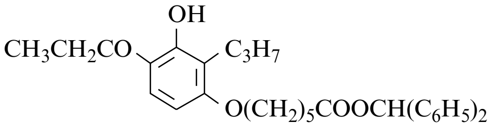
Syntheses

- Preparation by method E (compound **114**) (25%) [7482].
- Also refer to: [7112,7453,7684].

m.p. 117° [7482].

6-[3-Hydroxy-4-(1-oxopropyl)-2-propylphenoxy]hexanoic acid diphenylmethyl ester

[106627-30-9]



$C_{31}H_{36}O_5$ mol.wt. 488.62

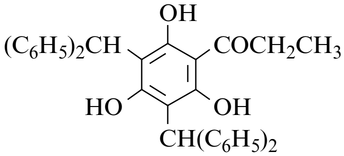
Synthesis

- This ketone (compound **43**) was synthesized from 2,4-di hydroxy-3-propyl-propiophenone according to the method C (56%) [7450].

^{1}H NMR [7450].

1-[3,5-Bis(diphenylmethyl)-2,4,6-trihydroxyphenyl]-1-propanone

[105630-20-4]



$C_{35}H_{30}O_4$ mol.wt. 514.62

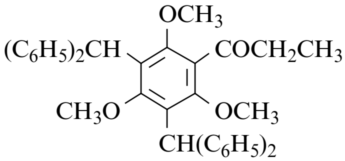
Synthesis

- Obtained by reaction of 2,4,6-trihydroxypropiophenone with diphenylcarbinol in dioxan in the presence of boron trifluoride etherate at 60–70° for 4 h (11%) [6886].

m.p. 249–250° [6886]; ^{1}H NMR [6886], IR [6886], UV [6886]; TLC [6886].

1-[3,5-Bis(diphenylmethyl)-2,4,6-trimethoxyphenyl]-1-propanone

[105630-21-5]



$C_{38}H_{36}O_4$ mol.wt. 556.70

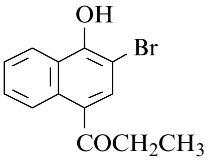
Synthesis

- Obtained by reaction of dimethyl sulfate with 3,5-bis-(diphenylmethyl)-2,4,6-trihydroxypropiophenone in the presence of potassium carbonate in refluxing acetone for 10 h [6886].

m.p. 198–199° [6886]; ^{1}H NMR [6886], UV [6886]; TLC [6886].

23.2 Naphthalene Derivatives

1-(3-Bromo-4-hydroxy-1-naphthalenyl)-1-propanone



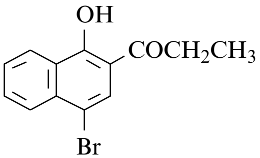
$C_{13}H_{11}BrO_2$ mol.wt. 279.13

Synthesis

- Obtained by reaction of bromine with 4-propionyl-1-naphthol in chloroform [7756].

m.p. 111° [7756].

1-(4-Bromo-1-hydroxy-2-naphthalenyl)-1-propanone



$C_{13}H_{11}BrO_2$ mol.wt. 279.13

Syntheses

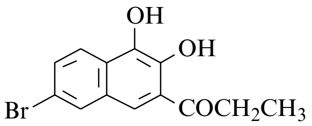
- Preparation by reaction of bromine with 2-propionyl-1-naphthol in acetic acid [7757].

- Also obtained by reaction of bromine with 4,4′-dihydroxy-3,3′-dipropionyldinaphthyl trisulfide (m.p. 190°) in acetic acid [7758].
- Also refer to: [6730,7759,7760].

m.p. 98–99° [7759], 98° [6730,7758], 97° [7757].

1-(7-Bromo-3,4-dihydroxy-2-naphthalenyl)-1-propanone

[52749-67-4]



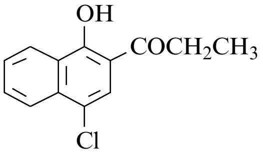
$C_{13}H_{11}BrO_3$ mol.wt. 295.13

Syntheses

- Obtained by irradiation of 6-bromo-1,2-naphthoquinone and propionaldehyde in benzene with a 300 W high-pressure mercury arc lamp between 15° and 20° for 2–10 days [7761], (26%) [7762].

m.p. 143–144.5° [7762]; ^{1}H NMR [7762], IR [7762].

1-(4-Chloro-1-hydroxy-2-naphthalenyl)-1-propanone



$C_{13}H_{11}ClO_2$ mol.wt. 234.68

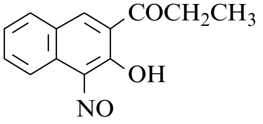
Syntheses

- Obtained by Fries rearrangement of 4-chloro-1-naphthyl propionate with aluminium chloride on heating in a water bath for 1.5 h (61%) [7763].
- Also obtained by action of chlorine with 4,4′-dihydroxy-3,3′-dipropionyldinaphthyl trisulfide (m.p. 190°) in acetic acid [7758].
- Also obtained by action of sulfuryl chloride with 2-propionyl-α-naphthol in the presence of bismuth chloride in ethyl ether [7758].
- Also obtained by action of chlorine with 2-propionyl-α-naphthol in acetic acid [7758].

m.p. 93° [7758], 90–91° [7763].

1-(3-Hydroxy-4-nitroso-2-naphthalenyl)-1-propanone

[256335-72-5]



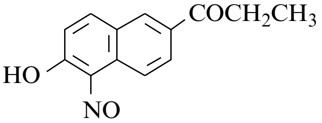
$C_{13}H_{11}NO_3$ mol.wt. 229.24

Synthesis

- Refer to: [7764] (Japanese patent).

1-(6-Hydroxy-5-nitroso-2-naphthalenyl)-1-propanone

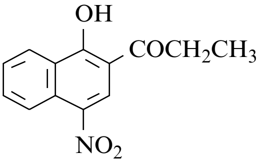
[219661-16-2] $C_{13}H_{11}NO_3$ mol.wt. 229.24



Syntheses

– Refer to: [7765,7766].

1-(1-Hydroxy-4-nitro-2-naphthalenyl)-1-propanone



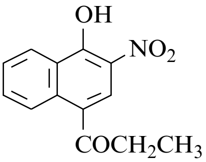
$C_{13}H_{11}NO_4$ mol.wt. 245.23

Syntheses

- – Preparation by reaction of fuming nitric acid (d = 1.5) with 2-propionyl-1-naphthol (85%) [7760].
- – Also obtained by reaction of fuming nitric acid with 2,4-di-propionyl-1-naphthol in acetic acid [7767].
- – Also obtained by reaction of fuming nitric acid with 4-acetyl-2-propionyl-1-naphthol in acetic acid [7767].
- – Also obtained by reaction of nitric acid (d = 1.42) with 2-propionyl-α-naphthol in acetic acid [7758].
- – Also obtained by action of nitric acid (d = 1.42) with 4,4′-dihydroxy-3,3′-dipropionyldinaphthyl trisulfide (m.p. 190°) in acetic acid [7758].
- – Also refer to: [7759].

m.p. 162–163° [7759], 162° [7760], 158° [7758].

1-(4-Hydroxy-3-nitro-1-naphthalenyl)-1-propanone



$C_{13}H_{11}NO_4$ mol.wt. 245.23

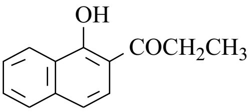
Syntheses

– Obtained by reaction of fuming nitric acid with 4-propionyl-1-naphthol [7756] or with 2,4-dipropionyl-1-naphthol in acetic acid [7767].

m.p. 1 00° [7756].

1-(1-Hydroxy-2-naphthalenyl)-1-propanone

[24490-31-1] $C_{13}H_{12}O_2$ mol.wt. 200.24



Syntheses

– Preparation by Fries rearrangement of α-naphthyl propionate,

- in the presence of aluminium chloride (40%) [7768], first at 100° for 2 h, then at 120° for 1 h (54%) [7769];

  - in the presence of stannic chloride (100%) [7768], at 55° for 16 h (73%) [7770];
  - in the presence of zinc chloride (100%) [7768];
  - in the presence of a catalytic amount of scandium triflate—Sc(OTf)$_3$—(5 mol%) in toluene at 100° for 6 h [7771], (89%) [7772,7773].

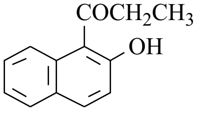
- Preparation by reaction of propionic acid with α-naphthol,
  - in the presence of zinc chloride (Nencki reaction) [6376,6730], (65%) [7760], (48%) [7759];
  - in the presence of boron trifluoride [6819,7774], at 70° for 2 h (83%) [7775] at 100° for 5 h (80%) [7639], at 28–30° for 18 h (65%) [7639] or at r.t. (20%) [7776];
  - in the presence of polyphosphoric acid at 100° for 15 min (42%) [7777];
  - in the presence of hafnium triflate (20 mol %) in toluene-nitromethane at 100° for 6 h (80%) [7778].
- Also obtained by reaction of propionyl chloride with α-naphthol,
  - in the presence of a catalytic amount of scandium triflate—Sc(OTf)$_3$—(5 mol%) in toluene/nitromethane at 100° for 6 h (98%) [7772,7779];
  - in the presence of zinc chloride in nitrobenzene (22%) [7756].
- Also obtained by boiling a 4-hydroxy-3-(1-oxopropyl)-1-naphthalenesulfonic acid and concentrated sulfuric acid mixture, followed by distillation [7757].
- Also obtained by heating 4-propionyl-α-naphthol, 2,4-dipropionyl-α-naphthol or 4-acetyl-2-propionyl-α-naphthol with zinc chloride in acetic acid or propionic acid on a sand bath for 3 h [7767].
- Also obtained by dye-sensitized photooxidation of enolic tautomer of 1-(3,4-dihydro-1-hydroxy-2-naphthalenyl)-1-propanone [134643-89-3] (19–24%) [7780].
- Also refer to: [6684,7781–7785].

m.p. 86° [7775,7777], 85–87° [7776], 85° [7760], 84–85° [7772], 83–84° [7769], 82° [6819,7639,7770], 81–82° [7759], 81° [6376,6730,7757];
^{1}H NMR [7772], ^{13}C NMR [7772], IR [7772];
fuorescence spectrum data [6960].

Oxime *(2-Pronapox)* [21660-75-3] $C_{13}H_{13}NO_2$
mol.wt. 215.25 [7786–7789].

1-(2-Hydroxy-1-naphthalenyl)-1-propanone

[33828-93-2] $C_{13}H_{12}O_2$ mol.wt. 200.24



Syntheses

- Preparation by Fries rearrangement of β-naphthyl propionate (m.p. 46–47°) [7790], in the presence of aluminium chloride,
  - first in boiling carbon disulfide for 1 h, then at 120° for 4 h after solvent elimination [7791], (72%) [7792];
  - in nitrobenzene at r.t. for 48 h (66%) [7793];
  - in tetrachloroethane at higher temperatures (60–100%) [7790].

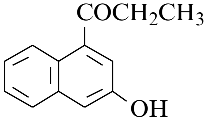
- Preparation by reaction of propionic acid with β-naphthol in the presence of boron trifluoride [6819,7774], at 28–30° for 18 h (75%) or at 100° for 5 h (85%) [7639].
- Preparation by reaction of propionic anhydride with β-naphthol in the presence of aluminium chloride in ethylene dichloride (95%) [7300].
- Also obtained by treatment of 2-methoxynaphthalene with propionyl chloride in refluxing carbon disulfide for 2 h (30%) [7794].
- Also refer to: [7795–7797].

b.p.$_{13}$ 171–175° [7793];
m.p. 82–84° [7791], 78.8–79.8° [7794], 72° [6819], 71° [7639], 70–72° [7793], 70–71° [7792], 64–66° [7790]. There is a large dispersion of melting points pointed out in literature.
^{13}C NMR [7794], ^{17}O NMR [7798]; MNDO heat of formation [7799].

Oxime [350026-68-5] $C_{13}H_{13}NO_2$ mol.wt. 215.25 (m.p. 112–116°) [7797].

1-(3-Hydroxy-1-naphthalenyl)-1-propanone

[175226-44-5] $C_{13}H_{12}O_2$ mol.wt. 200.24

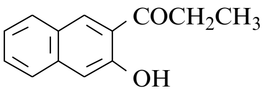


Synthesis
- Refer to: [7799].

Note: MNDO heat of formation [7799].

1-(3-Hydroxy-2-naphthalenyl)-1-propanone

[91902-70-4] $C_{13}H_{12}O_2$ mol.wt. 200.24

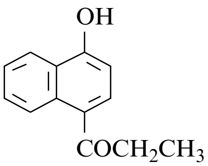


Synthesis
- Refer to: [7799].

Note: MNDO heat of formation [7799].

1-(4-Hydroxy-1-naphthalenyl)-1-propanone

[133181-63-2] $C_{13}H_{12}O_2$ mol.wt. 200.24



Syntheses
- Preparation from 4-methoxy-1-propionylnaphthalene with boiling pyridinium chloride for 30 min [6667] or by heating at 240–250° for 1 h in a sealed tube (55%) [7770].

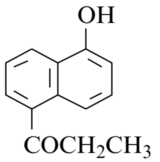
- Also obtained by reaction of propionic acid with α-naphthol,
  - in the presence of boron trifluoride at r.t. (20%) [7639], (10%) [7776];
  - in the presence of polyphosphoric acid at 100° for 15 min (1%) [7777].

- Also obtained by Fries rearrangement of 1-naphthyl propionate with aluminium chloride (4%) [7768], at 100° for 2 h, then at 120° for 1 h (6%) [7769] or in nitrobenzene at r.t. (23%) [7800].
- Also obtained by reaction of propionyl chloride with α-naphthol in the presence of zinc chloride in nitrobenzene (43%) [7756].
- Also obtained by heating 1-hydroxy-4-propionyl-2-naphthoic acid at >205° for 30 min [7767]. There is a carboxyl group elimination.

m.p. 190° [7776], 189° [7770], 188–189° [7769,7800], 187° [6667,7639], 185° [7777].

1-(5-Hydroxy-1-naphthalenyl)-1-propanone

[91307-45-8] $C_{13}H_{12}O_2$ mol.wt. 200.24

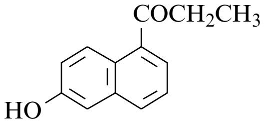


Synthesis

- Refer to: [7801].

1-(6-Hydroxy-1-naphthalenyl)-1-propanone

[175226-45-6] $C_{13}H_{12}O_2$ mol.wt. 200.24



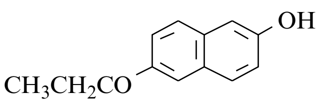
Synthesis

- Refer to: [7799].

Note: MNDO heat of formation [7799].

1-(6-Hydroxy-2-naphthalenyl)-1-propanone

[33828-92-1] $C_{13}H_{12}O_2$ mol.wt. 200.24



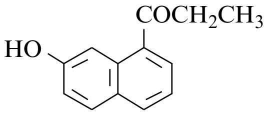
Syntheses

- Preparation by treatment of 6-propionylnerolin (2-methoxy-6-propionylnaphthalene) with boiling pyridinium chloride for 20 min (86%) [6667].
- Preparation by Fries rearrangement of 2-naphthyl propionate [7802] using hydrogen fluoride as catalyst [7803].
- Also refer to: [7804–7809].

m.p. 164° [6667]; MNDO heat of formation [7799].

1-(7-Hydroxy-1-naphthalenyl)-1-propanone

[175226-47-8] $C_{13}H_{12}O_2$ mol.wt. 200.24



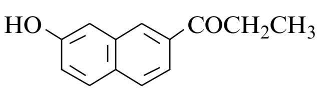
Synthesis

- Refer to: [7799].

Note: MNDO heat of formation [7799].

1-(7-Hydroxy-2-naphthalenyl)-1-propanone

[175226-46-7] $C_{13}H_{12}O_2$ mol.wt. 200.24



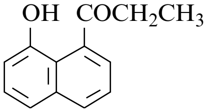
Synthesis

- Refer to: [7799].

Note: MNDO heat of formation [7799].

1-(8-Hydroxy-1-naphthalenyl)-1-propanone

[131421-22-2] $C_{13}H_{12}O_2$ mol.wt. 200.24



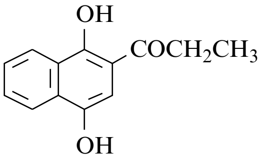
Syntheses

- Obtained by adding dropwise, at −78° under nitrogen, a solution of ethylmagnesium bromide in ethyl ether to a solution of 2*H*-naphtho[1,8-*bc*]furan-2-one (m.p. 99–101°) [7810] in THF over a period of 3 h. After heating the mixture to 0°, a saturated aqueous ammonium chloride solution was added (33%) [7811].
- Also refer to: [7812].

m.p. 114–117° [7811]; ^{1}H NMR [7811].

1-(1,4-Dihydroxy-2-naphthalenyl)-1-propanone

[75859-15-3] $C_{13}H_{12}O_3$ mol.wt. 216.24

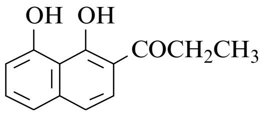


Syntheses

- Obtained by demethylation of the corresponding 4-methyl ether with concentrated hydrobromic acid in acetic acid [7813].
- Also obtained by thermal reaction of 2-propionyl-1,4-naphthoquinone with allyltrimethylstannane in benzene under argon atmosphere (7%) [7814].
- Also obtained by photochemical reaction (409–429 nm) between 1,4-naphthoquinone and propionaldehyde, in benzene at r.t. for 5 days [7815] under argon (79%) [7816] according to the procedure [7817].

m.p. 186–188° [7815], 182–184° [7814];
^{1}H NMR [7814–7816], ^{13}C NMR [7816],
IR [7814–7816], MS [7814,7816].

1-(1,8-Dihydroxy-2-naphthalenyl)-1-propanone



$C_{13}H_{12}O_3$ mol.wt. 216.24

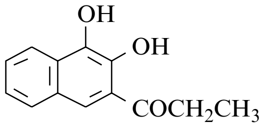
Syntheses

- Obtained by reaction of propionic acid with 1,8-dihydroxy-naphthalene in the presence of zinc chloride at 145–150° [7818].
- Also obtained by reaction of zinc chloride with 1,8-di-hydroxynaphthalene dipropionate in nitrobenzene at 140–150° [7819].

m.p. 101–102° [7818].

1-(3,4-Dihydroxy-2-naphthalenyl)-1-propanone

[61983-10-6]



$C_{13}H_{12}O_3$ mol.wt. 216.24

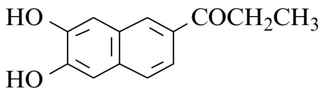
Syntheses

- Obtained by irradiation of 1,2-naphthoquinone and propionaldehyde,
  - in acetonitrile-benzene by UV in the presence of magnesium perchlorate (29%) [7820];
  - in benzene by a 300 W high-pressure mercury arc lamp between 15° and 20° for 2–10 days [7761], (19%) [7762].
- Also refer to: [7821].

m.p. 129–130.5° [7762]; ^{1}H NMR [7762], IR [7762].

1-(6,7-Dihydroxy-2-naphthalenyl)-1-propanone

[144289-53-2]



$C_{13}H_{12}O_3$ mol.wt. 216.24

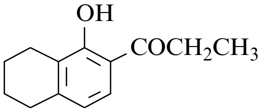
Synthesis

- Obtained by total demethylation of its dimethyl ether (SM) with 1 M boron tribromide at 0°. SM was prepared by reaction of propionic anhydride with 2,3-dimethoxy-naphthalene in nitrobenzene at 0° in the presence of aluminium chloride [7822].

BIOLOGICAL ACTIVITY: As lipoxygenase inhibitor [7822].

1-(5,6,7,8-Tetrahydro-1-hydroxy-2-naphthalenyl)-1-propanone

[100612-28-0] $C_{13}H_{16}O_2$ mol.wt. 204.27



Syntheses

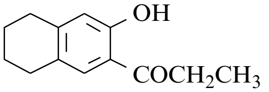
- Obtained by Fries rearrangement of 5,6,7,8-tetrahydro-1-naphthyl propionate (b.p.$_{12}$ 152°) with aluminium chloride in nitrobenzene, first at 0° for 2–3 h, then at r.t. for 15–17 h (68%) [7800].
- Also obtained by Friedel–Crafts acylation of 5,6,7,8-tetrahydro-1-naphthol with propionyl chloride in the presence of titanium tetrachloride neat at 120° for 1 h (85%) [7823].

m.p. 87.5–88.5° [7800], 86–88° [7823];
^{1}H NMR [7823], ^{13}C NMR [7823], IR [7823], MS [7823].

Oxime $C_{13}H_{17}NO_2$ mol.wt. 219.28 (m.p. 141–141.5°) [7800].

1-(5,6,7,8-Tetrahydro-3-hydroxy-2-naphthalenyl)-1-propanone

[60401-57-2] $C_{13}H_{16}O_2$ mol.wt. 204.27



Syntheses

- Obtained by Fries rearrangement of 5,6,7,8-tetrahydro-2-naphthyl propionate (b.p.$_{12}$ 160°) with aluminium chloride at 120° for 4 h (48%) [7824].
- Also obtained by Fries rearrangement of 5,6,7,8-tetrahydro-2-naphthyl propionate with aluminium chloride in nitrobenzene at r.t. overnight [7824].
- Also refer to: [7825,7826].

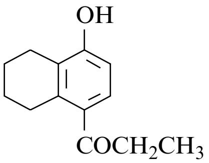
m.p. 58–59° [7824].

USE: Intermediate for the preparation of allergy inhibitor [7825].

Oxime $C_{13}H_{17}NO_2$ mol.wt. 219.28 (m.p. 134.5–135.5°) [7824].

1-(5,6,7,8-Tetrahydro-4-hydroxy-1-naphthalenyl)-1-propanone

$C_{13}H_{16}O_2$ mol.wt. 204.27



Syntheses

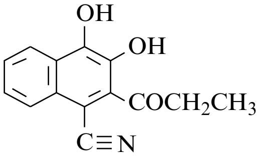
- Obtained by Fries rearrangement of 5,6,7,8-tetrahydro-1-naphthyl propionate (b.p.$_{12}$ 152°) with aluminium chloride in nitrobenzene, first at 0° for 2–3 h, then at r.t. for 15–17 h [7800].
- Also obtained by treatment of its methyl ether with aluminium chloride in refluxing benzene for 6 h (55%) [7403].

m.p. 152–153° [7403], 146–147.5° [7800].

Semicarbazone $C_{14}H_{19}N_3O_2$ mol.wt. 261.32 (m.p. 225–226.5°) [7800].

3,4-Dihydroxy-2-(1-oxopropyl)-1-naphthalenecarbonitrile

[61983-21-9] $C_{14}H_{11}NO_3$ mol.wt. 241.25



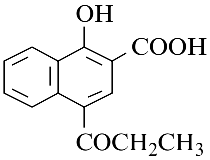
Syntheses

- Obtained by irradiation of 4-cyano-1,2-naphthoquinone and propionaldehyde in benzene by a 300 W high-pressure mercury arc lamp between 15° and 20° for 2–10 days [7761], (50%) [7762] or by exposition to sunlight for a fortnight (18%) [7827].

m.p. 171–174° [7762], 168° [7827]; ^{1}H NMR [7762], IR [7762].

1-Hydroxy-4-(1-oxopropyl)-2-naphthoic acid

$C_{14}H_{12}O_4$ mol.wt. 244.25



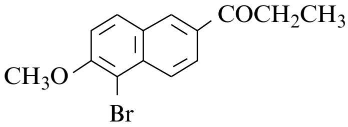
Syntheses

- Obtained from 2,3-dimethyl-6-propionyl-1,4-α-naphthopyrone (m.p. 168°) by refluxing with 5% aqueous sodium hydroxide for 3 h [7767].
- Also refer to: [7756].

m.p. 205° [7756,7767].

1-(5-Bromo-6-methoxy-2-naphthalenyl)-1-propanone

[92189-66-7] $C_{14}H_{13}BrO_2$ mol.wt. 293.16



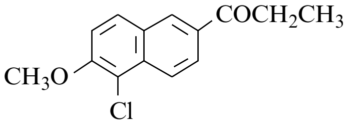
Syntheses

- Preparation by bromination of 1-(6-methoxy-2-naphthalenyl)-1-propanone with 1,3-dibromo-5,5-dimethylhydantoin in ethanol (99%) [7828].
- Also obtained by heating 5-bromo-6-methoxy-2-(α-bromopropionyl)naphthalene (m.p. 162°) with zinc in acetic acid for 3 h (75%) [7829].
- Also refer to: [7830–7833].

m.p. 127–128° [7829].

1-(5-Chloro-6-methoxy-2-naphthalenyl)-1-propanone

[69750-45-4] $C_{14}H_{13}ClO_2$ mol.wt. 248.71



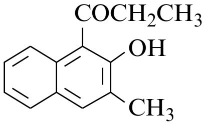
Syntheses

- Preparation by reaction of propionyl chloride with 1-chloro-2-methoxynaphthalene (m.p. 66°) in the presence of aluminium chloride in methylene chloride, first at 0°, then at 5° for 15 min (97%) [7834].
- Also refer to: [7835] (Chinese paper).

m.p. 129–131° [7834]; crystal data [7834].

1-(2-Hydroxy-3-methyl-1-naphthalenyl)-1-propanone

[185413-93-8] $C_{14}H_{14}O_2$ mol.wt. 214.26



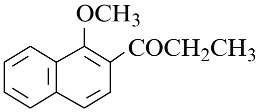
Synthesis

- Obtained (poor yield) from 2-methyl-1-phenylheptane-3,5-dione by treatment with manganese (III) acetate in acetic acid at 60° (ca. 4%) [7836].

GC-MS [7836]; MS [7836].

1-(1-Methoxy-2-naphthalenyl)-1-propanone

$C_{14}H_{14}O_2$ mol.wt. 214.26



Syntheses

- Preparation by reaction of dimethyl sulfate with 2-propionyl-1-naphthol in the presence of alkali [7760].
- Preparation by reaction of ethylmagnesium bromide with 1-methoxy-2-naphthonitrile (59%) [6973].
- Also obtained by treatment of 2-propionyl-1-naphthol with methyl iodide in the presence of potassium carbonate in refluxing acetone for 8 h (35%) [7837].
- Also refer to: [6755,7838].

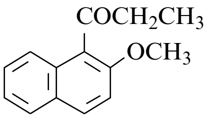
b.p.$_{0.3}$ 125–126° [6973];
m.p. 50–51° [6973], 48–49.5° [7837], 45° [7760], 42–43° [7838].

Oxime $C_{14}H_{15}NO_2$ mol.wt. 229.28 (m.p. 112–113°) [7838].

Semicarbazone $C_{15}H_{17}N_3O_2$ mol.wt. 271.32 (m.p. 192°) [7838].

1-(2-Methoxy-1-naphthalenyl)-1-propanone

[25801-58-5] $C_{14}H_{14}O_2$ mol.wt. 214.26



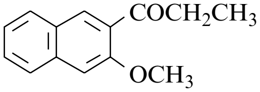
Syntheses

- Obtained by reaction of propionyl chloride with 2-methoxy-naphthalene in the presence of aluminium chloride in methylene chloride for 5 min at 0° under nitrogen (74%) [7839].
- Also obtained by acylation of 2-methoxynaphthalene with propionic anhydride in nitrobenzene in the presence of zeolite-beta catalysts [7840].
- Also obtained by electrochemical acylation of 2-methoxynaphthalene with propionic anhydride in methylene chloride in the presence of $LiClO_4$ (74%) [7841].
- Also refer to: [7358,7842,7843].

b.p.$_{2-3}$ 141–143° [7791];
^1H NMR [7839], IR [7839]; HPLC [7839].

1-(3-Methoxy-2-naphthalenyl)-1-propanone

[17295-05-5] $C_{14}H_{14}O_2$ mol.wt. 214.26



Syntheses

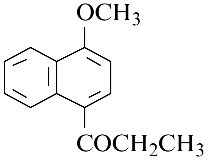
- Obtained by adding methyllithium in diethyl ether to a solution of 4,4-dimethyl-2-[2-(3-methoxynaphthyl)]-oxazoline (m.p. 125–127°) in THF/diethyl ether at −95°. Then, the resulting orange clear solution was stirred at −78° for 6 h, quenched with neat methyl iodide and warmed to r.t. overnight (47%) [7844].
- Also obtained by condensation of 3-methoxy-2-naphthoyl chloride with diethylcadmium in benzene (63%) [7837].

yellowish oil [7844]; m.p. 48–49° [7837];
^{1}H NMR [7844], ^{13}C NMR [7844], MS [7844]; TLC [7844].

Semicarbazone $C_{15}H_{17}N_3O_2$ mol.wt. 271.32 (m.p. 212–213°) [7837].

1-(4-Methoxy-1-naphthalenyl)-1-propanone

[5471-38-5] $C_{14}H_{14}O_2$ mol.wt. 214.26



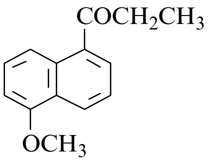
Syntheses

- Preparation by reaction of propionic acid with 1-methoxy-naphthalene in the presence of boron trifluoride, first at 0°, then at 70° for 10–15 h (84%) [7845].
- Also obtained by reduction of 3-(dibutylamino)-1-(4-methoxynaphthalen-1-yl)-1-propanone with Adam′s catalyst (22%) [7846].
- Also refer to: [7292,7756,7847–7849].

m.p. 58° [7756,7845], 56–57° [7846]; ^{1}H NMR [7849].

1-(5-Methoxy-1-naphthalenyl)-1-propanone

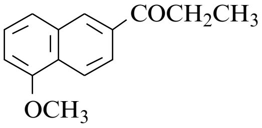
[904923-39-3] $C_{14}H_{14}O_2$ mol.wt. 214.26



Synthesis

- Refer to: [7847].

1-(5-Methoxy-2-naphthalenyl)-1-propanone



$C_{14}H_{14}O_2$ mol.wt. 214.26

Synthesis

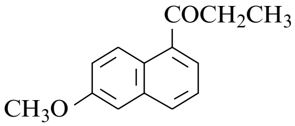
- Preparation by reaction of ethylmagnesium bromide with 5-methoxy-2-naphthonitrile in benzene (84%) [7850].

m.p. 92–93° [7850].

Semicarbazone $C_{15}H_{17}N_3O_2$ mol.wt. 271.32 (m.p. 212°) [7850].

1-(6-Methoxy-1-naphthalenyl)-1-propanone

[81336-21-2]



$C_{14}H_{14}O_2$ mol.wt. 214.26

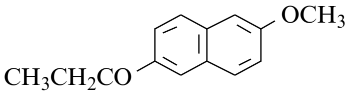
Synthesis

- Obtained from sodium-liquid ammonia reduction of 4,6-dimethoxy-1-naphthyl ethyl ketone in THF/EtOH mixture in presence of excess ammonium chloride [7851].

pale yellow oil [7851]; b.p.$_{0.1}$ 80° [7851];
^{1}H NMR [7851]; IR [7851].

1-(6-Methoxy-2-naphthalenyl)-1-propanone

[2700-47-2]



$C_{14}H_{14}O_2$ mol.wt. 214.26

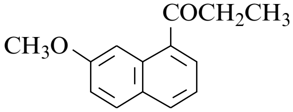
Syntheses

- Obtained by reaction of propionyl chloride with 2-methoxynaphthalene in the presence of aluminium chloride,
  - in methylene chloride and nitromethane (74%) [7852];
  - in methylene chloride and nitrobenzene at 15° for 20 h (71%) [7839];
  - in nitrobenzene (64%) [7853] at r.t. for 24 h (80%) [7854].
- Also obtained by reaction of propionyl chloride with 2-methoxynaphthalene (neroline) in the presence of stannic chloride in nitrobenzene, first at 0°, then at r.t. for 12 h (32–41%) [7855].
- Also obtained by acylation of 2-methoxynaphthalene with propionic anhydride in nitrobenzene in the presence of zeolite-beta catalysts [7840].
- Also refer to: [7829,7831–7833,7843,7856–7861].

b.p.$_{0.3}$ 180–185° [7854], b.p.$_{0.5}$ 190–220° [7855];
m.p. 114–115° [7839], 111.5–113.5° [7853], 109° [7854,7855];
^{1}H NMR [7839], IR [7839]; HPLC [7839].

1-(7-Methoxy-1-naphthalenyl)-1-propanone

[507272-84-6] $C_{14}H_{14}O_2$ mol.wt. 214.26

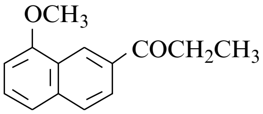


Syntheses

– Refer to: [7847,7862].

1-(8-Methoxy-2-naphthalenyl)-1-propanone

$C_{14}H_{14}O_2$ mol.wt. 214.26



Synthesis

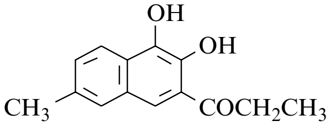
– Preparation by reaction of ethylmagnesium bromide with 8-methoxy-2-naphthonitrile in benzene (84%) [7850].

m.p. 69–70° [7850].

Semicarbazone $C_{15}H_{17}N_3O_2$ mol.wt. 271.32 (m.p. 188–189°) [7850].

1-(3,4-Dihydroxy-7-methyl-2-naphthalenyl)-1-propanone

[61983-39-9] $C_{14}H_{14}O_3$ mol.wt. 230.26



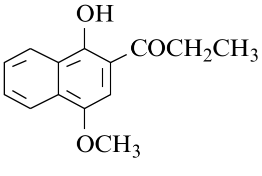
Syntheses

– Obtained by irradiation of 6-methyl-1, 2-naphthoquinone and propionaldehyde in benzene by a 300 W high-pressure mercury arc lamp between 15° and 20° for 2–10 days [7761], (15%) [7762].

m.p. 148–149° [7762]; ^{1}H NMR [7762], IR [7762].

1-(1-Hydroxy-4-methoxy-2-naphthalenyl)-1-propanone

[128462-64-6] $C_{14}H_{14}O_3$ mol.wt. 230.26

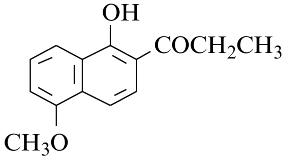


Synthesis

– Obtained by reaction of 2-propionyl-1,3-indandione with diazomethane in ethyl ether (66–72%) [7813].

1-(1-Hydroxy-5-methoxy-2-naphthalenyl)-1-propanone

[68047-77-8] $C_{14}H_{14}O_3$ mol.wt. 230.26



Syntheses

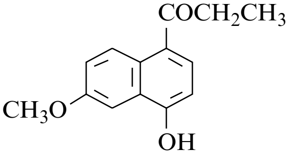
– Preparation by Fries rearrangement of 5-methoxy-1-naphthyl propionate with boron trifluoride etherate at 120° for 1 h (80%) [7863].

- Also obtained by photo-Fries rearrangement of 5-methoxy-1-naphthyl propionate in methanol [7864].

m.p. 102–103° [7864], 92–95° [7863]; IR [7864], MS [7864].

1-(4-Hydroxy-6-methoxy-1-naphthalenyl)-1-propanone

[81336-23-4] $C_{14}H_{14}O_3$ mol.wt. 230.26



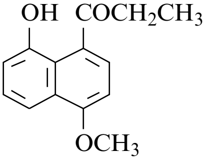
Synthesis

- Obtained from sodium-liquid ammonia reduction of 4,6-dimethoxy-1-naphthyl ethyl ketone in THF (6%) [7851].

m.p. 130–132° [7851]; IR [7851], MS [7851].

1-(8-Hydroxy-4-methoxy-1-naphthalenyl)-1-propanone

[70340-72-6] $C_{14}H_{14}O_3$ mol.wt. 230.26



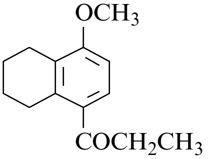
Syntheses

- Obtained by propionylation of 5-methoxy-1-propionoxy-naphthalene with subsequent ester cleavage [7865], (96%) [7866].
- Also refer to: [7867].

m.p. 135–136° [7866]; ^{1}H NMR [7866], IR [7866].

1-(5,6,7,8-Tetrahydro-4-methoxy-1-naphthalenyl)-1-propanone

$C_{14}H_{18}O_2$ mol.wt. 218.30



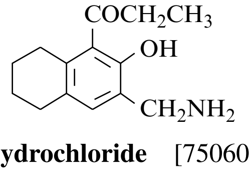
Synthesis

- Obtained by Friedel–Crafts acylation of 5,6,7,8-tetrahydro-1-methoxynaphthalene with propionyl chloride in the presence of aluminium chloride in nitrobenzene, first at 0° for 4 h, then at r.t. for 24 h (33%) [7403].

b.p.$_{10}$ 169–171° [7403]; m.p. 36–37° [7403].

1-[3-(Aminomethyl)-5,6,7,8-tetrahydro-2-hydroxy-1-naphthalenyl]-1-propanone

[75061-06-2] $C_{14}H_{19}NO_2$ mol.wt. 233.31



Synthesis

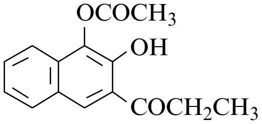
- Refer to: [7106].

Hydrochloride [75060-82-1] $C_{14}H_{19}NO_2$, HCl mol.wt. 269.77.

USE: Antiinflammatory [7106].

1-[4-(Acetyloxy)-3-hydroxy-2-naphthalenyl]-1-propanone

[75089-89-3] $C_{15}H_{14}O_4$ mol.wt. 258.27



Synthesis

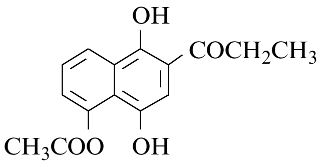
- Obtained by keeping a benzene solution of 1,2-naphthoquinone, 3-propionylnaphthalene-1,2-diol, DTBP and acetaldehyde in the dark at 30° for 7 days (16%) [7821].

m.p. 130–131° [7821];

^{1}H NMR [7821], IR [7821], MS [7821]; TLC [7821].

1-[5-(Acetyloxy)-1,4-dihydroxy-2-naphthalenyl]-1-propanone

[360790-46-1] $C_{15}H_{14}O_5$ mol.wt. 274.27



Synthesis

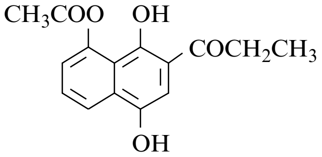
- Obtained by irradiation (409–429 nm) of 5,8-dioxo-5,8-dihydro-1-naphthalenyl acetate in the presence of propionaldehyde in benzene for 18 h at r.t. (41%) [7868].

m.p. 170–171° [7868];

^{1}H NMR [7868], ^{13}C NMR [7868], IR [7868], UV [7868], MS [7868]; HPLC [7868].

1-[8-(Acetyloxy)-1,4-dihydroxy-2-naphthalenyl]-1-propanone

[360790-41-6] $C_{15}H_{14}O_5$ mol.wt. 274.27



Synthesis

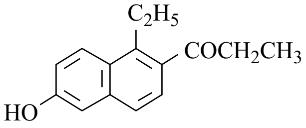
- Obtained by irradiation (409–429 nm) of 5,8-dioxo-5,8-dihydro-1-naphthalenyl acetate in the presence of propionaldehyde in benzene for 18 h at r.t. (43%) [7868].

m.p. 179–181° [7868];

^{1}H NMR [7868], ^{13}C NMR [7868], IR [7868], UV [7868], MS [7868]; HPLC [7868].

1-(1-Ethyl-6-hydroxy-2-naphthalenyl)-1-propanone

[195729-19-2] $C_{15}H_{16}O_2$ mol.wt. 228.29

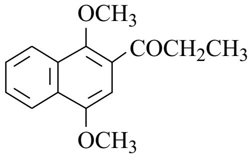


Synthesis

- Refer to: [7809].

1-(1,4-Dimethoxy-2-naphthalenyl)-1-propanone

[477904-75-9] $C_{15}H_{16}O_3$ mol.wt. 244.29

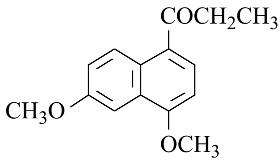


Synthesis

- Refer to: [7869].

1-(4,6-Dimethoxy-1-naphthalenyl)-1-propanone

[81336-14-3] $C_{15}H_{16}O_3$ mol.wt. 244.29



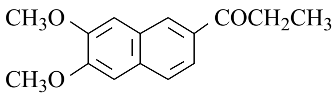
Synthesis

- Preparation by reaction of propionyl chloride with 1,7-dimethoxynaphthalene in the presence of aluminium chloride in nitrobenzene at r.t. for 16 h (75%) [7851].

b.p.$_{0.4}$ 190–195° [7851]; m.p. 126–127° [7851]; ^{1}H NMR [7851], IR [7851].

1-(6,7-Dimethoxy-2-naphthalenyl)-1-propanone

[72337-80-5] $C_{15}H_{16}O_3$ mol.wt. 244.29



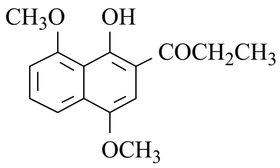
Syntheses

- Preparation by reaction of propionic anhydride with 2,3-dimethoxynaphthalene in the presence of aluminium chloride in nitrobenzene at 0° [7822].
- Also obtained by reaction of propionyl chloride with 2,3-dimethoxynaphthalene in the presence of aluminium chloride in nitrobenzene at r.t. overnight (77%) [7870].
- Also refer to: [7871,7872].

b.p.$_{17}$ 243–244° [7870]; m.p. 102° [7870], 101–103° [7872]; ^{1}H NMR [7872], IR [7872].

1-(1-Hydroxy-4,8-dimethoxy-2-naphthalenyl)-1-propanone

[88792-61-4] $C_{15}H_{16}O_4$ mol.wt. 260.29



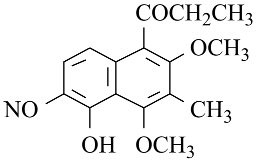
Synthesis

- Obtained by Fries rearrangement of 1-propionoxy-4,8-dimethoxynaphthalene (m.p. 109–110°) [7873] with boron trifluoride etherate (1.1 equiv) at 120° for 5 min (92%) [7873], (good yield) [7874] according to the procedure [7875].

m.p. 129–130° [7873,7874]; ^{1}H NMR [7873], IR [7873], MS [7873].

1-(5-Hydroxy-2,4-dimethoxy-3-methyl-6-nitroso-1-naphthalenyl)-1-propanone

[78377-72-7] $C_{16}H_{17}NO_5$ mol.wt. 303.31



Synthesis

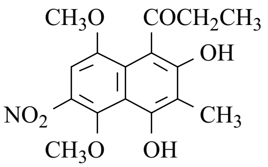
- Obtained by treatment of 6,8-dimethoxy-7-methyl-5-(1-oxopropyl)-1,2-naphthalenedione [78377-71-6] (compound **9**) with hydroxylamine hydrochloride (excess) in ethanol at r.t. [7876].

USE: As intermediate in total synthesis of rifamycine S segment (compound **16b**) [7876].

m.p. 139–140° [7876]; ^{1}H NMR [7876].

1-(2,4-Dihydroxy-5,8-dimethoxy-3-methyl-6-nitro-1-naphthalenyl)-1-propanone

[98291-50-0] $C_{16}H_{17}NO_7$ mol.wt. 335.31



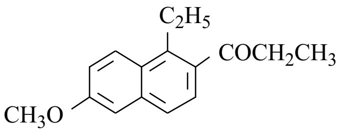
Synthesis

- Obtained by treatment of 1-[5,8-dimethoxy-2,4-bis-(methoxymethoxy)-3-methyl-6-nitro-1-naphthyl]-1-propanone (m.p. 89.5–90.5°) in THF with 1 N hydrochloric acid at 23° for 3 days (quantitative yield) [7877].

orange-yellow crystals [7877]; ^{1}H NMR [7877]; TLC [7877].

1-(1-Ethyl-6-methoxy-2-naphthalenyl)-1-propanone

[195729-50-1] $C_{16}H_{18}O_2$ mol.wt. 242.32

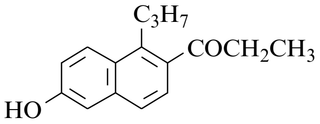


Synthesis

- Refer to: [7809].

1-(6-Hydroxy-1-propyl-2-naphthalenyl)-1-propanone

[195729-60-3] $C_{16}H_{18}O_2$ mol.wt. 242.32

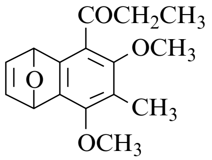


Synthesis

- Refer to: [7809].

1-(1,4-Dihydro-6,8-dimethoxy-7-methyl-1,4-epoxynaphthalene-5-yl)-1-propanone

[90363-43-2] $C_{16}H_{18}O_4$ mol.wt. 274.32



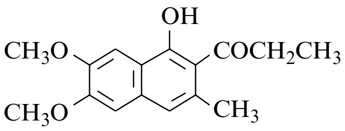
Synthesis

- Obtained by Jones oxidation of 1-(1,4-dihydro-6,8-dimethoxy-7-methyl-1,4-epoxynaphthalene-5-yl)-1-propanol (80%) [7878].

m.p. 74–75° [7878]; ^{1}H NMR [7878].

1-(1-Hydroxy-6,7-dimethoxy-3-methyl-2-naphthalenyl)-1-propanone

[125575-68-0] $C_{16}H_{18}O_4$ mol.wt. 274.32



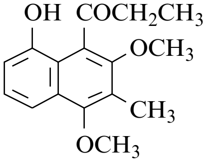
Synthesis

- Preparation by treatment of ethyl 3,4-dimethoxy-6-(phenylsulfinyl)methylbenzoate (m.p. 102–104°), first with LDA in THF at −78°, then 4-hexen-3-one and the mixture refluxed for 12 h (80%) [7879].

^{1}H NMR [7879].

1-(8-Hydroxy-2,4-dimethoxy-3-methyl-1-naphthalenyl)-1-propanone

[90363-44-3] $C_{16}H_{18}O_4$ mol.wt. 274.32



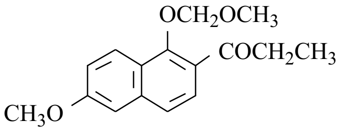
Syntheses

- Preparation by reaction of 60% perchloric acid with 1-(1,4-dihydro-6,8-dimethoxy-7-methyl-1,4-epoxynaphthalen-5-yl)-1-propanone (m.p. 74–75°) in THF at 50° for 50 min (86%) [7878].
- Also obtained by treatment of 3,5-dimethoxy-4-methyl-2*H*-naphtho[1,8-*bc*] furan-2-one [90363-46-5] (m.p. 107–108°) with ethyllithium (1.05 equiv) in ethyl ether at 0° for 1.5 h [7878].

m.p. 179–181° [7878]; ^{1}H NMR [7878], IR [7878], UV [7878].

1-[6-Methoxy-1-(methoxymethoxy)-2-naphthalenyl]-1-propanone

[195730-04-2] $C_{16}H_{18}O_4$ mol.wt. 274.32

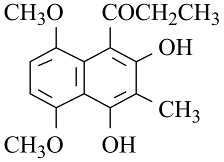


Synthesis

- Refer to: [7809].

1-(2,4-Dihydroxy-5,8-dimethoxy-3-methyl-1-naphthalenyl)-1-propanone

[98291-47-5] $C_{16}H_{18}O_5$ mol.wt. 290.32



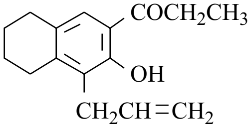
Synthesis

- Preparation by stirring a solution of 1-[2,4-bis (benzyloxy)-5,8-dimethoxy-3-methyl-1-naphthyl]-1-propanone in ethyl acetate with Pd in methanol at 25° for 20 min under hydrogen bubbling (quantitative yield) [7877].

m.p. 111–112° [7877];
^{1}H NMR [7877], IR [7877], UV [7877]; TLC [7877].

1-[5,6,7,8-Tetrahydro-3-hydroxy-4-(2-propenyl)-2-naphthalenyl]-1-propanone

[60401-59-4] $C_{16}H_{20}O_2$ mol.wt. 244.33



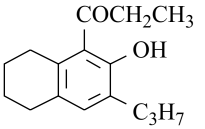
Syntheses

- Preparation by Claisen rearrangement of 1-[5,6,7,8-tetra-hydro-3-(2-propenyloxy)-2-naphthalenyl]-1-propanone [7825,7826].

USE: Intermediate for the preparation of allergy inhibitor [7825].

1-(5,6,7,8-Tetrahydro-2-hydroxy-3-propyl-1-naphthalenyl)-1-propanone

$C_{16}H_{22}O_2$ mol.wt. 246.35



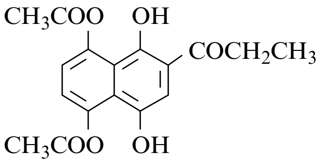
Synthesis

- Obtained by Fries rearrangement of 5,6,7,8-tetrahydro-3-propyl-2-naphthalenyl propionate (b.p.$_{20}$ 192°) with aluminium chloride at 150° for 3 h [7824].

m.p. 83–83.5° [7824].

1-[5,8-Bis(acetyloxy)-1,4-dihydroxy-2-naphthalenyl]-1-propanone

[360790-49-4] $C_{17}H_{16}O_7$ mol.wt. 332.31



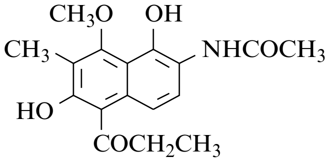
Synthesis

- Obtained by irradiation (409–429 nm) of 4-(acetyloxy)-5,8-dioxo-5,8-dihydro-1-naphthalenyl acetate in the presence of propionaldehyde in benzene for 18 h at r.t. (60%) [7868].

m.p. 159–160° [7868];
^{1}H NMR [7868], ^{13}C NMR [7868], IR [7868], MS [7868]; HPLC [7868].

N-[1,6-Dihydroxy-8-methoxy-7-methyl-5-(1-oxopropyl)-2-naphthalenyl]acetamide

[78377-74-9]



$C_{17}H_{19}NO_5$ mol.wt. 317.34

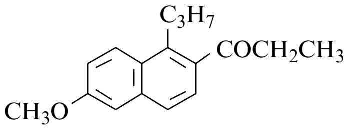
Synthesis

- Obtained by reaction of boron trichloride with N-[1-(acetyloxy)-6,8-dimethoxy-7-methyl-5-(1-oxopropyl)-2-naphthalenyl]acetamide in methylene chloride at –20° (94%) [7876].

m.p. 119–123° [7876].

1-(6-Methoxy-1-propyl-2-naphthalenyl)-1-propanone

[195729-54-5]



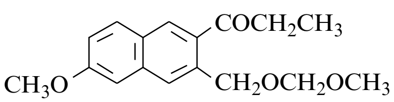
$C_{17}H_{20}O_2$ mol.wt. 256.34

Synthesis

- Refer to: [7809].

1-[6-Methoxy-3-[(methoxymethoxy)methyl]-2-naphthalenyl]-1-propanone

[195730-12-2]



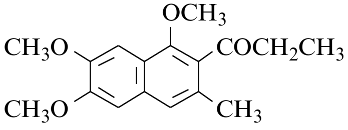
$C_{17}H_{20}O_4$ mol.wt. 288.34

Synthesis

- Refer to: [7809].

1-(1,6,7-Trimethoxy-3-methyl-2-naphthalenyl)-1-propanone

[125575-55-5]



$C_{17}H_{20}O_4$ mol.wt. 288.34

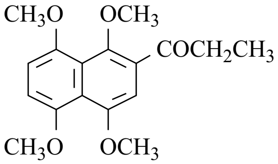
Synthesis

- Preparation by treatment of 1-(1-hydroxy-6,7-di-methoxy-3-methyl-2-naphthalenyl)-1-propanone with methyl iodide in the presence of potassium carbonate in refluxing acetone (95%) [7879].

m.p. 81–82° [7879]; ^{1}H NMR [7879], MS [7879].

1-(1,4,5,8-Tetramethoxy-2-naphthalenyl)-1-propanone

[275803-01-5]



$C_{17}H_{20}O_5$ mol.wt. 304.34

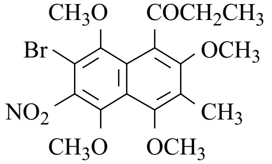
Syntheses

- Obtained from 2-(1-hydroxypropyl)-1,4,5,8-tetramethoxy-naphthalene by oxidation with MnO_2 in refluxing benzene for 10 h (90%) [7880].
- Also refer to: [7881].

m.p. 68.3–69.5° [7880]; ^{1}H NMR [7880], IR [7880].

1-(7-Bromo-2,4,5,8-tetramethoxy-3-methyl-6-nitro-1-naphthalenyl)-1-propanone

[90363-51-2] $C_{18}H_{20}BrNO_7$ mol.wt. 442.26



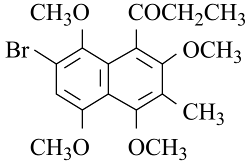
Synthesis

- Obtained by nitration of 1-(7-bromo-2,4,5,8-tetramethoxy-3-methyl-1-naphthalenyl)-1-propanone with copper(II) nitrate in acetic anhydride at −20° for 2 h (56%) [7878].

m.p. 154–155° [7878]; ^{1}H NMR [7878].

1-(7-Bromo-2,4,5,8-tetramethoxy-3-methyl-1-naphthalenyl)-1-propanone

[90363-49-8] $C_{18}H_{21}BrO_5$ mol.wt. 397.27



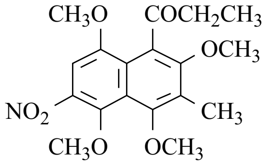
Synthesis

- Obtained by methylation of 1-(7-bromo-5,8-dihydroxy-2,4-dimethoxy-3-methyl-1-naphthalenyl)-1-propanone with dimethyl sulfate in acetone in the presence of potassium carbonate (74%) [7878].

m.p. 87–88° [7878]; ^{1}H NMR [7878].

1-(2,4,5,8-Tetramethoxy-3-methyl-6-nitro-1-naphthalenyl)-1-propanone

[90363-52-3] $C_{18}H_{21}NO_7$ mol.wt. 363.37



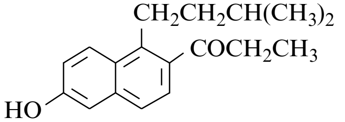
Syntheses

- Preparation by nitration of 1-(2,4,5,8-tetramethoxy-3-methyl-1-naphthalenyl)-1-propanone with copper (II) nitrate in acetic anhydride at −20° (80%) [7878].
- Also obtained by debromination of 1-(7-bromo-2,4,5,8-tetramethoxy-3-methyl-6-nitro-1-naphthalenyl)-1-propanone with sodium formate [$(Ph_3P)_4Pd$, DMF, 100°, 3 h] [7878].
- Also obtained by methylation of 1-(2,4-dihydroxy-5,8-dimethoxy-3-methyl-6-nitro-1-naphthalenyl)-1-propanone (SM) with dimethyl sulfate in the presence of potassium carbonate in acetone at 20° for 5 h (94%). SM was obtained by treatment of 1-(5,8-dimethoxy-2,4-bis(methoxymethoxy)-3-methyl-6-nitro-1-naphthalenyl)-1-propanone in THF with 1 N HCl at 23° for 3 days (100%) [7877].

m.p. 126–127° [7878]; ^{1}H NMR [7877,7878],
IR [7877,7878], UV [7877,7878],
X-ray crystallography [7878]; TLC [7877,7878].

1-[6-Hydroxy-1-(3-methylbutyl)-2-naphthalenyl]-1-propanone

[195729-79-4]



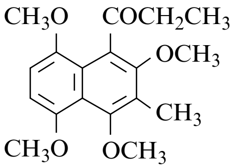
$C_{18}H_{22}O_2$ mol.wt. 270.37

Synthesis

– Refer to: [7809] (Japanese patent).

1-(2,4,5,8-Tetramethoxy-3-methyl-1-naphthalenyl)-1-propanone

[90363-50-1]



$C_{18}H_{22}O_5$ mol.wt. 318.37

Syntheses

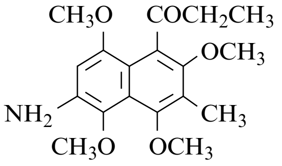
– Preparation by debromination of 1-(7-bromo-2,4,5,8-tetramethoxy-3-methyl-1-naphthalenyl)-1-propanone with zinc powder in 10% aqueous sodium hydroxide-dioxane (1:2 v/v) at 70° for 12 h (100%) [7878].

– Also obtained by reductive methylation of 5,7-dimethoxy-6-methyl-8-propionyl-1,4-naphthoquinone (60%) [7878].

m.p. 76–77° [7878]; ^{1}H NMR [7878].

1-(6-Amino-2,4,5,8-tetramethoxy-3-methyl-1-naphthalenyl)-1-propanone

[90363-53-4]



$C_{18}H_{23}NO_5$ mol.wt. 333.38

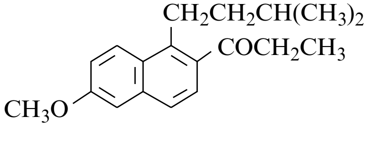
Synthesis

– Obtained by catalytic reduction of 1-(2,4,5,8-tetramethoxy-3-methyl-6-nitro-1-naphthalenyl)-1-propanone with hydrogen in methanol in the presence of 5% Pd/C [7878].

^{1}H NMR [7878].

1-[6-Methoxy-1-(3-methylbutyl)-2-naphthalenyl]-1-propanone

[195729-78-3]



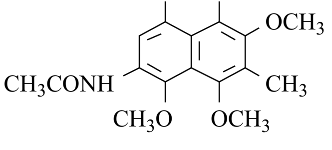
$C_{19}H_{24}O_2$ mol.wt. 284.40

Synthesis

– Refer to: [7809] (Japanese patent).

N-[1,4,6,8-Tetramethoxy-7-methyl-5-(1-oxopropyl)-2-naphthalenyl]acetamide

[90363-54-5]



$C_{20}H_{25}NO_6$ mol.wt. 375.42

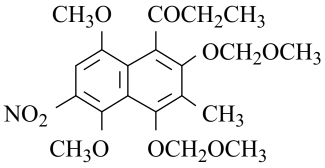
Synthesis

– Obtained by acetylation of 1-(6-amino-2,4,5,8-tetra-methoxy-3-methyl-1-naphthalenyl)-1-propanone with acetic anhydride in the presence of pyridine (95%) [7878].

m.p. 193–194° [7878]; ^{1}H NMR [7878].

1-[5,8-Dimethoxy-2,4-bis(methoxymethoxy)-3-methyl-6-nitro-1-naphthalenyl]-1-propanone

[98291-49-7]



$C_{20}H_{25}NO_9$ mol.wt. 423.42

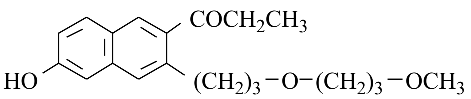
Synthesis

- Obtained by adding a solution of 1-[5,8-dimethoxy-2,4-bis(methoxymethoxy)-3-methyl-1-naphthalenyl]-1-propanone in acetic anhydride to a mixture of copper(II) nitrate trihydrate in acetic anhydride at –40° and stirring at –40° for 1 h (77%) [7877].

m.p. 89.5–90.5° [7877]; ^{1}H NMR [7877], IR [7877], UV [7877].

1-[6-Hydroxy-3-[3-(3-methoxypropoxy)propyl]-2-naphthalenyl]-1-propanone

[181236-19-1]



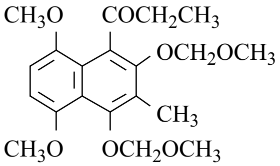
$C_{20}H_{26}O_4$ mol.wt. 330.42

Synthesis

- Refer to: [7882] (Japanese patent).

1-[5,8-Dimethoxy-2,4-bis(methoxymethoxy)-3-methyl-1-naphthalenyl]-1-propanone

[98291-48-6]



$C_{20}H_{26}O_7$ mol.wt. 378.42

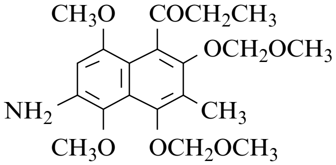
Synthesis

- Preparation by adding chloromethyl methyl ether to an ice-cold solution of 1-(2,4-dihydroxy-5,8-dimethoxy-3-methyl-1-naphthalenyl)-1-propanone in THF, solution previously treated with sodium hydride (95%) [7877].

pale yellow syrup [7877];
^{1}H NMR [7877], IR [7877], UV [7877]; TLC [7877].

1-[6-Amino-5,8-dimethoxy-2,4-bis(methoxymethoxy)-3-methyl-1-naphthalenyl]-1-propanone

[98291-51-1]



$C_{20}H_{27}NO_7$ mol.wt. 394.44

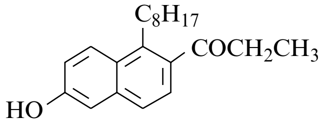
Synthesis

- Preparation by reaction of 1-[5,8-dimethoxy-2,4-bis-(methoxymethoxy)-3-methyl-6-nitro-1-naphthalenyl]-1-propanone with hydrogen in methanol in the presence of 5% Pd/C (100%) [7877].

yellow-brown syrup [7877].

1-(6-Hydroxy-1-octyl-2-naphthalenyl)-1-propanone

[195729-93-2]



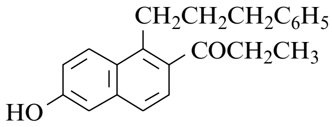
$C_{21}H_{28}O_2$ mol.wt. 312.45

Synthesis

- Refer to: [7809].

1-[6-Hydroxy-1-(3-phenylpropyl)-2-naphthalenyl]-1-propanone

[195729-70-5]



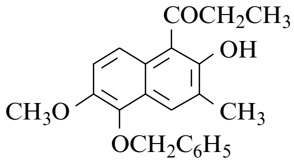
$C_{22}H_{22}O_2$ mol.wt. 318.42

Synthesis

- Refer to: [7809].

1-[2-Hydroxy-6-methoxy-3-methyl-5-(phenylmethoxy)-1-naphthalenyl]-1-propanone

[185413-94-9]



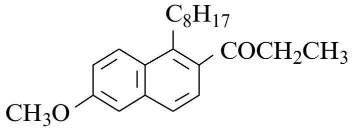
$C_{22}H_{22}O_4$ mol.wt. 350.41

Synthesis

- Preparation by treatment of the tetralone 24—two diastereoisomeric 1-acetoxy-5-benzyloxy-3,4-dihydro-6-methoxy-3-methyl-1-propionylnaphthalen-2(1*H*)-ones—in methylene chloride with silica gel for 16 h (81%) [7836].

1-(6-Methoxy-1-octyl-2-naphthalenyl)-1-propanone

[195729-92-1]



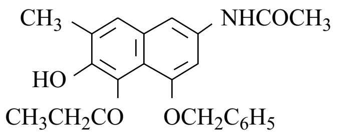
$C_{22}H_{30}O_2$ mol.wt. 326.48

Synthesis

- Refer to: [7809].

N-[6-Hydroxy-7-methyl-5-(1-oxopropyl)-4-(phenylmethoxy)-2-naphthalenyl] acetamide

[185413-96-1]



$C_{23}H_{23}NO_4$ mol.wt. 377.44

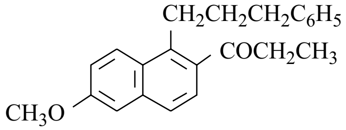
Synthesis

- Obtained by treatment of the tetralone 27—two diastereoisomeric 1-acetoxy-6-acetylamino-8-benzyloxy-3,4-dihydro-3-methyl-1-propionyl-naphthalen-2(1*H*)-ones — in methylene chloride with silica gel (pH 10) (80%) [7836].

m.p. 93–95° [7836];
^{1}H NMR [7836], ^{13}C NMR [7836], IR [7836], MS [7836].

1-[6-Methoxy-1-(3-phenylpropyl)-2-naphthalenyl]-1-propanone

[195729-68-1] $C_{23}H_{24}O_2$ mol.wt. 332.44

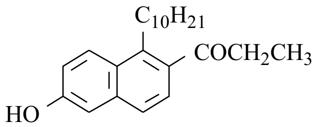


Synthesis

- Refer to: [7809].

1-(1-Dodecyl-6-hydroxy-2-naphthalenyl)-1-propanone

[195729-86-3] $C_{23}H_{32}O_2$ mol.wt. 340.51

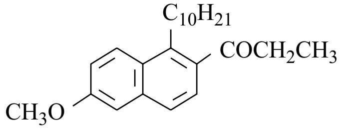


Synthesis

- Refer to: [7809].

1-(1-Dodecyl-6-methoxy-2-naphthalenyl)-1-propanone

[195729-85-2] $C_{24}H_{34}O_2$ mol.wt. 354.53

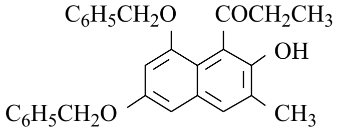


Synthesis

- Refer to: [7809].

1-[2-Hydroxy-3-methyl-6,8-bis(phenylmethoxy)-1-naphthalenyl]-1-propanone

[185413-95-0] $C_{28}H_{26}O_4$ mol.wt. 426.51



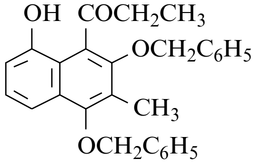
Synthesis

- Obtained from 1-(3,5-dibenzyloxyphenyl)-2-methylheptane-3,5-dione by treatment with manganese (III) acetate in acetic acid (68%) [7836].

m.p. 36–38° [7836]; ^{1}H NMR [7836], IR [7836], MS [7836].

1-[8-Hydroxy-3-methyl-2,4-bis(phenylmethoxy)-1-naphthalenyl]-1-propanone

[98291-44-2] $C_{28}H_{26}O_4$ mol.wt. 426.51



Syntheses

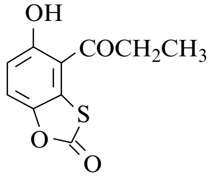
- Preparation by treatment of 1-[2,4-bis(benzyloxy)-5,8-dihydro-3-methyl-5,8-epoxy-1-naphthyl]-1-propanone (m.p. 112–114°) in THF with 60% perchloric acid at 25° for 35 h (81%) [7877].
- Also obtained by adding dropwise 1.60 M ethyllithium in ethyl ether to an ice-cold suspension of 3,5-bis(benzyloxy)-4-methyl-2*H*-naphtho[1,8-*bc*]furan-2-one (m.p. 136–137°) in ethyl ether. After being stirred at 0° for 1 h, a saturated aqueous ammonium chloride solution was added to the mixture (26%) [7877].

m.p. 188–190° (d) [7877];
^{1}H NMR [7877], IR [7877], UV [7877]; TLC [7877].

23.3 Heterocyclic Derivatives

5-Hydroxy-4-(1-oxopropyl)-1,3-benzoxathiol-2-one

[112450-17-6] $C_{10}H_8O_4S$ mol.wt. 224.24



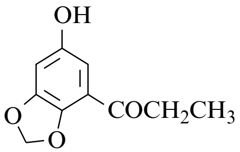
Synthesis

- Preparation by adding a solution of 2-propionyl-1,4-benzoquinone in acetic acid to a solution of thiourea in 2 N hydrochloric acid, stirring at r.t. for 30 min, then heating on a steam bath for 1 h (74%) [7883].

m.p. 194–195° [7883]; ^{1}H NMR [7883], ^{13}C NMR [7883].

1-(6-Hydroxy-1,3-benzodioxol-4-yl)-1-propanone

$C_{10}H_{10}O_4$ mol.wt. 194.19



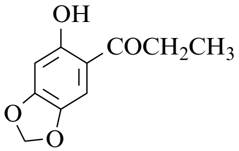
Synthesis

- Refer to: [7884] *(Hypothetical compound).*

1-(6-Hydroxy-1,3-benzodioxol-5-yl)-1-propanone *(Kakuol)*

2-Hydroxy-4,5-methylenedioxypropiophenone

[18607-90-4] $C_{10}H_{10}O_4$ mol.wt. 194.19



Syntheses

- Obtained from sesamolyl propionate with aluminium chloride in refluxing carbon disulfide for 1 h [7885].
- Synthesized from sesamol [7886].

Isolation from natural sources

- From *Asarum* root oil [7887] of China [7888].
- From *Asarum forbesii* [7889].
- From the essential oils from five species of *Asarum* namely, *Asarum heterotropoides* variety mandshuricum, *Asarum sieboldii* (cultivated), *Asarum caudigerellum* (from Sichuan), *Asarum sieboldii* (from Shandong), *Asarum sieboldii* (wild) [7890] and *Asarum sieboldii* rhizome [7891].
- From *Asarum sieboldii* forma seoulense, *Asarum forbesii*, *Asarum inflatum* variety dinghugense and *Asarum caudigerum* variety cardiophyllum [7892].
- From *Asiasarum* [7886].
- From *Asiasarum heterotropoides* MAEK. variety *mandshuricum* MAEK. [7893].

- From the root and rhizomes of *Asarum heterotropoides* variety *mandshuricum* [7894].
- From the leaves of *Piper marginatum* Jacq. (Piperaceae) [7895,7896].
- From volatile oil of the Chinese drug chichin, *Asiasarum heterotropoides* variety *seoulense* [7885].
- From essential oil of *Asiasari Radix* [7897].
- Absorption, distribution and excretion of *[3H]-labeled Kakuol* in mice and rats [7898].
- Also refer to: [7899–7901].

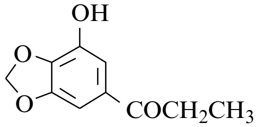
N.B.: The formula of *Kakuol* represented in Chem. Abstr., **104**, 199940a (1986) was erroneous.

m.p. 110° [7885]; ^{1}H NMR [7902], ^{13}C NMR [7902];
GC-MS [7888,7890,7892,7896,7897]; GC [7885,7887].
USE: Fungicide [7886,7891].

BIOLOGICAL ACTIVITY: Antiviral [7894].

1-(7-Hydroxy-1,3-benzodioxol-5-yl)-1-propanone ***(Demethyllatifolon) (Radiatinol)***
3-Hydroxy-4,5-methylenedioxypropiophenone
3,4-Methylenedioxy-5-hydroxypropiophenone
1-(3,4-Methylenedioxy-5-hydroxyphenyl)-propan-1-one
4-Hydroxy-5,6-methylenedioxy-2-(1-oxopropyl)benzene

[83016-68-6] $C_{10}H_{10}O_4$ mol.wt. 194.19



Syntheses

- Refer to: [7903] (compound **12**), [7904].

Isolation from natural sources

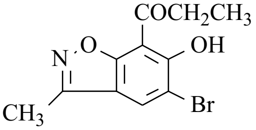
- From *Ferula communis* subsp *communis* (compound **20**) [7905].
- From *Ferula elaeochytris* (Umbelliferae) (compound **2**) [7884].
- From *Sphallerocarpus gracilis* (Umbelliferae) (compound **1**) [7906].
- From the underground parts of *Laserpitium siler L.* of Slovenian origin [7907].
- From the underground parts of *Laser trilobum* (L.) Borkh. species [7908].
- From the roots of *Peucedanum pauciradiatum* (Umbelliferae) [7909].

USE: Antifeedant activity against granary pests [7910] and insect [7904].

m.p. 169–170° [7909], 161–164° [7907], 161–162° [7906], 159–162° [7908], 154–156° [7884];
^{1}H NMR [7884,7906,7907,7909], ^{13}C NMR [7884,7906],
IR [7884,7907,7909], UV [7884], MS [7884,7907];
GC [7911]; GC/MS [7911].

1-(5-Bromo-6-hydroxy-3-methyl-1,2-benzisoxazol-7-yl)-1-propanone

[73860-03-4] $C_{11}H_{10}BrNO_3$ mol.wt. 284.11



Syntheses

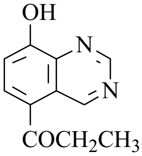
- Preparation by reaction of bromine with 6-hydroxy-3-methyl-7-propionyl-1,2-benzisoxazole in acetic acid at r.t. for 2 h (87%) [7912].
- Also refer to: [7913].

m.p. 146–147° [7912,7913]; ^{1}H NMR [7912], IR [7912,7913].

Oxime [73860-07-8] $C_{11}H_{11}BrN_2O_3$ mol.wt. 299.12 (m.p. 216–217°) [7912].

1-(8-Hydroxy-5-quinazolinyl)-1-propanone

[185437-45-0] $C_{11}H_{10}N_2O_2$ mol.wt. 202.22

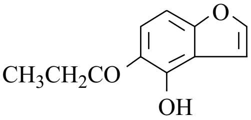


Synthesis

- Refer to: [7914] (Japanese patent).

1-(4-Hydroxy-5-benzofuranyl)-1-propanone

[124210-94-2] $C_{11}H_{10}O_3$ mol.wt. 190.20



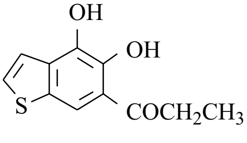
Synthesis

- Obtained by ozonolysis of 3-allyl-2,4-dihydroxypropiophenone in ethyl acetate at −10° for 1 h and subsequent ring closure of the resulting aldehyde using orthophosphoric acid (50%) [7915].

m.p. 92–94° [7915]; ^{1}H NMR [7915], IR [7915].

1-(4,5-Dihydroxybenzo[*b*]thien-6-yl)-1-propanone

[912952-35-3] $C_{11}H_{10}O_3S$ mol.wt. 222.26



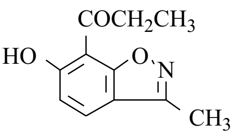
Synthesis

- Obtained by photoacylation of benzo[*b*]thiophene-4,5-dione with propionaldehyde in benzene (31%) [7916].

m.p. 138–140° [7916];
^{1}H NMR [7916], IR [7916].

1-(6-Hydroxy-3-methyl-1,2-benzisoxazol-7-yl)-1-propanone

[68241-22-5] $C_{11}H_{11}NO_3$ mol.wt. 205.21



Syntheses

- Preparation by Fries rearrangement of 6-propionoxy-3-methyl-1,2-benzisoxazole with aluminium chloride at 140° for 2 h [7917], (70%) [7918].

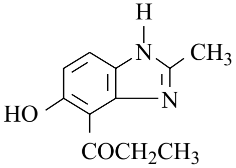
- Also refer to: [7267,7912,7913,7919].

m.p. 88° [7918]; ^{1}H NMR [7918].

Oxime [68241-40-7] $C_{11}H_{12}N_2O_3$ mol.wt. 220.23 (m.p. 184°) [7918].

1-(5-Hydroxy-2-methyl-1*H*-benzimidazol-4-yl)-1-propanone

[137538-58-0] $C_{11}H_{12}N_2O_2$ mol.wt. 204.23

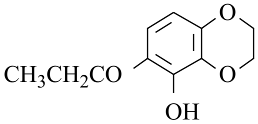


Synthesis

- Obtained by Friedel–Crafts acylation of 5-hydroxy-2-methylbenzimidazole with propionic anhydride [7920].

1-(2,3-Dihydro-5-hydroxy-1,4-benzodioxin-6-yl)-1-propanone

$C_{11}H_{12}O_4$ mol.wt. 208.21



Synthesis

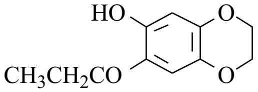
- Refer to: [7921].

Oxime [34747-40-5] $C_{11}H_{13}NO_4$ mol.wt. 223.23.

- A reagent for photometric determination of titanium [7921].

1-(2,3-Dihydro-7-hydroxy-1,4-benzodioxin-6-yl)-1-propanone

[859403-57-9] $C_{11}H_{12}O_4$ mol.wt. 208.21

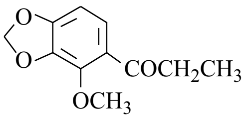


Synthesis

- Obtained by Fries rearrangement of 2,3-dihydro-6-propionyloxy-1,4-benzodioxin with titanium tetrachloride in 1,2-dichloroethane at r.t. [7922].

1-(4-Methoxy-1,3-benzodioxol-5-yl)-1-propanone

[340016-41-3] $C_{11}H_{12}O_4$ mol.wt. 208.21



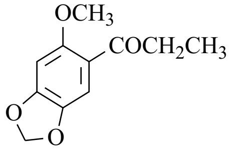
Isolation from natural sources

- From *Anethum sowa* Roxb. (Apiaceae) [7923].

m.p. 67° [7923];
^{1}H NMR [7923], IR [7923], MS [7923].

1-(6-Methoxy-1,3-benzodioxol-5-yl)-1-propanone

[70342-29-9] $C_{11}H_{12}O_4$ mol.wt. 208.21



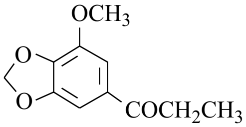
Isolation from natural sources

- From the *Piper aleyreanum* [7924] (Piperaceae).
- From the roots of *Piper marginatum* [7902].
- From *Asiasarum sieboldi* roots [7925].

m.p. 69–71° [7895].
USE: Fungicide [7886].

1-(7-Methoxy-1,3-benzodioxol-5-yl)-1-propanone *(Crocatone) (Latifolone)*

[19937-86-1] $C_{11}H_{12}O_4$ mol.wt. 208.21



Syntheses

- Preparation by methylation of 1-(3,4-methylenedioxy-5-hydroxyphenyl)-1-propanone with diazomethane [7909].
- Preparation by Jones oxidation of the intermediate alcohol obtained by reaction of ethylmagnesium bromide with 3-methoxy-4,5-(methylenedioxy) benzaldehyde [7926].

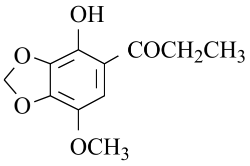
Isolation from natural sources

- From the root of *Laserpitium latifolium* L. (Umbelliferae) [7927–7930].
- From the roots of *Laserpitium siler* L. (Umbelliferae) [7907].
- From *Laser trilobum* L. BORKH (Umbelliferae) [7929].
- From the roots of *Ferula tunshanica Su* [7931].
- From *Ferula sinaica* (Umbellifereae) [7932,7933].
- From *Ferula arrigoni* (Umbellifereae) [7934].
- From *Ferula licentiana* variety *tunshanica* and *Ferula kingdonwardii* [7935,7936].
- From the roots of *Thapsia garganica* L. (Umbellifereae) [7911].
- From the roots of *Thapsia villosa* (Umbellifereae) [7937–7939].
- Also refer to: [7433,7940].

m.p. 89–90° [7929], 88–89° [7926], 88° [7927–7930], 86–88° [7937];
^{1}H NMR [7907,7937,7941], ^{13}C NMR [7932],
IR [7928,7930,7937,7942],
UV [7928,7930,7942], MS [7932,7937];
Chromatography [7928]; polarography [7928,7930]; TLC [7937].

1-(4-Hydroxy-7-methoxy-1,3-benzodioxol-5-yl)-1-propanone

[119426-01-6] $C_{11}H_{12}O_5$ mol.wt. 224.21



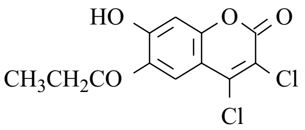
Isolation from natural sources

- From the roots and aerial parts of *Ferula latipinna* (Umbelliferae) [7943].

m.p. 95–97° [7943];
^{1}H NMR [7943], IR [7943], MS [7943].

3,4-Dichloro-7-hydroxy-6-(1-oxopropyl)-2*H*-1-benzopyran-2-one

[63876-51-7] $C_{12}H_8Cl_2O_4$ mol.wt. 287.10



Synthesis

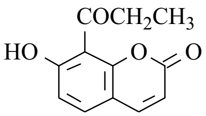
- Obtained in two steps: First, reaction of aluminium chloride (6 mol) with respropiophenone (2 mol) in methylene chloride, the stirring continued till the evolution of hydrogen chloride gas ceased (3 h). Then, hexachloropropene (2 mol) was added and the reaction mixture was stirred for 3 more hours [7944].

m.p. 148–149° [7944]; ^{1}H NMR [7944], IR [7944], MS [7944].

7-Hydroxy-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one

7-Hydroxy-8-propionylcoumarin
8-Propionylumbelliferone

[67752-18-5] $C_{12}H_{10}O_4$ mol.wt. 218.21



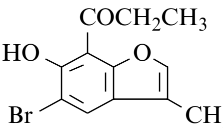
Synthesis

- Obtained by Fries rearrangement of 7-propionoxycoumarin (m.p. 94°) with aluminium chloride for 1 h at 150–160° (45%) [6806].

m.p. 168° [6806].

5-Bromo-6-hydroxy-3-methyl-7-(1-oxopropyl)benzofuran

[100953-75-1] $C_{12}H_{11}BrO_3$ mol.wt. 283.12



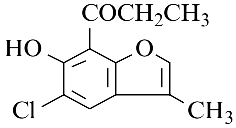
Synthesis

- Obtained by decarboxylation of 5-Bromo-6-hydroxy-3-methyl-7-propionylcoumarilic acid on heating for 10 min at 255° [7945].

m.p. 104–105° [7945].

5-Chloro-6-hydroxy-3-methyl-7-(1-oxopropyl)benzofuran

[104095-32-1] $C_{12}H_{11}ClO_3$ mol.wt. 238.67



Synthesis

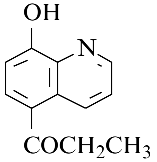
- Obtained by decarboxylation of 5-chloro-6-hydroxy-3-methyl-7-propionylcoumarilic acid on heating for 10 min at 255° [7945].

m.p. 114° [7945].

8-Hydroxy-5-(1-oxopropyl)quinoline

1-(8-Hydroxy-[5]quinolyl)-propane-1-one

[91569-25-4] $C_{12}H_{11}NO_2$ mol.wt. 201.22



Syntheses

- Obtained by Friedel–Crafts acylation of 8-hydroxy-quinoline with propionyl chloride in the presence of aluminium chloride in nitrobenzene [7946,7947] at 70–80° (20%) [7948,7949].

m.p. 127–128° [7949], 124–125° [7946], 91° [7948]. One of the reported melting points is obviously wrong.

USE: Fungicide [7946].

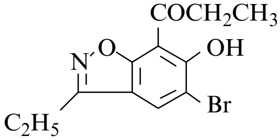
Hydrochloride $C_{12}H_{11}NO_2$, HCl mol.wt. 237.68 (m.p. 279–280° (d) [7948], 224° [7949]).

One of the reported melting points is obviously wrong.

- Refer to: [7948,7949].

1-(5-Bromo-3-ethyl-6-hydroxy-1,2-benzisoxazol-7-yl)-1-propanone

[73860-04-5] $C_{12}H_{12}BrNO_3$ mol.wt. 298.16



Synthesis

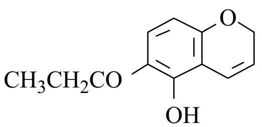
- Preparation by reaction of bromine with 3-ethyl-6-hydroxy-7-propionyl-1,2-benzisoxazole in acetic acid at r.t. for 2 h (80%) [7912].

m.p. 109–110° [7912]; ^{1}H NMR [7912], IR [7912].

Oxime [73860-08-9] $C_{12}H_{13}BrN_2O_3$ mol.wt. 313.15 (m.p. 185°) [7912].

1-(5-Hydroxy-2*H*-1-benzopyran-6-yl)-1-propanone

[457628-04-5] $C_{12}H_{12}O_3$ mol.wt. 204.23



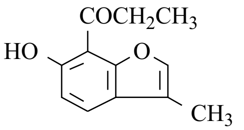
Syntheses

- Refer to: [6524–6526].

1-(6-Hydroxy-3-methyl-7-benzofuranyl)-1-propanone

6-Hydroxy-3-methyl-7-propionylbenzofuran

[35093-15-3] $C_{12}H_{12}O_3$ mol.wt. 204.23



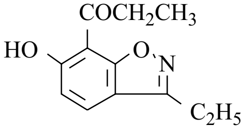
Syntheses

- Obtained by decarboxylation of 6-hydroxy-3-methyl-7-propionylcoumarilic acid on heating for 10 min at 248° [7950].
- Also obtained from 3-chloro-4-methyl-8-propionyl-coumarin by heating with 10% aqueous sodium carbonate for 2 h on a water bath [7950].
- Also obtained by treatment of 3-bromo-7-hydroxy-8-propionyl-4-methylcoumarin with refluxing 7% aqueous sodium carbonate for 1 h (25%) [7951].

m.p. 98° [7950], 92° [7951].

1-(3-Ethyl-6-hydroxy-1,2-benzisoxazol-7-yl)-1-propanone

[68241-23-6] $C_{12}H_{13}NO_3$ mol.wt. 219.24



Syntheses

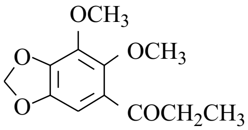
- Preparation by Fries rearrangement of 6-propionoxy-3-ethyl-1,2-benzisoxazole with aluminium chloride at 135–140° for 2 h (65%) [7918].
- Also refer to: [7912].

m.p. 65° [7918]; ^{1}H NMR [7918].

Oxime [68241-42-9] $C_{12}H_{14}N_2O_3$ mol.wt. 234.25 (m.p. 146°) [7918].

1-(4,5-Dimethoxy-1,3-benzodioxol-6-yl)-1-propanone

$C_{12}H_{14}O_5$ mol.wt. 238.24



Synthesis

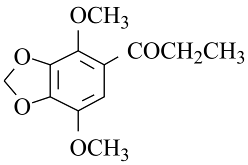
- Obtained by treatment of isoapiole dibromide (m.p. 105°) with potassium hydroxide [7952].

N.B.: This isoapiole was prepared from apiole (Dill) [6640] by allylic-propenylic rearrangement (Claisen).
b.p.$_{760}$ 260° [7952].

Semicarbazone $C_{13}H_{17}N_3O_5$ mol.wt. 295.30 (m.p. 159–160°) [7952].

1-(4,7-Dimethoxy-1,3-benzodioxol-5-yl)-1-propanone *(Methoxylatifolone)*

[107882-48-4] $C_{12}H_{14}O_5$ mol.wt. 238.24



Syntheses

- Obtained by treatment of isoapiole dibromide (m.p. 70–72°) with sodium methoxide in refluxing methanol for 5 h [7243].

N.B. This isoapiole is also named apiole (Parsley) [6640].

- Also refer to: [7953].

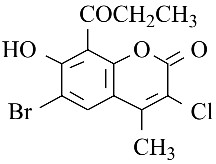
Isolation from natural sources

- From roots of *Ferula ovina* Boiss. (Umbelliferae) [7954] and *Ferula sinaica* Boiss growing in Egypt [7933].

m.p. 95.5° [7243].

6-Bromo-3-chloro-7-hydroxy-4-methyl-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one

6-Bromo-3-chloro-7-hydroxy-4-methyl-8-propionylcoumarin



$C_{13}H_{10}BrClO_4$ mol.wt. 345.58

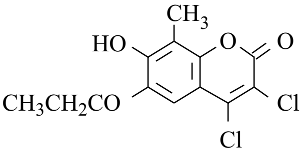
Synthesis

- Obtained by Fries rearrangement of 6-bromo-3-chloro-4-methyl-7-propionyloxycoumarin (m.p. 136°) with aluminium chloride for 90 min at 155° [7945].

m.p. 190° [7945].

3,4-Dichloro-7-hydroxy-8-methyl-6-(1-oxopropyl)-2*H*-1-benzopyran-2-one

[63876-52-8]



$C_{13}H_{10}Cl_2O_4$ mol.wt. 301.13

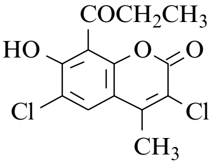
Synthesis

- Obtained in two steps: First, reaction of aluminium chloride (6 mol) with 2,4-dihydroxy-3-methylpropiophenone (2 mol) in methylene chloride, the stirring continued till the evolution of hydrogen chloride gas ceased (3 h). Then, hexachloropropene (2 mol) was added and the reaction mixture was stirred for 3 more hours [7944].

m.p. 191–192° [7944]; ^{1}H NMR [7944], IR [7944], MS [7944].

3,6-Dichloro-7-hydroxy-4-methyl-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one

3,6-Dichloro-7-hydroxy-4-methyl-8-propionylcoumarin



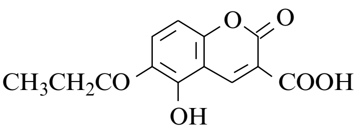
$C_{13}H_{10}Cl_2O_4$ mol.wt. 301.13

Synthesis

- Obtained by Fries rearrangement of 3,6-dichloro-4-methyl-7-propionyloxycoumarin with aluminium chloride for 90 min at 155° [7945].

5-Hydroxy-6-(1-oxopropyl)-2*H*-1-benzopyran-2-one-3-carboxylic acid

5-Hydroxy-6-propionylcoumarin-3-carboxylic acid



$C_{13}H_{10}O_6$ mol.wt. 262.22

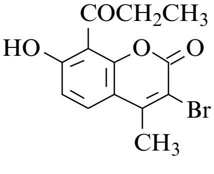
Syntheses

- Preparation from cyanoacetic acid and 2,4-dihydroxy-3-formylpropiophenone in 20% sodium hydroxide solution (59%) [6768].
- Also refer to: [7955].

m.p. 185–186° [6768].

3-Bromo-7-hydroxy-4-methyl-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one

[35093-14-2]



$C_{13}H_{11}BrO_4$ mol.wt. 311.13

Synthesis

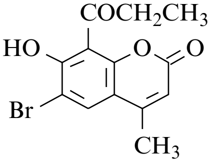
- Obtained by reaction of bromine with 7-hydroxy-8-propionyl-4-methylcoumarin in hot acetic acid for 10 min (49%) [7951].

m.p. 214° [7951].

6-Bromo-7-hydroxy-4-methyl-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one

6-Bromo-7-hydroxy-4-methyl-8-propionylcoumarin

[109099-37-8]



$C_{13}H_{11}BrO_4$ mol.wt. 311.13

Syntheses

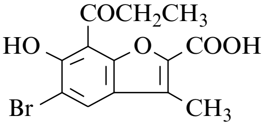
- Obtained by Pechmann condensation of 4-bromo-2-propionylresorcinol with ethyl acetoacetate [6450].
- Also obtained by Fries rearrangement of 7-(pro pionyloxy)-6-bromo-4-methylcoumarin (m.p. 128°) with aluminium chloride (3.3 mol) at 150° for 1 h (36%) [6450].

m.p. 198° [6450].

5-Bromo-6-hydroxy-3-methyl-7-(1-oxopropyl)benzofuran-2-carboxylic acid

5-Bromo-6-hydroxy-3-methyl-7-propionylcoumarilic acid

[108245-61-0]



$C_{13}H_{11}BrO_5$ mol.wt. 327.13

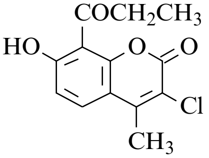
Synthesis

- Obtained by Fries rearrangement of 5-bromo-3-methyl-6-(propionyloxy)coumarilic acid (m.p. 207–208°) with aluminium chloride by heating for 1 h at 150–160° [7945].

m.p. 273–274° [7945].

3-Chloro-7-hydroxy-4-methyl-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one

[110054-61-0] $C_{13}H_{11}ClO_4$ mol.wt. 266.68



Synthesis

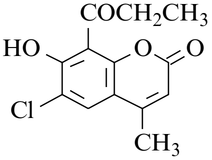
- Obtained by Fries rearrangement of 3-chloro-4-methyl-7-(propionyloxy)coumarin (m.p. 140°) with aluminium chloride at 150–155° for 1 h [7950].

m.p. 206–207° [7950].

6-Chloro-7-hydroxy-4-methyl-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one

6-Chloro-7-hydroxy-4-methyl-8-propionylcoumarin

[110030-74-5] $C_{13}H_{11}ClO_4$ mol.wt. 266.68



Syntheses

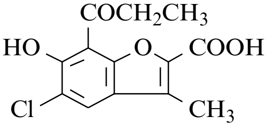
- Obtained by Pechmann condensation of 4-chloro-2-propionylresorcinol with ethyl acetoacetate (60%) [6450].
- Also obtained by Fries rearrangement of 6-chloro-4-methyl-7-(propionyloxy)coumarin (m.p. 111°) with aluminium chloride at 140–150° for 1 h (40%) [6450].

m.p. 181° [6450].

5-Chloro-6-hydroxy-3-methyl-7-(1-oxopropyl)benzofuran-2-carboxylic acid

5-Chloro-6-hydroxy-3-methyl-7-propionylcoumarilic acid

[109218-76-0] $C_{13}H_{11}ClO_5$ mol.wt. 282.68



Synthesis

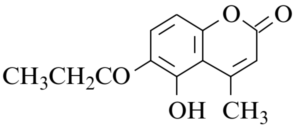
- Obtained by Fries rearrangement of 5-chloro-3-methyl-6-(propionyloxy)coumarilic acid (m.p. 201°) with aluminium chloride by heating for 1 h at 150–160° [7945].

m.p. 276° [7945].

5-Hydroxy-4-methyl-6-(1-oxopropyl)-2*H*-1-benzopyran-2-one

5-Hydroxy-4-methyl-6-propionylcoumarin

[39818-44-5] $C_{13}H_{12}O_4$ mol.wt. 232.24



Syntheses

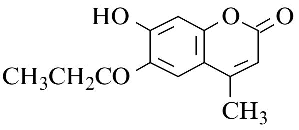
- Obtained by condensation of respropiophenone with ethyl acetoacetate in the presence of aluminium chloride in nitrobenzene at 120–130° (39%) [6750].
- Also obtained by Fries rearrangement of 4-methyl-5-(propionyloxy)coumarin with aluminium chloride at 120–130° for 2 h [6750].

m.p. 164–165° [6750].

7-Hydroxy-4-methyl-6-(1-oxopropyl)-2*H*-1-benzopyran-2-one

7-Hydroxy-4-methyl-6-propionylcoumarin
6-Propionyl-4-methylumbelliferone

[39818-42-3] $C_{13}H_{12}O_4$ mol.wt. 232.24



Syntheses

- Obtained by Fries rearrangement of 4-methylumbelliferone propionate with aluminium chloride at 160–165° for 1 h [6800].
- Also obtained from 2,4-dihydroxy-5-propionyl-β-methyl-cinnamic acid by cyclisation with sulfuric acid or on heating above its melting point. It is partly dehydrated yielding the titled ketone [6800].
- Also obtained by reaction of acetoacetic ester with respropiophenone in the presence of phosphorous oxychloride in refluxing benzene for 3 h (25%) [6752].

m.p. 228° [6800], 227–228° [6752].

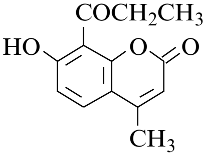
Acetate $C_{15}H_{14}O_5$ mol.wt. 274.27 (m.p. 137°) [6800].

Benzoate $C_{20}H_{16}O_5$ mol.wt. 336.34 (m.p. 138°) [6800].

7-Hydroxy-4-methyl-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one

7-Hydroxy-4-methyl-8-propionylcoumarin
8-Propionyl-4-methylumbelliferone

[3361-71-5] $C_{13}H_{12}O_4$ mol.wt. 232.24



Syntheses

- Obtained by Fries rearrangement of 4-methylumbelliferone propionate (m.p. 145–146°) [6801] with aluminium chloride for 60–70 min at 100–155° (68%) [6801], at 120–170° for 2–3 h [6802,6803], (69%) [6805] or at 160–165° for 1 h (29%) [6800].
- Also obtained from 2-propionylresorcin by condensation with acetoacetic ester in the presence of concentrated sulfuric acid [6800].

m.p. 200° [6800], 197–198° [6801], 192–195° [6802,6805], 187° [6803].

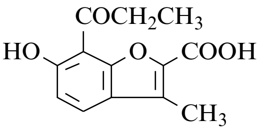
Acetate $C_{15}H_{14}O_5$ mol.wt. 274.27 (m.p. 168°) [6800].

Benzoate $C_{20}H_{16}O_5$ mol.wt. 336.34 (m.p. 134°) [6800].

6-Hydroxy-3-methyl-7-(1-oxopropyl)benzofuran-2-carboxylic acid

6-Hydroxy-3-methyl-7-propionylcoumarilic acid

[108881-73-8] $C_{13}H_{12}O_5$ mol.wt. 248.24



Syntheses

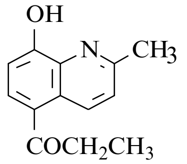
- Obtained by Fries rearrangement of 3-methyl-6-propionyl-oxycoumarilic acid (m.p. 229°) with aluminium chloride by heating for 1 h, first at 120–125°, then at 150–160° [7950].

- Also obtained by treatment of 3-bromo-7-hydroxy-8-propionyl-4-methylcoumarin with refluxing 7% aqueous sodium carbonate for 1 h (42%) [7951].

m.p. 240° [7950].

8-Hydroxy-2-methyl-5-(1-oxopropyl)quinoline

[100393-42-8] $C_{13}H_{13}NO_2$ mol.wt. 215.25



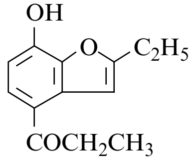
Synthesis

- Obtained by reaction of propionyl chloride with 8-quinaldinol (8-hydroxy-2-methylquinoline) in nitrobenzene in the presence of aluminium chloride [7946].

m.p. 97–98° [7946].
USE: Fungicide [7946].

1-(2-Ethyl-7-hydroxy-4-benzofuranyl)-1-propanone

[39544-07-5] $C_{13}H_{14}O_3$ mol.wt. 218.25

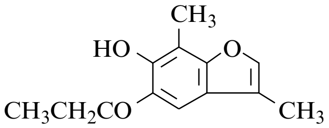


Synthesis

- Refer to: [7956].

1-(6-Hydroxy-3,7-dimethyl-5-benzofuranyl)-1-propanone

[39874-76-5] $C_{13}H_{14}O_3$ mol.wt. 218.25



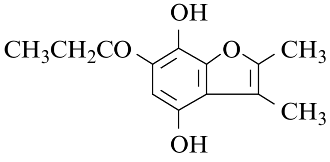
Synthesis

- Obtained from 3-bromo-7-hydroxy-6-propionyl-4,8-di-methylcoumarin with refluxing 10% aqueous sodium carbonate solution for 1 h (25%) [7957].

m.p. 120° [7957].

1-(4,7-Dihydroxy-2,3-dimethyl-6-benzofuranyl)-1-propanone

[49812-94-4] $C_{13}H_{14}O_4$ mol.wt. 234.25



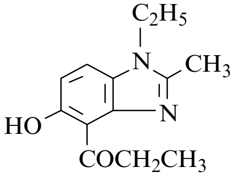
Synthesis

- Preparation by treatment of its dimethyl ether with pyridinium chloride (compound **10**) (63%) [7148].

m.p. 190° [7148].
USE: Radioprotective activity [7148].

1-(1-Ethyl-5-hydroxy-2-methyl-1*H*-benzimidazol-4-yl)-1-propanone

[137538-60-4] $C_{13}H_{16}N_2O_2$ mol.wt. 232.28

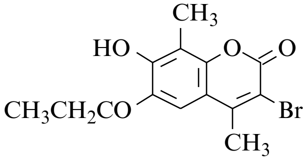


Synthesis

- Obtained by Friedel–Crafts acylation of 1-ethyl-5-hydroxy-2-methylbenzimidazole with propionic anhydride [7920].

3-Bromo-7-hydroxy-4,8-dimethyl-6-(1-oxopropyl)-2*H*-1-benzopyran-2-one

[39874-75-4] $C_{14}H_{13}BrO_4$ mol.wt. 246.26



Synthesis

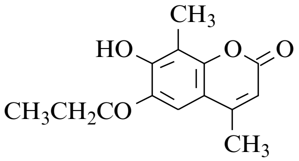
- Obtained by reaction of bromine with 7-hydroxy-6-propionyl-4,8-dimethylcoumarin in hot acetic acid (45%) [7957].

m.p. 223° [7957].

7-Hydroxy-4,8-dimethyl-6-(1-oxopropyl)-2*H*-1-benzopyran-2-one

7-Hydroxy-4,8-dimethyl-6-propionylcoumarin

[25944-43-8] $C_{14}H_{14}O_4$ mol.wt. 246.26



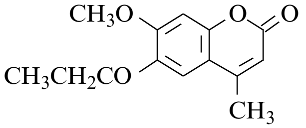
Syntheses

- Obtained by Fries rearrangement of 4,8-dimethyl-7-propionoxycoumarin (m.p. 129°) [7958] with aluminium chloride at 160° for 2 h (60%) [7959].
- Also refer to: [7957].

m.p. 200° [7959].

7-Methoxy-4-methyl-6-(1-oxopropyl)-2*H*-1-benzopyran-2-one

$C_{14}H_{14}O_4$ mol.wt. 246.26

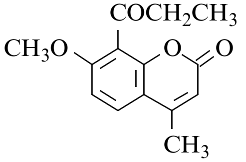


Syntheses

- Obtained by reaction of dimethyl sulfate with 7-hydroxy-4-methyl-6-propionylcoumarin in aqueous alkaline solution [6800].
- Also obtained by oxidation of 7-methoxy-4-methyl-6-propylcoumarin in diethyl ether with ceric ammonium nitrate in aqueous acetic acid (94%) [7960].

m.p. 180° [6800], 172° [7960]; ^1H NMR [7960], IR [7960].

7-Methoxy-4-methyl-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one



$C_{14}H_{14}O_4$ mol.wt. 246.26

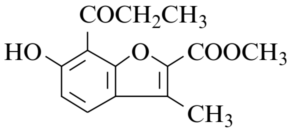
Synthesis

- Obtained by reaction of dimethyl sulfate with 8-propionyl-4-methylumbelliferone in the presence of a N sodium hydroxide solution at r.t. for 2 h [6800].

m.p. 150° [6800].

6-Hydroxy-3-methyl-7-(1-oxopropyl)benzofuran-2-carboxylic acid methyl ester

[35093-16-4]



$C_{14}H_{14}O_5$ mol.wt. 262.26

Synthesis

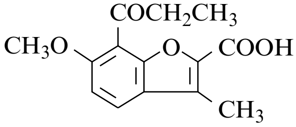
- Obtained by esterification of the corresponding carboxylic acid with dimethyl sulfate and sodium bicarbonate in refluxing acetone for 2 h [7951].

m.p. 154° [7951].

6-Methoxy-3-methyl-7-(1-oxopropyl)benzofuran-2-carboxylic acid

6-Methoxy-3-methyl-7-propionylcoumarilic acid

[109103-25-5]



$C_{14}H_{14}O_5$ mol.wt. 262.26

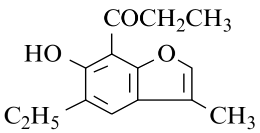
Synthesis

- Preparation by reaction of methyl iodide with 6-hydroxy-3-methyl-7-propionylcoumarilic acid in the presence of potassium carbonate in acetone [7950].

m.p. 93° [7950].

5-Ethyl-6-hydroxy-3-methyl-7-(1-oxopropyl)benzofuran

[106379-22-0]



$C_{14}H_{16}O_3$ mol.wt. 232.28

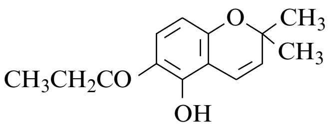
Synthesis

- Obtained by decarboxylation of 5-ethyl-6-hydroxy-3-methyl-7-propionylcoumarilic acid on heating for 10 min at 255° [7945].

m.p. 64–65° [7945].

1-(5-Hydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-1-propanone

[118585-45-8]



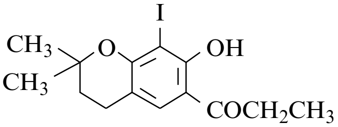
$C_{14}H_{16}O_3$ mol.wt. 232.28

Synthesis

- Refer to: [7961].

1-(3,4-Dihydro-7-hydroxy-8-iodo-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-1-propanone

[117844-80-1] $C_{14}H_{17}IO_3$ mol.wt. 360.19



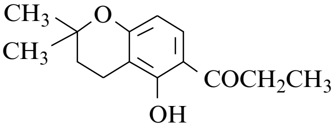
Synthesis

- Obtained by reaction of isoprene (2-methyl-1,3-butadiene) with 2,4-dihydroxy-3-iodopropiophenone in petroleum ether in the presence of 85% phosphoric acid for 20 h at 30–35° (89%) [6530].

m.p. 91–92° [6530]; ^{1}H NMR [6530].

1-(3,4-Dihydro-5-hydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-1-propanone

[117844-76-5] $C_{14}H_{18}O_3$ mol.wt. 234.30



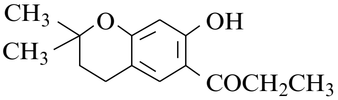
Synthesis

- Obtained by reaction of isoprene (2-methyl-1,3-butadiene) with respropiophenone in petroleum ether in the presence of 85% phosphoric acid for 8 h at 30–35° (25%) [6530].

m.p. 95–96° [6530]; ^{1}H NMR [6530].

1-(3,4-Dihydro-7-hydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-1-propanone

[117844-77-6] $C_{14}H_{18}O_3$ mol.wt. 234.30



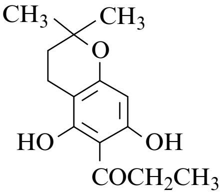
Syntheses

- Obtained by reaction of isoprene (2-methyl-1,3-butadiene) with respropiophenone in petroleum ether in the presence of 85% phosphoric acid for 8 h at 30–35° (32%) [6530].
- Also obtained by refluxing the 8-iodo derivative with N,N-dimethylaniline for 2 h at 190–200° (98%) [6530].

m.p. 148–149° [6530]; ^{1}H NMR [6530].

1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-1-propanone

[201035-07-6] $C_{14}H_{18}O_4$ mol.wt. 250.29

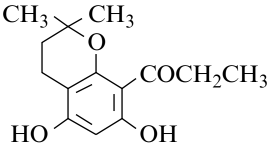


Synthesis

- Refer to: [6887].

1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl)-1-propanone
2,2-Dimethyl-8-propionylchroman-5,7-diol

[201035-08-7] $C_{14}H_{18}O_4$ mol.wt. 250.29

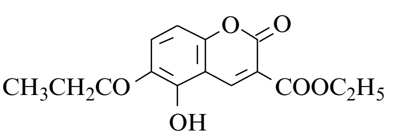


Syntheses

- Obtained by reaction of 2-methyl-2-buten-1-ol with phloropropiophenone in dioxane in the presence of Amberlite IR-120 resin (H^+ form), first at 20° during 0.5 h, then at reflux for 24 h [7739].
- Also refer to: [6887].

5-Hydroxy-6-(1-oxopropyl)-2*H*-1-benzopyran-2-one-3-carboxylic acid ethyl ester
5-Hydroxy-2-oxo-6-propionyl-2*H*-chromene-3-carboxylic acid ethyl ester
5-Hydroxy-6-propionylcoumarin-3-carboxylic acid ethyl ester

$C_{15}H_{14}O_6$ mol.wt. 290.27



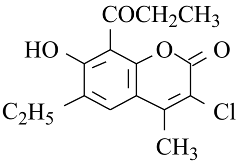
Synthesis

- Obtained by reaction of 5-hydroxy-6-propionyl-coumarin-3-carboxylic acid with ethanol in the presence of concentrated sulfuric acid for 18 h at reflux [7955].

m.p. 152–154° [7955].

3-Chloro-6-ethyl-7-hydroxy-4-methyl-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one
3-Chloro-6-ethyl-7-hydroxy-4-methyl-8-propionylcoumarin

$C_{15}H_{15}ClO_4$ mol.wt. 294.73

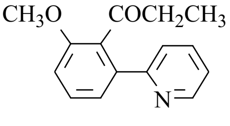


Synthesis

- Obtained by Fries rearrangement of 3-chloro-6-ethyl-4-methyl-7-propionyloxycoumarin with aluminium chloride for 90 min at 155° [7945].

1-[2-Methoxy-6-(2-pyridinyl)phenyl]-1-propanone

[872630-73-4] $C_{15}H_{15}NO_2$ mol.wt. 241.29



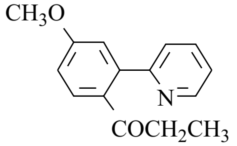
Synthesis

- Obtained by acylation of 2-(3-methoxyphenyl)pyridine (2 mmol) with ethylene (7 atm) and carbon monoxide (20 atm) in the presence of catalytic amounts of $Rh_4(CO)_{12}$ in DMA at 160° for 10 h (20%) [7962].

^1H NMR [7962], ^{13}C NMR [7962], IR [7962], MS [7962]; GC [7962].

1-[4-Methoxy-2-(2-pyridinyl)phenyl]-1-propanone

[188527-69-7] $C_{15}H_{15}NO_2$ mol.wt. 241.29



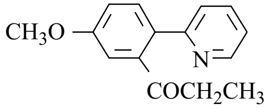
Synthesis

- Obtained by acylation of 2-(3-methoxyphenyl)pyridine (2 mmol) with ethylene (7 atm) and carbon monoxide (20 atm) in the presence of catalytic amounts of $Rh_4(CO)_{12}$ in DMA at 160° for 10 h (53%) [7962].

^{1}H NMR [7962], ^{13}C NMR [7962], IR [7962], MS [7962]; GC [7962].

1-[5-Methoxy-2-(2-pyridinyl)phenyl]-1-propanone

[872630-76-7] $C_{15}H_{15}NO_2$ mol.wt. 241.29



Synthesis

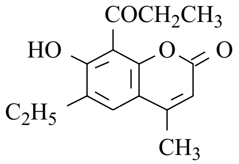
- Obtained by acylation of 2-(4-methoxyphenyl) pyridine (2 mmol) with ethylene (7 atm) and carbon monoxide (20 atm) in the presence of catalytic amounts of $Rh_4(CO)_{12}$ in DMA at 180° for 10 h (62%) [7962].

light yellow solid [7962]; m.p. 125–130° [7962];
^{1}H NMR [7962], ^{13}C NMR [7962], IR [7962], MS [7962];
GC [7962].

6-Ethyl-7-hydroxy-4-methyl-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one

6-Ethyl-7-hydroxy-4-methyl-8-propionylcoumarin

[109402-62-2] $C_{15}H_{16}O_4$ mol.wt. 260.29



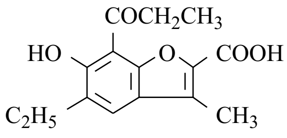
Syntheses

- Preparation by reaction of 4-ethyl-2-propionylresorcinol with ethyl acetoacetate in the presence of 80% sulfuric acid at r.t. overnight [6450].
- Also obtained by Fries rearrangement of 7-propionoxy-6-ethyl-4-methylcoumarin (m.p. 154°) with aluminium chloride (3.3 mol) at 140–150° for 1 h (40%) [6450].

m.p. 111° [6450].

5-Ethyl-6-hydroxy-3-methyl-7-(1-oxopropyl)-2-benzofurancarboxylic acid

[109402-64-4] $C_{15}H_{16}O_5$ mol.wt. 276.29



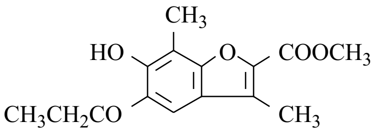
Synthesis

- Obtained by Fries rearrangement of 5-ethyl-3-methyl-6-propionyloxycoumarilic acid (m.p. 175°) with aluminium chloride by heating for 1 h at 150–160° [7945].

m.p. 243° [7945].

6-Hydroxy-3,7-dimethyl-5-(1-oxopropyl)-2-benzofurancarboxylic acid methyl ester

[39874-77-6]



$C_{15}H_{16}O_5$ mol.wt. 276.29

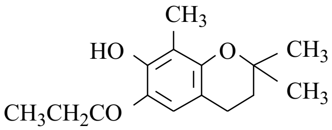
Synthesis

- Obtained by reaction of dimethyl sulfate with the corresponding carboxylic acid in the presence of sodium bicarbonate in refluxing acetone (24%) [7957].

m.p. 172° [7957].

1-(3,4-Dihydro-7-hydroxy-2,2,8-trimethyl-2*H*-1-benzopyran-6-yl)-1-propanone

[117844-90-3]



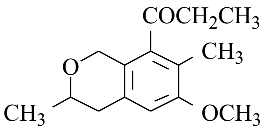
$C_{15}H_{20}O_3$ mol.wt. 248.32

Synthesis

- Refer to: [6530].

1-(3,4-Dihydro-6-methoxy-3,7-dimethyl-1*H*-2-benzopyran-8-yl)-1-propanone

[229003-30-9]



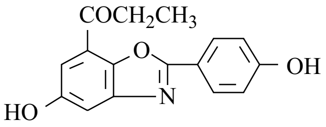
$C_{15}H_{20}O_3$ mol.wt. 248.32

Synthesis

- Refer to: [7963].

1-[5-Hydroxy-2-(4-hydroxyphenyl)-7-benzoxazolyl]-1-propanone

[868853-56-9]



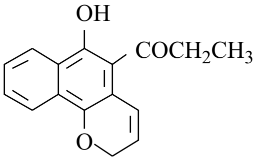
$C_{16}H_{13}NO_4$ mol.wt. 283.28

Synthesis

- Refer to: [7964] (compound **72**).

1-(6-Hydroxy-2*H*-naphtho[1,2-*b*]pyran-5-yl)-1-propanone

[127869-99-2]



$C_{16}H_{14}O_3$ mol.wt. 254.29

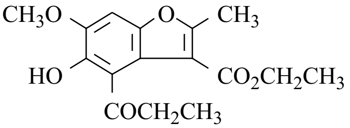
Synthesis

- Obtained by thermal reaction of 2-propionyl-1,4-naphthoquinone with allyltrimethylstannane in benzene under argon atmosphere (3%) [7814].

m.p. 69–72° [7814];
^1H NMR [7814], IR [7814], MS [7814].

5-Hydroxy-6-methoxy-2-methyl-4-(1-oxopropyl)-3-benzofurancarboxylic acid ethyl ester

[78094-46-9]



$C_{16}H_{18}O_6$ mol.wt. 306.32

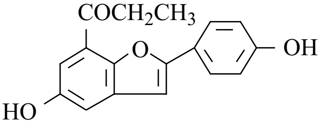
Synthesis

- Obtained by Michael addition of ethyl acetoacetate to 2-(2-ethyl-1,3-dioxolan-2-yl)-6-methoxy-2,5-cyclo-hexadiene-1,4-dione [78094-44-7] in ethanol in the presence of 0.1 equiv of sodium ethoxide, at r.t. for 30 min; then, addition of 6 N hydrochloric acid which effected deketalization and dehydration of the adduct (37%) [7128].

m.p. 97–99° [7128];
^{1}H NMR [7128], IR [7128]; X-ray crystallography [7128].

1-[5-Hydroxy-2-(4-hydroxyphenyl)-7-benzofuranyl]-1-propanone

[551002-14-3]



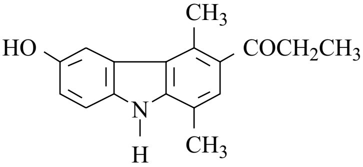
$C_{17}H_{14}O_4$ mol.wt. 282.30

Synthesis

- Refer to: [7965].

1-(6-Hydroxy-1,4-dimethyl-9*H*-carbazol-3-yl)-1-propanone

[136950-73-7]



$C_{17}H_{17}NO_2$ mol.wt. 267.33

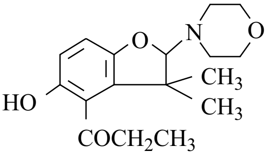
Synthesis

- Obtained directly by reaction of propionic anhydride with 6-methoxy-1,4-dimethyl-9*H*-carbazole in refluxing toluene (55%) [7966].

m.p. 248° [7966];
^{1}H NMR [7966], IR [7966].

1-[2,3-Dihydro-5-hydroxy-3,3-dimethyl-2-(4-morpholinyl)-4-benzofuranyl]-1-propanone

[116074-75-0]



$C_{17}H_{23}NO_4$ mol.wt. 305.37

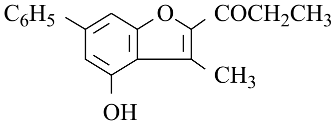
Synthesis

- Obtained by adding dropwise a solution of 2-propionyl-1,4-benzoquinone in methylene chloride to a stirred solution of 1-(N-morpholino)-2-methyl-1-propene *(enamine)* in methylene chloride at 5°, then the mixture stirred for 1 h at r.t. (50%) [7967].

m.p. 133–134° [7967]; ^{1}H NMR [7967], IR [7967].

1-(4-Hydroxy-3-methyl-6-phenyl-2-benzofuranyl)-1-propanone

[687184-53-7]



$C_{18}H_{16}O_3$ mol.wt. 280.32

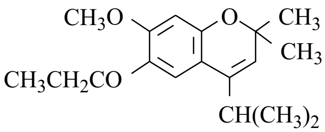
Synthesis

– Refer to: [7968] (compound **11h**).

BIOLOGICAL ACTIVITY: Anticancer [7968].

1-[7-Methoxy-2,2-dimethyl-4-(1-methylethyl)-2*H*-1-benzopyran-6-yl]-1-propanone

[851036-32-3]



$C_{18}H_{24}O_3$ mol.wt. 288.39

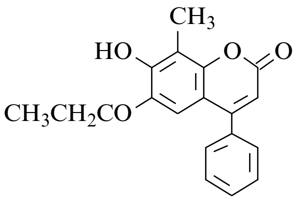
Synthesis

– Refer to: [7969].

7-Hydroxy-8-methyl-6-(1-oxopropyl)-4-phenyl-2*H*-1-benzopyran-2-one

7-Hydroxy-8-methyl-4-phenyl-6-propionylcoumarin

[25944-41-6]



$C_{19}H_{16}O_4$ mol.wt. 308.33

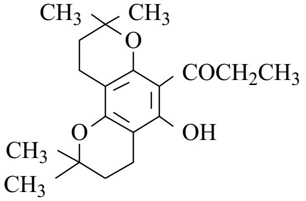
Synthesis

– Obtained by Fries rearrangement of 8-methyl-4-phenyl-7-(propionyloxy)coumarin (m.p. 109°) [7958] with aluminium chloride at 160° for 2 h (55%) [7959].

m.p. 179° [7959].

1-(3,4,9,10-Tetrahydro-5-hydroxy-2,2,8,8-tetramethyl-2*H*,8*H*-benzo[1,2-*b*:3,4-*b*′]dipyran-6-yl)-1-propanone

3,4,9,10-Tetrahydro-2,2,8,8-tetramethyl-6-propionyl-2*H*,8*H*-benzo[1,2-*b*;3,4-*b*′] dipyran-5-ol



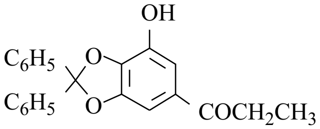
$C_{19}H_{26}O_4$ mol.wt. 318.41

Synthesis

– Obtained by reaction of 2-methyl-2-buten-1-ol with phloropropiophenone in dioxane in the presence of Amberlite IR-120 resin (4$^+$ form), first at 20° during 0.5 h, then at reflux for 24 h [7739].

1-(7-Hydroxy-2,2-diphenyl-1,3-benzodioxol-5-yl)-1-propanone

[178678-85-8] $C_{22}H_{18}O_4$ mol.wt. 346.38

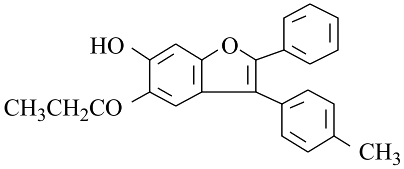


Synthesis

- Refer to: [7970].

1-[6-Hydroxy-3-(4-methylphenyl)-2-phenyl-5-benzofuranyl]-1-propanone

[438490-68-7] $C_{24}H_{20}O_3$ mol.wt. 356.42

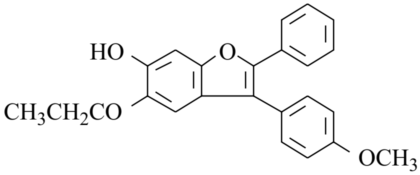


Synthesis

- Refer to: [7971].

1-[6-Hydroxy-3-(4-methoxyphenyl)-2-phenyl-5-benzofuranyl]-1-propanone

[438490-65-4] $C_{24}H_{20}O_4$ mol.wt. 372.42



Syntheses

- Refer to: [7971,7972].

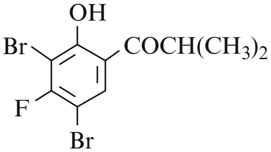
USE: Antifeedant [7971].

Chapter 24
Aromatic Ketones Containing One Isobutyryl Group

24.1 Benzene Derivatives

1-(3,5-Dibromo-4-fluoro-2-hydroxyphenyl)-2-methyl-1-propanone

[881190-43-8] $C_{10}H_9Br_2FO_2$ mol.wt. 339.99



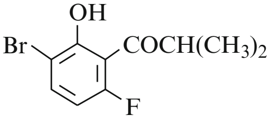
Synthesis

– Refer to: [7973].

USE: Preparation of piperidine compounds as NMDA receptor antagonists for treatment of dementia [7973].

1-(3-Bromo-6-fluoro-2-hydroxyphenyl)-2-methyl-1-propanone

[881190-64-3] $C_{10}H_{10}BrFO_2$ mol.wt. 261.09



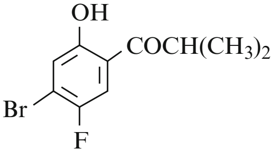
Synthesis

– Refer to: [7973].

USE: Preparation of piperidine compounds as NMDA receptor antagonists for treatment of dementia [7973].

1-(4-Bromo-5-fluoro-2-hydroxyphenyl)-2-methyl-1-propanone

[140896-93-1] $C_{10}H_{10}BrFO_2$ mol.wt. 261.09

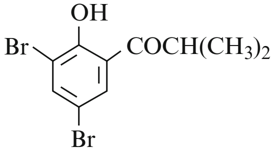


Synthesis

– Obtained by Fries rearrangement of 3-bromo-4-fluoro-phenyl isobutyrate [7974].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_24,

1-(3,5-Dibromo-2-hydroxyphenyl)-2-methyl-1-propanone



$C_{10}H_{10}Br_2O_2$ mol.wt. 322.00

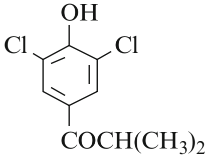
Synthesis

- Obtained by reaction of bromine with o-isobutyrophenone in acetic acid [7975].

m.p. 97° [7975].

1-(3,5-Dichloro-4-hydroxyphenyl)-2-methyl-1-propanone

[124500-38-5] $C_{10}H_{10}Cl_2O_2$ mol.wt. 233.09



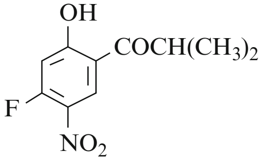
Syntheses

- Obtained by Fries rearrangement of 2,6-dichlorophenyl isobutyrate with aluminium chloride at 130–135° for 4.5 h (42%) [6401].
- Also refer to: [7976].

m.p. 112–113° [6401].

1-(4-Fluoro-2-hydroxy-5-nitrophenyl)-2-methyl-1-propanone

[119994-05-7] $C_{10}H_{10}FNO_4$ mol.wt. 227.19



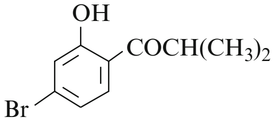
Syntheses

- Obtained by reaction of fuming nitric acid with 4-fluoro-2-hydroxyisobutyrophenone in acetic acid at 0° for 30 min [6412].
- Also refer to: [7977].

Oxime [119994-06-8] $C_{10}H_{11}FN_2O_4$ mol.wt. 242.21 [7978].

1-(4-Bromo-2-hydroxyphenyl)-2-methyl-1-propanone

[140896-90-8] $C_{10}H_{11}BrO_2$ mol.wt. 243.10

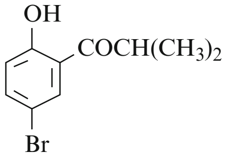


Synthesis

- Obtained by Fries rearrangement of 3-bromophenyl isobutyrate [7974].

1-(5-Bromo-2-hydroxyphenyl)-2-methyl-1-propanone

[934524-37-5] $C_{10}H_{11}BrO_2$ mol.wt. 243.10

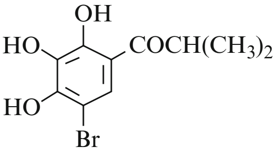


Syntheses

- Obtained by reaction of isobutyryl chloride with 4-bromo-anisole in the presence of aluminium chloride in carbon disulfide for 48 h at r.t. (35%) [7979].
- Also refer to: [7980].

b.p.$_{12}$ 144–145° [7979].

1-(5-Bromo-2,3,4-trihydroxyphenyl)-2-methyl-1-propanone



$C_{10}H_{11}BrO_4$ mol.wt. 275.10

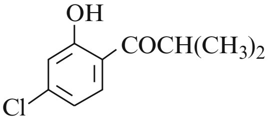
Synthesis

- Preparation by reaction of bromine with 2,3,4-trihydroxy-isobutyrophenone in acetic acid [6454].

m.p. 135° [6454].

1-(4-Chloro-2-hydroxyphenyl)-2-methyl-1-propanone

[6618-61-7]



$C_{10}H_{11}ClO_2$ mol.wt. 198.65

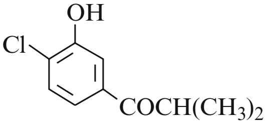
Syntheses

- Obtained by using standard Friedel–Crafts acylation [7976].
- Also refer to: [6460,6463].

b.p.$_{11}$ 133–138° [6460].

1-(4-Chloro-3-hydroxyphenyl)-2-methyl-1-propanone

[124500-32-9]



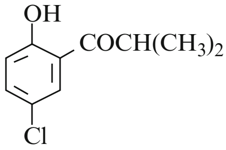
$C_{10}H_{11}ClO_2$ mol.wt. 198.65

Synthesis

- Obtained by using standard Friedel–Crafts acylation [7976].

1-(5-Chloro-2-hydroxyphenyl)-2-methyl-1-propanone

[90743-04-7]



$C_{10}H_{11}ClO_2$ mol.wt. 198.65

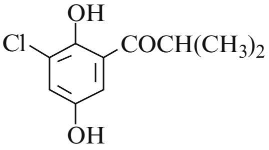
Syntheses

- Obtained by Fries rearrangement of 4-chlorophenyl isobutyrate (b.p.$_{11}$ 120°; m.p. 29°) with aluminium chloride at 110° for 3 h [7981] or in nitrobenzene at 140° for 2 h (55%) [7979].
- Also refer to: [6489,7980,7982,7983].

yellow oil [7981]; b.p.$_{20}$ 130° [7981], b.p.$_{18}$ 140–142° [7979].

1-(3-Chloro-2,5-dihydroxyphenyl)-2-methyl-1-propanone

[918310-93-7]



$C_{10}H_{11}ClO_3$ mol.wt. 214.65

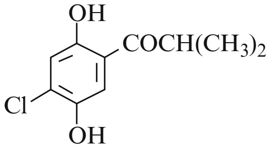
Synthesis

- Refer to: [7984].

USE: For preparation of agrochemical pesticides [7984].

1-(4-Chloro-2,5-dihydroxyphenyl)-2-methyl-1-propanone

[88772-49-0] $C_{10}H_{11}ClO_3$ mol.wt. 214.65

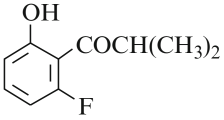


Synthesis

- Refer to: [7985].

1-(2-Fluoro-6-hydroxyphenyl)-2-methyl-1-propanone

[881190-63-2] $C_{10}H_{11}FO_2$ mol.wt. 182.19



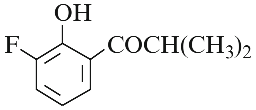
Synthesis

- Refer to: [7973].

USE: Preparation of piperidine compounds as NMDA receptor antagonists for treatment of dementia [7973].

1-(3-Fluoro-2-hydroxyphenyl)-2-methyl-1-propanone

[879339-62-5] $C_{10}H_{11}FO_2$ mol.wt. 182.19



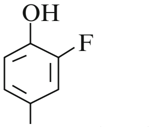
Synthesis

- Refer to: [6517].

USE: Preparation of heteroaryl alkylidenetetrahydro-naphthalenamines as anti-inflammatories [6517].

1-(3-Fluoro-4-hydroxyphenyl)-2-methyl-1-propanone

[879339-65-8] $C_{10}H_{11}FO_2$ mol.wt. 182.19



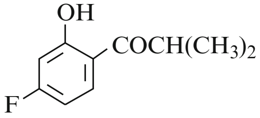
Syntheses

- Refer to: [6517,7986].

USE: Preparation of heteroaryl alkylidenetetrahydro-naphthalenamines as anti-inflammatory agents [6517]; preparation of δ-methylenebenzenebutanamines as anti-inflammatory agents [7986].

1-(4-Fluoro-2-hydroxyphenyl)-2-methyl-1-propanone

[123450-86-2] $C_{10}H_{11}FO_2$ mol.wt. 182.19



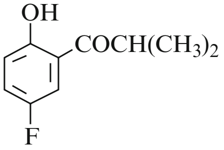
Syntheses

- Refer to: [6412] (Japanese patent) and [7973].

USE: Preparation of piperidine compounds as NMDA receptor antagonists for treatment of dementia [7973].

1-(5-Fluoro-2-hydroxyphenyl)-2-methyl-1-propanone

[183280-17-3] $C_{10}H_{11}FO_2$ mol.wt. 182.19



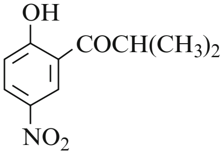
Synthesis

- Obtained by Fries rearrangement of p-fluorophenyl isobutyrate with aluminium chloride between 150° and 180° for 20 min (38%) [7987].

oil [7987]; ^{1}H NMR [7987], MS [7987].

1-(2-Hydroxy-5-nitrophenyl)-2-methyl-1-propanone

[88521-75-9] $C_{10}H_{11}NO_4$ mol.wt. 209.20



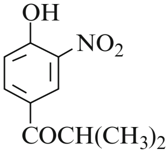
Syntheses

- Obtained by reaction of isobutyryl chloride with p-nitrophenol in nitrobenzene in the presence of aluminium chloride (9%) [6538], according to the modified method [7988].
- Also refer to: [7989].

m.p. 74–74.5° [6538]; ^{1}H NMR [6538], IR [6538].

1-(4-Hydroxy-3-nitrophenyl)-2-methyl-1-propanone

[82350-84-3] $C_{10}H_{11}NO_4$ mol.wt. 209.20

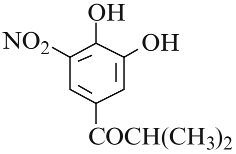


Synthesis

- Refer to: [7990].

1-(3,4-Dihydroxy-5-nitrophenyl)-2-methyl-1-propanone

[134610-34-7] $C_{10}H_{11}NO_5$ mol.wt. 225.20



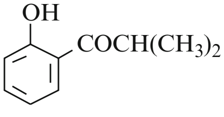
Synthesis

- Obtained by demethylation of 4-hydroxy-3-methoxy-5-nitroisobutyrophenone with pyridinium chloride at 180° for 45 min [7991].

BIOLOGICAL ACTIVITY: As catechol-O-methyltransferase inhibitor [7991].
m.p. 98–99° [7991].

1-(2-Hydroxyphenyl)-2-methyl-1-propanone

[6640-69-3] $C_{10}H_{12}O_2$ mol.wt. 164.20



Syntheses

- Obtained by Fries rearrangement of phenyl isobutyrate,
  - with aluminium chloride [7992],

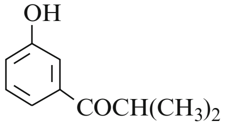
  - without solvent at 140–150° for 1 h [7794], (40%) [7993] or at 170° for 40 min (40%) [7994];
  - in nitrobenzene at 50° for 18 h (25%) [6567] or in carbon disulfide (17%) [6683];
    - with ferric chloride in a boilin g water bath for 6 h [7975].
- Also obtained by reaction of isobutyryl chloride with phenol in nitrobenzene on a steam-cone for 30 min (7%) [7993].
- Also obtained by reaction of sec-butyllithium (1.1 equiv) with 2-bromo-4-methylphenyl isobutyrate in tetrahydrofuran/ethyl ether/hexane at −95° for 30 min and −78° for 30 min, then hydrolysis with saturated ammonium chloride (62%) (metal-promoted Fries rearrangement) [6590].
- Also obtained by hydrolysis of 2-{2-methyl-1-[(1*R*)-1-phenylethyl]iminopropyl}phenol (4ea) with aqueous acetic acid/THF at 40° for 4 h (77%) [6593].
- Also refer to: [7980,7995–7998].

N.B.: The formula of the ketone obtained by [7999] is not compatible with the o-hydroxyketone of the title. In fact, the mentioned melting point (64–66°) [7999] should be much lower than the one of the p-isomer, that is of 56° [7975].

colourless oil [6593,6683];
b.p.$_{0.4}$ 68° [7993], b.p.$_{3.5}$ 79.3–79.5° [7994], b.p.$_{1}$ 102–105° [7992],
b.p.$_{10}$ 110° [7975], b.p.$_{21}$ 121–123° [7997], b.p.$_{16}$ 130° [6683];
^{1}H NMR [6590,6593], ^{13}C NMR [6590,6593,7794],
IR [6590,6593,6624], MS [6593,6628].

1-(3-Hydroxyphenyl)-2-methyl-1-propanone

[103323-37-1] $C_{10}H_{12}O_2$ mol.wt. 164.20



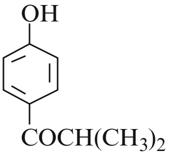
Syntheses

- Obtained by treatment of 3-methoxyisobutyrophenone with pyridinium chloride at 210° for 30 min [6659].
- Also obtained by treatment of 1-hydroxy-1-(3-hydroxy-phenyl)-2-methylpropane (m.p. 132–134°) with DDQ in dioxane for 72 h [6659].
- Also obtained by diazotization of 3-aminoisobutyrophenone, followed by hydrolysis of the diazonium salt obtained (19%) [8000].
- Also obtained by reaction of diisopropylcadmium with 3-acetoxybenzoyl chloride in benzene, followed by treatment of the acetate obtained with refluxing 10% sodium hydroxide for 2–3 h (15%) [6657].

b.p.$_{1}$ 129° [6657]; MS [6628].

1-(4-Hydroxyphenyl)-2-methyl-1-propanone

[34917-91-4] $C_{10}H_{12}O_2$ mol.wt. 164.20



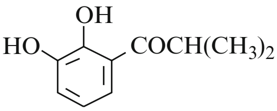
Syntheses

- Preparation by reaction of isobutyronitrile with phenol in the presence of triflic acid at r.t. for 15 days (32%) [8001].
- Also obtained by Fries rearrangement of phenyl isobutyrate,
  - with aluminium chloride [7992,8002],
    - in nitrobenzene at 25° for 40 h (86%) [7993], at 40° for 48 h under an argon atmosphere [8003] or at 50° for 18 h (62%) [6567];
    - in carbon disulfide (15%) [6683];
    - without solvent at 140–150° for 1 h (11%) [7993];
  - with ferric chloride in a boiling water bath for 6 h (16%) [7975].
- Also obtained by reaction of isobutyryl chloride with phenol in nitrobenzene on a steam-cone for 30 min (73%) [7993].
- Also obtained by demethylation of 4-isobutyrylanisole with pyridinium chloride at 180° for 10 h (quantitative yield) [8004] or at reflux for 15 min (92%) [6672].
- Also refer to: [6664,6834,7985,7997,8005–8017].

b.p.$_{0.1}$ 128° [7993], b.p.$_{21}$ 196–198° [7997], b.p.$_{17}$ 200° [6683];
m.p. 58–59° [6683], 56° [7975], 54° [6567,7993]; MS [6628].

1-(2,3-Dihydroxyphenyl)-2-methyl-1-propanone

[862666-40-8] $C_{10}H_{12}O_3$ mol.wt. 180.20



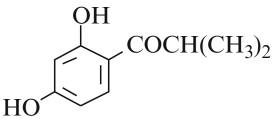
Syntheses

- Preparation by total demethylation of 2,3-dimethoxy-isobutyrophenone with boron tribromide in methylene chloride at r.t. overnight (62%) [6726].
- Also refer to: [6727].

Brown wax [6726]; m.p. 93.5° [6727];
^{1}H NMR [6726], ^{13}C NMR [6726], IR [6726], MS [6726].

1-(2,4-Dihydroxyphenyl)-2-methyl-1-propanone

[29048-54-2] $C_{10}H_{12}O_3$ mol.wt. 180.20



Syntheses

- Preparation by reaction of isobutyronitrile with resorcinol in the presence of triflic acid at r.t. for 14 days (81%) [8001].
- Also obtained by condensation of isobutyric acid with resorcinol,
  - in the presence of zinc chloride at 125–135° [8018];
  - in the presence of boron trifluoride at 70° for 2 h (79%) [6736];

  - in the presence of Amberlite IR-120 or Zeokarb 225 (cation-exchange resin, sulfonic acid type) at 160° for 2–3 h (69%) [6740].

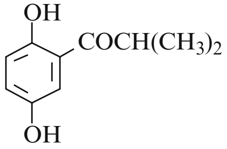
- Also obtained by reaction of isobutyryl chloride with resorcinol in the presence of aluminium chloride at 85–90° (79%) [8019] or in nitrobenzene at 40–50° (59%) [8020].
- Other preparation (Japanese paper) (75%) [8021].
- Also refer to: [8022].

b.p.$_{0.3}$ 150° [8020], b.p.$_{0.4}$ 150–151° [6736], b.p.$_{6\text{–}7}$ 173–175° [8018];
m.p. 67–69° [6740], 67–68.5° [8018], 67–68° [8021], 66–67° [8001];
^{1}H NMR [8001], IR [8001], MS [8001].
USE: In determination of uranium by spectrophotometry [8023].

BIOLOGICAL ACTIVITY: Antimelanoma and skin depigmentation [6778]; catechol O-methyl-transferase inhibitor [8024,8025].
Uranium complex [68079-14-1] [8023].

1-(2,5-Dihydroxyphenyl)-2-methyl-1-propanone

[112450-27-8] $C_{10}H_{12}O_3$ mol.wt. 180.20

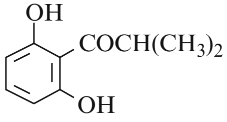


Syntheses

- Refer to: [7883, 8026].

1-(2,6-Dihydroxyphenyl)-2-methyl-1-propanone

[101386-02-1] $C_{10}H_{12}O_3$ mol.wt. 180.20



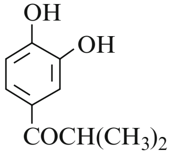
Synthesis

- Obtained from 2-chloro-2-isobutyryl-1,3-cyclohexanedione by rearrangement in 10–20% hydrogen chloride-DMF at 120–130° for 15–20 min, according to the procedure [6810], (66%) [6811].

b.p.$_{0.02}$ 105–110° [6811]; m.p. 112° [6811];
^{1}H NMR [6811], ^{13}C NMR [6811], IR [6811].

1-(3,4-Dihydroxyphenyl)-2-methyl-1-propanone *(U-0521)*

[5466-89-7] $C_{10}H_{12}O_3$ mol.wt. 180.20



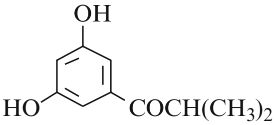
Syntheses

- Obtained by reaction of isobutyryl chloride with pyrocatechol in the presence of aluminium chloride in chlorobenzene, first at 55° for 30 min, then at 110–115° for 3 h (39%) [8027].
- Also obtained by treatment of a pyrocatechol and its diisobutyrate mixture with aluminium chloride in chlorobenzene at 110° for 3 h (18%) [8027].
- Also obtained by Fries rearrangement of pyrocatechol diisobutyrate (69%) [6727].
- Also obtained (35%) [8028] according to the procedure [8029].
- Also refer to: [6828,8030–8039].

b.p.$_{0.05}$ 150–155° [8027], b.p.$_4$ 200–210° [6727];
m.p. 106.5–107.5° [6727], 94–96° [8027,8029].

BIOLOGICAL ACTIVITY: Catechol O-methyltransferase inhibitor [8024,8025, 8040]; pharmacology [6828]; antimelanoma and skin depigmentation [6778]; antihypertensive [8041]; tyrosine hydroxylase inhibition [8042].

1-(3,5-Dihydroxyphenyl)-2-methyl-1-propanone

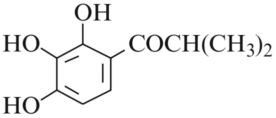


$C_{10}H_{12}O_3$ mol.wt. 180.20

Synthesis

- Refer to: [8043] (Japanese patent).

2-Methyl-1-(2,3,4-trihydroxyphenyl)-1-propanone



$C_{10}H_{12}O_4$ mol.wt. 196.20

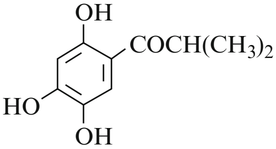
Synthesis

- Obtained by reaction of isobutyric acid with pyrogallol in the presence of zinc chloride at 140–145° (40%) (Nencki reaction) [6454].

b.p.$_{19}$ 198–200° [6454]; m.p. 118° [6454].

2-Methyl-1-(2,4,5-trihydroxyphenyl)-1-propanone

[99186-85-3]



$C_{10}H_{12}O_4$ mol.wt. 196.20

Syntheses

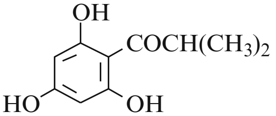
- Preparation by reaction of isobutyryl chloride with 1,2,4-benzenetriol in nitrobenzene in the presence of aluminium chloride [8044–8047].
- Also obtained by Fries rearrangement of 1,2,4-benzenetriol triisobutyrate with aluminium chloride in nitrobenzene [8045].
- Also refer to: [8048].

m.p. 136–138° [8044–8047].

USE: Antioxidizing agent [8048] for fats and oils [8044–8046], lard [8049] and paraffin waxes [8046].

2-Methyl-1-(2,4,6-trihydroxyphenyl)-1-propanone

[35458-21-0]



$C_{10}H_{12}O_4$ mol.wt. 196.20

Syntheses

- Obtained by reaction of isobutyronitrile with phloroglucinol (Hoesch reaction) [8050], (34%) [8051] according to the procedure [8052] or in the presence of triflic acid at r.t. for 13 days (41%) [8001].

- Preparation by reaction of isobutyryl chloride with phloroglucinol [7740] in the presence of aluminium chloride in a nitrobenzene/carbon disulfide solution (80%) [8053], (52–54%) [6880,8054,8055], (44%) [8056], (38%) [8057] or in a methylene chloride/nitromethane solution [8058].
- Also obtained by reaction of isobutyric acid with phloroglucinol,
  - in the presence of boron trifluoride etherate and the mixture heated on a steam bath for 2 h [8059,8060];
  - in the presence of phosphorous oxychloride and aluminium chloride under nitrogen, at 0° for 8 h (40–54%) [8061,8062].
- Also refer to: [8063–8071].
- Also obtained by reaction performed by VPS, (valerophenone synthase), a polyketide synthase, with isobutyryl-CoA [8072–8075].
- Also refer to: [6889, 8076–8078].

Isolation from natural sources

- From *Helichrysum species* [8079,8080].
- From Hops *Humulus lupulus* L. (Cannabinaceae) [8081].
- Formation in the biosynthesis of hop bitter acids in *Humulus lupulus* (Cannabaceae) [8082].

colourless oil [8079], yellowish oil [8081];

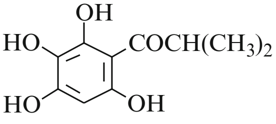
m.p. (hydrates) 78–82° [8056], 71–74° [8068], 70° [8055], 68° [8052,8083], 65–66.5° [8001]; (anhydrous) 177–178° [8050], 140° [6880], 138–140° [8052,8056], 138–139° [8057], 138° [8054,8055, 8084], 134–136° [8068]. One of the reported melting points is obviously wrong.

^{1}H NMR [8001,8057,8059,8079,8081],
^{13}C NMR [8059,8081], IR [8001,8059,8068,8079,8081], UV [8057,8059,8075, 8085],
MS [8001,8057,8068,8073,8075,8079,8081];
GC [6895]; TLC [8057]; HPLC [8057,8073,8075].
USE: Fungicide [6880].

BIOLOGICAL ACTIVITY: Antibiotic [8086]; antibiotic-resistant bacteria [8062]; for biliary and urinary tract disorder treatment [8087]; antagonist against both thromboxane A_2 and leukotriene D_4 [8088]; antimicrobial for staphylococcus aureus [6891]; as allergy inhibitor [8089]; vesicular stomatitis virus inhibition [8059].

2-Methyl-1-(2,3,4,6-tetrahydroxyphenyl)-1-propanone

[198879-05-9] $C_{10}H_{12}O_5$ mol.wt. 212.20

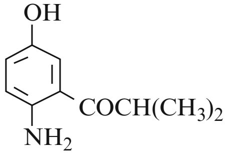


Syntheses

- Refer to: [8077,8078].

1-(2-Amino-5-hydroxyphenyl)-2-methyl-1-propanone

[404918-98-5] $C_{10}H_{13}NO_2$ mol.wt. 179.22

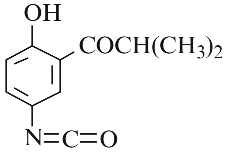


Synthesis

- Refer to: [8090].

1-(2-Hydroxy-5-isocyanatophenyl)-2-methyl-1-propanone

[150129-35-4] $C_{11}H_{11}NO_3$ mol.wt. 205.21

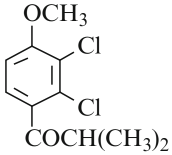


Synthesis

- Refer to: [8091].

1-(2,3-Dichloro-4-methoxyphenyl)-2-methyl-1-propanone

[53107-35-0] $C_{11}H_{12}Cl_2O_2$ mol.wt. 247.12



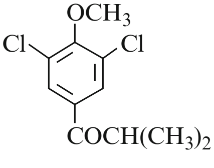
Syntheses

- Preparation by reaction of isobutyryl chloride with 2,3-dichloroanisole in the presence of aluminium chloride in methylene chloride,
  - first at 0° for 30 min, then at r.t. for 15 h (79%) [8092];
  - first at 5° for 1 h, then at r.t. for 20–25 h (49%) [8093].
- Also refer to: [8094–8100].

b.p.$_{0.5}$ 120–130° [8093–8100],
b.p.$_{0.5}$ 137–142° [8092].

1-(3,5-Dichloro-4-methoxyphenyl)-2-methyl-1-propanone

[124500-34-1] $C_{11}H_{12}Cl_2O_2$ mol.wt. 247.12

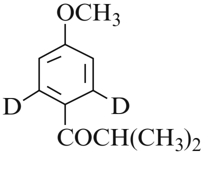


Synthesis

- Refer to: [7976].

1-(4-Methoxyphenyl-*2,6-d₂*)-2-methyl-1-propanone

[870456-80-7] $C_{11}H_{12}D_2O_2$ mol.wt. 180.24



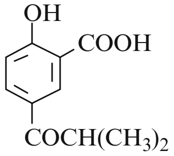
Synthesis

- Obtained (low yield) by regioselective *ortho*-deuteration of 4-methoxyisobutyrophenone in the presence of $[Ir(PPh_3)_3(cod)]^+ . BF_4^-$ [8101].

^1H NMR [8101].

2-Hydroxy-5-(2-methyl-1-oxopropyl)benzoic acid

2-Hydroxy-5-isobutyrylbenzoic acid



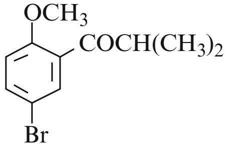
$C_{11}H_{12}O_4$ mol.wt. 208.21

Synthesis

- Obtained by saponification of methyl 2-hydroxy-5-isobutyrylbenzoate with sodium hydroxide in refluxing dilute ethanol for 2.5 h (81%) [8102].

m.p. 168–170° [8102].

1-(5-Bromo-2-methoxyphenyl)-2-methyl-1-propanone



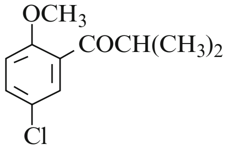
$C_{11}H_{13}BrO_2$ mol.wt. 257.13

Synthesis

- Obtained by reaction of dimethyl sulfate with 5-bromo-2-hydroxyisobutyrophenone in the presence of aqueous sodium hydroxide [7979].

b.p.$_{10}$ 158–163° [7979].

1-(5-Chloro-2-methoxyphenyl)-2-methyl-1-propanone



$C_{11}H_{13}ClO_2$ mol.wt. 212.68

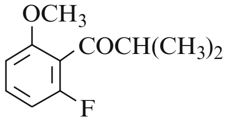
Synthesis

- Refer to: [7979].

b.p.$_9$ 150–152° [7979].

1-(2-Fluoro-6-methoxyphenyl)-2-methyl-1-propanone

[881190-66-5] $C_{11}H_{13}FO_2$ mol.wt. 196.22



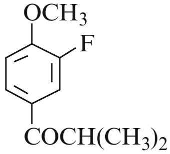
Synthesis

- Refer to: [7973].

USE: Preparation of piperidine compounds as NMDA receptor antagonists for treatment of dementia [7973].

1-(3-Fluoro-4-methoxyphenyl)-2-methyl-1-propanone

[879339-67-0] $C_{11}H_{13}FO_2$ mol.wt. 196.22



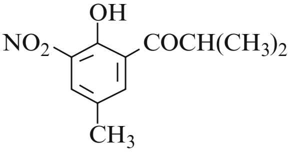
Syntheses

– Refer to: [6517, 7986].

USE: Preparation of heteroaryl alkylidenetetrahydro-naphthalenamines as antiinflammatory agents [6517]; preparation of δ-methylenebenzene-butanamines as antiinflammatory agents [7986].

1-(2-Hydroxy-5-methyl-3-nitrophenyl)-2-methyl-1-propanone

[70978-42-6] $C_{11}H_{13}NO_4$ mol.wt. 223.23



Syntheses

– Preparation by nitration of 2-hydroxy-5-methyl-isobutyrophenone at –20° (76%) [6926].

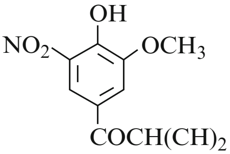
– Also obtained by nitration of 2-hydroxy-5-methyl-isobutyrophenone in methylene chloride using fuming nitric acid (d = 1.5) in acetic acid at r.t. for 6 h (76%) [6993].

– Also obtained by reaction of phenyllithium with 2-bromo-4-methyl-6-nitrophenyl isobutyrate in THF at –78° (65%) [8103].

m.p. 75–77° [6926,6993].

1-(4-Hydroxy-3-methoxy-5-nitrophenyl)-2-methyl-1-propanone

[134610-33-6] $C_{11}H_{13}NO_5$ mol.wt. 239.23



Synthesis

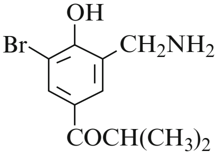
– Obtained by reaction of 50% nitric acid with 4-hydroxy-3-methoxyisobutyrophenone in acetic acid at r.t. for 15 min [7991].

m.p. 85–87° [7991].

BIOLOGICAL ACTIVITY: As catechol-O-methyltransferase inhibitor [7991].

1-[3-(Aminomethyl)-5-bromo-4-hydroxyphenyl]-2-methyl-1-propanone

[104129-15-9] $C_{11}H_{14}BrNO_2$ mol.wt. 272.14



Syntheses

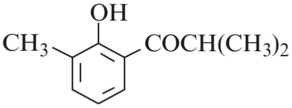
– Refer to: [6476,7008].

Oxime [104129-14-8] $C_{11}H_{15}BrN_2O_2$ mol.wt. 287.16.

BIOLOGICAL ACTIVITY: Diuretic and hypohypertensive agent [6476,7008].

1-(2-Hydroxy-3-methylphenyl)-2-methyl-1-propanone

[128291-79-2] $C_{11}H_{14}O_2$ mol.wt. 178.23



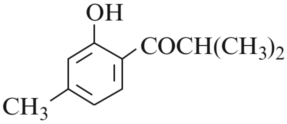
Syntheses

- Obtained by Fries rearrangement of o-tolyl isobutyrate [8104], (b.p.$_{15}$ 112°) [7981] with aluminium chloride at a higher temperature (56%) [8105], at 140° for 4 h (45%) [8106] or at 100–110° (20%) [7981].
- Also obtained by reaction of isobutyryl chloride with o-cresol in the presence of aluminium chloride [8107].

lightly yellow greenish oil [7981]; b.p.$_{23}$ 135–137° [8106]; IR [7078].

1-(2-Hydroxy-4-methylphenyl)-2-methyl-1-propanone

[116557-45-0] $C_{11}H_{14}O_2$ mol.wt. 178.23



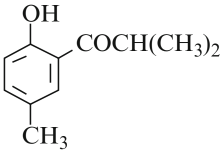
Syntheses

- Obtained by Fries rearrangement of 3-methylphenyl isobutyrate in the presence of aluminium chloride (70%) [7992], (66%) [7019].
- Also obtained by reaction of isobutyric acid with m-cresol in the presence of P_2O_5/SiO_2 at 100° for 24 h (52%) [8108].
- Also refer to: [7276].

b.p.$_{11}$ 120–121° [7019], b.p.$_{20}$ 130° [7992], b.p. 235° [8108];
^{1}H NMR [8108], IR [8108].

1-(2-Hydroxy-5-methylphenyl)-2-methyl-1-propanone

[64207-03-0] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

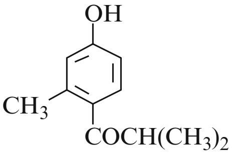
- Preparation by Fries rearrangement of p-cresyl isobutyrate,
  - with aluminium chloride (92%) [7992], at 170° for 1.5 h (80–96%) [6492];
  - with aluminium chloride at 140–150° for 4–5 h (80%) [7981].
- Also obtained by reaction of isobutyric acid with p-cresol in the presence of boron trifluoride at 70° for 2 h (79%) [6583].
- Also obtained from 2-bromo-2-methyl-1-(2-hydroxy-5-methylphenyl)-1-propanone, named o-[α-bromoisobutyro]-p-cresol in the paper,
  - by treatment with zinc powder in acetic acid [8109];
  - by treatment with boiling dimethylaniline or diethylaniline [8110].
- Also refer to: [6926,7033,7052,7980,8109,8111,8112].

colourless oil [7981];

b.p.$_{10}$ 124–125° [7981], b.p.$_{10}$ 125° [7992], b.p.$_{11}$ 125–125.3° [7033],
b.p.$_{18}$ 131–132° [6583], b.p.$_{760}$ 250–251° [7981], b.p.$_{763}$ 250.5–251.5° [7033];
UV [7033,8111].

1-(4-Hydroxy-2-methylphenyl)-2-methyl-1-propanone

[761459-40-9] $C_{11}H_{14}O_2$ mol.wt. 178.23



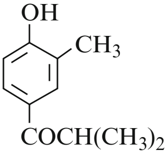
Syntheses

- Obtained (by-product) by Fries rearrangement of 3-methyl-phenyl isobutyrate in the presence of aluminium chloride (7%) [7992].
- Also obtained by reaction of isobutyric acid with m-cresol in the presence of P_2O_5/SiO_2 at 100° for 24 h (26%) [8108].
- Preparation by heating 4-methoxy-2-methylisobutyrophenone with pyridinium chloride at reflux for 15 min (80%) [6672].

b.p.$_1$ 160° [7992].
USE: Intermediate in preparation of fungicidal acid amide derivatives [8113].

1-(4-Hydroxy-3-methylphenyl)-2-methyl-1-propanone

[73206-57-2] $C_{11}H_{14}O_2$ mol.wt. 178.23



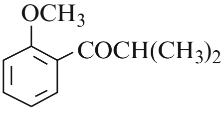
Syntheses

- Obtained by Fries rearrangement of o-tolyl isobutyrate (b.p.$_{15}$ 112°) [7981] with aluminium chloride at 100–110° (major compound) [7981] or at 140° for 4 h (44%) [8106].

b.p.$_{12}$ 182° [7981];
m.p. 123–124° [8106], 122° [7981].

1-(2-Methoxyphenyl)-2-methyl-1-propanone

[74786-53-1] $C_{11}H_{14}O_2$ mol.wt. 178.23

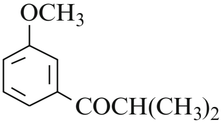


Syntheses

- Preparation by reaction of dimethyl sulfate with 2-hydroxy-isobutyrophenone [7992].
- Also refer to: [8114–8117].

1-(3-Methoxyphenyl)-2-methyl-1-propanone

[6026-75-1] $C_{11}H_{14}O_2$ mol.wt. 178.23



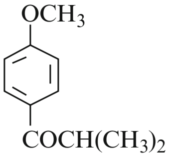
Syntheses

- Preparation by a Grignard reaction of isobutyryl chloride with (3-methoxyphenyl)magnesium chloride at –70° in THF according to the method [8118], (60%) [7356].
- Also refer to: [8115,8119–8121].

oil [7356].

1-(4-Methoxyphenyl)-2-methyl-1-propanone

[2040-20-2] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

- Preparation by reaction of isobutyryl chloride with anisole,
  - in the presence of aluminium chloride [8122];
  - in the presence of graphite in refluxing ethylene dichloride for 3 h (89%) [8123,8124];
  - in the presence of bismuth triflate at 80° for 1 h (99%) [8125];
  - in the presence of bismuth (III) chloride at 80° for 2 h (71%) [8126];
  - in the presence of trifluoroacetic acid [8127].
- Also obtained by reaction of isobutyric acid with anisole,
  - on the solid surface of alumina in the presence of trifluoroacetic anhydride (90%) [7090];
  - in the presence of $Eu(NTf_2)_3$ (15 mol %) at 250° for 16 h (77%) (compound **9f**) [7091].
- Also obtained by reaction of isobutyric anhydride with anisole,
  - in the presence of chloroacetic acid for 48 h at 170–180° (93–97%) [7089];
  - in the presence of $TiCl(OTf)_3$/TfOH in acetonitrile at r.t. for 12 h (90%) [8128];
  - in the presence of iodine for 3 h at reflux (42%) [6721].
- Also obtained by photo-oxidative fragmentation of 2-methyl-1-(4-methoxy phenyl)-1-propanol sensitized by titanium dioxide in acetonitrile in the presence of silver sulfate (18%) [8129].
- Also obtained by treatment of ethyl 2,2-dimethyl-3-oxo-3-(4-methoxyphenyl) propionate with sulfuric acid in refluxing acetic acid (41%) [8130].
- Also refer to: [6672,7033,7090,7095,8115,8121,8131–8133].

Isolation from natural sources

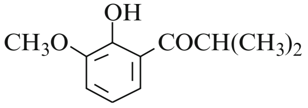
- From *Betula Alba* L., also named *Betula verrucosa* Ehr or *Betula pendula* Roth [8122].

b.p.$_{2.1}$ 12–114° [7095], b.p.$_{13}$ 148° [8122], b.p.$_{14}$ 149–150° [7033],
b.p.$_{14}$ 150° [8130], b.p.$_{16}$ 152.5–153° [7089], b.p.$_{40}$ 188–190° [6721],
b.p. 276–277° [8122]; ^{13}C NMR [7095], UV [7033].

Semicarbazone $C_{12}H_{17}N_3O_2$ mol.wt. 235.29 (m.p. 193–194° [8122], 188–189° [8130]).

1-(2-Hydroxy-3-methoxyphenyl)-2-methyl-1-propanone

[266310-09-2] $C_{11}H_{14}O_3$ mol.wt. 194.23

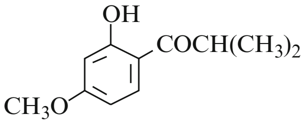


Synthesis

- Refer to: [7131] (Chinese paper).

1-(2-Hydroxy-4-methoxyphenyl)-2-methyl-1-propanone

[29048-55-3] $C_{11}H_{14}O_3$ mol.wt. 194.23



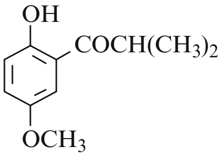
Syntheses

- Obtained by partial methylation of 2,4-dihydroxy-isobutyrophenone with dimethyl sulfate in the presence of sodium methoxide in refluxing methanol [7276], (66%) [8020].
- Other preparation (Japanese paper) (93%) [8021].

b.p.$_{0.2}$ 120–123° [8020], b.p.$_5$ 127–128° [8021]; m.p. 27° [8020].

1-(2-Hydroxy-5-methoxyphenyl)-2-methyl-1-propanone

[176642-56-1] $C_{11}H_{14}O_3$ mol.wt. 194.23



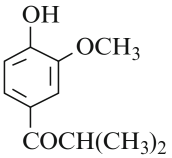
Syntheses

- Refer to: [7150,7984].

USE: For preparation of agrochemical pesticides [7984].

1-(4-Hydroxy-3-methoxyphenyl)-2-methyl-1-propanone

[14046-53-8] $C_{11}H_{14}O_3$ mol.wt. 194.23



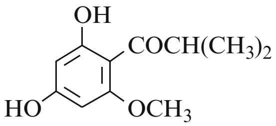
Syntheses

- Preparation by reaction of isobutyric anhydride with guaiacol in the presence of zinc chloride at 155° for 3 min [7991].
- Also obtained by Fries rearrangement of guaiacol isobutyrate with aluminium chloride in nitrobenzene, first at r.t. for 3 h, then at 90° for 30 min (14%) [6828].
- Also refer to: [8134].

b.p.$_{0.07}$ 118° [6828];
m.p. 95–97.5° [6828], 86–87° [7991];
^{1}H NMR [6828], IR [6828].
BIOLOGICAL ACTIVITY: As catechol-O-methyltransferase inhibitor [7991]; pharmacology [6828].

1-(2,4-Dihydroxy-6-methoxyphenyl)-2-methyl-1-propanone *(Robustaol B)*

[102092-19-3] $C_{11}H_{14}O_4$ mol.wt. 210.23



Syntheses

- Obtained by reaction of isobutyronitrile with 5-methoxy-resorcinol (Hoesch reaction) [8135].
- Total synthesis [8136] (Chinese paper).

Isolation from natural sources

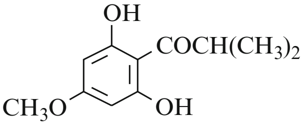
- From *Eucalyptus robusta* Sm. [8136].

- From *Kunzea sinclairii* and *Kunzea ericoides* (Myrtaceae) [8135].

  ^{1}H NMR [8135], ^{13}C NMR [8135], spectral analyses [8136] (Chinese paper).
  BIOLOGICAL ACTIVITY: Antiviral [8135,8137].

1-(2,6-Dihydroxy-4-methoxyphenyl)-2-methyl-1-propanone

[42541-62-8] $C_{11}H_{14}O_4$ mol.wt. 210.23



Syntheses

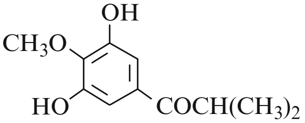
- Preparation by reaction of diazomethane with phloroisobutyrophenone in ethyl ether at 0° for 5 days (59%) [8051].
- Also obtained (poor yield) by reaction of dimethyl sulfate with phloroisobutyrophenone in the presence of potassium bicarbonate in refluxing benzene overnight (6%) [8138].
- Also obtained by reaction of isobutyronitrile with 5-methoxyresorcinol (Hoesch reaction) [8135].

Isolation from natural sources

- From *Kunzea sinclairii* and *Kunzea ericoides* (Myrtaceae) [8135].
  m.p. 162–163° [8138], 153–155° (d) [7175];
  ^{1}H NMR [8051,8135,8138], IR [8051,8138], UV [8051].
  BIOLOGICAL ACTIVITY: Antiviral [8135].

1-(3,5-Dihydroxy-4-methoxyphenyl)-2-methyl-1-propanone

[148204-60-8] $C_{11}H_{14}O_4$ mol.wt. 210.23



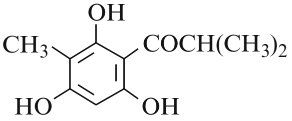
Isolation from natural sources

- Identification in liquid wastes from eucalyptus wood and kraft lignin charring [7259].

GC [7259], GC-MS [7259].

2-Methyl-1-(2,4,6-trihydroxy-3-methylphenyl)-1-propanone

[69480-03-1] $C_{11}H_{14}O_4$ mol.wt. 210.23



Syntheses

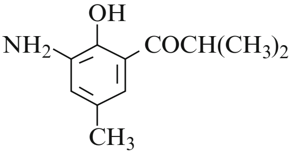
- Preparation by reaction of isobutyronitrile with 2-methyl-phloroglucinol (Hoesch reaction) [6895,7255], (32%) [8139], (35%) [8140].
- Preparation by reaction of isobutyryl chloride with 2-methyl-phloroglucinol in the presence of aluminium chloride in carbon disulfide/nitrobenzene mixture (51%) [8054].
- Also refer to: [6891].

m.p. (monohydrate) 160–161° [8140]; (anhydrous) 161–162° [7255,8140], 160° [8054], 158–159° [6895];
CG [6895]; MS [6895].

BIOLOGICAL ACTIVITY: Antimicrobial for staphylococcus aureus [6891].

1-(3-Amino-2-hydroxy-5-methylphenyl)-2-methyl-1-propanone

[70977-80-9] $C_{11}H_{15}NO_2$ mol.wt. 193.25



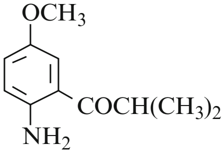
Syntheses

– Preparation by hydrogenation of the 2-hydroxy-5-methyl-3-nitroisobutyrophenone using 5% Pd/C as catalyst in ethanol [6993], (89%) [6932].

m.p. 41–42° [6926,6993].

1-(2-Amino-5-methoxyphenyl)-2-methyl-1-propanone

[166973-19-9] $C_{11}H_{15}NO_2$ mol.wt. 193.25



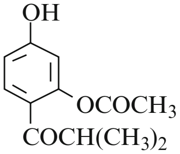
Synthesis

– Refer to: [8141].

USE: Intermediate; preparation of triazoloquinolines which increase apo-A1 production for use in treatment of diseases or conditions caused by elevated levels of LDL-cholesterol or by inflammation.

1-[2-(Acetyloxy)-4-hydroxyphenyl]-2-methyl-1-propanone

[251463-58-8] $C_{12}H_{14}O_4$ mol.wt. 222.24



Synthesis

– Obtained by partial enzymatic regioselective deacetylation of 2,4-diacetoxyisobutyrophenone (oil) with porcine pancreatic lipase in THF in the presence of n-butanol for 48 h at 42–45° (55%) [8022].

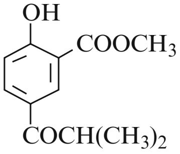
semi-solid [8022]:

^{1}H NMR [8022], ^{13}C NMR [8022], IR [8022], UV [8022], MS [8022]; TLC [8022].

2-Hydroxy-5-(2-methyl-1-oxopropyl)benzoic acid methyl ester

Methyl 2-hydroxy-5-isobutyrylbenzoate

$C_{12}H_{14}O_4$ mol.wt. 222.24



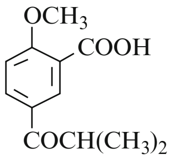
Syntheses

– Obtained by reaction of isobutyryl chloride with methyl o-anisate in carbon disulfide in the presence of aluminium chloride, first at 0°, then at r.t. for 30 h (23%) [8102].

– Also obtained by Fries rearrangement of o-carbomethoxyphenyl isobutyrate (b.p.$_{10-12}$ 150–154.5°) [8102].

m.p. 97–99° [8102].

2-Methoxy-5-(2-methyl-1-oxopropyl)benzoic acid



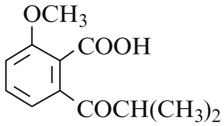
$C_{12}H_{14}O_4$ mol.wt. 222.24

Synthesis

- Obtained by treatment of methyl 2-methoxy-5-isobutyrylbenzoate with sodium hydroxide in dilute ethanol (37%) [8102].

m.p. 97.5–99.5° [8102].

2-Methoxy-6-(2-methyl-1-oxopropyl)benzoic acid

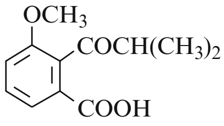


$C_{12}H_{14}O_4$ mol.wt. 222.24

Synthesis

- Obtained by reaction of diisopropylcadmium with 3-methoxyphthalic anhydride (a mixture with its isomer below) [8102].

3-Methoxy-2-(2-methyl-1-oxopropyl)benzoic acid



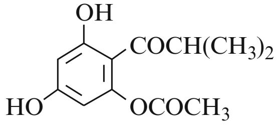
$C_{12}H_{14}O_4$ mol.wt. 222.24

Synthesis

- Obtained by reaction of diisopropylcadmium with 3-methoxyphthalic anhydride (a mixture with its isomer above) [8102].

1-[2-(Acetyloxy)-4,6-dihydroxyphenyl]-2-methyl-1-propanone

[62545-34-0]



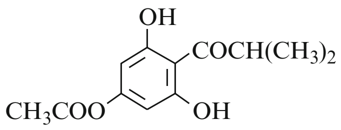
$C_{12}H_{14}O_5$ mol.wt. 238.24

Synthesis

- Obtained by acetylation of phloroisobutyrophenone [8058].

1-[4-(Acetyloxy)-2,6-dihydroxyphenyl]-2-methyl-1-propanone

[62545-45-3]



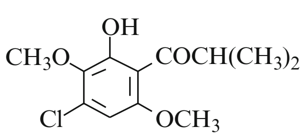
$C_{12}H_{14}O_5$ mol.wt. 238.24

Synthesis

- Obtained by acetylation of phloroisobutyrophenone [8058].

1-(4-Chloro-2-hydroxy-3,6-dimethoxyphenyl)-2-methyl-1-propanone

[88771-65-7]



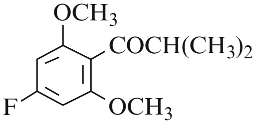
$C_{12}H_{15}ClO_4$ mol.wt. 258.70

Synthesis

- Refer to: [7985].

1-(4-Fluoro-2,6-dimethoxyphenyl)-2-methyl-1-propanone

[263010-96-4] $C_{12}H_{15}FO_3$ mol.wt. 226.24

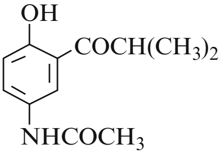


Syntheses

– Refer to: [8142,8143].

4-Hydroxy-3-(2-methyl-1-oxopropyl)acetanilide

$C_{12}H_{15}NO_3$ mol.wt. 221.26

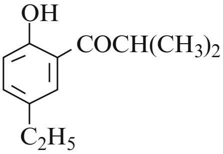


Synthesis

– Refer to: [7283].

1-(5-Ethyl-2-hydroxyphenyl)-2-methyl-1-propanone

[847344-76-7] $C_{12}H_{16}O_2$ mol.wt. 192.26

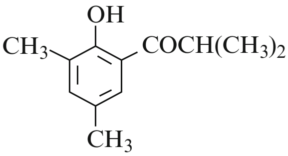


Syntheses

– Refer to: [7980,8144].

1-(2-Hydroxy-3,5-dimethylphenyl)-2-methyl-1-propanone

[106141-17-7] $C_{12}H_{16}O_2$ mol.wt. 192.26



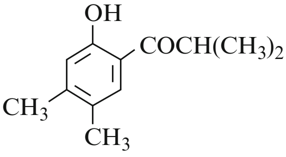
Syntheses

- Obtained by Fries rearrangement of 2,4-dimethylphenyl isobutyrate (b.p. 245°) with aluminium chloride at 120° for 3 h (70%) [7981].
- Also obtained by reaction of sec-butyllithium (1.1 equiv) with 2-bromo-4,6-dimethylphenyl isobutyrate in tetrahydrofuran/ethyl ether/hexane at −95° for 30 min and −78°for 30 min, then hydrolysis with saturated ammonium chloride (72%) (metal-promoted Fries rearrangement) [6590].
- Also refer to: [8110].

yellow oil [7981]; b.p.$_{13}$ 129° [7981];
^{1}H NMR [6590], ^{13}C NMR [6590], IR [6590].

1-(2-Hydroxy-4,5-dimethylphenyl)-2-methyl-1-propanone

[50342-15-9] $C_{12}H_{16}O_2$ mol.wt. 192.26



Synthesis

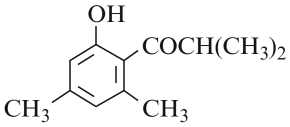
– Obtained by Fries rearrangement of 3,4-dimethylphenyl isobutyrate with aluminium chloride at 130° for 5 h [7276].

b.p.$_{20}$ 150° [7276].

Oxime $C_{12}H_{17}NO_2$ mol.wt. 207.27 (m.p. 136°) [7276].

1-(2-Hydroxy-4,6-dimethylphenyl)-2-methyl-1-propanone

[21009-91-6] $C_{12}H_{16}O_2$ mol.wt. 192.26



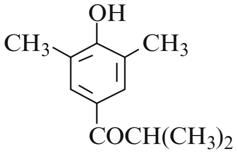
Syntheses

- Obtained by reaction of isobutyryl chloride with 3,5-di-methylanisole in the presence of aluminium chloride in boiling carbon disulfide [7276,8110], (70–75%) [7052].
- Also obtained by Fries rearrangement of 3,5-dimethylphenyl isobutyrate with aluminium chloride at 140° for 30 min (25%) [8145] or at 130° for 5 h [7276].
- Also obtained by reaction of isobutyric acid with 3,5-dimethylphenol in the presence of boron trifluoride at 95° [6583].
- Also refer to: [8110].

b.p.$_{20}$ 150° [7276].
m.p. 93–94° [7052,8110], 93° [6583], 82–82.5° and 91–91.5° (dual m.p.) [8145];
^{1}H NMR [8145], IR [8145].

1-(4-Hydroxy-3,5-dimethylphenyl)-2-methyl-1-propanone

$C_{12}H_{16}O_2$ mol.wt. 192.26



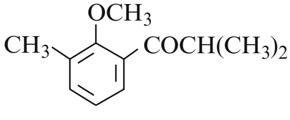
Synthesis

- Preparation by Fries rearrangement of 2,6-dimethylphenyl isobutyrate with aluminium chloride at 125° for 3.5 h (94%) [6401].

m.p. 106.5–107° [6401].

1-(2-Methoxy-3-methylphenyl)-2-methyl-1-propanone

[82053-90-5] $C_{12}H_{16}O_2$ mol.wt. 192.26



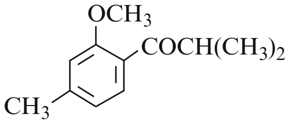
Syntheses

- Preparation by reaction of dimethyl sulfate with 2-hydroxy-3-methylisobutyrophenone in the presence of potassium carbonate in boiling acetone for 10 h (93%) [8106].
- Also refer to: [8105].

b.p.$_{15}$ 124–125° [8106].

1-(2-Methoxy-4-methylphenyl)-2-methyl-1-propanone

[143428-35-7] $C_{12}H_{16}O_2$ mol.wt. 192.26



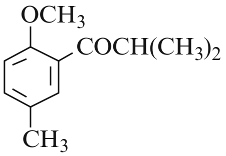
Synthesis

- Obtained by reaction of dimethyl sulfate with 2-hydroxy-4-methylisobutyrophenone in the presence of sodium hydroxide [7992].

b.p.$_{15}$ 136–138° [7992].

1-(2-Methoxy-5-methylphenyl)-2-methyl-1-propanone

[30574-34-6] $C_{12}H_{16}O_2$ mol.wt. 192.26



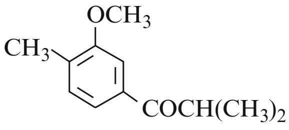
Syntheses

- Preparation by reaction of dimethyl sulfate with 2-hydroxy-5-methylisobutyrophenone in the presence of sodium hydroxide [7033,7992].
- Also obtained by oxidation of 1-(2-methoxy-5-methylphenyl)-2-methyl-1-propanol with PCC (90%) [8146] according to the process [8147].
- Also refer to: [7313,8148].

b.p.$_5$ 105° [8146], b.p.$_{10}$ 136–137.5° [7033], b.p.$_{14}$ 138° [7992];
^{1}H NMR [8146], IR [8146], UV [7033].

1-(3-Methoxy-4-methylphenyl)-2-methyl-1-propanone

[74654-51-6] $C_{12}H_{16}O_2$ mol.wt. 192.26



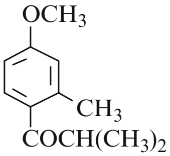
Synthesis

- Preparation by nucleophilic substitution reaction via arene-chromium tricarbonyl complex (83%) [8149].

b.p.$_{0.04}$ 92–110° [8149];
^{1}H NMR [8149], IR [8149]; GLC [8149].

1-(4-Methoxy-2-methylphenyl)-2-methyl-1-propanone

[54696-07-0] $C_{12}H_{16}O_2$ mol.wt. 192.26



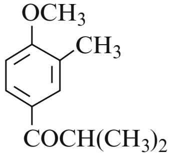
Syntheses

- Refer to: [6672,8113].

USE: Intermediate in preparation of fungicidal acid amide derivatives [8113].

1-(4-Methoxy-3-methylphenyl)-2-methyl-1-propanone

[2954-63-4] $C_{12}H_{16}O_2$ mol.wt. 192.26



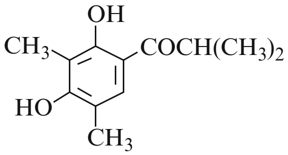
Syntheses

- Preparation by acylation of 2-methylanisole,
  - with isobutyric acid in the presence of triflic acid [8150];
  - with isobutyric anhydride in acetonitrile in the presence of $TiCl(OTf)_3/TfOH$ at r.t. for 12 h (96%) [8128];
  - with isobutyryl chloride in the presence of aluminium chloride in carbon disulfide (70%) [7319], first at 0°, then at r.t. (81%) [8151].
- Also refer to: [8152–8154].

b.p.$_{0.5}$ 110–115° [8151], b.p. 275° [7319]; m.p. 22–23° [8151].

1-(2,4-Dihydroxy-3,5-dimethylphenyl)-2-methyl-1-propanone

[267001-71-8] $C_{12}H_{16}O_3$ mol.wt. 208.26



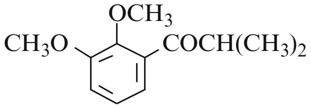
Synthesis

- Obtained by reaction of isobutyric acid with 2,4-di-methylresorcinol in the presence of boron trifluoride etherate at 120° for 1–2 h under an argon atmosphere. Then, the resulting complex was refluxed for 30 min to 1 h in an aqueous THF (70–80%) [8155].

m.p. 81–82° [8155];
^{1}H NMR [8155], ^{13}C NMR [8155], IR [8155], MS [8155]; TLC [8155].

1-(2,3-Dimethoxyphenyl)-2-methyl-1-propanone

[105329-87-1] $C_{12}H_{16}O_3$ mol.wt. 208.26



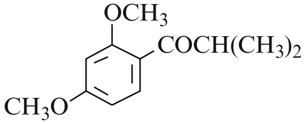
Syntheses

- Obtained by oxidation of 1-(2,3-dimethoxyphenyl)-2-methyl-1-propanol in acetone with sodium dichromate in dilute sulfuric acid at r.t. for 3 h (60%) [6726].
- Also refer to: [7131].

Colourless oil [6726];
^{1}H NMR [6726], ^{13}C NMR [6726], IR [6726], MS [6726].

1-(2,4-Dimethoxyphenyl)-2-methyl-1-propanone

[86774-65-4] $C_{12}H_{16}O_3$ mol.wt. 208.26



Syntheses

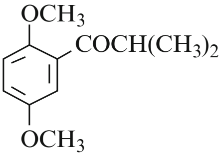
- Preparation by reaction of isobutyryl chloride with 1,3-di-methoxybenzene [6796] in the presence of aluminium chloride in petroleum ether [7992].

- Preparation by reaction of N,N-diethylisobutyramide with 1,3-dimethoxybenzene in the presence of phosphorous oxychloride (63%) [8156,8157].
- Preparation by reaction of isobutyronitrile with 1,3-dimethoxybenzene in the presence of triflic acid [8001].
- Also refer to: [8158].

b.p.$_1$ 120–125° [8156,8157], b.p.$_2$ 126–130° [6796], b.p.$_2$ 128° [7992];
m.p. 35–37° [8001]; ^{1}H NMR [8001], IR [8001].

1-(2,5-Dimethoxyphenyl)-2-methyl-1-propanone

[89556-60-5] $C_{12}H_{16}O_3$ mol.wt. 208.26



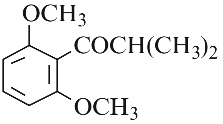
Syntheses

- Preparation by reaction of isobutyryl chloride with 1,4-di-methoxybenzene [7697] in the presence of aluminium chloride in petroleum ether [7697,7992] or in carbon disulfide [7336].

b.p.$_2$ 125° [7697,7992];
^{1}H NMR [7336], IR [7336], UV [7336], MS [7336].

1-(2,6-Dimethoxyphenyl)-2-methyl-1-propanone

[52856-18-5] $C_{12}H_{16}O_3$ mol.wt. 208.26



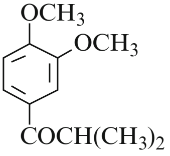
Syntheses

- Preparation by condensation of 2,6-dimethoxyphenyllithium,
  - with isobutyryl chloride in ethyl ether at −78° (78%) [8158];
  - with methyl isobutyrate in refluxing ethyl ether for 5 h (46%) [8159].

m.p. 38.9–39.2° [8159].

1-(3,4-Dimethoxyphenyl)-2-methyl-1-propanone

[14046-55-0] $C_{12}H_{16}O_3$ mol.wt. 208.26



Syntheses

- Preparation by acylation of veratrole,
  - with isobutyryl chloride in the presence of aluminium chloride [7350], (85%) [6796], in benzene [8083], in ethylene dichloride (73%) [7356];
  - with isobutyric acid in the presence of PPA at 60° for 2.5 h [8160], (87%) [7360] or for 15 h (68%) [8161];
  - with isobutyric anhydride in acetonitrile in the presence of $TiCl(OTf)_3/TfOH$ at r.t. for 12 h (89%) [8128].

- Also obtained by oxidation of 4-methoxyphenylpropane with DDQ in wet dioxane/silica gel under sonication (61%) [7093].
- Also obtained by reaction of 3,4-dimethoxybenzoic acid with isopropylmagnesium bromide (72%) [8162].
- Also obtained by oxidation of 1-(3,4-dimethoxyphenyl)-2-methyl-1-propanol with chromium trioxide in dilute sulfuric acid (Jones's reaction) (82%) [8163].
- Also refer to: [6828,7539,8164–8166].

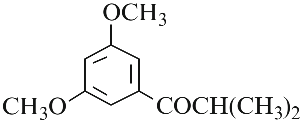
clear oil [8162]; b.p.$_{0.2}$ 116–117° [6796,8083], b.p.$_{0.1-0.7}$ 117–126° [7360]; ^{1}H NMR [7093,8162,8163], ^{13}C NMR [7093,8162].

USE: Fungicide [7539] or intermediate in preparation of fungicidal acid amide derivatives [8113].

BIOLOGICAL ACTIVITY: Antiinflammatory [7350].

1-(3,5-Dimethoxyphenyl)-2-methyl-1-propanone

[73109-77-0] $C_{12}H_{16}O_3$ mol.wt. 208.26



Syntheses

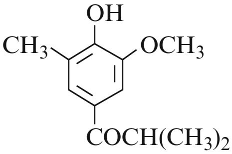
- Obtained by reaction of isobutylmagnesium bromide on 3,5-dimethoxybenzonitrile (m.p. 87–88°) (88%) [8167].
- Also obtained by reaction of isopropylmagnesium bromide with 3,5-dimethoxybenzamide (71%) [8168].

oil [8168]; b.p.$_1$ 117–119° [8168], b.p.$_2$ 143–145° [8167].

Semicarbazone $C_{13}H_{19}N_3O_3$ mol.wt. 265.31 (m.p. 195–196°) [8167].

1-(4-Hydroxy-3-methoxy-5-methylphenyl)-2-methyl-1-propanone

$C_{12}H_{16}O_3$ mol.wt. 208.26



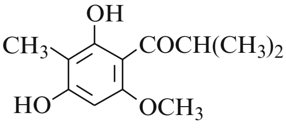
Synthesis

- Preparation by Fries rearrangement of 2-methoxy-6-methylphenyl isobutyrate with aluminium chloride in refluxing carbon disulfide for 3 h (60%) [7390].

b.p.$_{0.35}$ 122–130° [7390]; m.p. 52–53° [7390].

1-(2,4-Dihydroxy-6-methoxy-3-methylphenyl)-2-methyl-1-propanone

[91555-68-9] $C_{12}H_{16}O_4$ mol.wt. 224.26



Synthesis

- Preparation by condensation of isobutyronitrile with 2,6-dihydroxy-4-methoxytoluene (Hoesch reaction) (63%) [7255].

Isolation from natural sources

- From the aerial parts of *Hypericum beanii* (Guttiferae) [8169].

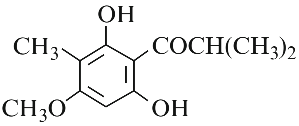
pale yellow oil [8169]; b.p.$_{0.2}$ 135–140° [7255];

m.p. 148–149° [7255];
^{1}H NMR [8169], ^{13}C NMR [8169], IR [8169], UV [7255,8169], MS [8169].

BIOLOGICAL ACTIVITY: Anti-staphylococcal [8169].

1-(2,6-Dihydroxy-4-methoxy-3-methylphenyl)-2-methyl-1-propanone
(Aspidinol-iB)

[42541-64-0] $C_{12}H_{16}O_4$ mol.wt. 224.26



Syntheses

- Obtained by reaction of isobutyronitrile with 2-methyl-phloroglucinol 1-methyl ether (Hoesch reaction) (37%) [8170].
- Also obtained by reaction of isobutyric acid with 2,4-di-hydroxy-6-methoxytoluene (m.p. 114–117°) in the presence of boron trifluoride for 45 min (47%) [8171].
- Preparation by reaction of isobutyryl chloride with 3-methoxy-2-methylphloroglucinol in the presence of aluminium chloride in carbon disulfide/nitrobenzene mixture (69%) [8054].
- Also obtained by reaction of methyl iodide with 2,6-dihydroxy-4-methoxy-isobutyrophenone in methanol in the presence of sodium methoxide at 0° for 5 days (9%) [8051].
- Also obtained by partial methylation of 3-methylphloroisobutyrophenone with diazomethane in ethyl ether for 4 days at r.t. (24%) [7255].
- Also refer to: [8172].

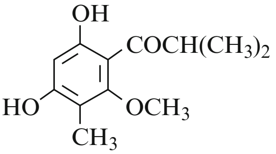
Isolation from natural source

- From *Eucalyptus pulverulenta* [8173].

m.p. 145–147° [8051], 142° [8054], 141–142° [7255,8170], 136–138° [8171];
^{1}H NMR [8051,8171], ^{13}C NMR [8171], IR [8051,8171],
UV [7255,8051], MS [8171].

1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)-2-methyl-1-propanone
(Pseudoaspidinol-iB)

[55382-30-4] $C_{12}H_{16}O_4$ mol.wt. 224.26



Syntheses

- Obtained by alkaline cleavage of kosotoxin. Kosotoxin (m.p. 119–122°) was isolated from *Hagenia abyssinica* (Bruce) Gmel [6932].
- Also obtained by treatment of methyl 2,6-dihydroxy-4-methoxy-3-methyl-5-isobutyrylbenzoate (m.p. 88–90°) with refluxing 5–6% aqueous potassium hydroxide for 30 min (63%) [7255] or for 1 h (94%) [8171].

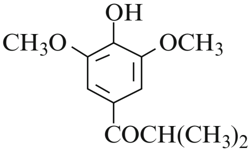
- Also obtained by treatment of 2,6-dihydroxy-4-methoxy-3-methyl-5-isobutyrylbenzoic acid with refluxing 2 N aqueous sodium carbonate for 90 min (86%) [7255].
- Also refer to: [8174–8177].

Isolation from natural sources

- From *Hagenia abyssinica* [8178].

m.p. 81–82° [8171], 79–80° [7255], 59–61° [6932];
^{13}C NMR [8175,8179,8180], UV [7255]; GC [6895].

1-(4-Hydroxy-3,5-dimethoxyphenyl)-2-methyl-1-propanone



$C_{12}H_{16}O_4$ mol.wt. 224.26

Syntheses

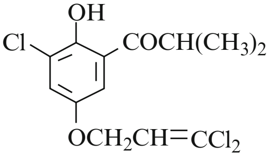
- Obtained by partial demethylation of 3,4,5-trimethoxy-isobutyrophenone in concentrated sulfuric acid at 35–40° for 20 h (85%) [8181].
- Also obtained (by-product) on reaction of isobutylmagnesium bromide with 3,4,5-trimethoxybenzonitrile (45%) [8181], 27% [8167].

m.p. 94° [8167], 93–93.5° [8181].

Semicarbazone $C_{13}H_{19}N_3O_4$ mol.wt. 281.31 (m.p. 162.5°) [8167].

1-[3-Chloro-5-[(3,3-dichloro-2-propen-1-yl)oxy]-2-hydroxyphenyl]-2-methyl-1-propanone

[918311-03-2]



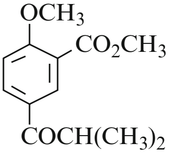
$C_{13}H_{13}Cl_3O_3$ mol.wt. 323.60

Synthesis

- Refer to: [7984].

USE: For preparation of agrochemical pesticides [7984].

2-Methoxy-5-(2-methyl-1-oxopropyl)benzoic acid methyl ester



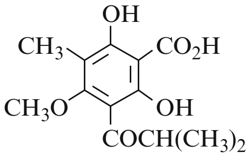
$C_{13}H_{16}O_4$ mol.wt. 236.27

Synthesis

- Preparation by reaction of dimethyl sulfate with methyl 2-hydroxy-5-isobutyrylbenzoate in the presence of sodium methoxide in methanol at 50–60° (98%) [8102].

m.p. 50–52° [8102].

2,6-Dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl)benzoic acid



$C_{13}H_{16}O_6$ mol.wt. 268.27

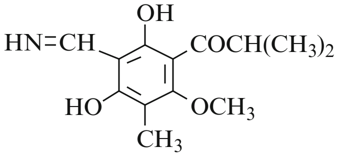
Synthesis

– Obtained by treatment of methyl 2,6-dihydroxy-4-methoxy-3-methyl-5-isobutyrylbenzoate with an aqueous 6% potassium hydroxide solution (32%) [7255].

m.p. 103–105° [7255]; UV [7255].

1-[2,4-Dihydroxy-3-(iminomethyl)-6-methoxy-5-methylphenyl]-2-methyl-1-propanone

[98442-56-9]



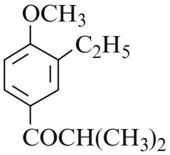
$C_{13}H_{17}NO_4$ mol.wt. 251.28

Synthesis

– Refer to: [8171].

1-(3-Ethyl-4-methoxyphenyl)-2-methyl-1-propanone

[111039-01-1]



$C_{13}H_{18}O_2$ mol.wt. 206.28

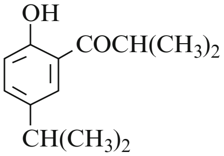
Synthesis

– Preparation by Friedel–Crafts acylation of 2-ethylanisole with isobutyryl chloride in the presence of aluminium chloride [7350].

BIOLOGICAL ACTIVITY: Antiinflammatory [7350].

1-[2-Hydroxy-5-(1-methylethyl)phenyl]-2-methyl-1-propanone

[934524-36-4]



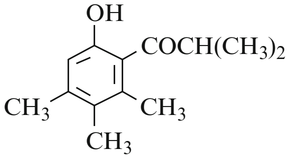
$C_{13}H_{18}O_2$ mol.wt. 206.28

Synthesis

– Refer to: [7980].

1-(6-Hydroxy-2,3,4-trimethylphenyl)-2-methyl-1-propanone

[82490-57-1]



$C_{13}H_{18}O_2$ mol.wt. 206.28

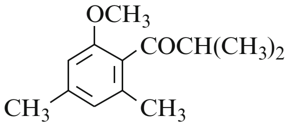
Synthesis

– Obtained by Fries rearrangement of 3,4,5-trimethylphenyl isobutyrate with aluminium chloride, first at 90°, then at 140° for 30 min (14%) [8182].

m.p. 86–87° [8182]; ^{1}H NMR [8182].

1-(2-Methoxy-4,6-dimethylphenyl)-2-methyl-1-propanone

[38319-72-1] $C_{13}H_{18}O_2$ mol.wt. 206.28



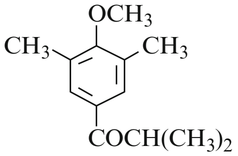
Synthesis

- Obtained by methylation of 2-hydroxy-4,6-dimethyl-isobutyrophenone with dimethyl sulfate [8145].

b.p.$_2$ 109–110° [8145]; ^{1}H NMR [8145], IR [8145].

1-(4-Methoxy-3,5-dimethylphenyl)-2-methyl-1-propanone

[124500-33-0] $C_{13}H_{18}O_2$ mol.wt. 206.28

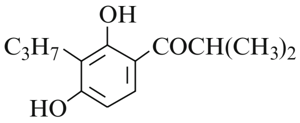


Synthesis

- Refer to: [7976].

1-(2,4-Dihydroxy-3-propylphenyl)-2-methyl-1-propanone

[120072-80-2] $C_{13}H_{18}O_3$ mol.wt. 222.28

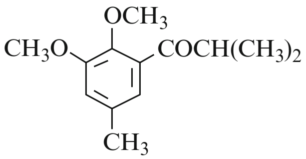


Synthesis

- Refer to: [8183].

1-(2,3-Dimethoxy-5-methylphenyl)-2-methyl-1-propanone

[882512-69-8] $C_{13}H_{18}O_3$ mol.wt. 222.28



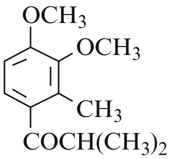
Synthesis

- Obtained by treatment of 1-(2,3-dimethoxy-5-methylphenyl)-2-methyl-1-propanol with PCC and silica gel in methylene chloride for 2 h at r.t. (96%) [8184].

oil [8184];
^{1}H NMR [8184], ^{13}C NMR [8184], IR [8184], MS [8184].

1-(3,4-Dimethoxy-2-methylphenyl)-2-methyl-1-propanone

$C_{13}H_{18}O_3$ mol.wt. 222.28



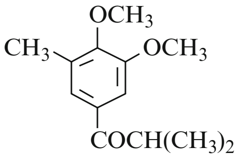
Synthesis

- Obtained by reaction of isobutyryl chloride with 3,4-dimethoxy-2-methylphenylmagnesium bromide in THF (51%) [7390].

b.p.$_{0.15}$ 99° [7390]; IR [7390], UV [7390].

2,4-Dinitrophenylhydrazone $C_{19}H_{22}N_4O_6$ mol.wt. 402.41 (m.p. 111–112°) [7390].

1-(3,4-Dimethoxy-5-methylphenyl)-2-methyl-1-propanone



$C_{13}H_{18}O_3$ mol.wt. 222.28

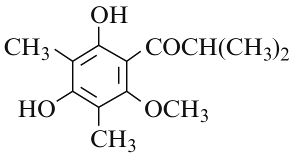
Synthesis

– Preparation by reaction of dimethyl sulfate with 4-hydroxy-3-methoxy-5-methylisobutyrophenone in methanol in the presence of sodium methoxide (40%) [7390].

b.p.$_{0.06}$ 103° [7390]; IR [7390], UV [7390].

1-(2,4-Dihydroxy-6-methoxy-3,5-dimethylphenyl)-2-methyl-1-propanone

[97761-90-5]



$C_{13}H_{18}O_4$ mol.wt. 238.28

Synthesis

– Obtained by hydrogenation of 2,6-dihydroxy-3-isobutyryl-4-methoxy-5-methylbenzaldehyde in acetic acid in the presence of 10% Pd/C for 1.5 h (61%) [8171].

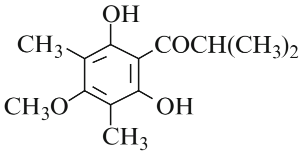
Isolation from natural sources

– From *Hagenia abyssinica* (Rosaceae) [8171,8178].

m.p. 73° [8171];
^{1}H NMR [8171,8178], ^{13}C NMR [8171,8178], IR [8171],
MS [8171,8178].

1-(2,6-Dihydroxy-4-methoxy-3,5-dimethylphenyl)-2-methyl-1-propanone

[138690-38-7]



$C_{13}H_{18}O_4$ mol.wt. 238.28

Isolation from natural sources

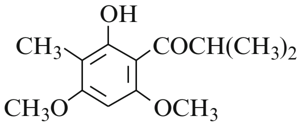
– From *Eucalyptus robusta* Smith leaves (Myrtaceae) [8185].
– Also refer to: [8172].

^{1}H NMR [8185], ^{13}C NMR [8185], IR [8185],
UV [8185], MS [8185]; HPLC [8185].

BIOLOGICAL ACTIVITY: Phosphodiesterase inhibitory [8185].

1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-2-methyl-1-propanone *(Baeckeol)*

[479-88-9]



$C_{13}H_{18}O_4$ mol.wt. 238.28

Syntheses

– Preparation in two steps: First, reaction of isobutyric acid with 2,4,6-trimethoxytoluene (m.p. 24–26°) in the presence of boron trifluoride. Then, the complex obtained* (76%, m.p. 180°) was hydrolyzed in dilute methanol at 50° for 1 h (96%) [8139].

- 2,2-Difluor-1,2-dihydro-4-isopropyl-5,7-dimethoxy-8-methyl-1-oxa-3-oxonia-2-boratanaphthalin (5a) (in german).

- Also obtained by direct methylation of phloroisobutyrophenone with methyl iodide in the presence of potassium carbonate in refluxing acetone for 2 h (29%) [8186].
- Also obtained by reaction of diazomethane with 2,4,6-trihydroxy-3-methylisobutyrophenone in ethyl ether (20%) [8140], (13%) [7255,8139].

Isolation from natural sources

- From the essential oils of *Baeckea crenulata* and *Darwinia grandiflora* (Myrtaceae) [8140,8186–8188].
- From the leaves and terminal branches of *Thryptomene saxicola* (Myrtaceae) [8189].
- From *Calythrix angulata* Lindl. [8190].
- From *Baeckea frutescens* L. (Myrtaceae) [8191–8193] and *Baeckea gunniana* variety *latifolia* [8194].
- From steam volatile leaf oils of some *Melaleuca* species from western Australia (Myrtaceae) [8195].
- From the leaf oil of *Xanthostemon eucalyptoides* (Myrtaceae) [8196].
- Also refer to: [8172,8197].

oil [8189]; b.p.$_{0.1}$ 140° [7255];

m.p. 104° [8190], 103.5–104° [8194], 103–104° [7255,8186–8188,8193], 103° [8139], 102–103° [8140];

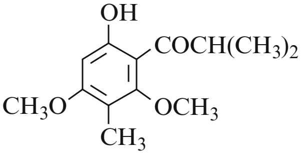
^{1}H NMR [8139,8189,8191], ^{13}C NMR [8189,8191], UV [8085,8198], [7255,8139], MS [8191]; GC [8190,8196]; GC-MS [8195,8196].

BIOLOGICAL ACTIVITY: Antitumor [8191]; cytotoxic [8191].

1-(6-Hydroxy-2,4-dimethoxy-3-methylphenyl)-2-methyl-1-propanone

(o-Isobaeckeol, Isobaeckeol)

[98442-62-7] $C_{13}H_{18}O_4$ mol.wt. 238.28



Synthesis

- Obtained by reaction of isobutyric acid with 4-hydroxy-2,6-dimethoxytoluene (m.p. 144–146°) in the presence of boron trifluoride (51%) [8171].

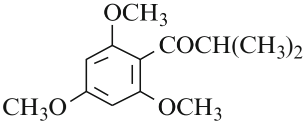
Isolation from natural sources

- From the leaves and terminal branches of *Thryptomene saxicola* (Myrtaceae) [8189].
- From Australian Austromyrtus (*Austromyrtus dulcis* and *Austromyrtus tenuifolia*) (Myrtaceae) [8199].
- From the leaf essential oils of *Eucalypyus miniata* and *Eucalypyus chartaboma* [8200].
- From the leaf oil of *Xanthostemon crenulatus* and *Xanthostemon umbrosus* (Myrtaceae) [8196].
- Also refer to: [8172].

N.B.: This ketone gives a stable complex with boron trifluoride [8172], named "2,2-difluor-4-isopropyl-5,7-dimethoxy-6-methyl-1-oxa-3-oxonia-2-boratanaphthalin" (in German in the paper) (m.p. 141–142°) [8171].
oil [8189]; m.p. 56° [8171];
^{1}H NMR [8171,8189,8200], ^{13}C NMR [8189,8200],
IR [8171,8189], MS [8189,8200];
GC [8196], GC-MS [8189,8196,8199].

2-Methyl-1-(2,4,6-trimethoxyphenyl)-1-propanone (*Conglomerone*)

[480-25-1] $C_{13}H_{18}O_4$ mol.wt. 238.28



Synthesis

- Preparation by reaction of isobutyronitrile with 1,3,5-tri-methoxybenzene (Hoesch reaction) [7515].

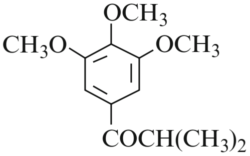
Isolation from natural sources

- From *Eucalyptus conglomerata* (Myrtaceae) [8201].
- Also refer to: [8197].

m.p. 61–62° [7515], 57–60° [8001]; ^{1}H NMR [8001], IR [8001].

2-Methyl-1-(3,4,5-trimethoxyphenyl)-1-propanone

$C_{13}H_{18}O_4$ mol.wt. 238.28



Syntheses

- Obtained by reaction of isobutylmagnesium bromide on 3,4,5-trimethoxybenzonitrile (27%) [8167], (16%) [8181].
- Also obtained by reaction of diisobutylcadmium with 3,4,5-trimethoxybenzoyl chloride (50%) [7538], according to the method [7315].

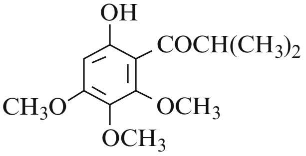
Isolation from natural source

- From the essential oil of *Ocotea comoriensis* Kostermans (Lauraceae) [8202].

b.p.$_{0.9}$ 34–136° [7538], b.p.$_{1}$ 147–150° [8167], b.p.$_{6}$ 164–166° [8181];
m.p. 37–39° [8181]; GC [8202]; GC-MS [8202].

1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-2-methyl-1-propanone

[853577-59-0] $C_{13}H_{18}O_5$ mol.wt. 254.28

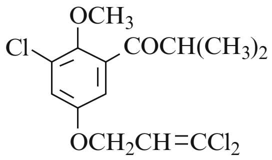


Syntheses

- Preparation by reaction of isobutyryl chloride with 3,4,5-trimethoxyphenol using boron trifluoride etherate as catalyst (80%) [8203].
- Also refer to: [8204].

USE: Preparation of chromenones as inhibitors of antiapoptotic BCL-2 family members for treatment of cancer [8204].

1-[3-Chloro-5-[(3,3-dichloro-2-propen-1-yl)oxy]-2-methoxyphenyl]-2-methyl-1-propanone



$C_{14}H_{15}Cl_3O_3$ mol.wt. 337.63

Synthesis

- Refer to: [7984].

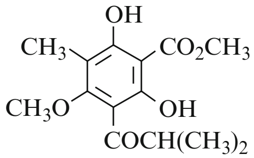
USE: For preparation of agrochemical pesticides [7984].

Oxime [918311-05-4] $C_{14}H_{16}Cl_3NO_3$ mol.wt. 352.64.

USE: For preparation of agrochemical pesticides [7984].

Methyl 2,6-dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl)benzoate

[67771-05-5] $C_{14}H_{18}O_6$ mol.wt. 282.29



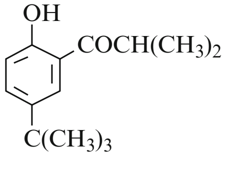
Syntheses

- Preparation by treatment of methyl 2,6-dihydroxy-4-methoxy-3-methylbenzoate with an isobutyric anhydride and isobutyric acid mixture in the presence of boron trifluoride for 5 h at r.t. (87%) [7255], (77%) [8171].

b.p.$_{0.001}$ 130–135° [7255]; m.p. 88–90° [7255,8171];
^{1}H NMR [8171], ^{13}C NMR [8171], IR [8171], UV [7255,8171].

1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-2-methyl-1-propanone

[75060-54-7] $C_{14}H_{20}O_2$ mol.wt. 220.31

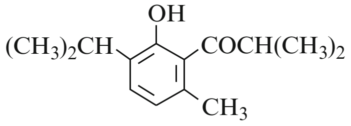


Synthesis

- Refer to: [7106].

1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-2-methyl-1-propanone

[106477-03-6] $C_{14}H_{20}O_2$ mol.wt. 220.31

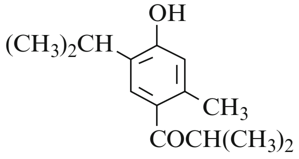


Syntheses

- Obtained by Friedel–Crafts acylation of thymol with isobutyric acid in the presence of aluminium chloride at reflux for 12 h (79%) [7581].
- Also obtained by Fries rearrangement of thymyl isobutyrate with aluminium chloride [7580].

b.p.$_{13}$ 118–120° [7580,7581]; m.p. 49° [7580,7581].

1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-2-methyl-1-propanone



$C_{14}H_{20}O_2$ mol.wt. 220.31

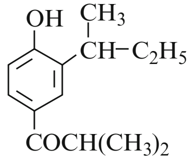
Syntheses

- Obtained by reaction of isobutyryl chloride with thymol in the presence of aluminium chloride in nitrobenzene [7470].
- Also obtained by treatment of its methyl ether (b.p.$_{16}$ 168°) with pyridinium chloride at reflux for 3 h (22%) [7583].

b.p.$_{13}$ 194° [7583]; m.p. 80° [7470], 79° [7583].

1-[4-Hydroxy-3-(1-methylpropyl)phenyl]-2-methyl-1-propanone

[16648-76-3] $C_{14}H_{20}O_2$ mol.wt. 220.31



Syntheses

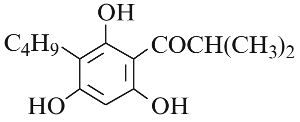
- Preparation by Fries rearrangement of 2-sec-butylphenyl isobutyrate with aluminium chloride in nitrobenzene at 50° for 24 h [7589].
- Also refer to: [7590].

b.p.$_{0.8}$ 172° [7589]; m.p. 88–89° [7589].

USE: Fungicide [7590].

1-(3-Butyl-2,4,6-trihydroxyphenyl)-2-methyl-1-propanone

[66711-57-7] $C_{14}H_{20}O_4$ mol.wt. 252.31



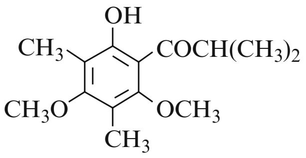
Synthesis

- Refer to: [7640].

USE: Fungicide [7640].

1-(2-Hydroxy-4,6-dimethoxy-3,5-dimethylphenyl)-2-methyl-1-propanone

[173867-31-7] $C_{14}H_{20}O_4$ mol.wt. 252.31



Isolation from natural sources

- From the leaves of *Baeckea frutescens* L. (Myrtaceae) [8191,8192].
- From the leaf oil of the Genus *Xanthostemon* (Myrtaceae) [8196].

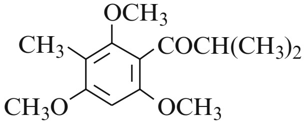
oil [8192]; ^{1}H NMR [8191,8192], ^{13}C NMR [8191,8192], MS [8191,8192]; GC [8196], GC-MS [8196].

BIOLOGICAL ACTIVITY: Antitumor [8191]; cytotoxic against leukaemia L 1210 cells [8191,8192].

2-Methyl-1-(2,4,6-trimethoxy-3-methylphenyl)-1-propanone

(Baeckeol methyl ether)

[92301-90-1]



$C_{14}H_{20}O_4$ mol.wt. 252.31

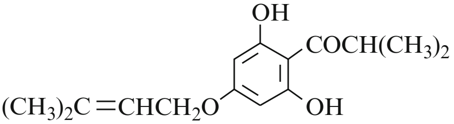
Isolation from natural sources

- From leaf essential oils of *Eucalyptus chartaboma* and *Eucalyptus miniata* (Myrtaceae) [8200].
- From the leaf oil of *Xanthostemon eucalyptoides* (Myryaceae) [8196].

^{1}H NMR [8200], ^{13}C NMR [8200], MS [8200]; GC [8196], GC-MS [8196].

1-[2,6-Dihydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-2-methyl-1-propanone

[70219-81-7]



$C_{15}H_{20}O_4$ mol.wt. 264.32

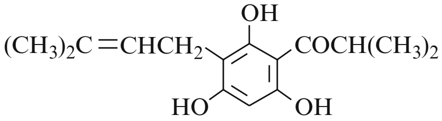
Isolation from natural sources

- From *Helichrysum crispum* (Compositae) [8205].
- From *Leontonyx squarrosus* DC (Compositae) (tribe Inulae) [7620].
- From *Helichrysum asperum* (Thunb.) Hilliard et Burtt. var. *albidulum* (DC) Hilliard (compound **7**) (Compositae) [8206].

colourless oil [7620]; ^{1}H NMR [7620], IR [7620], MS [7620].

2-Methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-propanone

[35932-36-6]



$C_{15}H_{20}O_4$ mol.wt. 264.32

Syntheses

- Obtained by reaction of isopentenyl bromide (prenyl bromide) with phloroisobutyrophenone [8207],
  - in a benzene/ethyl ether solution in the presence of sodium methoxide, first at 5°, then at r.t. for 6 h (18%) [6880];
  - in dioxane in the presence of sodium hydride at 60° for 3.5 h (32%) [8067];
  - in aqueous potassium hydroxide [8076], (54%) [8060], (7%) [8057].
- Also refer to: [8065,8138,8208], [8077,8078,8082].

Isolation from natural sources

- From *Helichrysum gymnoconum* (Compositae) [8209].
- From *Helichrysum odoratissimum* and *Helichrysum cephaloideum* (compound **14**) [8210].
- From *Helichrysum infaustum* [8205].
- From *Helichrysum platypterum* DC [8080,8211,8212].
- From *Helichrysum stenopterum* [8080].

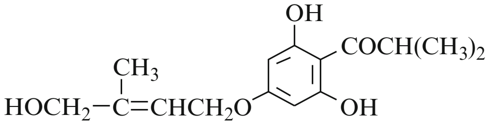
- From *Helichrysum flanaganii* Bolus [8212].
- From *Helichrysum asperum* (Thunb.) Hilliard et Burtt. var. *albidulum* (DC) Hilliard [8206].
- From *Helichrysum indicum* (L.) Grieson [8206].
- From *Helichrysum moeserianum* Thell. [8206].
- From *Helichrysum species* endemic to Madagascar [8213].
- From *Helichrysum kraussii* (Asteraceae) [8214].
- From *Helichrysum paronychioides* [8215].
- Formation from in vitro prenylation of phloroisobutyrophenone (SM) in *Humulus lupulus* (Cannabaceae). The first step, formation of SM from malonyl-CoA and isobutyryl-CoA, is catalyzed by valerophenone synthase (VPS) [8216].

m.p. 166° [8060], 165° [8067], 160° [6880], 159–160° [8057];
^{1}H NMR [8057,8060,8067,8076,8209,8214,8215,8217],
^{13}C NMR [8214], IR [8060,8209,8215], UV [8057,8215],
MS [8057,8076,8209,8214,8215];
TLC [8057,8214]; HPLC [8057,8216]; GC-MS [8214].
USE: Fungicide, [6880]; antioxidizing agent [8215].

BIOLOGICAL ACTIVITY: Antibacterial [8214].

1-[2,6-Dihydroxy-4-[(4-hydroxy-3-methyl-2-butenyl)oxy]phenyl]-2-methyl-1-propanone *(E)*

[122585-52-8]



$C_{15}H_{20}O_5$ mol.wt. 280.32

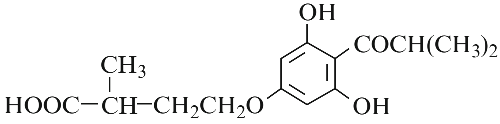
Isolation from natural sources

- From *Helichrysum asperum* (Thunb.) Hilliard et Burtt. var. *albidulum* (DC) Hilliard (compound **8**) [8206].

^{1}H NMR [8206], IR [8206], MS [8206].

4-[3,5-Dihydroxy-4-(2-methyl-1-oxopropyl)phenoxy]-2-methylbutanoic acid

[122585-57-3]



$C_{15}H_{20}O_6$ mol.wt. 296.32

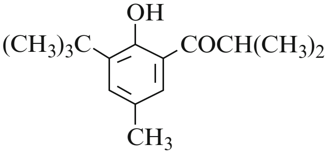
Isolation from natural sources

- From *Helichrysum asperum* (Thunb.) Hilliard et Burtt. var. *albidulum* (DC) Hilliard (compound **14**) [8206].

^{1}H NMR [8206], IR [8206], MS [8206].

1-[3-(1,1-Dimethylethyl)-2-hydroxy-5-methylphenyl]-2-methyl-1-propanone

[133903-10-3]



$C_{15}H_{22}O_2$ mol.wt. 234.34

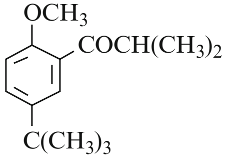
Synthesis

- The reaction of [AlMe(dbmp)$_2$] (Hdbmp = 2,6-di-tert-butyl-4-methylphenol) with O:C(Cl)Pr^i leads to acylation of one of the dbmp ligands and affords [AlMe(dbmp)-(bhmpp)] (SM). (Hbhmpp = 3-tert-butyl-2-hydroxy-5-methylisobutyrophenone). Hydrolysis of SM yields uncomplexed Hbhmpp, the titled ketone [7629].

^{1}H NMR [7629], ^{13}C NMR [7629], IR [7629]; X-ray crystallography [7629].

1-[5-(1,1-Dimethylethyl)-2-methoxyphenyl]-2-methyl-1-propanone

[75060-47-8]

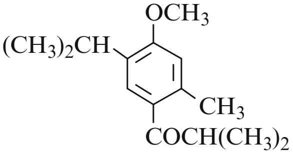


$C_{15}H_{22}O_2$ mol.wt. 234.34

Synthesis

- Refer to: [7106].

1-[4-Methoxy-2-methyl-5-(1-methylethyl)phenyl]-2-methyl-1-propanone



$C_{15}H_{22}O_2$ mol.wt. 234.34

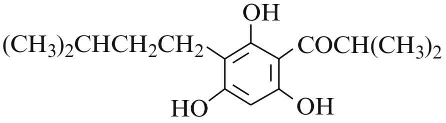
Syntheses

- Preparation by acylation of 2-isopropyl-5-methylanisole with isobutyryl chloride in the presence of aluminium chloride in carbon disulfide [7319].
- Also refer to: [7583].

b.p.$_{20}$ 178° [7319], b.p.$_{16}$ 186° [7583].

2-Methyl-1-[2,4,6-trihydroxy-3-(3-methylbutyl)phenyl]-1-propanone

[22628-86-0]



$C_{15}H_{22}O_4$ mol.wt. 266.34

Syntheses

- Obtained by reaction of isobutyryl chloride with isopentylphloroglucinol (m.p. 125–126.5°) in the presence of aluminium chloride in nitrobenzene for 4 days at 0° (17%) [8218].
- Preparation by hydrogenation of 2,4,6-trihydroxy-3-isopentenylisobutyrophenone in the presence of PtO$_2$ in methanol under an hydrogen atmosphere at r.t. for 1 h (87%) [6880].

- Also refer to: [8219,8220].

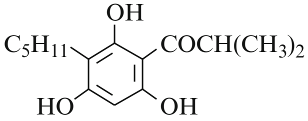
m.p. 184° [6880], 183–184° [8218];
^{1}H NMR [8218], UV [8218], MS [8220].

BIOLOGICAL ACTIVITY: Fungicide [6880].

2-Methyl-1-(2,4,6-trihydroxy-3-pentylphenyl)-1-propanone

[96756-26-2]

$C_{15}H_{22}O_4$ mol.wt. 266.34



Synthesis

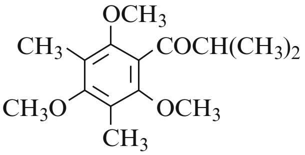
- Preparation by reaction of isobutyryl chloride with 2,4,6-trihydroxypentylbenzene in the presence of aluminium chloride in a nitrobenzene/carbon disulfide solution, first at r.t., then at 30–35° for 6 h (31%) [6880].

m.p. 164° [6880].
USE: Fungicide [6880].

2-Methyl-1-(2,4,6-trimethoxy-3,5-dimethylphenyl)-1-propanone
(Apodophyllone, Camfieldone)

[25677-09-2]

$C_{15}H_{22}O_4$ mol.wt. 266.34



Isolation from natural sources

- From the leaves of *Eucalyptus apodophylla* (Myrtaceae) [8221], *Eucalyptus miniata* and *Eucalyptus Chartaboma* [8200], *Xanthostemon eucalyptoides* [8196].
- From the leaf oil of *Xanthostemon crenulatus* (Myrtaceae) [8196].
- Also refer to: [8197,8222].

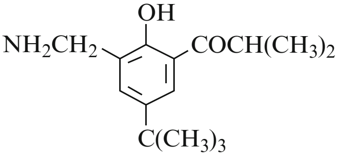
^{1}H NMR [8200,8221], ^{13}C NMR [8200,8221],
MS [8200,8221]; GC [8196,8221], GC-MS [8196,8221].

USE: Antiherbivore chem. of Eucalyptus involved cues and deterrents for marsupial folivores [8222].

1-[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-2-methyl-1-propanone

[75060-94-5]

$C_{15}H_{23}NO_2$ mol.wt. 249.35

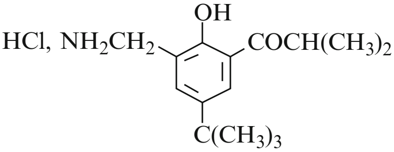


Synthesis

- Refer to: [7106].

1-[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-2-methyl-1-propanone *(Hydrochloride)*

[75060-72-9] $C_{15}H_{23}NO_2$, HCl mol.wt. 285.81



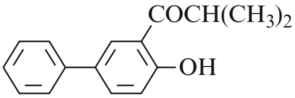
Synthesis

– Refer to: [7106].

BIOLOGICAL ACTIVITY: Inflammation inhibitor [7106].

1-(4-Hydroxy[1,1′-biphenyl]-3-yl)-2-methyl-1-propanone

[868266-16-4] $C_{16}H_{16}O_2$ mol.wt. 240.30

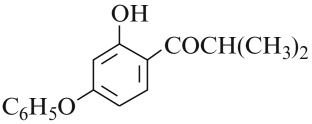


Synthesis

– Refer to: [8223].

1-(2-Hydroxy-4-phenoxyphenyl)-2-methyl-1-propanone

[307000-32-4] $C_{16}H_{16}O_3$ mol.wt. 256.30



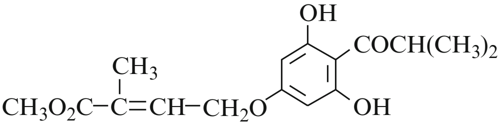
Synthesis

– Refer to: [7678].

USE: For inhibiting development of body odours in cosmetic compositions [7679–7683].

4-[3,5-Dihydroxy-4-(2-methyl-1-oxopropyl)phenoxy]-2-methyl-2-butenoic acid methyl ester

[122585-60-8] $C_{16}H_{20}O_6$ mol.wt. 308.33



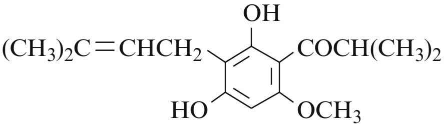
Isolation from natural sources

– From *Helichrysum asperum* (Thunb.)
Hilliard et Burtt. var. *albidulum* (DC)
Hilliard (Compositae) [8206].

^{1}H NMR [8206], IR [8206], MS [8206].

1-[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-methyl-1-propanone

[71539-57-6] $C_{16}H_{22}O_4$ mol.wt. 278.35



Synthesis

– Obtained by reaction of diazomethane with 3-isopentenyl-2,4,6-trihydroxy-isobutyrophenone [8067].

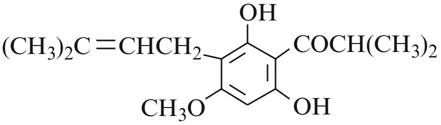
Isolation from natural sources

- From *Helichrysum gymnoconum* (Compositae) [8209].
- From *Helichrysum platypterum* [8080].

m.p. 122° [8067], 120–122° [8080];
^{1}H NMR [8080,8209], IR [8080,8209], MS [8080,8209].

1-[2,6-Dihydroxy-4-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-methyl-1-propanone

[103771-68-2]



$C_{16}H_{22}O_4$ mol.wt. 278.35

Isolation from natural sources

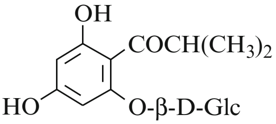
- From *Helichrysum cephaloideum* (compound **19**) [8080].

N.B.: This methyl ether has been reported previously (compound **3**) in mixture with the 2-methylbutyryl isomer (compound **7**) [8209], but were erroneously assigned as 6-O-methyl ethers.

^{1}H NMR [8209], IR [8209], MS [8209].

1-[2-(β-D-Glucopyranosyloxy)-4,6-dihydroxyphenyl]-2-methyl-1-propanone

[17004-75-0]



$C_{16}H_{22}O_9$ mol.wt. 358.35

Isolation from natural sources

- From Hops *Humulus lupulus* L. (Cannabinaceae) [8081,8224].

white powder [8081];

$(\alpha)_D^{20} = -55.6^\circ$

(methanol) [8081];

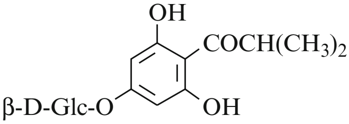
^{1}H NMR [8081], ^{13}C NMR [8081], IR [8081], MS [8081].

USE: Antioxidizing agent [8224]

BIOLOGICAL ACTIVITY: Antiallergic agent [8224].

1-[4-(β-D-Glucopyranosyloxy)-2,6-dihydroxyphenyl]-2-methyl-1-propanone

[868634-82-6]



$C_{16}H_{22}O_9$ mol.wt. 358.35

Isolation from natural sources

- From Hops *Humulus lupulus* L. (Cannabinaceae) [8081].

beige powder [8081];

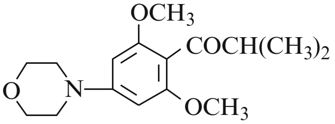
$(\alpha)_D^{20} = -17.4^\circ$

(methanol) [8081];

^{1}H NMR [8081], ^{13}C NMR [8081], IR [8081], MS [8081].

1-[2,6-Dimethoxy-4-(4-morpholinyl)phenyl]-2-methyl-1-propanone

[263010-97-5]

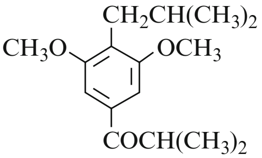


$C_{16}H_{23}NO_4$ mol.wt. 293.36

Syntheses

- Refer to: [8142,8143].

1-[3,5-Dimethoxy-4-(2-methylpropyl)phenyl]-2-methyl-1-propanone



$C_{16}H_{24}O_3$ mol.wt. 264.36

Syntheses

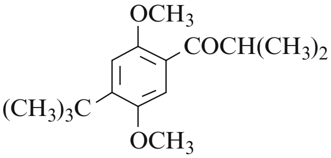
- Obtained (by-product) by reaction of isobutylmagnesium bromide on 3,4,5-trimethoxybenzonitrile [8167], (6%) [8181].

m.p. 183–184° [8181].

Semicarbazone $C_{17}H_{27}N_3O_3$ mol.wt. 321.42 (m.p. 184°) [8167].

1-[4-(1,1-Dimethylethyl)-2,5-dimethoxyphenyl]-2-methyl-1-propanone

[120350-21-2]



$C_{16}H_{24}O_3$ mol.wt. 264.36

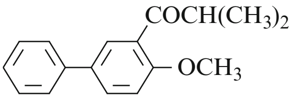
Synthesis

- Preparation by reaction of isobutyryl chloride with 2-tert-butylhydroquinone dimethyl ether in the presence of aluminium chloride in carbon disulfide [7336].

^{1}H NMR [7336], IR [7336], UV [7336], MS [7336].

1-(4-Methoxy[1,1′-biphenyl]-3-yl)-2-methyl-1-propanone

[868266-15-3]



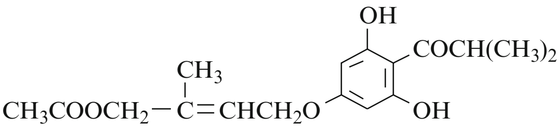
$C_{17}H_{18}O_2$ mol.wt. 254.33

Synthesis

- Refer to: [8223].

1-[4-[[4-(Acetyloxy)-3-methyl-2-butenyl]oxy]-2,6-dihydroxyphenyl]-2-methyl-1-propanone *(E)*

[122585-53-9]



$C_{17}H_{22}O_6$ mol.wt. 322.36

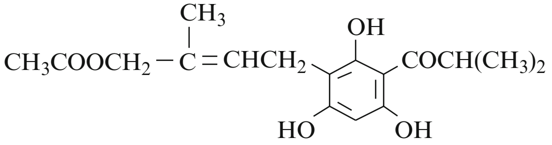
Isolation from natural sources

- From *Helichrysum asperum* (Thunb.) Hilliard et Burtt. var. *albidulum* (DC) Hilliard (compound **9**) [8206].

^{1}H NMR [8206].

1-[3-[4-(Acetyloxy)-3-methyl-2-butenyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-propanone *(E)*

[122585-62-0]



$C_{17}H_{22}O_6$ mol.wt. 322.36

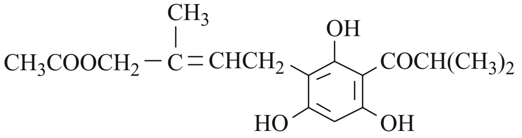
Isolation from natural sources

- From *Helichrysum asperum* (Thunb.) Hilliard et Burtt. (compound **24**) [8206].

^{1}H NMR [8206], IR [8206], MS [8206].

1-[3-[4-(Acetyloxy)-3-methyl-2-butenyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-propanone

[261928-44-3]



$C_{17}H_{22}O_6$ mol.wt. 322.36

Isolation from natural sources

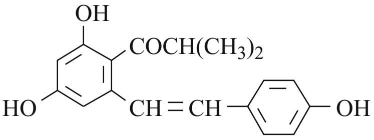
- From *Helichrysum caespititium* (Asteraceae) [8225].

BIOLOGICAL ACTIVITY: Antimicrobial properties [8225]; for treatment of tuberculosis and other infections [8226].

m.p. 140° [8225]; ^{1}H NMR [8225], ^{13}C NMR [8225], MS [8225].

1-[2,4-Dihydroxy-6-[(1*E*)-2-(4-hydroxyphenyl)ethenyl]phenyl]-2-methyl-1-propanone

[352276-35-8]



$C_{18}H_{18}O_4$ mol.wt. 298.34

Synthesis

- Refer to: [8227].

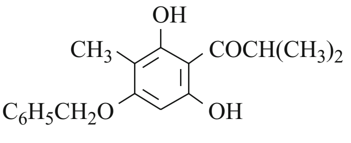
Isolation from natural sources

- From the root bark of *Ekebergia benguelensis* C. DC. (Meliaceae) (compound **4**) [8228].

pale yellow amorphous powder [8228]; m.p. 80–72° (such as in the paper) [8228];

^{1}H NMR [8228], ^{13}C NMR [8228], IR [8228], UV [8228], MS [8228].

1-[2,6-Dihydroxy-3-methyl-4-(phenylmethoxy)phenyl]-2-methyl-1-propanone



$C_{18}H_{20}O_4$ mol.wt. 300.35

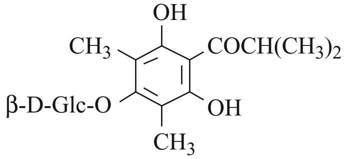
Synthesis

- Obtained by reaction of phenyldiazomethane with 3-methylphloroisobutyrophenone in ethyl ether, first at r.t. for 2 h, then at reflux for 30 min (15%) [7255].

b.p.$_{0.3}$ 150° [7255]; m.p. 135° [7255]; UV [7255].

1-[4-(β-D-Glucopyranosyloxy)-2,6-dihydroxy-3,5-dimethylphenyl]-2-methyl-1-propanone

[241131-36-2]



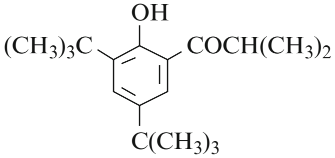
$C_{18}H_{26}O_9$ mol.wt. 386.40

Isolation from natural sources

- From *Hypericum japonicum* [8229].

1-[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]-2-methyl-1-propanone

[102520-04-7]



$C_{18}H_{28}O_2$ mol.wt. 276.42

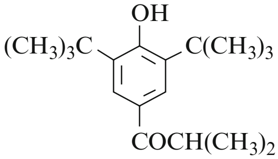
Synthesis

- Preparation by reaction of isopropylmagnesium chloride and 2,4-di-tert-butyl-6-cyanophenol in ethyl ether, first at 0° for 30 min, then at r.t. overnight (83%) [8230].

m.p. 33.5–34.5° [8230]; ^{1}H NMR [8230], IR [8230].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-2-methyl-1-propanone

[14035-36-0]



$C_{18}H_{28}O_2$ mol.wt. 276.42

Syntheses

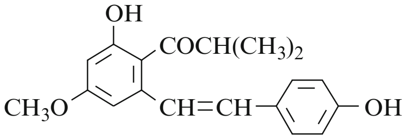
- Obtained in oxygenation of 2,4-dimethyl-3-[3,5-bis-(1,1-dimethylethyl)-4-hydroxyphenyl]-2-pentene (m.p. 66.2–67.0°) in the presence of Co(Salpr) in methylene chloride at 0° (20%) [8230].
- Preparation by acylation of 2,6-di-tert-butylphenol with isobutyric acid in trifluoroacetic anhydride at r.t. [8231], first at 0°, then at r.t. for 3 h (92%) [7727].
- Preparation by acylation of 2,6-di-tert-butylphenol with isobutyryl chloride in the presence of aluminium chloride [8232].
- Also refer to: [8233–8236].

m.p. 145–147° [7727,8231];
^{1}H NMR [7727,8230], IR [7727]; TLC [8230].
USE: Antioxidizing agent [8237].

O-methyloxime [81389-57-3] $C_{19}H_{31}NO_2$ mol.wt. 305.46 [7821].

1-[2-Hydroxy-6-[(1*E*)-2-(4-hydroxyphenyl)ethenyl]-4-methoxyphenyl]-2-methyl-1-propanone

[352276-34-7]



$C_{19}H_{20}O_4$ mol.wt. 312.37

Synthesis

- Refer to: [8227].

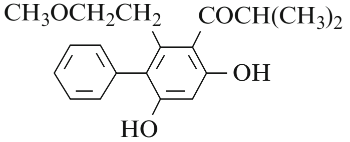
Isolation from natural sources

- From the root bark of *Ekebergia benguelensis* C. DC. (Meliaceae) (compound **3**) [8228].

reddish amorphous powder [8228]; m.p. 173–175° [8228];
^{1}H NMR [8228], ^{13}C NMR [8228], IR [8228], UV [8228], MS [8228].

1-[4,6-Dihydroxy-2-(2-methoxyethyl)[1,1′-biphenyl]-3-yl]-2-methyl-1-propanone

[860152-81-1] $C_{19}H_{22}O_4$ mol.wt. 314.38

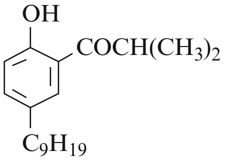


Synthesis

- Refer to: [7664].

1-(2-Hydroxy-5-nonylphenyl)-2-methyl-1-propanone

[219906-66-8] $C_{19}H_{30}O_2$ mol.wt. 290.45



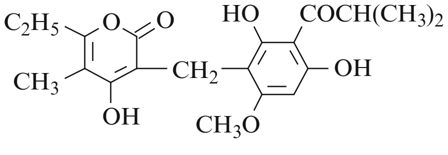
Synthesis

- Obtained in two steps: First, 4-nonylphenol was heated with aluminium isopropoxide in toluene/isopropanol with distillation of isopropanol and some toluene. Then, the mixture was cooled to 50° and treated with isobutyraldehyde over 50 min followed by stirring for 1 h [8238].

3-[[2,4-Dihydroxy-6-methoxy-3-(2-methyl-1-oxopropyl) phenyl]methyl]-6-ethyl-4-hydroxy-5-methyl-2*H*-pyran-2-one

(Nor-auricepyrone)

[103766-16-1] $C_{20}H_{24}O_7$ mol.wt. 376.41



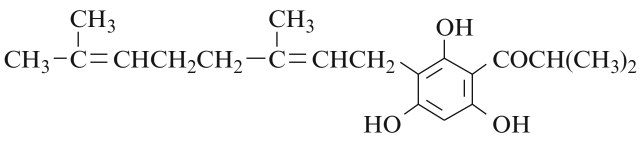
Isolation from natural sources

- From *Helichrysum cephaloideum* (Compositae) (compound **25**) [8080].

yellow oil [8080]; ^{1}H NMR [8080],
IR [8080], MS [8080]; TLC [8080].

1-[3-[(2*E*)-3,7-Dimethyl-2,6-octadienyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-propanone

(E) [72008-03-8] $C_{20}H_{28}O_4$ mol.wt. 332.44
(Z) [72008-05-0]



Synthesis

- Obtained by alkylation of phloroisobutyrophenone with geranyl bromide in dioxane in the presence of sodium hydride (28%) [8067].

Isolation from natural sources

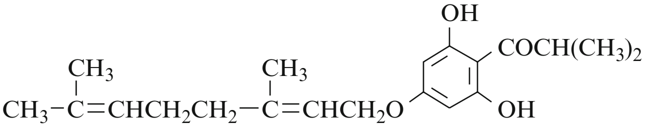
- From *Helichrysum infaustum* (Compositae) [8205].
- From *Helichrysum stenopterum* [8080].
- From *Helichrysum oxofilum* [8067].
- From *Helichrysum krookii* Moeser [8212].
- From *Helichrysum natalitium* DC [8211].
- From *Hypericum jovis* (Guttiferae) [6882,8239].
- From *Helichrysum monticola* Hilliard [8206].
- From *Esenbeckia nesiotica* (Rutaceae) [8240].
- From the aerial parts of *Achyrocline alata* (HBK) DC (tribe Inulae) [8241].
- From the leaves of *Hypericum styphelioide*s [8242].

USE: Antioxidizing agent [8239]; taxonomy in relation to [8241].

m.p. 128–130° [8240];
^{1}H NMR [8211,8240,8242], ^{13}C NMR [8211,8239,8240,8242],
IR [8240], UV [8240], MS [8211,8240].

1-[4-[[(2*E*)-3,7-Dimethyl-2,6-octadienyl]oxy]-2,6-dihydroxyphenyl]-2-methyl-1-propanone

[71539-65-6] $C_{20}H_{28}O_4$ mol.wt. 332.44



Isolation from natural sources

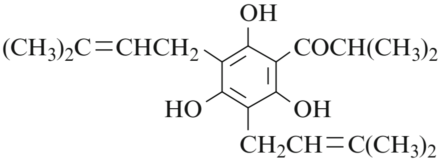
- From *Helichrysum gymnoconum* (Compositae) [8209].
- From *Hypericum jovis* (Guttiferae) [8239].
- From *Helichrysum anomalum* Less (compound **7a**) (Compositae) [8206].

USE: Antioxidizing agent [8239].

^{1}H NMR [8209], ^{13}C NMR [8239], IR [8209], MS [8209].

2-Methyl-1-[2,4,6-trihydroxy-3,5-bis(3-methyl-2-butenyl) phenyl]-1-propanone *(Deoxycohumulone, Desoxycohumulone, 4-Deoxycohumulone)*

[5880-42-2] $C_{20}H_{28}O_4$ mol.wt. 332.44



Syntheses

- Obtained by reaction of 2-methyl-3-buten-2-ol (3-hydroxy-3-methyl-1-butene) with phloroisobutyrophenone in the presence of zinc chloride [8058] or boron trifluoride etherate in dioxane [8064], at 20° for 8–9 h (18%) [8063], (11–14%) [8243].
- Also obtained by reaction of 1-chloro-3-methyl-2-butene with phloroisobutyrophenone in the presence of magnesium oxide and potassium iodide in refluxing acetone [8070] under nitrogen or argon [8071].
- Also obtained by reaction of 1-bromo-3-methyl-2-butene with phloroisobutyrophenone in the presence of a weakly basic resin DeAcidite 4-1P (OH form) in boiling benzene (14%) [7739].
- Also obtained by prenylation of 3-prenyl-2,4,6-trihydroxyisobutyrophenone [8208].
- Also obtained by phase transfer-catalyzed prenylation of 2,4,6-trihydroxyisobutyrophenone [7740].
- Also obtained by photolysis of colupulone [8243,8244] in isopropanol or methanol at 350 nm under nitrogen for 7 days (65%) [8245].
- Also refer to: [7739,8246,8247], [8077,8078,8082,8248,8249].

Isolation from natural sources

- In cohumulone formation in hop plant [8250].
- Formation from isobutyric acid and isovaleric acid in hop [8251].
- Formation from in vitro prenylation of phloroisobutyrophenone (SM) in *Humulus lupulus* (Cannabaceae). The first step, formation of SM from malonyl-CoA and isobutyryl-CoA, is catalyzed by valerophenone synthase (VPS) [8216].

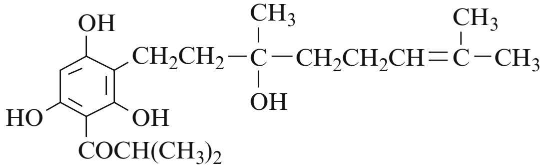
m.p. 89° [7739], 88–90° [8063,8243], 88–89° [8245,8249];
^{1}H NMR [8245], IR [8245], UV [8243,8245], MS [8245,8247];
GLC [8247]; HPLC [8216,8252,8253].
Note: Humulone formation [8244].

BIOLOGICAL ACTIVITY: Antibiotic [8086].

2-Methyl-1-[2,4,6-trihydroxy-3-(3-hydroxy-3,7-dimethyl-6-octenyl) phenyl]-1-propanone *(Hyperjovinol A)*

[722457-94-5] (+)

$C_{20}H_{30}O_5$ mol.wt. 350.46



Isolation from natural sources

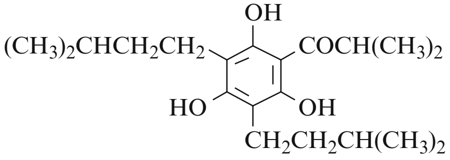
- From *Hypericum jovis* (Guttiferae) [6882,8239].

yellow amorphous solid [8239];
$(\alpha)_D = +5^\circ$ c = 0.1 in chloroform) [8239];
^{1}H NMR [8239], ^{13}C NMR [8239], IR [8239], UV [8239], MS [8239].

USE: Antioxidizing agent [6882,8239].

2-Methyl-1-[2,4,6-trihydroxy-3,5-bis(3-methylbutyl)phenyl]-1-propanone

[33759-61-4] $C_{20}H_{32}O_4$ mol.wt. 336.47



Syntheses

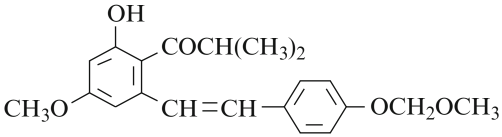
- Obtained by hydrogenation of colupulone in methanol in the presence of $PdCl_2$ (87%) [8254].
- Also refer to: [8086,8255].

clear gum unstable in air [8254]; ^1H NMR [8254], IR [8254], UV [8254].

BIOLOGICAL ACTIVITY: Antibiotic [8086].

1-[2-Hydroxy-4-methoxy-6-[(1*E*)-2-[4-(methoxymethoxy)phenyl]ethenyl]phenyl]-2-methyl-1-propanone

[852612-18-1] $C_{21}H_{24}O_5$ mol.wt. 356.42

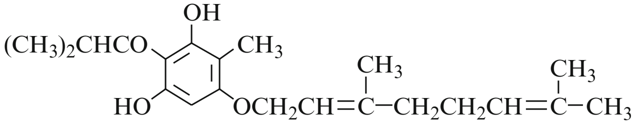


Synthesis

- Refer to: [8227].

1-[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2,6-dihydroxy-3-methylphenyl]-2-methyl-1-propanone *(Otogirin)*

[137251-97-9] $C_{21}H_{30}O_4$ mol.wt. 346.47



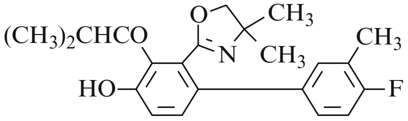
Isolation from natural sources

- From *Hypericum erectum* (Hypericaceae) [8256], (Guttiferae) [8088].
  BIOLOGICAL ACTIVITY: Antibiotic [8088,8256] and antiallergenic [8256].

m.p. 66–68° [8256];
^1H NMR [8256], ^{13}C NMR [8256], IR [8256], UV [8256], HRMS [8256].

1-[2-(4,5-Dihydro-4,4-dimethyl-2-oxazolyl)-4′-fluoro-4-hydroxy-3′-methyl[1,1′-biphenyl]-3-yl]-2-methyl-1-propanone

[136553-42-9] $C_{22}H_{24}FNO_3$ mol.wt. 369.44



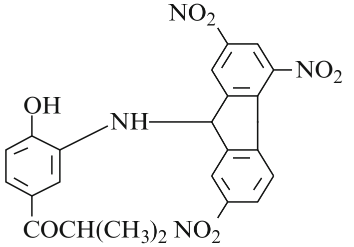
Synthesis

- Refer to: [8257].

USE: In preparation of HMG-CoA reductase inhibitors [8257].

1-[4-Hydroxy-3-[(2,4,7-trinitro-9*H*-fluorene-9-ylidene)amino]phenyl]-2-methyl-1-propanone

[161319-29-5] $C_{23}H_{18}N_4O_8$ mol.wt. 478.42

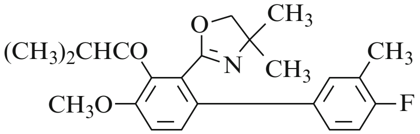


Synthesis

– Refer to: [8258].

1-[2-(4,5-Dihydro-4,4-dimethyl-2-oxazolyl)-4′-fluoro-4-methoxy-3′-methyl [1,1′-biphenyl]-3-yl]-2-methyl-1-propanone

[136577-47-4] $C_{23}H_{26}FNO_3$ mol.wt. 383.46



Synthesis

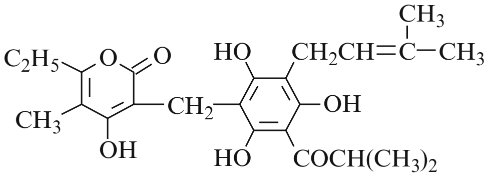
– Refer to: [8257].

USE: In preparation of HMG-CoA reductase inhibitors [8257].

6-Ethyl-4-hydroxy-5-methyl-3-[[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)-5-(2-methyl-1-oxopropyl)phenyl]methyl]-2*H*-pyran-2-one

(6-O-Desmethylauricepyron)

[75680-08-9] $C_{24}H_{30}O_7$ mol.wt. 430.50



Isolation from natural sources

– From the roots of *Helichrysum mixtum* (Compositae) (compound **21**) [8080].

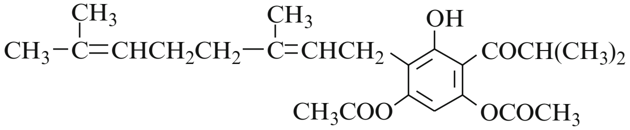
– From the aerial parts of *Helichrysum stenopterum* (Compositae) (compound **21**) [8080].

– From *Helichrysum odoratissimum* and *Helichrysum cephaloideum* (compound **1**) [8210].

^{1}H NMR [8210], IR [8210], MS [8210].

1-[4,6-Bis(acetyloxy)-3-(3,7-dimethyl-2,6-octadienyl)-2-hydroxyphenyl]-2-methyl-1-propanone *(E)*

[144785-83-1] $C_{24}H_{32}O_6$ mol.wt. 416.51

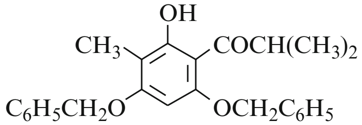


Synthesis

- Obtained by partial acylation of the corresponding substituted phloroglucinol with acetic anhydride in the presence of pyridine [8240].

oil [8240]; ¹H NMR [8240], IR [8240], MS [8240].

1-[2-Hydroxy-3-methyl-4,6-bis(phenylmethoxy)phenyl]-2-methyl-1-propanone



$C_{25}H_{26}O_4$ mol.wt. 390.48

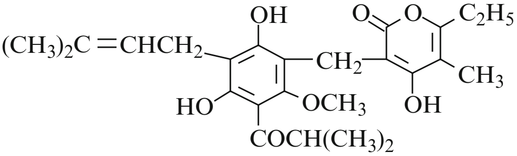
Synthesis

- Obtained by partial benzylation of 3-methylphloroisobutyrophenone with benzyl bromide in the presence of potassium carbonate in refluxing acetone (13%) [7255], according to the method [8259,8260].

oil [7255]; b.p.$_{0.2}$ 150–160° [7255].

3-[[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)-5-(2-methyl-1-oxopropyl) phenyl]-methyl]-6-ethyl-4-hydroxy-5-methyl-2*H*-pyran-2-one *(Auricepyron)*

[74048-81-0]



$C_{25}H_{32}O_7$ mol.wt. 444.52

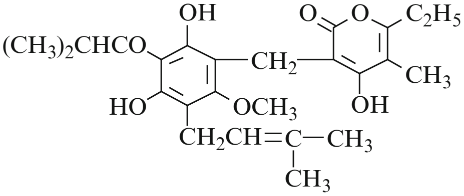
Isolation from natural sources

- From the roots of *Helichrysum auriceps* and *Helichrysum cephaloideum* (Compositae) [8210,8261].

m.p. 115° [8261]; ¹H NMR [8210,8261], IR [8261], MS [8261].

3-[[2,4-Dihydroxy-6-methoxy-5-(3-methyl-2-butenyl)-3-(2-methyl-1-oxopropyl) phenyl]-methyl]-6-ethyl-4-hydroxy-5-methyl-2*H*-pyran-2-one

[103771-70-6]



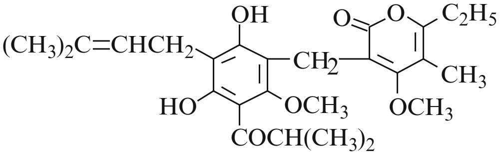
$C_{25}H_{32}O_7$ mol.wt. 444.52

Isolation from natural sources

- From the roots of *Helichrysum cephaloideum* (Compositae) (compound **23**) [8080].

3-[[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)-5-(2-methyl-1-oxopropyl)phenyl]-methyl]-6-ethyl-4-methoxy-5-methyl-2*H*-pyran-2-one

[74948-75-2]



$C_{26}H_{34}O_7$ mol.wt. 458.55

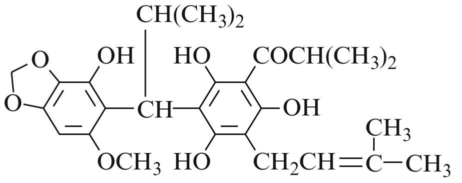
Synthesis

- Obtained by reaction of diazomethane with *Auricepyron* in ethyl ether at r.t. for 1 h [8261].

oil [8261];
¹H NMR [8261], ¹³C NMR [8261]; TLC [8261].

2-Methyl-1-[2,4,6-trihydroxy-3-[1-(4-hydroxy-6-methoxy-1,3-benzodioxol-5-yl)-2-methylpropyl]-5-(3-methyl-2-butenyl)phenyl]-1-propanone (S) *(Helinudifolin)*

[103766-15-0] $C_{27}H_{34}O_8$ mol.wt. 486.56



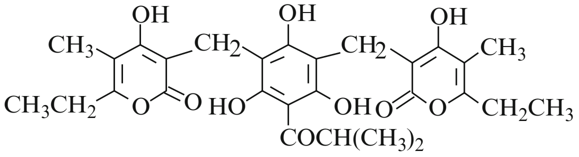
Isolation from natural sources

- From the roots of *Helichrysum nudifolium* (Compositae) (compound **27**) [8080].

colourless oil [8080];
^{1}H NMR [8080], IR [8080], MS [8080];
TLC [8080].

3,3′-[[2,4,6-Trihydroxy-5-(2-methyl-1-oxopropyl)-1,3-phenylene]-bis(methylene)] bis-[6-ethyl-4-hydroxy-5-methyl-2*H*-pyran-2-one *(23-Methylitalidipyron)*

[75680-22-7] $C_{28}H_{32}O_{10}$ mol.wt. 528.56



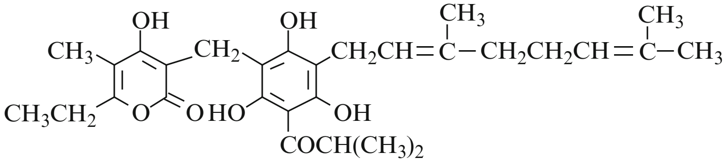
Isolation from natural sources

- From *Helichrysum italicum* (Compositae) [8210].

m.p. 178–182° [8210];
^{1}H NMR [8210], IR [8210], UV [8210], MS [8210].

3-[[3-(3,7-Dimethyl-2,6-octadienyl)-2,4,6-trihydroxy-5-(2-methyl-1-oxopropyl) phenyl]-methyl]-6-ethyl-4-hydroxy-5-methyl-2*H*-pyran-2-one *(E) (Achyroclinopyrone)*

[77820-39-4] $C_{29}H_{38}O_7$ mol.wt. 498.62



Isolation from natural sources

- From the aerial parts of *Achyrocline alata* (HBK) DC, but only isolated as its tetraacetate by treatment of the crude phloroglucinol derivative with acetic anhydride for 1 h at 70° [8241].

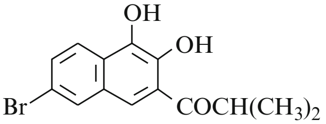
Tetraacetate $C_{37}H_{46}O_{11}$ mol.wt. 666.30 [8241].

colourless gum [8241]; ^{1}H NMR [8241], IR [8241], MS [8241].

24.2 Naphthalene Derivatives

1-(7-Bromo-3,4-dihydroxy-2-naphthalenyl)-2-methyl-1-propanone

[61983-31-1] $C_{14}H_{13}BrO_3$ mol.wt. 309.16



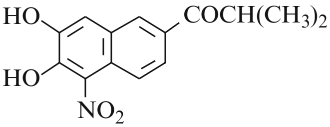
Synthesis

– Obtained by irradiation of 6-bromo-1,2-naphthoquinone and isobutyraldehyde in benzene by a 300 W high-pressure mercury arc lamp between 15° and 20° for 2–10 days (15%) [7762].

m.p. 116.5–118° [7762]; ^{1}H NMR [7762], IR [7762].

1-(6,7-Dihydroxy-5-nitro-2-naphthalenyl)-2-methyl-1-propanone

[404964-71-2] $C_{14}H_{13}NO_5$ mol.wt. 275.26

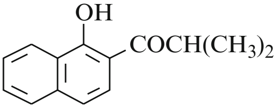


Synthesis

– Refer to: [8262].

1-(1-Hydroxy-2-naphthalenyl)-2-methyl-1-propanone

[79387-88-5] $C_{14}H_{14}O_2$ mol.wt. 214.26



Syntheses

– Obtained by Fries rearrangement of 1-naphthyl isobutyrate [8104],

- with aluminium chloride (53%) [7768], at 100–120° for 3 h (60%) [8263];
- with zinc chloride or stannic chloride (75%) [7768];
- with yttrium triflate-methane sulfonic acid in toluene at 100° for 5 h under argon [8264], (87%) [8265].

– Preparation by reaction of isobutyric acid with α-naphthol,

- in the presence of boron trifluoride at 70° for 2 h (95%) [7775];
- in the presence of zinc chloride at 170° (good yield) [6376];
- in the presence of P_2O_5/SiO_2 at 130° for 24 h (74%) [8108].

– The 4-isobutyryl-1-naphthol was quantitatively rearranged to the 2-isomer by refluxing with 35% sodium hydroxide solution for 2 h [7768].

– Also obtained by irradiation of 2-isobutyryl-3,4-dihydronaphthalen-1(2*H*)-one (b.p.$_{0.3}$ 110°) in various solvents (hexane, acetonitrile or methanol) under bubbling air in 10–30% yield depending on the solvent used [8266].

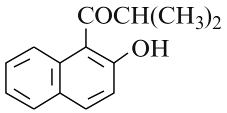
– Also obtained by dye-sensitized photooxidation of enolic tautomer of 1-(3,4-dihydro-1-hydroxy-2-naphthalenyl)-2-methyl-1-propanone [128838-13-1] (26–29%) [7780].

– Also refer to: [7784,7785].

m.p. 89–90° [8108], 87–88° [8266], 87° [7775], 79° [6376];
^{1}H NMR [8108,8266], IR [8108,8266], UV [8266].

1-(2-Hydroxy-1-naphthalenyl)-2-methyl-1-propanone

[95455-11-1] $C_{14}H_{14}O_2$ mol.wt. 214.26



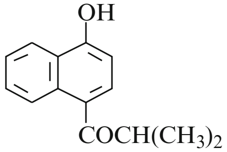
Syntheses

- Preparation by Fries rearrangement of 2-naphthyl isobutyrate in the presence of aluminium chloride [7992].
- Also obtained (poor yield) by treatment of 2-methoxy-naphthalene with isobutyryl chloride and aluminium chloride in refluxing carbon disulfide for 2 h (2%) [7794].

pure oil [7794]; b.p.$_3$ 164–166° [7992]; ^{13}C NMR [7794].

1-(4-Hydroxy-1-naphthalenyl)-2-methyl-1-propanone

[761459-41-0] $C_{14}H_{14}O_2$ mol.wt. 214.26

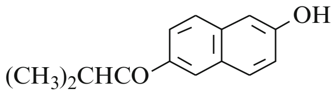


Syntheses

- Obtained by Fries rearrangement of α-naphthyl isobutyrate with aluminium chloride (34%) [7768].
- Also obtained by reaction of isobutyric acid with α-naphthol in the presence of P_2O_5/SiO_2 at 130° for 24 h (20%) [8108].

1-(6-Hydroxy-2-naphthalenyl)-2-methyl-1-propanone

[100886-06-4] $C_{14}H_{14}O_2$ mol.wt. 214.26

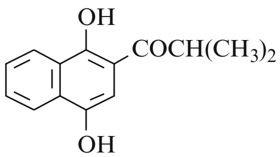


Syntheses

- Preparation by esterification of β-naphthol with isobutyryl fluoride in the presence of hydrogen fluoride at 0–40°, followed by Fries rearrangement of ester formed in situ [7802] at 60–100° [8267].

1-(1,4-Dihydroxy-2-naphthalenyl)-2-methyl-1-propanone

[128462-67-9] $C_{14}H_{14}O_3$ mol.wt. 230.26

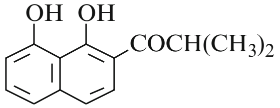


Synthesis

- Obtained by demethylation of the corresponding 4-methyl ether with concentrated hydrobromic acid in acetic acid [7813].

1-(1,8-Dihydroxy-2-naphthalenyl)-2-methyl-1-propanone

$C_{14}H_{14}O_3$ mol.wt. 230.26



Syntheses

- Obtained by reaction of isobutyric acid with 1,8-dihydroxy-naphthalene in the presence of zinc chloride at 145–150° [7818].

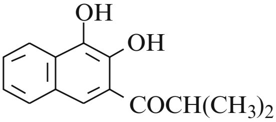
– Also obtained by reaction of zinc chloride with 1,8-dihydroxynaphthalene diisobutyrate in nitrobenzene at 140–150° [7819].

m.p. 88° [7818].

Diacetate $C_{18}H_{18}O_5$ mol.wt. 314.34 (m.p. 105–106°) [7818].

1-(3,4-Dihydroxy-2-naphthalenyl)-2-methyl-1-propanone

[61983-12-8] $C_{14}H_{14}O_3$ mol.wt. 230.26



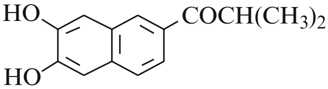
Syntheses

– Obtained by irradiation of 1,2-naphthoquinone and isobutyraldehyde in benzene by a 300 W high-pressure mercury arc lamp between 15° and 20° for 2–10 days [7761], (5%) [7762].

m.p. 105–107° [7762]; ^{1}H NMR [7762], IR [7762].

1-(6,7-Dihydroxy-2-naphthalenyl)-2-methyl-1-propanone

[404964-94-9] $C_{14}H_{14}O_3$ mol.wt. 230.26

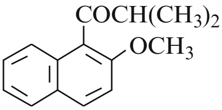


Synthesis

– Refer to: [8262].

1-(2-Methoxy-1-naphthalenyl)-2-methyl-1-propanone

$C_{15}H_{16}O_2$ mol.wt. 228.29



Syntheses

– Preparation by reaction of dimethyl sulfate with the sodium salt of 1-(2-Hydroxy-1-naphthalenyl)-2-methyl-1-propanone [7992].

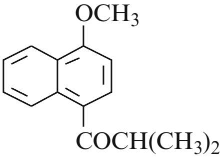
– Also obtained by treatment of 2-methoxynaphthalene,

- with isobutyric anhydride and aluminium chloride in refluxing carbon disulfide (20%) [7794];
- with isobutyryl chloride and aluminium chloride in refluxing carbon disulfide for 5 h (2%) [7794].

yellow oil [7992]; b.p.$_3$ 176–178° [7992].

1-(4-Methoxy-1-naphthalenyl)-2-methyl-1-propanone

[36198-81-9] $C_{15}H_{16}O_2$ mol.wt. 228.29



Syntheses

– Preparation by reaction of isobutyric acid with 1-methoxy-naphthalene in the presence of boron trifluoride, first at 0°, then at 70° for 10–15 h (93%) [7845].

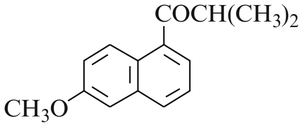
– Also obtained by Fries rearrangement of α-naphthyl isobutyrate with aluminium chloride (34%) [7768].

- Also obtained by reaction of isobutyryl chloride with 1-methoxynaphthalene in the presence of aluminium chloride in carbon disulfide (72%) [7846,7849] according to the procedure [8268].

m.p. 100–101° [7849], 97–97.5° [7846], 96° [7845];
^{1}H NMR [7849].

1-(6-Methoxy-1-naphthalenyl)-2-methyl-1-propanone

[69769-76-2] $C_{15}H_{16}O_2$ mol.wt. 228.29



Synthesis

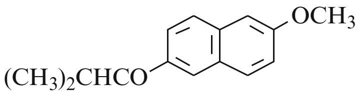
- Obtained from sodium-liquid ammonia reduction of 4,6-dimethoxy-1-naphthyl isopropyl ketone in THF/EtOH mixture in presence of excess ammonium chloride [7851].

b.p.$_{0.1}$ 140° [7851]; ^{1}H NMR [7851], IR [7851].

1-(6-Methoxy-2-naphthalenyl)-2-methyl-1-propanone

6-isobutyrylnerolin

[179930-43-9] $C_{15}H_{16}O_2$ mol.wt. 228.29



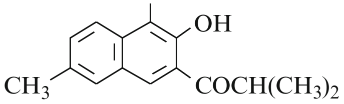
Syntheses

- Preparation by action of potassium tert-butoxide and methyl iodide with 6-propionylneroline (methylation) (74%) [7855].
- Also obtained by condensation of isobutyryl chloride with neroline in the presence of aluminium chloride in nitrobenzene at r.t. for 24 h [7855,8269], according to [7854].
- Also obtained by hydrolysis of 2-[1-(N-isobutyrylimino)-1-isopropyl]-6-methoxynaphthalene ($C_{19}H_{23}NO_2$, m.p. 141.5–142°) [8269].
- Also refer to: [8270,8271].

b.p.$_{15}$ 213–214° [7855], b.p.$_{14}$ 220° [8269];
m.p. 57–58° [7855,8269].

1-(3,4-Dihydroxy-7-methyl-2-naphthalenyl)-2-methyl-1-propanone

[61983-40-2] $C_{15}H_{16}O_3$ mol.wt. 244.29



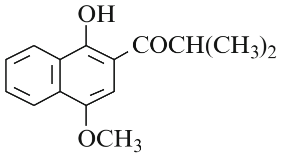
Synthesis

- Obtained by irradiation of 6-methyl-1,2-naphthoquinone and isobutyraldehyde in benzene by a 300 W high-pressure mercury arc lamp between 15° and 20° for 2–10 days (8%) [7762].

m.p. 155–156° [7762]; ^{1}H NMR [7762], IR [7762].

1-(1-Hydroxy-4-methoxy-2-naphthalenyl)-2-methyl-1-propanone

[128462-65-7] $C_{15}H_{16}O_3$ mol.wt. 244.29

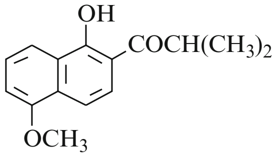


Synthesis

- Obtained by reaction of 2-isobutyryl-1,3-indandione with diazomethane in ethyl ether (66–72%) [7813].

1-(1-Hydroxy-5-methoxy-2-naphthalenyl)-2-methyl-1-propanone

[92920-81-5] $C_{15}H_{16}O_3$ mol.wt. 244.29



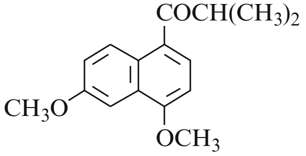
Synthesis

- Obtained by photo-Fries rearrangement of 5-methoxy-1-naphthyl isobutyrate (m.p. 57–58°) in methanol [7864].

m.p. 92–93° [7864]; IR [7864], MS [7864].

1-(4,6-Dimethoxy-1-naphthalenyl)-2-methyl-1-propanone

[69769-73-9] $C_{16}H_{18}O_3$ mol.wt. 258.32



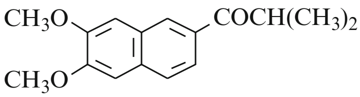
Synthesis

- Preparation by reaction of isobutyryl chloride with 1,7-dimethoxynaphthalene in the presence of aluminium chloride in nitrobenzene at r.t. for 16 h (81%) [7851].

b.p.$_{0.1}$ 180° [7851];
m.p. 73–74° [7851]; ^{1}H NMR [7851], IR [7851].

1-(6,7-Dimethoxy-2-naphthalenyl)-2-methyl-1-propanone

[337522-30-2] $C_{16}H_{18}O_3$ mol.wt. 258.32



Syntheses

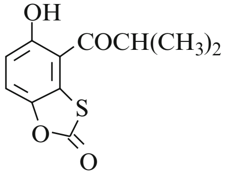
- Preparation by Friedel–Crafts acylation of 2,3-di-methoxynaphthalene with isobutyryl chloride in the presence of aluminium chloride in methylene chloride (71%) [7872].
- Also refer to: [8272] (Japanese patent).

m.p. 107° [7872]; ^{1}H NMR [7872], IR [7872].

24.3 Heterocyclic Derivatives

5-Hydroxy-4-(2-methyl-1-oxopropyl)-1,3-benzoxathiol-2-one

[112450-18-7] $C_{11}H_{10}O_4S$ mol.wt. 238.26



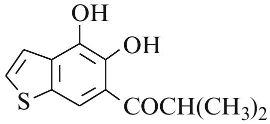
Synthesis

- Preparation by adding a solution of 2-isobutyryl-1,4-benzo-quinone in acetic acid to a solution of thiourea in 2 N hydrochloric acid, stirring at r.t. for 30 min, then heating on a steam bath for 1 h (61%) [7883].

m.p. 182–184° [7883];
^{1}H NMR [7883], ^{13}C NMR [7883].

1-(4,5-Dihydroxybenzo[*b*]thien-6-yl)-2-methyl-1-propanone

[912952-37-5] $C_{12}H_{12}O_3S$ mol.wt. 263.29



Synthesis

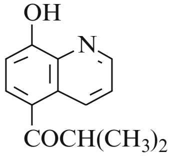
- Obtained by photoacylation of benzo[*b*]thiophene-4,5-dione with isobutyraldehyde in benzene (33%) [7916].

m.p. 117–120° [7916];
^{1}H NMR [7916], IR [7916].

8-Hydroxy-5-(2-methyl-1-oxopropyl)quinoline

1-(8-Hydroxy-[5]quinolyl)-2-methylpropan-1-one

[101724-90-7] $C_{13}H_{13}NO_2$ mol.wt. 215.25



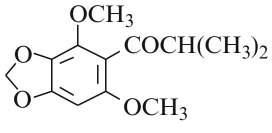
Synthesis

- Obtained by reaction of isobutyryl chloride with 8-quinolinol (8-hydroxyquinoline) in nitrobenzene in the presence of aluminium chloride [7946].

m.p. 75–76° [7946].
Note: Fungicide [7946].

1-(4,6-Dimethoxy-1,3-benzodioxol-5-yl)-2-methyl-1-propanone

[73213-22-6] $C_{13}H_{16}O_5$ mol.wt. 252.27



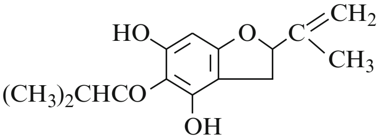
Isolation from natural sources

- From *Helichrysum chrysargyrum* (Compositae) [8273].

Colourless oil [8273];
^{1}H NMR [8273], IR [8273], MS [8273].

1-[2,3-Dihydro-4,6-dihydroxy-2-(1-methylethenyl)-5-benzofuranyl]-2-methyl-1-propanone *(4,6-Dihydroxy-9,9-dimethyltremeton)*

[103771-74-0]
[96552-58-8] (*R*)



$C_{15}H_{18}O_4$ mol.wt. 262.31

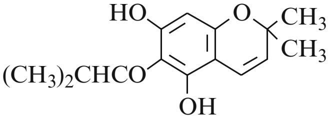
Isolation from natural sources

- From *Helichrysum* [8079].
- From *Helichrysum cephaloideum* and *Helichrysum mixtum* [8080].

colourless oil [8079];
$(\alpha)_D = -8.7^\circ$ (c = 0.9 in chloroform) [8079];
^{1}H NMR [8079,8217] IR [8079], MS [8079].

1-(5,7-Dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-2-methyl-1-propanone

[72935-07-0]



$C_{15}H_{18}O_4$ mol.wt. 262.31

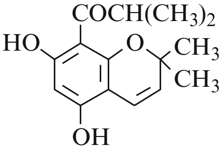
Synthesis

- Obtained (poor yield) by reaction of DDQ with 2,4,6-tri-hydroxy-3-(3-methyl-2-butenyl)isobutyrophenone in refluxing benzene for 20 min (3%) [8138].

m.p. 120–121° [8138]; ^{1}H NMR [8138], IR [8138], UV [8138].

1-(5,7-Dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl)-2-methyl-1-propanone

[72935-08-1]



$C_{15}H_{18}O_4$ mol.wt. 262.31

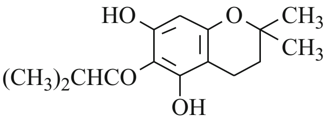
Synthesis

- Obtained (poor yield) by reaction of DDQ with 2,4,6-tri-hydroxy-3-(3-methyl-2-butenyl)isobutyrophenone in refluxing benzene for 20 min (8%) [8138].

m.p. 102–103° [8138];
^{1}H NMR [8138], IR [8138], UV [8138].

1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-2-methyl-1-propanone

[35932-38-8]



$C_{15}H_{20}O_4$ mol.wt. 264.32

Syntheses

- Obtained by treatment of 3-isoprenylphloroisobutyrophenone with p-toluenesulfonic acid in refluxing benzene [8060, 8207].
- Also refer to: [8138,8209].

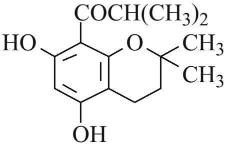
Isolation from natural sources

- From *Helichrysum platypterum* DC (Compositae) [8080].

m.p. 142–143° [8060], 142° [8080,8207];
^{1}H NMR [8060,8080,8207,8209], IR [8060,8080],
UV [8060,8207], MS [8080]; TLC [8080].

1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl)-2-methyl-1-propanone

[35932-37-7] $C_{15}H_{20}O_4$ mol.wt. 264.32



Syntheses

- Obtained by hydrolysis of 8-isobutyryl-7-hydroxy-5-methoxymethyleneoxy-2,2-dimethylchroman in refluxing methanol containing 1 drop of concentrated sulfuric acid for 3 h (90%) [8138].
- Also obtained by treatment of 3-isoprenylphloro-isobutyrophenone with p-toluenesulfonic acid in refluxing benzene [8207], (40%) [8060].
- Also obtained from a mixture of phloroisobutyrophenone, 2-methyl-3-buten-2-ol and Amberlite IR 120 resin (H^+ form) in refluxing dioxane for 24 h (5%) [7739].
- Also obtained by treatment of 3-(dimethylallyl)phloroisobutyrophenone in benzene in the presence of 10% sulfuric acid [8209].

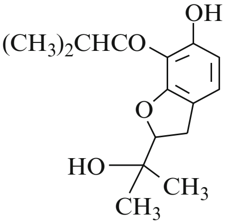
Isolation from natural sources

- From *Helichrysum platypterum* DC (Compositae) [8080].

m.p. 147° [7739], 145–146° [8060], 145° [8207], 144° [8080];
^{1}H NMR [8060,8080,8207,8209], ^{13}C NMR [8080],
IR [8060,8080,8209], UV [8060,8207], MS [8080,8209].

1-[2,3-Dihydro-6-hydroxy-2-(1-hydroxy-1-methylethyl)-7-benzofuranyl]-2-methyl-1-propanone *(Lupulone F)*

[842121-75-9] $C_{15}H_{20}O_4$ mol.wt. 264.32



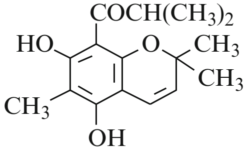
Isolation from natural sources

- From *Humulus lupulus* L. (Cannabinaceae) (compound **13**) [8274].

yellow oil [8274]; ^{1}H NMR [8274],
^{13}C NMR [8274], IR [8274], UV [8274],
MS [8274].

1-(5,7-Dihydroxy-2,2,6-trimethyl-2*H*-1-benzopyran-8-yl)-2-methyl-1-propanone

[111983-96-1] $C_{16}H_{20}O_4$ mol.wt. 276.33



Isolation from natural sources

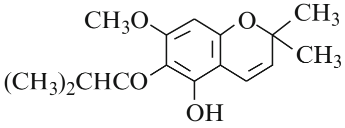
- From *Hypericum revolutum* Vahl (Guttiferae) [8275–8278].

m.p. 79–81° [8276];
^{1}H NMR [8276], ^{13}C NMR [8276], IR [8276],
UV [8276], MS [8276]; TLC [8276]; HPLC [8275,8277].
USE: Fungicide [8276].

BIOLOGICAL ACTIVITY: [8278].

1-(5-Hydroxy-7-methoxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-2-methyl-1-propanone

[365947-78-0] $C_{16}H_{20}O_4$ mol.wt. 276.33



Isolation from natural sources

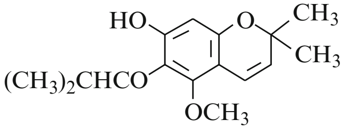
- From *Hypericum polyanthemum* [8279, 8280] Klotzsch ex Reichardt (Guttiferae) [8281].

viscous oil [8281];
^{1}H NMR [8281], ^{13}C NMR [8281], IR [8281], UV [8281], MS [8281].

BIOLOGICAL ACTIVITY: MAO inhibitory on rat brain mitochondria [8280]; antimicrobial [8282].

1-(7-Hydroxy-5-methoxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-2-methyl-1-propanone

[365947-74-6] $C_{16}H_{20}O_4$ mol.wt. 276.33



Isolation from natural sources

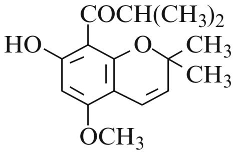
- From *Hypericum polyanthemum* [8279, 8280] Klotzsch ex Reichardt (Guttiferae) [8281].

viscous oil [8281];
^{1}H NMR [8281], ^{13}C NMR [8281], IR [8281], UV [8281], MS [8281].

BIOLOGICAL ACTIVITY: MAO inhibitory on rat brain mitochondria [8280]; antimicrobial [8282].

1-(7-Hydroxy-5-methoxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl)-2-methyl-1-propanone

[72934-97-5] $C_{16}H_{20}O_4$ mol.wt. 276.33



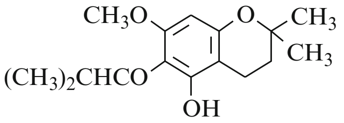
Synthesis

- Obtained by reaction of 3-chloro-3-methyl-1-butyne with 2,6-dihydroxy-4-methoxyisobutyrophenone in the presence of potassium carbonate and potassium iodide in refluxing acetone for 48 h (23%) [8138].

m.p. 67–68° [8138];
^{1}H NMR [8138], IR [8138], MS [8138].

1-(3,4-Dihydro-5-hydroxy-7-methoxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-2-methyl-1-propanone

[71539-69-0] $C_{16}H_{22}O_4$ mol.wt. 278.35



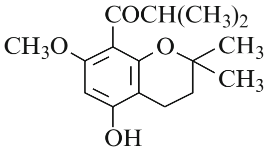
Isolation from natural sources

- From *Helichrysum gymnoconum* (Compositae) [8209].
- From *Helichrysum platypterum* DC [8080].

yellow oil [8080];
^{1}H NMR [8080,8209], IR [8080,8209], MS [8080,8209]; TLC [8080].

1-(3,4-Dihydro-5-hydroxy-7-methoxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl)-2-methyl-1-propanone

$C_{16}H_{22}O_4$ mol.wt. 278.35



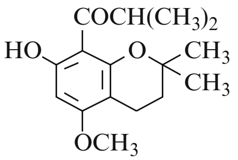
Isolation from natural sources

- From Helichrysum platypterum DC (Compositae) [8080].

yellow oil [8080];
^{1}H NMR [8080], IR [8080], MS [8080].

1-(3,4-Dihydro-7-hydroxy-5-methoxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl)-2-methyl-1-propanone

[72935-00-3] $C_{16}H_{22}O_4$ mol.wt. 278.35

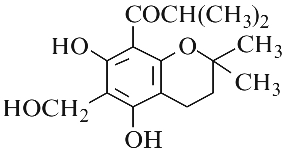


Synthesis

- Obtained by reaction of methyl iodide with 8-isobutyryl-5,7-dihydroxy-2,2-dimethylchroman in the presence of potassium carbonate in refluxing acetone for 1.5 h (66%) [8138].

m.p. 68–69° [8138]; ^{1}H NMR [8138], IR [8138].

1-[3,4-Dihydro-5,7-dihydroxy-6-(hydroxymethyl)-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone



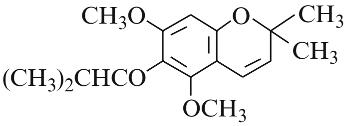
$C_{16}H_{22}O_5$ mol.wt. 294.35

Synthesis

- Refer to: [8060].

1-(5,7-Dimethoxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-2-methyl-1-propanone

[365947-69-9]



$C_{17}H_{22}O_4$ mol.wt. 290.36

Isolation from natural sources

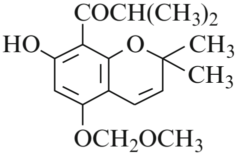
- From *Hypericum species* native to South Brazil (Guttiferae) [8280].
- From the aerial parts of *Hypericum polyanthemum* Klotzsch ex Reichardt [8279, 8281].

viscous oil [8281];
^{1}H NMR [8281], ^{13}C NMR [8281], IR [8281], UV [8281], MS [8281];
TLC [8280,8281]; HPLC [8280].

BIOLOGICAL ACTIVITY: MAO inhibitory on rat brain mitochondria [8280]; antitumor [8279]; antimicrobial [8282].

1-[7-Hydroxy-5-(methoxymethoxy)-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone

[72935-05-8]



$C_{17}H_{22}O_5$ mol.wt. 306.36

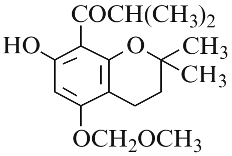
Synthesis

- Obtained by dehydrogenation of corresponding 3,4-dihydro derivative [72935-01-4] with DDQ in refluxing benzene for 1 h (83%) [8138].

m.p. 76–77° [8138]; ^{1}H NMR [8138].

1-[3,4-Dihydro-7-hydroxy-5-(methoxymethoxy)-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone

[72935-01-4]



$C_{17}H_{24}O_5$ mol.wt. 308.37

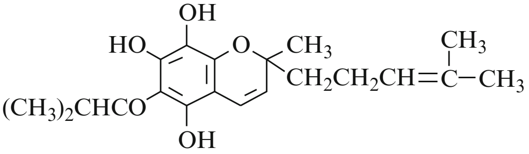
Syntheses

- Obtained by reaction of chloromethyl methyl ether with 8-isobutyryl-5,7-dihydroxy-2,2-dimethylchroman in the presence of potassium carbonate in acetone at r.t. overnight (82%) [8138].
- Also refer to: [8060].

m.p. 95–96° [8138]; ^{1}H NMR [8138], IR [8138].

2-Methyl-1-[5,7,8-trihydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2*H*-1-benzopyran-6-yl]-1-propanone

[103784-23-2]



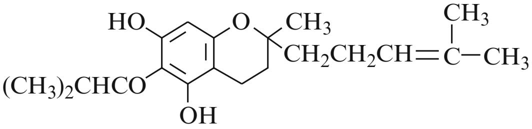
$C_{20}H_{26}O_5$ mol.wt. 346.42

Isolation from natural sources

- From South african *Helichrysum* species (Compositae) [8080].

1-[3,4-Dihydro-5,7-dihydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2*H*-1-benzopyran-6-yl]-2-methyl-1-propanone

[72008-16-3]
[722457-96-7] (-)



$C_{20}H_{28}O_4$ mol.wt. 332.44

Isolation from natural sources

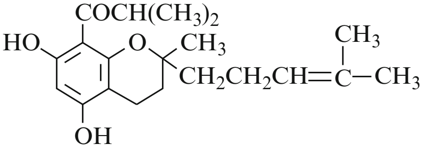
- From *Hypericum jovis* (Guttiferae) [8239].
- Also refer to: [8211].

$(\alpha)_D = -1^\circ$ (c = 0.1 in chloroform) [8239]; ^{1}H NMR [8211], ^{13}C NMR [8239], IR [8211], UV [8211], MS [8211].

USE: Antioxidizing agent [8239].

1-[3,4-Dihydro-5,7-dihydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone

[72008-14-1]



$C_{20}H_{28}O_4$ mol.wt. 332.44

Isolation from natural sources

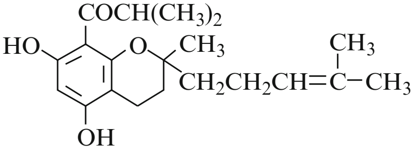
- From *Hypericum jovis* (Guttiferae) [8239].
- From *Hypericum amblycalyx* [8283].
- Also refer to: [8211].

USE: Antioxidizing agent [8239].

oil [8211]; ^{1}H NMR [8211], IR [8211], UV [8211], MS [8211].

1-[3,4-Dihydro-5,7-dihydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone (+)

[658702-62-6]



$C_{20}H_{28}O_4$ mol.wt. 332.44

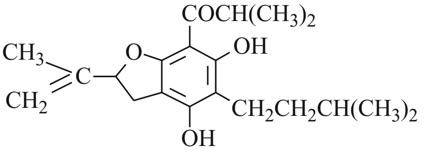
Isolation from natural sources

- From the aerial parts of *Hypericum amblycalyx* [8283].

Note: Cytotoxic [8283].

BIOLOGICAL ACTIVITY: Antibacterial [8283].

1-[2,3-Dihydro-4,6-dihydroxy-5-(3-methylbutyl)-2-(1-methylethenyl)-7-benzofuranyl]-2-methyl-1-propanone



$C_{20}H_{28}O_4$ mol.wt. 332.44

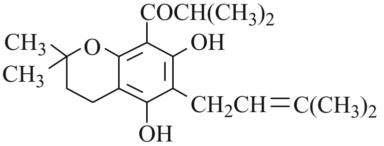
Synthesis

- Preparation by adding isoprene dibromide to a solution of 2,4,6-trihydroxy-3-isopentylisobutyrophenone disodium salt and sodium methoxide in methanol. The solution was stirred under nitrogen, in the dark, for 72 h [8218].

pale yellow oil [8218]; UV [8218], MS [8218]; TLC [8218].

1-[5,7-Dihydroxy-2,2-dimethyl-6-(3-methyl-2-butenyl)-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone

[31918-60-2]



$C_{20}H_{28}O_4$ mol.wt. 332.44

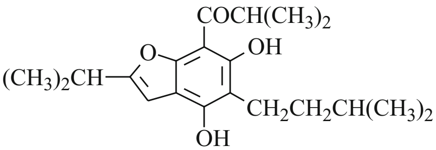
Synthesis

- Obtained from colupulone when refluxing with zinc dust in acetic acid containing some hydrochloric acid [8284].

^{1}H NMR [8284], IR [8284], UV [8284], MS [8284]; TLC [8284], GLC [8284].

1-[4,6-Dihydroxy-5-(3-methylbutyl)-2-(1-methylethyl)-7-benzofuranyl]-2-methyl-1-propanone

[18892-91-6]



$C_{20}H_{28}O_4$ mol.wt. 332.44

Syntheses

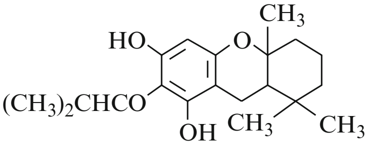
- Obtained by dehydration of 4,6-dihydroxy-2-(1-hydroxy-1-methylethyl)-7-isobutyryl-5-isopentyl-2,3-dihydrobenzo[*b*]furan (m.p. 150°) with p-toluenesulfonic acid in refluxing benzene under a stream of oxygen-free nitrogen for 20 h [8285], (54%) [8218].

- Also obtained by treatment of 4,6-dihydroxy-2-(1-methylethenyl)-7-isobutyryl-(2*H*)-benzofuran with p-toluenesulfonic acid in refluxing benzene for 3 h under nitrogen [8218].

m.p. 164–164.5° [8218], 163.5–164.5° [8285];
^{1}H NMR [8218,8285], IR [8218], UV [8218,8285],
MS [8218,8285,8286]; TLC [8218].

1-[(4a*R*,9a*R*)-2,3,4,4a,9,9a-Hexahydro-6,8-dihydroxy-1,1,4a-trimethyl-1*H*-xanthen-7-yl]-2-methyl-1-propanone *(Hyperjovinol B)*

[722457-95-6]



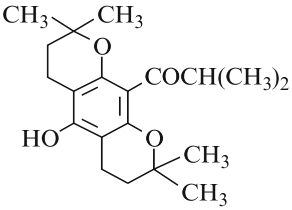
$C_{20}H_{28}O_4$ mol.wt. 332.44

Isolation from natural sources

- From *Hypericum jovis* (Guttiferae) [8239].

yellow amorphous solid [8239];
$(\alpha)_D = -2^{\circ}$ (c – 0.1 in chloroform) [8239];
^{1}H NMR [8239], ^{13}C NMR [8239], IR [8239], UV [8239], MS [8239].
USE: Antioxidizing agent [8239].

2-Methyl-1-(3,4,6,7-tetrahydro-5-hydroxy-2,2,8,8-tetramethyl-2*H*,8*H*-benzo[1,2-*b*:5,4-*b*′]-dipyran-10-yl)-1-propanone



$C_{20}H_{28}O_4$ mol.wt. 332.44

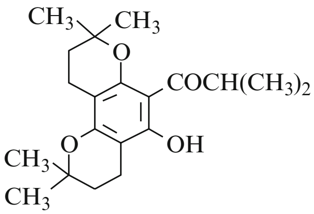
Syntheses

- Obtained from colupulone when refluxing with zinc dust in acetic acid containing some hydrochloric acid for 1.5 h [8284].
- Also obtained from colupulone when refluxing in methanol in the presence of 12 N hydrochloric acid for 3.5 h [8056].
- Also obtained by oxidation of colupulone (m.p. 92–94°) with ammonium persulfate in refluxing ethanol for 50 min [8219].
- Also obtained from isomerization of 6-isobutyryl-3,4,9,10-tetrahydro-5-hydroxy-2,2,8,8-tetra-methylbenzo-[1,2-*b*:3,4-*b*′]dipyran by heating with concentrated sulfuric acid at 60° for 5 min [8056], (40%) [8287].

m.p. 170–172° [8219], 169.5° [8056], 169–170.5° [8287], 168–169° [8284];
^{1}H NMR [8284], IR [8284,8287], UV [8056,8219,8284,8287];
TLC [8287]; GLC [8287].

2-Methyl-1-(3,4,9,10-tetrahydro-5-hydroxy-2,2,8,8-tetramethyl-2*H*,8*H*-benzo[1,2-*b*:3,4-*b′*]-dipyran-6-yl)-1-propanone

[23067-91-6] $C_{20}H_{28}O_4$ mol.wt. 332.44



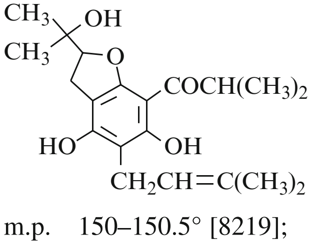
Syntheses

- Obtained by reaction of 1-bromo-3-methyl-2-butene with phloroisobutyrophenone in chloroform (6%) [8056].
- Also obtained (poor yield) in two steps: First, dialkenylation of phloroisobutyrophenone with 1-bromo-3-methyl-2-butene using powdered sodium, then cyclisation during work-up with 5 N hydrochloric acid (1%) [8288].
- Also obtained from a mixture of phloroisobutyrophenone, 2-methyl-3-buten-2-ol and Amberlite IR 120 (H^+ form) in refluxing dioxane for 24 h (6%) [7739].
- Also obtained from colupulone when refluxing with zinc dust in acetic acid containing some hydrochloric acid for 1.5 h [8284].
- Also obtained by oxidation of colupulone (m.p. 92–94°) with ammonium persulfate in refluxing ethanol for 50 min [8219].
- Also obtained by adding isobutyryl chloride to the solution of 3,4,9,10-tetrahydro-2,2,8,8-tetra-methylbenzo[1,2-*b*:3,4-*b′*]-dipyran-5-ol (compound VI) and aluminium chloride in nitrobenzene at −5° during 10 min. Then, the mixture was stirred at 20° for 4 days (16%) [8287].

m.p. 86–88° [8056], 85° [7739], 84° [8056,8288], 83–84° [8287], 82–83° [8284];
^{1}H NMR [8284,8288], IR [8056,8219,8284,8287],
UV [8056,8219,8284,8287], MS [8284]; TLC [8287]; GLC [8287].

1-[2,3-Dihydro-4,6-dihydroxy-2-(1-hydroxy-1-methylethyl)-5-(3-methyl-2-butenyl)-7-benzofuranyl]-2-methyl-1-propanone

[24416-50-0] $C_{20}H_{28}O_5$ mol.wt. 348.44



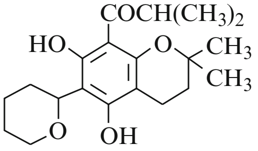
Syntheses

- Obtained by oxidation of colupulone (m.p. 92–94°) with ammonium persulfate in refluxing ethanol for 50 min [8219,8289].

m.p. 150–150.5° [8219];
^{1}H NMR [8219], IR [8219], UV [8219], MS [8219,8286].

1-[3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-6-(tetrahydro-2*H*-pyran-2-yl)-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone

[72935-03-6] $C_{20}H_{28}O_5$ mol.wt. 348.44



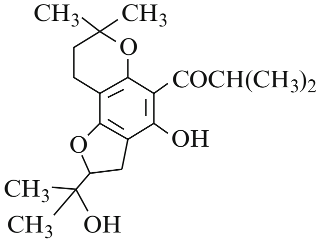
Synthesis

- Obtained by reaction of 2,3-dihydropyran with 8-isobutyryl-5,7-dihydroxy-2,2-dimethylchroman in the presence of p-toluenesulfonic acid in benzene at r.t. overnight (50%) [8138]. The same result was obtained in the presence of concentrated hydrochloric acid as catalyst.

m.p. 94–95° [8138]; ^{1}H NMR [8138], IR [8138].

1-[2,3,8,9-Tetrahydro-4-hydroxy-2-(1-hydroxy-1-methylethyl)-7,7-dimethyl-7*H*-furo[2,3-*f*]-1-benzopyran-5-yl]-2-methyl-1-propanone

[29525-25-5] $C_{20}H_{28}O_5$ mol.wt. 348.44



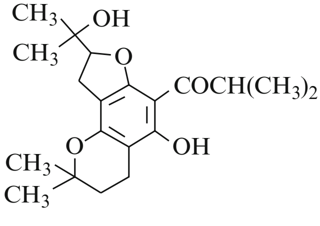
Syntheses

- Obtained by oxidation of colupulone (m.p. 92–94°) with ammonium persulfate in refluxing ethanol for 50 min [8219].
- Also obtained by treatment of 3,3a-dihydro-4-hydroxy-2-(1-hydroxy-1-methylethyl)-5-isobutyryl-3a,7-bis-(3-methyl-2-butenyl)-6-(2*H*)-benzofuranone [29525-23-3] with concentrated hydrochloric acid in refluxing methanol for 1 h [8219].

pale yellow oil [8219];
^{1}H NMR [8219], IR [8219], UV [8219], MS [8219].

1-[2,3,6,7-Tetrahydro-8-hydroxy-2-(1-hydroxy-1-methylethyl)-5,5-dimethyl-2*H*-furo[2,3-*h*]-1-benzopyran-9-yl]-2-methyl-1-propanone

[24416-48-6] $C_{20}H_{28}O_5$ mol.wt. 348.44



Syntheses

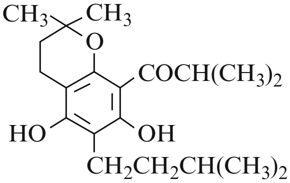
- Obtained by oxidation of colupulone (m.p. 92–94°) with ammonium persulfate in refluxing ethanol for 50 min [8219,8289].
- Also obtained by treatment of 1-[2,3-dihydro-4,6-di-hydroxy-2-(1-hydroxy-1-methylethyl)-5-(3-methyl-2-butenyl)-7-benzofuranyl]-2-methyl-1-propanone [24416-50-0] with concentrated hydrochloric acid in refluxing methanol for 2 h [8219].
- Also obtained by treatment of 3,5-dihydro-6-hydroxy-2-(1-hydroxy-1-methylethyl)-7-isobutyryl-5,5-bis(3-methyl-2-butenyl)-4-(2*H*)-benzofuranone [22592-22-9] with concentrated hydrochloric acid in refluxing methanol for 75 min [8219].

m.p. 107.5–109° [8219];
^{1}H NMR [8219], IR [8219], UV [8219], MS [8219,8286];
TLC [8219]; GLC [8219].

1-[3,4-Dihydro-5,7-dihydroxy-6-(3-methylbutyl)-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone

1-(5,7-Dihydroxy-6-isopentyl-2,2-dimethyl-8-chromanyl)-2-methyl-1-propanone

[29525-18-6] $C_{20}H_{30}O_4$ mol.wt. 334.46



Syntheses

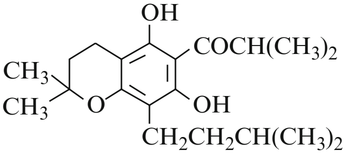
- Obtained by hydrogenolysis of 7-hydroxy-8-isobutyryl-2,2-dimethyl-6,6-bis(3-methyl-2-butenyl)-5-(6*H*)-chromanone [24416-46-4] in ethanol over 5% Pd/C with hydrogen [8219].
- Also obtained by reaction of 2-methyl-3-buten-2-ol with 2,4,6-trihydroxy-3-isopentylisobutyrophenone (m.p. 184–185°) in dioxan in the presence of boron trifluoride etherate under nitrogen [8219].

oil [8219]; ^{1}H NMR [8219], IR [8219], UV [8219], MS [8219].

1-[3,4-Dihydro-5,7-dihydroxy-8-(3-methylbutyl)-2,2-dimethyl-2*H*-1-benzopyran-6-yl]-2-methyl-1-propanone

1-(5,7-Dihydroxy-8-isopentyl-2,2-dimethyl-6-chromanyl)-2-methyl-1-propanone

[29373-18-0] $C_{20}H_{30}O_4$ mol.wt. 334.46



Syntheses

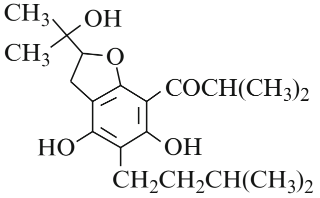
- Also obtained by reaction of 2-methyl-3-buten-2-ol with 2,4,6-trihydroxy-3-isopentylisobutyrophenone (m.p. 184–185°) in dioxan in the presence of boron trifluoride etherate at 55–60° for 4 h under nitrogen [8219].
- Obtained by hydrogenolysis of 7-hydroxy-6-isobutyryl-2,2-dimethyl-8,8-bis(3-methyl-2-butenyl)-5-(8*H*)-chromanone [29525-19-7] in ethanol over 5% Pd/C with hydrogen [8219].

oil [8219]; ^{1}H NMR [8219], IR [8219], UV [8219], MS [8219,8286].

1-[2,3-Dihydro-4,6-dihydroxy-2-(1-hydroxy-1-methylethyl)-5-(3-methylbutyl)-7-benzofuranyl]-2-methyl-1-propanone

[18944-22-4]
[22592-23-0]

$C_{20}H_{30}O_5$ mol.wt. 350.46



Syntheses

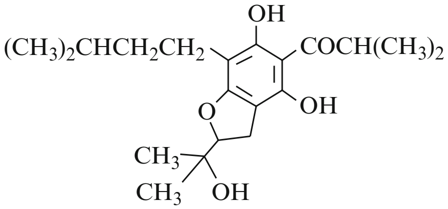
- Obtained by hydrogenation of 1-[2,3-dihydro-4,6-dihydroxy-2-(1-hydroxy-1-methylethyl)-5-(3-methyl-2-butenyl)-7-benzofuranyl]-2-methyl-1-propanone [24416-50-0] in ethanol with hydrogen in the presence of 5% Pd/C for 15 min [8219].
- Also obtained by hydrogenation of 3,5-dihydro-6-hydroxy-2-(1-hydroxy-1-methylethyl)-7-isobutyryl-5,5-bis(3-methyl-2-butenyl)-4-(2*H*)-benzofuranone (SM) with hydrogen in ethanol over Pd/C [8218,8285]. SM was obtained on mild air-oxidation of colupulone (m.p. 94.5–95.5°).

m.p. 152.5–153° [8219], 150.5–151.5° [8285], 150° [8218];
^{1}H NMR [8218,8285], IR [8218,8219],
UV [8218,8219,8285], MS [8218,8285,8286].

1-[2,3-Dihydro-4,6-dihydroxy-2-(1-hydroxy-1-methylethyl)-7-(3-methylbutyl)-5-benzofuranyl]-2-methyl-1-propanone

[29525-24-4]

$C_{20}H_{30}O_5$ mol.wt. 350.46



Syntheses

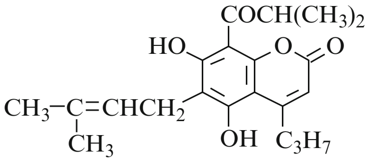
- Obtained by hydrogenolysis of 3,3a-dihydro-4-hydroxy-2-(1-hydroxy-1-methylethyl)-5-isobutyryl-3a,7-bis(3-methyl-2-butenyl)-6(2*H*)-benzofuranone [29525-23-3] in ethanol with hydrogen over 5% Pd/C [8219].
- Also refer to: [8218,8285].

m.p. 124–126° [8217,8219];
^{1}H NMR [8217,8219], IR [8219], UV [8219], MS [8219]; TLC [8219].

5,7-Dihydroxy-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxopropyl)-4-propyl-2*H*-1-benzopyran-2-one

[98216-05-8]

$C_{21}H_{26}O_5$ mol.wt. 358.43



Synthesis

- Refer to: [8053].

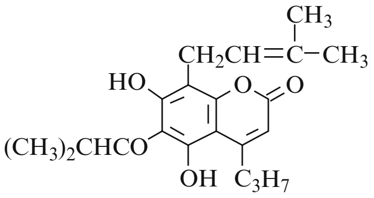
Isolation from natural sources

- From *Mammea americana* L. (Guttiferae) [8053].

USE: Insecticide [8053].

5,7-Dihydroxy-8-(3-methyl-2-butenyl)-6-(2-methyl-1-oxopropyl)-4-propyl-2*H*-1-benzopyran-2-one

[98192-71-3]



$C_{21}H_{26}O_5$ mol.wt. 358.43

Synthesis

- Refer to: [8053].

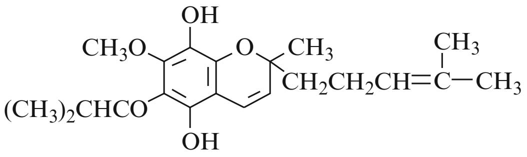
Isolation from natural sources

- From *Mammea americana* L. (Guttiferae) (compound mammea B/AD) [8053,8290].

m.p. 139–140° [8290];
^{1}H NMR [8290], IR [8290], UV [8290], MS [8290].
Note: Insecticide [8053].

1-[5,8-Dihydroxy-7-methoxy-2-methyl-2-(4-methyl-3-pentenyl)-2*H*-1-benzopyran-6-yl]-2-methyl-1-propanone *(Helicerestripyrone-6-O-methyl ether)*

[103771-77-3]



$C_{21}H_{28}O_5$ mol.wt. 360.45

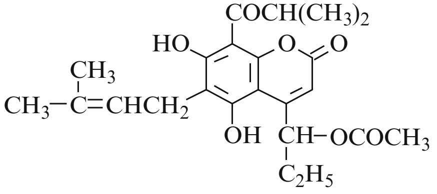
Isolation from natural sources

- From the aerial parts of *Helichrysum nudifolium* (Compositae) (compound **37**) [8080].

yellow oil [8080];
^{1}H NMR [8080], IR [8080], MS [8080]; TLC [8080].

4-(1-Acetoxypropyl)-5,7-dihydroxy-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-2-one (–)

[879420-45-8]



$C_{23}H_{28}O_7$ mol.wt. 416.47

Isolation from natural sources

- From the seed of *Mammea americana* L. (Guttiferae) (compound Xc) [8291], (Clusiaceae) (compound mammea E/BD) [8292].

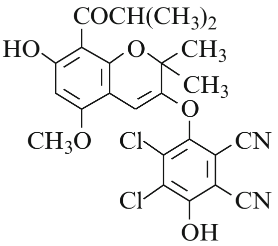
yellow semisolid [8292];
$(\alpha)_D^{25} = -46^\circ$ (methanol) [8292];
^{1}H NMR [8291,8292], ^{13}C NMR [8292], UV [8291,8292],
MS [8291,8292]; HPLC [8292].
Note: Cytotoxicity [8292]

USE: Insecticide [8291].

BIOLOGICAL ACTIVITY: apoptosis induction (human colon cancer SW-480 cells) [8292].

4,5-Dichloro-3-hydroxy-6-[[7-hydroxy-5-methoxy-2,2-dimethyl-8-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-3-yl]oxy]-1,2-benzenedicarbonitrile

[72935-04-7] $C_{24}H_{20}Cl_2N_2O_6$ mol.wt. 503.34



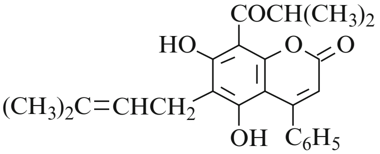
Synthesis

- Obtained by reaction of DDQ with 8-isobutyryl-7-hydroxy-5-methoxy-2,2-dimethyl-2*H*-chromen (m.p. 67–68°) in benzene at r.t. overnight [8138].

m.p. 246–247° [8138];
^{1}H NMR [8138], IR [8138], MS [8138].

5,7-Dihydroxy-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxopropyl)-4-phenyl-2*H*-1-benzopyran-2-one *(Isomesuol)*

[16981-21-8] $C_{24}H_{24}O_5$ mol.wt. 392.45



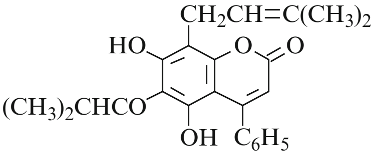
Syntheses

- Obtained by reaction of prenyl bromide with 5,7-di-hydroxy-4-phenyl-8-isobutyrylcoumarin in 10% aqueous potassium hydroxide at 0° (20–35%) [8053].
- Also obtained on treatment of *mesuol* with 5% methanolic or with 10% aqueous potassium hydroxide [8293]. *Mesuol* was isomerized to *Isomesuol* [8293].

m.p. 171° [8293]; ^{1}H NMR [8293], MS [8293].

5,7-Dihydroxy-8-(3-methyl-2-butenyl)-6-(2-methyl-1-oxopropyl)-4-phenyl-2*H*-1-benzopyran-2-one *(Mesuol)*

[16981-20-7] $C_{24}H_{24}O_5$ mol.wt. 392.45



Synthesis

- Obtained by prenylation of 5,7-dihydroxy-4-phenyl-6-isobutyrylcoumarin [8053].

Isolation from natural sources

- From the kernel of *Mammea americana* L. (Guttiferae) [8053,8294].
- From the bark of *Mammea aficana* G. Don (Guttiferae) [8295].
- From *Mesua ferrea* Linn [8293].

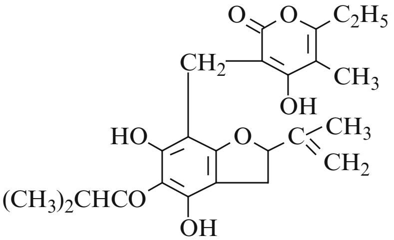
Note: Insecticidal material [8053].

BIOLOGICAL ACTIVITY: Antibiotic [8293].

m.p. 154° [8293];
^{1}H NMR [8293,8294], IR [8293], UV [8294], MS [8293,8294].

3-[[2,3-Dihydro-4,6-dihydroxy-2-(1-methylethenyl)-5-(2-methyl-1-oxopropyl)-7-benzofuranyl]methyl]-6-ethyl-4-hydroxy-5-methyl-2*H*-pyran-2-one

[75680-05-6]



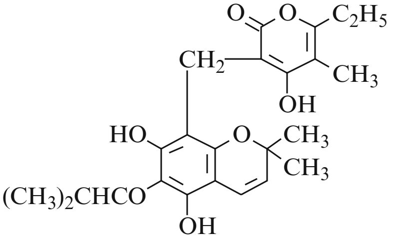
$C_{24}H_{28}O_7$ mol.wt. 428.48

Isolation from natural sources

- From the roots of *Helichrysum cephaloideum* and *Helichrysum mixtum* (Compositae) (compound **33**) [8080], (compound **8**) [8210].

3-[[5,7-Dihydroxy-2,2-dimethyl-6-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-8-yl]-methyl]-6-ethyl-4-hydroxy-5-methyl-2*H*-pyran-2-one

[103784-19-6]



$C_{24}H_{28}O_7$ mol.wt. 428.48

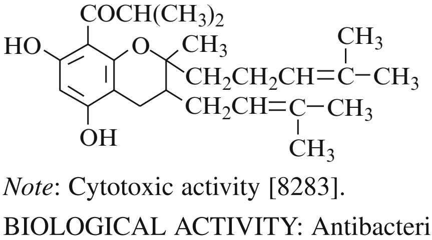
Isolation from natural sources

- From *Helichrysum mixtum* (Compositae) (compouund 35) [8080].

yellow oil [8080];
^{1}H NMR [8080], IR [8080], MS [8080].

1-[(2*R*,3*S*)-3,4-Dihydro-5,7-dihydroxy-2-methyl-3-(3-methyl-2-butenyl)-2-(4-methyl-3-pentenyl)-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone (+) *(Hypercalyxone)*

[658702-60-4]



$C_{25}H_{36}O_4$ mol.wt. 400.16

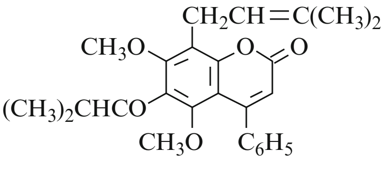
Isolation from natural sources

- From the aerial parts of *Hypericum amblycalyx* [8283].

Note: Cytotoxic activity [8283].

BIOLOGICAL ACTIVITY: Antibacterial [8283];

5,7-Dimethoxy-8-(3-methyl-2-butenyl)-6-(2-methyl-1-oxopropyl)-4-phenyl-2*H*-1-benzopyran-2-one



$C_{26}H_{28}O_5$ mol.wt. 420.51

Synthesis

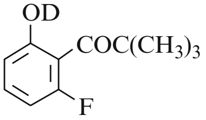
Refer to: [8293].

Chapter 25
Aromatic Ketones Containing One Pivaloyl Group

25.1 Benzene Derivatives

1-[2-Fluoro-6-(hydroxy-*d*)phenyl]-2,2-dimethyl-1-propanone

[195871-76-2] $C_{11}H_{12}DFO_2$ mol.wt. 181.23



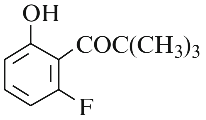
Syntheses

- Refer to: [7794,8296].

^{13}C NMR [8296], ^{19}F NMR [8296].

1-(2-Fluoro-6-hydroxyphenyl)-2,2-dimethyl-1-propanone

[158897-29-1] $C_{11}H_{13}FO_2$ mol.wt. 196.22



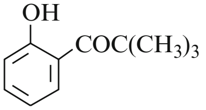
Synthesis

- Obtained (poor yield) by treatment of 6-fluoro-2-methoxy-benzonitrile with a threefold excess of tert-butyllithium in pentane for 1 h, followed by hydrolysis in 10% hydrochloric acid. After, treatment of the obtained 6-fluoro-2-methoxy-pivalophenone (compound 19) with a large excess of aluminium chloride in carbon disulfide overnight (ca. 1%) (compound 6) [7794], (compound 8) [8296].

Sublimation on a cold-finger at 50° and 0.1 mbar [7794];
^{13}C NMR [7794,8296], ^{19}F NMR [8296].

1-(2-Hydroxyphenyl)-2,2-dimethyl-1-propanone

[22526-25-6] $C_{11}H_{14}O_2$ mol.wt. 178.23



Syntheses

- Obtained by reaction of sec-butyllithium (1.1 equiv) with 2-bromophenyl pivalate in tetrahydrofuran/ethyl ether/hexane at −95° for 30 min and −78° for 30 min, then hydrolysis with saturated ammonium chloride (76%) (metal-promoted Fries rearrangement) [6590].

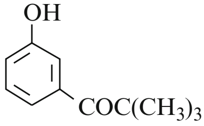
DOI 10.1007/978-1-4020-9787-4_25,

- Also obtained by action of aluminium chloride with 2-methoxypivalophenone [7794].

^{1}H NMR [6590], ^{13}C NMR [6590,7794], IR [6590], MS [6628].

1-(3-Hydroxyphenyl)-2,2-dimethyl-1-propanone

[32578-14-6] $C_{11}H_{14}O_2$ mol.wt. 178.23



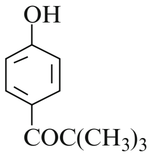
Syntheses

- Obtained by reaction of tricarbonyl-[(1,2,3,4-n)-1-acetoxy-5-*endo*-propionyl-1,3-cyclohexadiene]iron with trimethylamine N-oxide in DMA at r.t. for 1 h (16% and 59% of its acetoxy derivative) [8297].
- Also obtained by treatment of m-methoxypivalophenone with reluxing 45% hydrogen bromide in acetic acid for 4.5 h (72%) [8298].
- Also refer to: [8299].

b.p.$_{0.2}$ 126° [8298]; m.p. 65–67° [8298];
^{1}H NMR [8298], IR [8298], UV [8298], MS [6628,8298].

1-(4-Hydroxyphenyl)-2,2-dimethyl-1-propanone

[72569-10-9] $C_{11}H_{14}O_2$ mol.wt. 178.23



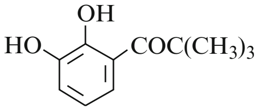
Syntheses

- Obtained by Fries rearrangement of phenyl pivalate (1 mol) in nitromethane at 20° for 6 days,
  - in the presence of hydrogen chloride (1 mol) and stannic chloride (2 mol) (23%) [8300];
  - in the presence of aluminium chloride (1.1 mol) (6%) [8300].
- Also obtained by Friedel–Crafts acylation of phenol (1 mol) with pivaloyl chloride (1 mol) in the presence of stannic chloride (2 mol) in nitromethane at 20° for 6 days (26%) [8300].
- Also obtained from p-acetoxybenzoyl chloride by treatment with lithium tert-butoxide and tert-butyllithium in a mixture pentane/tetrahydrofurane in the presence of cuprous iodide, followed by hydrolysis (80%) [6683].
- Also obtained by an ultrasound assisted iodine catalyzed Friedel–Crafts acylation of phenol with pivaloyl chloride for 10 min at r.t. (84%) [8301]. The same pivaloylation, carried out at silent (non-ultrasound) conditions, gave only, after 6 h at r.t., a yield of 62%.
- Also obtained by an ultrasound assisted aluminium chloride catalyzed Friedel–Crafts acylation of phenol with pivaloyl chloride in hexane for 10 min at r.t. (82%) [8302].
- Also refer to: [8303–8306].

b.p.$_{0\cdot 5}$ 157° [8302], b.p.$_5$ 170–174° [6683];
m.p. 90° [8300], 88–89° [6683];
^{1}H NMR (Sadtler: standard n° 35271M) [6683,8300,8302], ^{13}C NMR [6683], IR (Sadtler: standard n° 62639K) [6683,8300], UV [8300], MS [6628,6683,8300].

1-(2,3-Dihydroxyphenyl)-2,2-dimethyl-1-propanone

[100059-02-7] $C_{11}H_{14}O_3$ mol.wt. 194.23



Synthesis

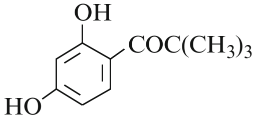
– Obtained from 2-pivaloylacetyl-2,5-dimethoxy-tetrahydrofuran (b.p.$_{1.5}$ 106°) by refluxing with 0.1 N hydrochloric acid for 2 h (81%) [8307].

viscous, yellow liquid [8307]; b.p.$_3$ 114–119° [8307].

Diacetate [101103-44-0] $C_{15}H_{18}O_5$ mol.wt. 278.30 (m.p. 70–71°) [8307].

1-(2,4-Dihydroxyphenyl)-2,2-dimethyl-1-propanone

[60884-07-3] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

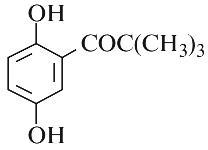
– Preparation by total demethylation of 2,4-dimethoxy-pivalophenone (m.p. 150°) with aluminium chloride (3 mol) in refluxing benzene for 5 h (80%) [8308].

– Also obtained by reaction of pivalic acid with resorcinol in the presence of boron trifluoride etherate at r.t. for 30 h (28%) [8308].

m.p. 129° [8308].
USE: Aldose reductase inhibition [8309].

1-(2,5-Dihydroxyphenyl)-2,2-dimethyl-1-propanone

[35459-97-3] $C_{11}H_{14}O_3$ mol.wt. 194.23



Syntheses

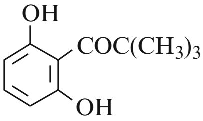
– Obtained by total demethylation of 2,5-dimethoxy-pivalophenone,
  - with 48% hydrobromic acid in refluxing acetic acid for 3 h (49%) [8310];
  - with 45% hydrobromic acid in refluxing acetic acid for 1.5 h (54%) [8311].

– Also obtained by irradiation of a 1,4-benzoquinone and trimethylacetaldehyde mixture for 18 days (56%) [8312].

– Also refer to: [8313].

m.p. 95–98° [8310], 95° [8312], 93–95° [8311];
^{1}H NMR [8311,8312], IR [8310–8312], MS [8311,8312,8314].
USE: Antioxidizing agent [8315,8316].

1-(2,6-Dihydroxyphenyl)-2,2-dimethyl-1-propanone



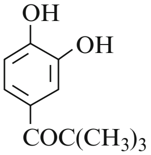
$C_{11}H_{14}O_3$ mol.wt. 194.23

Synthesis

- Refer to: [8308].

1-(3,4-Dihydroxyphenyl)-2,2-dimethyl-1-propanone

[72017-59-5] $C_{11}H_{14}O_3$ mol.wt. 194.23



Synthesis

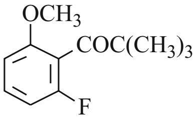
- Preparation by total demethylation of 3,4-dimethoxy-pivalophenone by heating with pyridinium chloride at 200–220° for 1 h (88%) [6828].

m.p. 127–129.5° [6828].

BIOLOGICAL ACTIVITY: Pharmacology [6828]; central nervous system depressant [6828].

1-(2-Fluoro-6-methoxyphenyl)-2,2-dimethyl-1-propanone

[158897-28-0] $C_{12}H_{15}FO_2$ mol.wt. 210.25



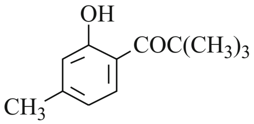
Synthesis

- Preparation by treatment of 6-fluoro-2-methoxybenzonitrile with a threefold excess of tert-butyllithium in pentane for 1 h, followed by hydrolysis [7794].

  ^{13}C NMR [7794].

1-(2-Hydroxy-4-methylphenyl)-2,2-dimethyl-1-propanone

[14194-55-9] $C_{12}H_{16}O_2$ mol.wt. 192.26



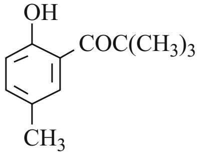
Syntheses

- Obtained by Fries rearrangement of 3-methylphenyl pivalate (1 mol) in nitromethane at 20° for 6 days,
  - in the presence of hydrogen chloride (1 mol) and stannic chloride (2 mol) (60%) [8300];
  - in the presence of aluminium chloride (1.1 mol) (27%) [8300].
- Also obtained by Fries rearrangement of 3-methylphenyl pivalate in nitrobenzene in the presence of aluminium chloride at r.t. for 142 h (22%) [6571].
- Also obtained by Friedel–Crafts acylation of 3-methylphenol (1 mol) with pivaloyl chloride (1 mol) in the presence of stannic chloride (2 mol) in nitromethane at 20° for 6 days (40%) [8300].

b.p.$_{7.5}$ 121–126° [6571], b.p.$_{21}$ 140–141° [8300];
^{1}H NMR (Sadtler: standard n° 30300M) [8300],
IR (Sadtler: standard n° 57345K) [8300], UV [8300], MS [8300].

1-(2-Hydroxy-5-methylphenyl)-2,2-dimethyl-1-propanone

[72569-13-2] $C_{12}H_{16}O_2$ mol.wt. 192.26



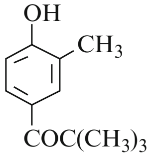
Syntheses

- Obtained by Fries rearrangement of 4-methylphenyl pivalate (1 mol) in nitromethane at 20° for 6 days,
    - in the presence of hydrogen chloride (1 mol) and stannic chloride (2 mol) (24%) [8300];
    - in the presence of aluminium chloride (1.1 mol) (9%) [8300].
- Also obtained by Fries rearrangement of 4-methylphenyl pivalate in nitrobenzene in the presence of aluminium chloride at r.t. for 162 h (3%) [6571].
- Also obtained by Friedel–Crafts acylation of 4-methylphenol (1 mol) with pivaloyl chloride (1 mol) in the presence of stannic chloride (2 mol) in nitromethane at 20° for 6 days (24%) [8300].
- Preparation by heating 2-methoxy-5-methylisobutyrophenone with pyridinium chloride at reflux for 2 h (83%) [6672].
- Also obtained by reaction of sec-butyllithium (1.0–1.1 equiv) with 2-bromo-4-methylphenyl pivalate (metal-promoted Fries rearrangement),
    - in tetrahydrofuran/ethyl ether/hexane at –95° for 30 min and –78° for 30 min, then hydrolysis with saturated ammonium chloride (71%) [6590];
    - in tetrahydrofuran at –100° for 2 h, followed by hydrolysis (25%) [8317].
- Also refer to: [7987].

oil [8300]; b.p.$_{0.3}$ 51° [8317], b.p.$_{7}$ 80° [6571];
n_D^{20} = 1.5341 [8317];
m.p. 32° [8317];
^{1}H NMR [6590,8300,8317], ^{13}C NMR [6590], IR [6590,8300,8317], UV [8300], MS [8300,8317].

1-(4-Hydroxy-3-methylphenyl)-2,2-dimethyl-1-propanone

[72569-11-0] $C_{12}H_{16}O_2$ mol.wt. 192.26



Syntheses

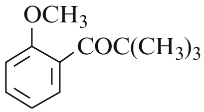
- Obtained by Fries rearrangement of 2-methylphenyl pivalate (1 mol) in nitromethane at 20° for 6 days,
    - in the presence of hydrogen chloride (1 mol) and stannic chloride (2 mol) (45%) [8300];
    - in the presence of aluminium chloride (1.1 mol) (6%) [8300].

- Also obtained by Friedel–Crafts acylation of 2-methylphenol (1 mol) with pivaloyl chloride (1 mol) in the presence of stannic chloride (2 mol) in nitromethane at 20° for 6 days (38%) [8300].

m.p. 118° [8300];
^{1}H NMR (Sadtler: standard n° 35272M) [8300],
IR (Sadtler: standard n° 62640K) [8300], UV [8300], MS [8300].

1-(2-Methoxyphenyl)-2,2-dimethyl-1-propanone

[22526-24-5] $C_{12}H_{16}O_2$ mol.wt. 192.26

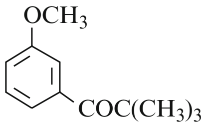


Syntheses

- Preparation by treatment of 2-methoxybenzoic acid with tert-butyllithium (3 equiv) in pentane (60%) [7794].
- Also refer to: [8318].

1-(3-Methoxyphenyl)-2,2-dimethyl-1-propanone

[32578-12-4] $C_{12}H_{16}O_2$ mol.wt. 192.26



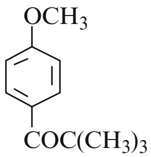
Syntheses

- Preparation by treatment of 1-(3-methoxyphenyl)-2,2-dimethylpropanol with manganese dioxide in refluxing benzene for 15 h (81%) [8298].
- Also obtained by reaction of m-anisic acid with tert-butylliyhium at –78° [8319].
- Also refer to: [8320].

b.p.$_{13}$ 129–130° [8298];
^{1}H NMR [8298,8319], IR [8298].

1-(4-Methoxyphenyl)-2,2-dimethyl-1-propanone

[2040-26-8] $C_{12}H_{16}O_2$ mol.wt. 192.26



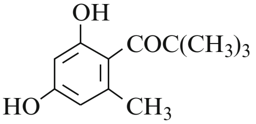
Syntheses

- Preparation by reaction of pivaloyl chloride with anisole,
  - in the presence of bismuth (III) chloride at 100° for 3 h (94%) [8126];
  - in the presence of indium metal (1 equiv) at 100° for 6 h without solvent (76%) or in dioxane for 4 h (80%) [8321].
- Also obtained by reaction of pivalic anhydride with anisole in acetonitrile in the presence of $TiCl(OTf)_3$/TfOH at r.t. for 12 h (68%) [8128].
- Also refer to: [7095,8129,8302,8322–8324].

b.p.$_2$ 116–118° [7095], b.p.$_8$ 138–140° [8325]; ^{13}C NMR [7095].

1-(2,4-Dihydroxy-6-methylphenyl)-2,2-dimethyl-1-propanone

[60883-92-3] $C_{12}H_{16}O_3$ mol.wt. 208.26



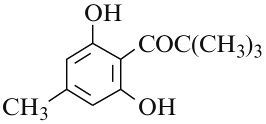
Synthesis

– Obtained by reaction of pivalic acid with orcinol in the presence of boron trifluoride etherate at r.t. for 5 days (15%) [8308].

m.p. 148° [8308]; MS [8308]; TLC [8308].

1-(2,6-Dihydroxy-4-methylphenyl)-2,2-dimethyl-1-propanone

[60884-09-5] $C_{12}H_{16}O_3$ mol.wt. 208.26



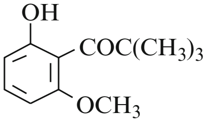
Synthesis

– Obtained by reaction of pivalic acid with orcinol in the presence of boron trifluoride etherate at r.t. for 5 days [8308].

pale yellow oil [8308]; b.p.$_1$ 220° [8308].

1-(2-Hydroxy-6-methoxyphenyl)-2,2-dimethyl-1-propanone

[60884-04-0] $C_{12}H_{16}O_3$ mol.wt. 208.26

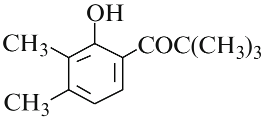


Synthesis

– Obtained by partial demethylation of 2,6-dimethoxypivalophenone (m.p. 86°) with aluminium chloride (3 mol) in refluxing benzene for 6 h (26%) [8308].
m.p. 82° [8308].

1-(2-Hydroxy-3,4-dimethylphenyl)-2,2-dimethyl-1-propanone

[72569-14-3] $C_{13}H_{18}O_2$ mol.wt. 206.28



Syntheses

– Obtained by Fries rearrangement of 2,3-dimethylphenyl pivalate (1 mol) in nitromethane at 20°,

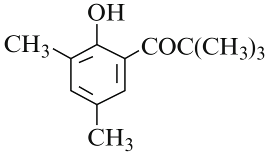
- in the presence of hydrogen chloride (1 mol) and stannic chloride (2 mol) for 24 h (47%), but only (41%) after 6 days of reaction [8300];
- in the presence of aluminium chloride (1.1 mol) for 6 days (29%) [8300].

– Also obtained by Friedel–Crafts acylation of 2,3-dimethylphenol (1 mol) with pivaloyl chloride (1 mol) in the presence of stannic chloride (2 mol) in nitromethane at 20° for 24 h (51%), but only (39%) after 6 days of reaction [8300].

m.p. 38° [8300];
^{1}H NMR (Sadtler: standard n° 35273M) [8300],
IR (Sadtler: standard n° 62641K) [8300], UV [8300], MS [8300].

1-(2-Hydroxy-3,5-dimethylphenyl)-2,2-dimethyl-1-propanone

[72569-15-4] $C_{13}H_{18}O_2$ mol.wt. 206.28



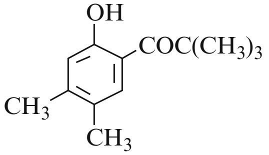
Syntheses

- Obtained by Fries rearrangement of 2,4-dimethylphenyl pivalate (1 mol) in nitromethane at 20° for 6 days,
  - in the presence of hydrogen chloride (1 mol) and stannic chloride (2 mol) (13%) [8300];
  - in the presence of aluminium chloride (1.1 mol) (4%) [8300].
- Also obtained by Friedel–Crafts acylation of 2,4-dimethylphenol (1 mol) with pivaloyl chloride (1 mol) in the presence of stannic chloride (2 mol) in nitromethane at 20° for 6 days (10%) [8300].

m.p. 41° [8300];
^{1}H NMR (Sadtler: standard n° 35274M) [8300],
IR (Sadtler: standard n° 62642K) [8300], UV [8300], MS [8300].

1-(2-Hydroxy-4,5-dimethylphenyl)-2,2-dimethyl-1-propanone

[72569-16-5] $C_{13}H_{18}O_2$ mol.wt. 206.28



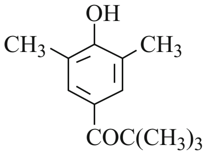
Syntheses

- Obtained by Fries rearrangement of 3,4-dimethylphenyl pivalate (1 mol) in nitromethane at 20° for 6 days,
  - in the presence of hydrogen chloride (1 mol) and stannic chloride (2 mol) (64%) [8300];
  - in the presence of aluminium chloride (1.1 mol) (47%) [8300].
- Also obtained by Friedel–Crafts acylation of 3,4-dimethylphenol (1 mol) with pivaloyl chloride (1 mol) in the presence of stannic chloride (2 mol) in nitromethane at 20° for 6 days (62%) [8300].

m.p. 69° [8300];
^{1}H NMR (Sadtler: standard n° 35275M) [8300],
IR (Sadtler: standard n° 62643K) [8300], UV [8300], MS [8300].

1-(4-Hydroxy-3,5-dimethylphenyl)-2,2-dimethyl-1-propanone

[72569-12-1] $C_{13}H_{18}O_2$ mol.wt. 206.28



Syntheses

- Obtained by Fries rearrangement of 2,6-dimethylphenyl pivalate (1 mol) in nitromethane at 20° for 6 days,
  - in the presence of hydrogen chloride (1 mol) and stannic chloride (2 mol) (42%) [8300];

- in the presence of aluminium chloride (1.1 mol) (3%) [8300].

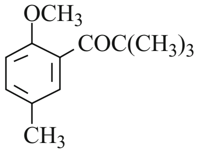
– Also obtained by Friedel–Crafts acylation of 2,6-dimethylphenol (1 mol) with pivaloyl chloride (1 mol) in the presence of stannic chloride (2 mol) in nitromethane at 20° for 6 days (28%) [8300].

N.B.: By irradiation of 2,4,6-trimethylphenyl pivalate (mesityl pivalate) in acetonitrile at 254 nm for 8 h at 0°, there is a photodecarboxylation and formation of 2-tert-butylmesitylene (16%) [8326].

m.p. 67° [8300]; ^{1}H NMR [8300], IR [8300], UV [8300], MS [8300].

1-(2-Methoxy-5-methylphenyl)-2,2-dimethyl-1-propanone

[54696-08-1] $C_{13}H_{18}O_2$ mol.wt. 206.28

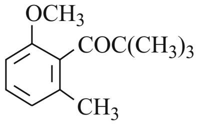


Synthesis

– Refer to: [6672].

1-(2-Methoxy-6-methylphenyl)-2,2-dimethyl-1-propanone

[162052-63-3] $C_{13}H_{18}O_2$ mol.wt. 206.28



Syntheses

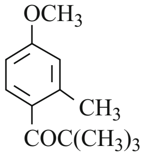
– Obtained by oxidation of 2,2-dimethyl-1-(2-methoxy-6-methylphenyl)-1-propanol with PCC in methylene chloride (90%) [8327].

– Also refer to: [8328].

yellow liquid [8327];
^{1}H NMR [8327], IR [8327], MS [8327]; TLC [8327].

1-(4-Methoxy-2-methylphenyl)-2,2-dimethyl-1-propanone

[2234-19-7] $C_{13}H_{18}O_2$ mol.wt. 206.28



Syntheses

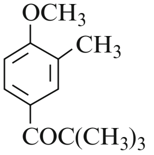
– Obtained by reaction of pivaloyl chloride with 3-methylanisole in petroleum ether in the presence of aluminium chloride, first at 0°, then at 55–60° for 15 min (63%) [8329].

– Also refer to: [7095].

b.p.$_{0.8}$ 100° [7095], b.p.$_4$ 126–128° [8329];
m.p. 73.5–74° [8329]; ^{13}C NMR [7095].

1-(4-Methoxy-3-methylphenyl)-2,2-dimethyl-1-propanone

[181260-67-3] $C_{13}H_{18}O_2$ mol.wt. 206.28



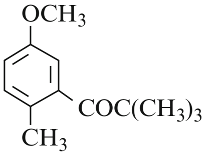
Syntheses

- Obtained by reaction of pivaloyl chloride with 2-methylanisole in petroleum ether in the presence of aluminium chloride, first at 0°, then at 55–60° for 15 min [8329].
- Also obtained by reaction of pivalic anhydride with 2-methylanisole in acetonitrile in the presence of $TiCl(OTf)_3$/TfOH at r.t. for 12 h (64%) [8128].

b.p.$_4$ 127–129° [8329]; m.p. 51–52° [8329].

1-(5-Methoxy-2-methylphenyl)-2,2-dimethyl-1-propanone

[2030-70-8] $C_{13}H_{18}O_2$ mol.wt. 206.28



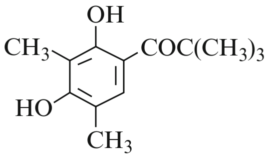
Syntheses

- Obtained by reaction of pivaloyl chloride with 4-methylanisole in petroleum ether in the presence of aluminium chloride, first at 0°, then at 55–60° for 15 min [8329].
- Also refer to: [7095].

b.p.$_4$ 130–132° [8329];
m.p. 51–52° [7095], 50–51° [8329]; ^{13}C NMR [7095].

1-(2,4-Dihydroxy-3,5-dimethylphenyl)-2,2-dimethyl-1-propanone

[267001-74-1] $C_{13}H_{18}O_3$ mol.wt. 222.28



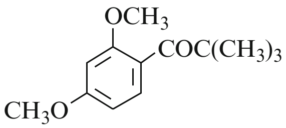
Synthesis

- Obtained by reaction of pivalic acid with 2,4-dimethyl-resorcinol in the presence of boron trifluoride etherate at 120° for 1–2 h under an argon atmosphere. Then, the resulting complex was refluxed for 30 min to 1 h in an aqueous THF (70–80%) [8155].

m.p. 61–62° [8155];
^{1}H NMR [8155], ^{13}C NMR [8155], IR [8155], MS [8155]; TLC [8155].

1-(2,4-Dimethoxyphenyl)-2,2-dimethyl-1-propanone

[60884-08-4] $C_{13}H_{18}O_3$ mol.wt. 222.28



Syntheses

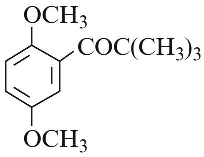
- Preparation by reaction of pivaloyl chloride with 1,3-di-methoxybenzene in the presence of stannic chloride/hydrochloric acid gas in nitromethane at r.t. for 6 days (quantitative yield) [8300].

- Also obtained on hydrolysis of 2-(2,4-dimethoxyphenyl)-2-(1,1-dimethylethyl)-1,3-benzoxathiole [111122-89-5] (m.p. 110–111°) by mercury(II) oxide/35% aqueous tetrafluoroboric acid at r.t. according to [8330], (quantitative yield) [8331].
- Also refer to: [8309].

b.p.$_{12}$ 171–173° [8331], b.p.$_{40}$ 193–194° [8300];
^{1}H NMR (Sadtler: standard n° 30301M) [8300], IR (Sadtler: standard n° 57346) [8300],
UV [8300], MS [8300].

1-(2,5-Dimethoxyphenyl)-2,2-dimethyl-1-propanone

[39868-14-9] $C_{13}H_{18}O_3$ mol.wt. 222.28



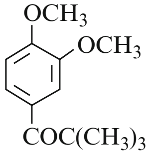
Syntheses

- Preparation by reaction of pivaloyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride in carbon disulfide [7336].
- Also refer to: [8332].

m.p. 46.5–48.5° [8310],
^{1}H NMR [7336], IR [7336], UV [7336], MS [7336].

1-(3,4-Dimethoxyphenyl)-2,2-dimethyl-1-propanone

[30314-46-6] $C_{13}H_{18}O_3$ mol.wt. 222.28

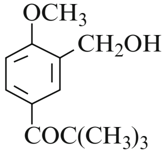


Synthesis

- Obtained by reaction of pivalic anhydride with veratrole in acetonitrile in the presence of TiCl(OTf)$_3$/TfOH at r.t. for 12 h (66%) [8128].

1-[3-(Hydroxymethyl)-4-methoxyphenyl]-2,2-dimethyl-1-propanone

[201150-91-6] $C_{13}H_{18}O_3$ mol.wt. 222.28



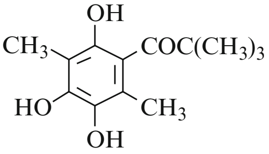
Syntheses

- Refer to: [8333,8334].

USE: Intermediate for the preparation of anticonvulsants [8333,8334].

2,2-Dimethyl-1-(2,4,5-trihydroxy-3,6-dimethylphenyl)-1-propanone

[267001-65-0] $C_{13}H_{18}O_4$ mol.wt. 238.28



Synthesis

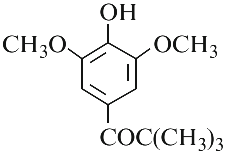
- Obtained by thermal rearrangement of 2,6-dimethyl-4-(trimethylacetyl)-3,6-dihydroxy-2,4-cyclohexadien-1-one in refluxing benzene for 12 h (93%) [8155].

C*aution*: The ketone obtained was stored under argon to prevent its oxidation to 3,6-dimethyl-5-(trimethylacetyl)-2-hydroxy-p-quinone.

yellow glass [8155];
^{1}H NMR [8155], ^{13}C NMR [8155], IR [8155], MS [8155]; TLC [8155].

1-(4-Hydroxy-3,5-dimethoxyphenyl)-2,2-dimethyl-1-propanone

[41247-25-0] $C_{13}H_{18}O_4$ mol.wt. 238.28



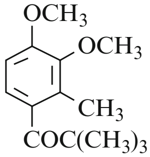
Synthesis

– Obtained by enzymic dehydration of 2,6-dimethoxy-4-(1-hydroxyneopentyl)phenol [38775-76-7] only (44% yield) or in mixture with 4-hydroxy-3,5-dimethoxycinnamyl alcohol [537-33-7] (poor yield) [8335].

oil [8335]; ^{1}H NMR [8335], MS [8335]; TLC [8335].

1-(3,4-Dimethoxy-2-methylphenyl)-2,2-dimethyl-1-propanone

$C_{14}H_{20}O_3$ mol.wt. 236.31



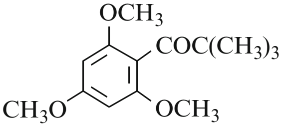
Synthesis

– Obtained by reaction of pivalonitrile with 3,4-dimethoxy-2-methylphenylmagnesium bromide in THF (54%) [7390].

b.p.$_{0.04}$ 81° [7390]; m.p. 28° [7390].

2,2-Dimethyl-1-(2,4,6-trimethoxyphenyl)-1-propanone

[111122-82-8] $C_{14}H_{20}O_4$ mol.wt. 252.31



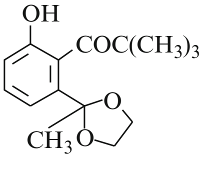
Syntheses

– Obtained on hydrolysis of 2-(1,1-dimethylethyl)-2-(2,4,6-trimethoxyphenyl)-1,3-benzoxathiole [111122-92-0] by mercury(II) oxide/35% aqueous tetrafluoroboric acid at r.t. according to [8330], (quantitative yield) [8331].

m.p. 70–71° [8331]; ^{1}H NMR [8331], IR [8331], MS [8331].

1-[2-Hydroxy-6-(2-methyl-1,3-dioxolan-2-yl)phenyl]-2,2-dimethyl-1-propanone

[171609-26-0] $C_{15}H_{20}O_4$ mol.wt. 264.32

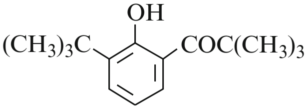


Synthesis

– Refer to: [8336] (Japanese patent).

1-[3-(1,1-Dimethylethyl)-2-hydroxyphenyl]-2,2-dimethyl-1-propanone

[5384-70-3] $C_{15}H_{22}O_2$ mol.wt. 234.34



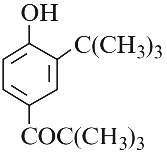
Syntheses

- Obtained by irradiation (450 W Hanovia lamp) of o-tert-butylphenyl pivalate (b.p.$_{0.7}$ 93°) in pentane or benzene solutions (14%) [8337].
- Also obtained by heating 6,11-di-tert-butyl-1,2,4-trioxa-spiro[4,6]undeca-6,8,10-triene (m.p. 78–79°) in methylcyclohexane at 100° for 1 h (60%) [8338].
- Also refer to: [8339].

m.p. 69–69.5° [8337]; IR [8337], UV [8337].

1-[3-(1,1-Dimethylethyl)-4-hydroxyphenyl]-2,2-dimethyl-1-propanone

[5384-64-5] $C_{15}H_{22}O_2$ mol.wt. 234.34



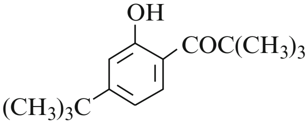
Synthesis

- Obtained by irradiation (450 W Hanovia lamp) of o-tert-butylphenyl pivalate (b.p.$_{0.7}$ 93°) in pentane or benzene solutions (3%) [8337].

m.p. 152–153° [8337]; IR [8337], UV [8337].

1-[4-(1,1-Dimethylethyl)-2-hydroxyphenyl]-2,2-dimethyl-1-propanone

[186962-25-4] $C_{15}H_{22}O_2$ mol.wt. 234.34



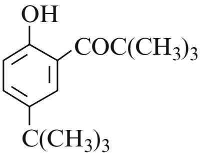
Synthesis

- Obtained by demethylation of 5-tert-butyl-2-pivaloyl-anisole with aluminium chloride in benzene at 50° for 43 h (34%) [8340].

colourless oil [8340]; ^{1}H NMR [8340], ^{13}C NMR [8340], IR [8340].

1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-2,2-dimethyl-1-propanone

[186962-23-2] $C_{15}H_{22}O_2$ mol.wt. 234.34



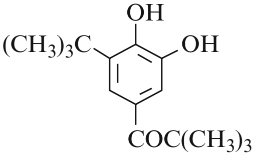
Synthesis

- Obtained by demethylation of 4-tert-butyl-2-pivaloylanisole with aluminium chloride in benzene at 50° for 27 h (63%) [8340].

colourless oil [8340];
^{1}H NMR [8340], ^{13}C NMR [8340], IR [8340].

1-[3-(1,1-Dimethylethyl)-4,5-dihydroxyphenyl]-2,2-dimethyl-1-propanone

[81389-67-5] $C_{15}H_{22}O_3$ mol.wt. 250.34



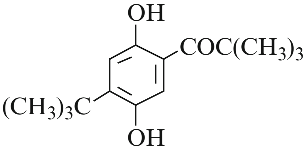
Syntheses

- Preparation by reduction of 4-pivaloyl-2,6-di-tert-butyl-6-hydroperoxy-2,4-cyclohexadienone (m.p. 112°) with dimethyl sulfide at r.t. (quantitative yield) [7727,8231].

m.p. 144–146° [8231]; ^{1}H NMR [8231].

1-[4-(1,1-Dimethylethyl)-2,5-dihydroxyphenyl]-2,2-dimethyl-1-propanone

[35460-03-8] $C_{15}H_{22}O_3$ mol.wt. 250.34



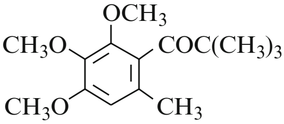
Synthesis

- Obtained by total demethylation of its dimethyl ether with refluxing pyridinium chloride for 2 h (63%) [8310].

m.p. 185–186° [8310]; IR [8310].

2,2-Dimethyl-1-(2,3,4-trimethoxy-6-methylphenyl)-1-propanone

[162052-64-4] $C_{15}H_{22}O_4$ mol.wt. 266.34



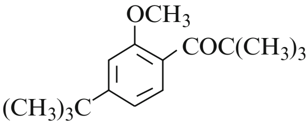
Synthesis

- Obtained by oxidation of 2,2-dimethyl-1-(2,3,4-trimethoxy-6-methylphenyl)-1-propanol (b.p.$_1$ 115–117°) with PCC in methylene chloride (92%) [8327].
- Also refer to: [8328].

m.p. 93–94° [8327]; ^{1}H NMR [8327], IR [8327], MS [8327].

1-[4-(1,1-Dimethylethyl)-2-methoxyphenyl]-2,2-dimethyl-1-propanone

[186962-24-3] $C_{16}H_{24}O_2$ mol.wt. 248.37



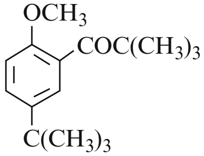
Synthesis

- Obtained by reaction of pivaloyl chloride with 3-tert-butylanisole in the presence of aluminium chloride in carbon disulfide at r.t. for 1 h (17%) [8340].

m.p. 45.5–46° [8340];
^{1}H NMR [8340], ^{13}C NMR [8340], IR [8340].

1-[5-(1,1-Dimethylethyl)-2-methoxyphenyl]-2,2-dimethyl-1-propanone

[186962-22-1] $C_{16}H_{24}O_2$ mol.wt. 248.37



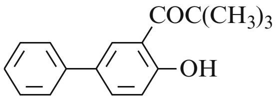
Synthesis

- Obtained by reaction of pivaloyl chloride with 4-tert-butylanisole in the presence of aluminium chloride in carbon disulfide at r.t. for 1 h (25%) [8340].

m.p. 0–31° [8340];
^{1}H NMR [8340], ^{13}C NMR [8340], IR [8340].

1-(4-Hydroxy[1,1′-biphenyl]-3-yl)-2,2-dimethyl-1-propanone

[868266-11-9] $C_{17}H_{18}O_2$ mol.wt. 254.33

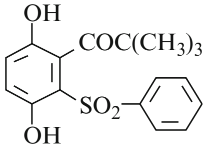


Synthesis

- Refer to: [8223].

1-[3,6-Dihydroxy-2-(phenylsulfonyl)phenyl]-2,2-dimethyl-1-propanone

[145746-56-1] $C_{17}H_{18}O_5S$ mol.wt. 334.39



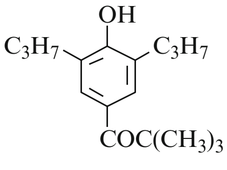
Synthesis

- Preparation by addition of sodium benzenesulfinate in water to pivaloyl-1,4-benzoquinone and trifluoroacetic acid in methylene chloride (66%) [8341].

m.p. 101.5–104° [8341];
^{1}H NMR [8341], IR [8341], MS [8341].

1-(4-Hydroxy-3,5-dipropylphenyl)-2,2-dimethyl-1-propanone

$C_{17}H_{26}O_2$ mol.wt. 262.39

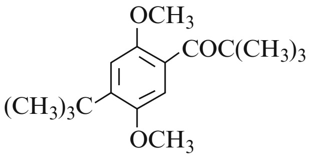


Synthesis

- Preparation by heating a suspension of sodium pivalate and 2,6-dipropylphenol in neat triflic acid [6402].

1-[4-(1,1-Dimethylethyl)-2,5-dimethoxyphenyl]-2,2-dimethyl-1-propanone

[39868-15-0] $C_{17}H_{26}O_3$ mol.wt. 278.39



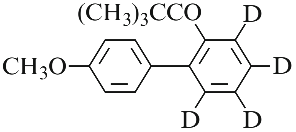
Syntheses

- Preparation by reaction of pivaloyl chloride with 2-tert-butylhydroquinone dimethyl ether in the presence of aluminium chloride in carbon disulfide [7336] or in a mixture of nitrobenzene/tetrachloroethane (6%) [8310].

m.p. 62.5–63.5° [8310];
^{1}H NMR [7336,8310], IR [7336,8310], UV [7336], MS [7336].

1-(4′-Methoxy[1,1′-biphenyl]-2-yl-*3,4,5,6-d*$_4$)-2,2-dimethyl-1-propanone

[852290-70-1] $C_{18}H_{16}D_4O_2$ mol.wt. 272.38

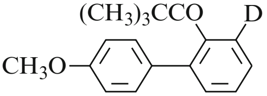


Synthesis

– Refer to: [8342].

1-(4′-Methoxy[1,1′-biphenyl]-2-yl-*3-d*)-2,2-dimethyl-1-propanone

[852290-72-3] $C_{18}H_{19}DO_2$ mol.wt. 269.36

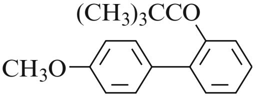


Synthesis

– Refer to: [8342].

1-(4′-Methoxy[1,1′-biphenyl]-2-yl)-2,2-dimethyl-1-propanone

[501374-20-5] $C_{18}H_{20}O_2$ mol.wt. 268.36

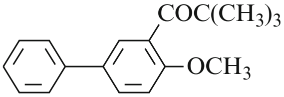


Synthesis

– Refer to: [8342].

1-(4-Methoxy[1,1′-biphenyl]-3-yl)-2,2-dimethyl-1-propanone

[868266-10-8] $C_{18}H_{20}O_2$ mol.wt. 268.36

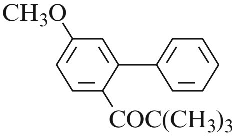


Synthesis

– Refer to: [8223].

1-(5-Methoxy[1,1′-biphenyl]-2-yl)-2,2-dimethyl-1-propanone

[501374-02-3] $C_{18}H_{20}O_2$ mol.wt. 268.36

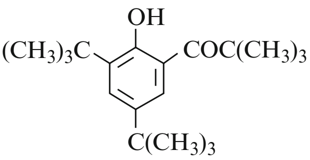


Syntheses

- Obtained from ruthenium-catalyzed phenylation of,
  - 2,4-dimethoxypivalophenone with phenylboronate via a carbon-oxygen bond cleavage (76%) [8318];
  - 4-methoxypivalophenone with phenylboronate [8342] (78%) [8343].

1-[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]-2,2-dimethyl-1-propanone

$C_{19}H_{30}O_2$ mol.wt. 290.45



Synthesis

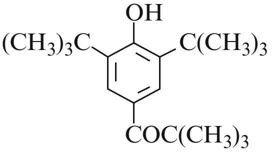
– Refer to: [8344].

Oxime [161429-78-3] $C_{19}H_{31}NO_2$ mol.wt. 305.46.

USE: Cosmetics containing this compound as chelating photoprotectants [8344].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-2,2-dimethyl-1-propanone

[14035-38-2] $C_{19}H_{30}O_2$ mol.wt. 290.45



Syntheses

- Preparation by acylation of 2,6-di-tert-butylphenol with pivalic acid in trifluoroacetic anhydride at r.t. [8231].
- Also refer to: [8234,8345].

m.p. 137–139° [8231].

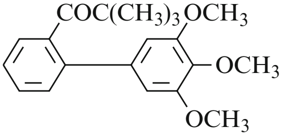
Note: Toxicity to mosquito [8231].

USE: Stabilizer for plastics, oils and fats against heat, light and oxidation [8345].

O-methyloxime [14446-95-8] $C_{20}H_{33}NO_2$ mol.wt. 319.49 [834**6**].

2,2-Dimethyl-1-(3′,4′,5′-trimethoxy[1,1′-biphenyl]-2-yl)-1-propanone

[878555-18-1] $C_{20}H_{24}O_4$ mol.wt. 328.41



Syntheses

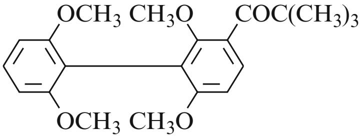
- Obtained from cobalt(II)-catalyzed cross-coupling between 2-fluoropivalophenone and 3,4,5-trimethoxyphenylcopper (formed by transmetallation of 3,4,5-trimethoxyphenylmagnesium chloride with CuCN.2LiCl) at 80° for 1 h (71%) [8347,8348].
- The same reaction realized with 2-chloropivalophenone at 80° for 30 min gives (81%) of the titled ketone [8347].
- Also refer to: [8349].

m.p. <30° [8348];

^{1}H NMR [8348], ^{13}C NMR [8348], IR [8348], MS [8348].

2,2-Dimethyl-1-(2,2′,6,6′-tetramethoxy[1,1′-biphenyl]-3-yl)-1-propanone

[475502-03-5] $C_{21}H_{26}O_5$ mol.wt. 358.43



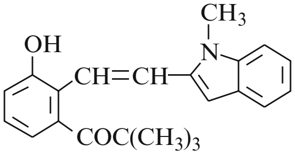
Synthesis

- Preparation by reaction of pivaloyl chloride with 2,2′,6,6′-tetramethoxydiphenyl in the presence of zinc in toluene at 90° for 12 h under nitrogen (65%) [8350].

m.p. 93–94° [8350], ^{1}H NM [8350], ^{13}C NMR [8350].

1-[3-Hydroxy-2-[2-(1-methyl-1*H*-indol-2-yl)ethenyl]phenyl]-2,2-dimethyl-1-propanone

[204569-03-9] $C_{22}H_{23}NO_2$ mol.wt. 333.43

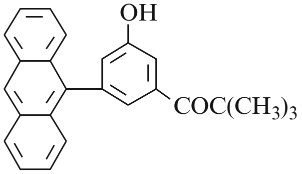


Synthesis

- Refer to: [8351].

1-[3-(9-Anthracenyl)-5-hydroxyphenyl]-2,2-dimethyl-1-propanone

[903583-32-4] $C_{25}H_{22}O_2$ mol.wt. 354.45



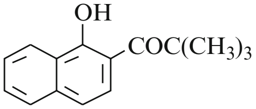
Synthesis

- Refer to: [8352].
  fluorescence spectra [8352].

25.2 Naphthalene Derivatives

1-(1-Hydroxy-2-naphthalenyl)-2,2-dimethyl-1-propanone

[100976-03-2] $C_{15}H_{16}O_2$ mol.wt. 228.29



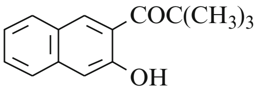
Syntheses

- Preparation by reaction of pivalic acid with α-naphthol in the presence of boron trifluoride at 70° for 2 h (72%) [7775].
- Also obtained by adding tert-butyllithium to 1-hydroxy-2-naphthoic acid at −78°; then the temperature was allowed to rise 25° (42%) [8263].
- Also obtained by Fries rearrangement of 1-naphthyl pivalate with stannic chloride [8353].

m.p. 68° [7775], 66–68° [8353]; IR [8353].

1-(3-Hydroxy-2-naphthalenyl)-2,2-dimethyl-1-propanone

[574001-78-8] $C_{15}H_{16}O_2$ mol.wt. 228.29



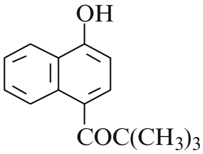
Synthesis

- Refer to: [8354] (compound 8u).

^{1}H NMR [8354], ^{13}C NMR [8354], IR [8354];
TLC [8354].

1-(4-Hydroxy-1-naphthalenyl)-2,2-dimethyl-1-propanone

[882698-66-0] $C_{15}H_{16}O_2$ mol.wt. 228.29



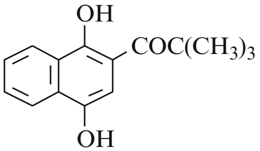
Synthesis

– Refer to: [8355] (Japanese patent).

USE: Preparation of azoquinone compound suitable as electron transport agent for electrophotographic photoconductor to improve sensitivity [8355].

1-(1,4-Dihydroxy-2-naphthalenyl)-2,2-dimethyl-1-propanone

[128462-68-0] $C_{15}H_{16}O_3$ mol.wt. 244.29

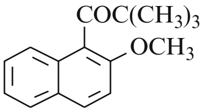


Synthesis

– Obtained by demethylation of the corresponding 4-methyl ether with concentrated hydrobromic acid in acetic acid [7813].

1-(2-Methoxy-1-naphthalenyl)-2,2-dimethyl-1-propanone

[130138-40-8] $C_{16}H_{18}O_2$ mol.wt. 242.32



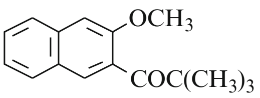
Synthesis

– Obtained by electrochemical acylation of 2-methoxynaphthalene with pivalic anhydride in methylene chloride in the presence of $LiClO_4$ at 35° (22%) [7841].

– Also refer to: [8356].

1-(3-Methoxy-2-naphthalenyl)-2,2-dimethyl-1-propanone

$C_{16}H_{18}O_2$ mol.wt. 242.32

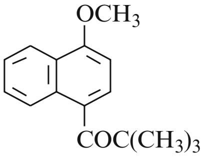


Synthesis

– Obtained by reaction of N-methoxy-N-methyl-2,2-dimethyl-propanamide with 2-methoxynaphthalene (multistage) [8354].

1-(4-Methoxy-1-naphthalenyl)-2,2-dimethyl-1-propanone

[36198-82-0] $C_{16}H_{18}O_2$ mol.wt. 242.32



Syntheses

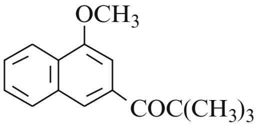
– Preparation by reaction of pivaloyl chloride with 1-methoxy-naphthalene in the presence of aluminium chloride in carbon disulfide [7849], according to the procedure [8268].

– Also obtained by methylation of 1-methoxy-4-isobutyryl-naphthalene [7845], according to the procedure [8357].

m.p. 94° [7845], 92–94° [7849]; ^1H NMR [7849].

1-(4-Methoxy-2-naphthalenyl)-2,2-dimethyl-1-propanone

[177028-19-2] $C_{16}H_{18}O_2$ mol.wt. 242.32



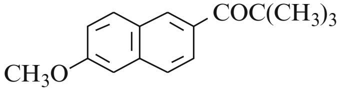
Synthesis

- Obtained (by-product) by reductive coupling of 2-pivaloyl-1,4-dimethoxynaphthalene (m.p. 93–94°) with $(TiCl_3)_2LiAlH_4$ in refluxing THF for 3 h under nitrogen (1%) [8358].

colourless oil [8358]; ^{1}H NMR [8358], MS [8358].

1-(6-Methoxy-2-naphthalenyl)-2,2-dimethyl-1-propanone

[67460-92-8] $C_{16}H_{18}O_2$ mol.wt. 242.32



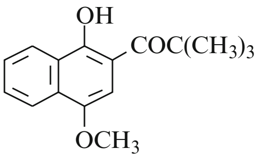
Syntheses

- Obtained by reaction of tert-butyllithium withN,N-dimethyl-6-methoxynaphthalene-2-carboxamide in THF at −78°, followed by quenching with ethyl iodide (73%) [8359].
- Also obtained by action of sodium amide and methyl iodide with 6-isobutyryl-neroline (methylation) (54%) [7855].
- Also obtained by action of tert-butylmagnesium chloride with 2-cyanoneroline in ethyl ether (25%) [7855], according to [8360].
- Also obtained by reaction of pivaloyl chloride with 2-methoxynaphthalene (neroline) in the presence of stannic chloride in nitrobenzene, first at 0°, then at r.t. for 1.5 h (12%) [7855].
- Also refer to: [7802,8356].

b.p.$_{0.5}$ 180–185° [7855]; m.p. 83–84° [7855];
^{1}H NMR [8359], MS [8359].

1-(1-Hydroxy-4-methoxy-2-naphthalenyl)-2,2-dimethyl-1-propanone

[128462-66-8] $C_{16}H_{18}O_3$ mol.wt. 258.32



Syntheses

- Obtained by reaction of 2-pivaloyl-1,3-indandione with diazomethane in ethyl ether (91%) [7813].
- Also obtained by reaction of pivaloyl chloride with 1-hydroxy-4-methoxynaphthalene in the presence of stannic chloride in 1,2-dichloroethane, first at −78° for 15 min, then at r.t. for 2 h under nitrogen (10%) [8358].

Isolation from Natural Sources

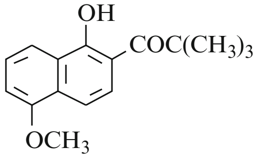
- From the sponge Petrosia seriata [8361].

m.p. 103–104° [8358]; ^{1}H NMR [8358], MS [8358].

BIOLOGICAL ACTIVITY: Natural cardioactive hydroquinone [8361].

1-(1-Hydroxy-5-methoxy-2-naphthalenyl)-2,2-dimethyl-1-propanone

[92920-82-6] $C_{16}H_{18}O_3$ mol.wt. 258.32



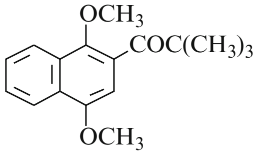
Syntheses

- Obtained by photo-Fries rearrangement of 5-methoxy-1-naphthyl pivalate (m.p. 123–124°) [7864], in methanol [7864,8362].
- Also refer to: [8363].

m.p. 76–77° [7864]; ^{1}H NMR [8363], IR [7864], MS [7864].

1-(1,4-Dimethoxy-2-naphthalenyl)-2,2-dimethyl-1-propanone

[177028-17-0] $C_{17}H_{20}O_3$ mol.wt. 272.34



Synthesis

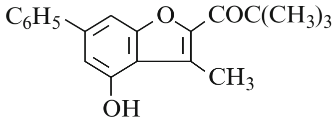
- Preparation: 1.6 M n-butyllithium in hexane was added to a solution of 1,4-dimethoxynaphthalene in THF at 0° under a nitrogen atmosphere, and the mixture stirred at r.t. for 5 h. Then, in nitrogen atmosphere and at −78°, the reaction mixture was added to pivaloyl chloride in THF and stirred 30 min (58%) [8358].

m.p. 93–94° [8358]; ^{1}H NMR [8358], ^{13}C NMR [8358]; TLC [8358].

25.3 Heterocyclic Derivatives

1-(4-Hydroxy-3-methyl-6-phenyl-2-benzofuranyl)-2,2-dimethyl-1-propanone

[687184-57-1] $C_{20}H_{20}O_3$ mol.wt. 308.38



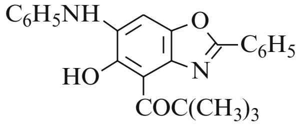
Synthesis

- Refer to: [7968] (compound 11i).

BIOLOGICAL ACTIVITY: Anticancer [7968].

1-[5-Hydroxy-2-phenyl-6-(phenylamino)-4-benzoxazolyl]-2,2-dimethyl-1-propanone

[65908-29-4] $C_{24}H_{22}N_2O_3$ mol.wt. 386.45



Synthesis

- Obtained by heating 2,5-dianilino-3-pivaloyl-1,4-benzoquinone with N-phenylbenzamidine at 120–125° for 1 h (26%) [8364].

m.p. 126° [8364];
^{1}H NMR [8364], IR [8364], UV [8364], MS [8364].

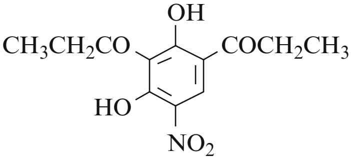
Part XI
Di- and Polyketones

Chapter 26
Aromatic Polyketones Containing Only Propionyl Groups

26.1 Propionyl Groups Located on the Same Ring

1,1′-(2,4-Dihydroxy-5-nitro-1,3-phenylene)bis-1-propanone

[105910-12-1]



$C_{12}H_{13}NO_6$ mol.wt. 267.24

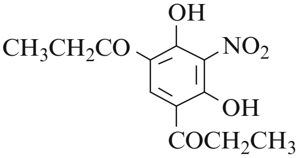
Synthesis

- Obtained (poor yield) by Fries rearrangement of 4-nitroresorcinol dipropionate with aluminium chloride (3.3 equiv) in nitrobenzene, first at 95–100° for 2 h, then at r.t. for 72 h (3%) [6560].

m.p. 132–133° [6560].

1,1′-(4,6-Dihydroxy-5-nitro-1,3-phenylene)bis-1-propanone

[105905-84-8]



$C_{12}H_{13}NO_6$ mol.wt. 267.24

Syntheses

- Preparation by Fries rearrangement of 2-nitroresorcinol dipropionate (m.p. 66°) with aluminium chloride (3.3 equiv) in nitrobenzene at 100° for 3 h (67–80%) or without solvent at 100–110° for 3 h (23%) [6554].
- Also obtained from 4,6-dipropionylresorcinol by treatment with a concentrated sulfuric acid/concentrated nitric acid mixture in acetic acid at 0° for 1 h (75%) [6554].
- Also obtained by Friedel–Crafts acylation of 2-nitroresorcinol with propionic anhydride in nitrobenzene in the presence of aluminium chloride (3.3 equiv) at 120–130° for 3 h (57%) [6554].

m.p. 248° [6554].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
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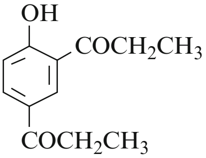
1,1′-(4-Hydroxy-1,3-phenylene)bis-1-propanone

[56481-70-0] $C_{12}H_{14}O_3$ mol.wt. 206.24



Syntheses

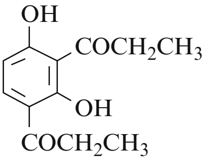
– Preparation by Friedel–Crafts acylation of o-hydroxypropiophenone with propionyl chloride in the presence of aluminium chloride in refluxing carbon disulfide for 2 h (94%) [8365].

– Also refer to: [6351].

m.p. 54–55° [8365], 53° [6351];
^{1}H NMR (Sadtler: standard n° 20025M) [8365], IR (Sadtler: standard n° 47033) [8365].

1,1′-(2,4-Dihydroxy-1,3-phenylene)bis-1-propanone

[2999-19-1] $C_{12}H_{14}O_4$ mol.wt. 222.24



Syntheses

– Preparation by Fries rearrangement of resorcinol dipropionate in the presence of aluminium chloride [6560],

- without solvent, $AlCl_3$ (2 equiv) at 130–135° for 4.5 h [7383] or at 180° for 30 min (55%) [6733], $AlCl_3$ (3 equiv) at 180–185° for 1.5 h (55%) [6745];
- in nitromethane, $AlCl_3$ (2 equiv) at r.t. for 3 h (24%) [6733].

– Preparation by Fries rearrangement of resorcinol dipropionate in the presence of titanium tetrachloride [6733],

- without solvent, $TiCl_4$ (2 or 5 equiv) at 100° for 2 h (63% and 74% yields, respectively) [6733];
- in nitromethane, $TiCl_4$ (2 equiv) at r.t. for 3 h (36%) [6733].

– Also obtained by acylation of resorcinol,

- with propionyl chloride (2 equiv) in nitromethane at r.t. for 5 h in the presence of titanium tetrachloride (5 equiv) (82%) or aluminium chloride (5 equiv) (17%) [6733];
- with propionic anhydride, by heating in the presence of concentrated sulfuric acid (23%) [8366];
- with propionic acid in the presence of boron trifluoride at 140° for 3 h in a sealed tube (poor yield) [6736].

– Also obtained by acylation of respropiophenone,

- with propionyl chloride (2 equiv) in nitromethane at 20° for 24 h in the presence of titanium tetrachloride (5 equiv) (78%) or aluminium chloride (5 equiv) (24%) [6733];

  - with propionic acid in the presence of boron trifluoride at 130–150° in a sealed tube (poor yield) [6736].

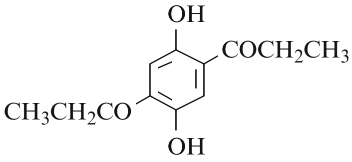
- Also obtained by action of sodium ethoxide in refluxing ethanol for 3 h,
  - with 2,3-dimethyl-7-hydroxy-8-propionylchromone (m.p. 161.5–162°) [8367];
  - with α,β,α′,β′-tetramethyl[benzo-1.6.3.2-di(γ-pyrone)] (m.p. 188–191°) [8367].

- Also refer to: [8368].

b.p.$_{24}$ 184–185° [7383],
m.p. 83° [6733], 82° [8367], 81° [8366], 78–79° [7383], 78° [6736];
^{1}H NMR (Sadtler: standard n° 24406M), IR (Sadtler: standard n° 52518) [6733],
UV [6733].

1,1′-(2,5-Dihydroxy-1,4-phenylene)bis-1-propanone

[91497-44-8] $C_{12}H_{14}O_4$ mol.wt. 222.24



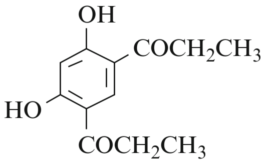
Synthesis

- Obtained (poor yield) by condensation of hydroquinone with propionic acid in xylene in the presence of BF_3-HF by heating on a boiling water bath (7%) [6820].

m.p. 151–152° [6820].

1,1′-(4,6-Dihydroxy-1,3-phenylene)bis-1-propanone

[2999-20-4] $C_{12}H_{14}O_4$ mol.wt. 222.24



Syntheses

- Preparation by Fries rearrangement of resorcinol dipropionate,
  - in the presence of zinc chloride at 130° [8369], (60%) [8367], (40–50%) [8370] or at 180° (33%) [6733];
  - in the presence of aluminium chloride (2 equiv) in nitromethane at r.t. for 2 h (41%) or without solvent at 180° for 30 min (24%) [6733];
  - in the presence of titanium tetrachloride (2 equiv) in nitromethane at r.t. for 2 h (16%) [6733].

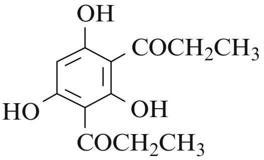
- Also obtained by reaction of propionic anhydride with resorcinol,
  - in the presence of stannic chloride (5 equiv) at reflux for 5 h (63%) [6733];
  - in the presence of zinc chloride (0.5 equiv) at reflux for 30 min (39%) [6733];
  - in the presence of concentrated sulfuric acid at reflux [8366].

- Also obtained by reaction of propionyl chloride with resorcinol in nitromethane at r.t. for 2 h in the presence of aluminium chloride (5 equiv) (29%) or titanium tetrachloride (5 equiv) (10%) [6733].
- Also obtained by reaction of propionyl chloride with respropiophenone in nitromethane at r.t. for 5 h in the presence of aluminium chloride (5 equiv) (44%) or titanium tetrachloride (5 equiv) (19%) [6733].
- Also obtained by reaction of propionic acid with respropiophenone,
  - in the presence of polyphosphoric acid for 10 min in a boiling water bath (34%) [6738];
  - in the presence of zinc chloride (Nencki reaction) [6554].
- Also refer to: [6452,8368,8371–8374].

m.p. 128° [6733], 127° [6554], 125–126° [8367], 125° [6738,8366,8370];
^{1}H NMR (Sadtler: standard n° 24408M), IR (Sadtler: standard n° 52520) [6733],
UV [6733].

1,1′-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-propanone
(Dipropionylphloroglucinol) (Phlorodipropiophenone)

[3145-11-7] $C_{12}H_{14}O_5$ mol.wt. 238.24



Syntheses

- Obtained by reaction of phloroglucinol with propionic anhydride,
  - by heating in the presence of concentrated sulfuric acid [8366];
  - in the presence of boron trifluoride etherate at reflux for 4 h [8375].
- Preparation by Friedel–Crafts acylation of phloroglucinol with propionic acid in the presence of boron trifluoride etherate (53–78%) [8059,8088,8376].
- Also obtained by reaction of 2-propionylphloroglucinol with propionic anhydride in the presence of boron trifluoride at 26–30° for 24–30 h [8377].
- Also refer to: [6888,8089,8378].

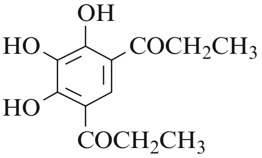
m.p. 152–154° [8375], 137–138° [8366]. One of the reported melting points is obviously wrong.
^{1}H NMR [8059,8088], ^{13}C NMR [8059,8088], IR [8059,8088], UV [8059, 8088].

USE: Fungicide [8377].

BIOLOGICAL ACTIVITY: Antimicrobial against *Bacillus subtilis* [8376]; vesicular stomatitis virus inhibition [8059]; as allergy inhibitor [8089]; for herpes virus control [8379]; as herpes virus inhibitor [8380].

1,1′-(4,5,6-Trihydroxy-1,3-phenylene)bis-1-propanone *(Gallodipropiophenone)* *4,6-Dipropionylpyrogallol*

[3811-86-7] $C_{12}H_{14}O_5$ mol.wt. 238.24



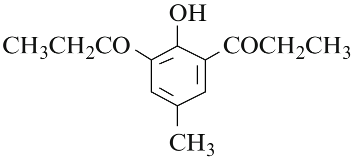
Synthesis

- Obtained by reaction of pyrogallol with propionic anhydride by heating in the presence of concentrated sulfuric acid [8366].

m.p. 186° [8366].

1,1′-(2-Hydroxy-5-methyl-1,3-phenylene)bis-1-propanone

[105290-18-4] $C_{13}H_{16}O_3$ mol.wt. 220.27



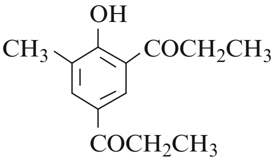
Synthesis

- Refer to: [8381].

Note: Lanthanum, lutetium and yttrium complexes [8381].

1,1′-(4-Hydroxy-5-methyl-1,3-phenylene)bis-1-propanone

[137937-49-6] $C_{13}H_{16}O_3$ mol.wt. 220.27



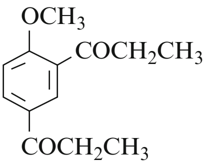
Synthesis

- Preparation by Fries rearrangement of 3-methyl-4-propionoxypropiophenone (m.p. 52–53°) [7070] with aluminium chloride without solvent at 160° for 2 h (80%) [7011].

m.p. 50° [7011]; ^1H NMR [7011], IR [7011], UV [7011], MS [7011].

1,1′-(4-Methoxy-1,3-phenylene)bis-1-propanone

[60278-78-6] $C_{13}H_{16}O_3$ mol.wt. 220.27



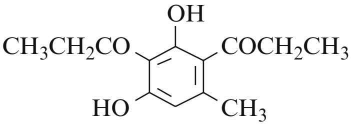
Syntheses

- Preparation by reaction of dimethyl sulfate with 2,4-dipropionylphenol in the presence of 20% sodium hydroxide in methanol for 1.5 h at r.t. (81%) [6733].
- Also refer to: [7611].

m.p. 94° [6733];
^1H NMR (Sadtler: standard 24410M), IR (Sadtler: standard 52523) [6733], UV [6733].

1,1′–(2,4-Dihydroxy-6-methyl-1,3-phenylene)bis-1-propanone

(Dipropionylorcinol)



$C_{13}H_{16}O_4$ mol.wt. 236.27

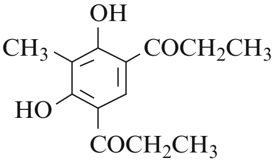
Syntheses

- Preparation by Fries rearrangement of orcinol dipropionate with aluminium chloride at 145–150° for 90 min (60%) [6745,7125].
- Also obtained by condensing propionic anhydride with γ-orcpropiophenone in the presence of aluminium chloride in nitrobenzene [7119,7126].

m.p. 85–86° [6745,7119,7125].

1,1′-(4,6-Dihydroxy-5-methyl-1,3-phenylene)bis-1-propanone

[218591-69-6]

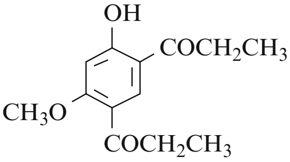


$C_{13}H_{16}O_4$ mol.wt. 236.27

Synthesis

- Refer to: [8368].

1,1′-(4-Hydroxy-6-methoxy-1,3-phenylene)bis-1-propanone



$C_{13}H_{16}O_4$ mol.wt. 236.27

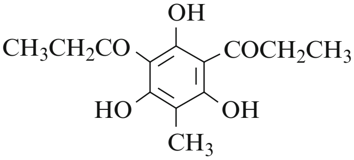
Syntheses

- Obtained by Fries rearrangement of 3-methoxy-4-propionylphenyl propionate [6351].
- Also obtained by partial methylation of 4,6-dipropionyl-resorcinol [6351].
- Also obtained by reaction of propionic acid with 4-hydroxy-2-methoxypropiophenone in the presence of polyphosphoric acid for 10 min in a boiling water bath (31%) [6738].
- Also obtained by reaction of propionic acid with resorcinol monomethyl ether (by-product) in the presence of polyphosphoric acid for 10 min in a boiling water bath (1%) [6738].

m.p. 130° [6351], 127° [6738];
^{1}H NMR (Sadtler: standard n° 30293M), IR (Sadtler: standard n° 57338).

1,1′-(2,4,6-Trihydroxy-5-methyl-1,3-phenylene)bis-1-propanone

[3098-43-9] $C_{13}H_{16}O_5$ mol.wt. 252.27



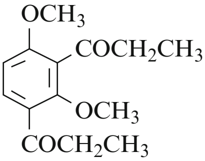
Syntheses

- Preparation by reaction of propionic anhydride with 2-methylphloroglucinol in the presence of boron trifluoride etherate at reflux for 4 h [8375].
- Also refer to: [8382].

m.p. 135–137° [8375].

1,1′-(2,4-Dimethoxy-1,3-phenylene)bis-1-propanone

[60302-89-8] $C_{14}H_{18}O_4$ mol.wt. 250.29



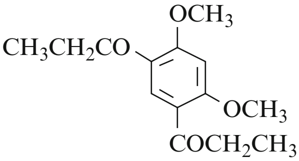
Syntheses

- Preparation by reaction of dimethyl sulfate with 2,4-dipropionylresorcinol in the presence of 20% sodium hydroxide in methanol for 1.5 h at r.t. (88%) [6733].
- Also refer to: [7611].

b.p.$_{18}$ 198° [6733]; m.p. 40.5° [6733];
^{1}H NMR (Sadtler: standard 24407M), IR (Sadtler: standard 52519) [6733], UV [6733].

1,1′-(4,6-Dimethoxy-1,3-phenylene)bis-1-propanone

[60278-79-7] $C_{14}H_{18}O_4$ mol.wt. 250.29



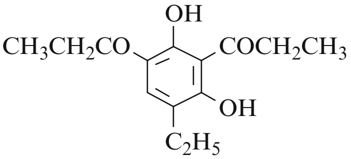
Syntheses

- Preparation by reaction of dimethyl sulfate with 4,6-dipropionylresorcinol in the presence of 20% sodium hydroxide in methanol for 1.5 h at r.t. (35%) [6733].
- Also refer to: [7611].

m.p. 165° [6733];
^{1}H NMR (Sadtler: standard 25359M), IR (Sadtler: standard 52521) [6733], UV [6733].

1,1′-(5-Ethyl-2,4-dihydroxy-1,3-phenylene)bis-1-propanone

$C_{14}H_{18}O_4$ mol.wt. 250.29



Synthesis

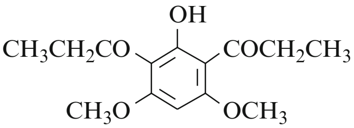
- Obtained by Fries rearrangement of 4-ethylresorcinol dipropionate in the presence of aluminium chloride (2 equiv) without solvent at 60–70° for 3–4 h or in nitrobenzene at 110° for 5 h [7383].

m.p. 81° [7383].

1,1′-(2-Hydroxy-4,6-dimethoxy-1,3-phenylene)bis-1-propanone

[94190-87-1]

$C_{14}H_{18}O_5$ mol.wt. 266.29



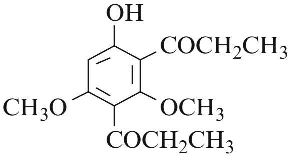
Syntheses

- Obtained by reaction of propionic acid with phloroglucinol dimethyl ether in the presence of polyphosphoric acid at 60° for 1 h (41%) [7438].
- Also refer to: [7439].

m.p. 75–80° [7438]; ^{1}H NMR [7438].

1,1′-(6-Hydroxy-2,4-dimethoxy-1,3-phenylene)bis-1-propanone

$C_{14}H_{18}O_5$ mol.wt. 266.29



Synthesis

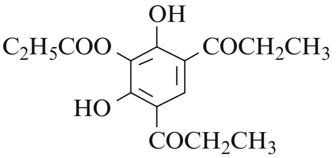
- Obtained by treatment of phloroglucinol dimethyl ether with propionic acid in the presence of polyphosphoric acid at 100° for 15 min (30%) [7514].

m.p. 101° [7514].

1,1′-[4,6-Dihydroxy-5-(1-oxopropoxy)-1,3-phenylene]bis-1-propanone

[52597-50-9]

$C_{15}H_{18}O_6$ mol.wt. 294.30

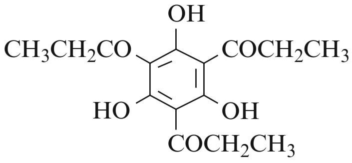


Synthesis

- Obtained by Fries rearrangement of pyrogallol tripropionyl ester with zinc chloride at 130° (40%) [8372].

m.p. 190° [8372]; ^{1}H NMR [8372], IR [8372].

1,1′,1″-(2,4,6-Trihydroxy-1,3,5-benzenetriyl)tris-1-propanone



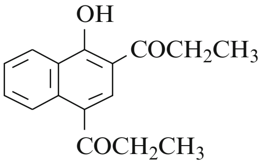
$C_{15}H_{18}O_6$ mol.wt. 294.30

Synthesis

- Obtained by treatment of phloroglucinol with propionic acid in the presence of polyphosphoric acid [8383] at 100° for 10 min (20%) [7514].

m.p. 143° [7514].

1,1′-(1-Hydroxy-2,4-naphthalene)bis-1-propanone



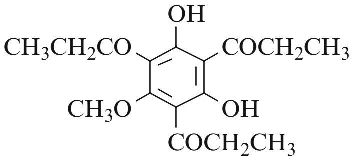
$C_{16}H_{16}O_3$ mol.wt. 256.30

Syntheses

- Preparation by reaction of propionyl chloride with 2-propionyl-1-naphthol,
  - in the presence of zinc chloride in nitrobenzene for 48 h at r.t. (nearly quantitative yield) [7767];
  - in the presence of aluminium chloride in the same conditions (45%) [7767].
- Also obtained (by-product) by Fries rearrangement of 1-naphthyl propionate with aluminium chloride, first at 100° for 2 h, then at 120° for 1 h (2%) [7769].
- Also refer to: [7756].

m.p. 103° [7756,7767], 100–101° [7769].
Na salt [7767].

1,1′,1″-(2,4-Dihydroxy-6-methoxy-1,3,5-benzenetriyl)tris-1-propanone



$C_{16}H_{20}O_6$ mol.wt. 308.33

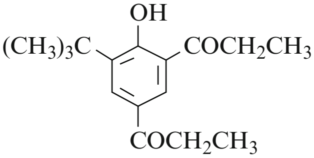
Synthesis

- Obtained by treatment of phloroglucinol monomethyl ether with propionic acid in the presence of polyphosphoric acid [8383] at 100° for 10 min (32%) [7514].

m.p. 104° [7514].

1,1′-[5-(1,1′-Dimethylethyl)-4-hydroxy-1,3-phenylene]bis-1-propanone

[54362-59-3] $C_{16}H_{22}O_3$ mol.wt. 262.35



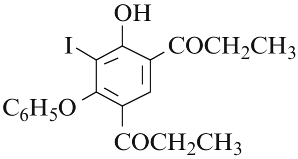
Syntheses

- Obtained (by-product) by Fries rearrangement of 2-tert-butylphenyl propionate in nitromethane at 20° with antimony pentachloride (7%) [6570,7561,7576] or rhenium pentachloride (7%) in the same conditions [7576].

m.p. 64° [6570,7561]; easy sublimation [6570,7561];
^{1}H NMR [6570,7561], IR [6570,7561], UV [6570,7561],
MS [6570,7561]; TLC [6570,7561].

1,1′-(4-Hydroxy-5-iodo-6-phenoxy-1,3-phenylene)bis-1-propanone

[245407-10-7] $C_{18}H_{17}IO_4$ mol.wt. 424.23



Syntheses

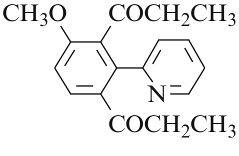
- Obtained by oxidation of 5-propionyl-2,4-dihydroxy-propiophenone [8374] with phenyliodonium diacetate (PIDA) under three conditions (compound 4e) [6452]:

(a) (*basic*): in the presence of potassium hydroxide in methanol at 0° and stirring overnight (75%);
(b) (*neutral*): in refluxing methanol (40%);
(c) (*acidic*): in refluxing acetic acid (60%).

m.p. 119–120° [6452]; ^{1}H NMR [6452], IR [6452]; TLC [6452].

1,1′-[4-Methoxy-2-(2-pyridinyl)-1,3-phenylene]bis-1-propanone

[205983-85-3] $C_{18}H_{19}NO_3$ mol.wt. 297.35



Synthesis

- Obtained by acylation of 2-(3-methoxyphenyl)pyridine (2 mmol) with ethylene (7 atm) and carbon monoxide (20 atm) in the presence of catalytic amounts of $Rh_4(CO)_{12}$ in DMA at 160° for 10 h (7%) [7962].

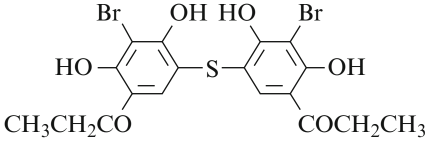
Dark brown solid [7962]; m.p. 148° [7962];
^{1}H NMR [7962], ^{13}C NMR [7962], IR [7962], MS [7962]; GC [7962].

26.2 Propionyl Groups Located on Different Rings

1,1′-[Thiobis(5-bromo-4,6-dihydroxy-3,1-phenylene)]bis-1-propanone

[103096-89-5]

$C_{18}H_{16}Br_2O_6S$ mol.wt. 520.20



Synthesis

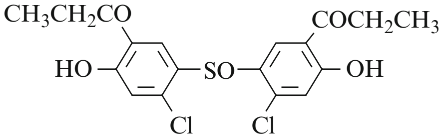
- Obtained by adding a 10% solution of bromine (wt/vol) in acetic acid to a hot solution of 3,3′-dipropionyl-4,4′,6,6′-tetrahydroxy diphenyl thioether in acetic acid [6368].

m.p. 212–213° [6368].

1,1′-[Sulfinylbis(6-chloro-4-hydroxy-3,1-phenylene)]bis-1-propanone

[50444-94-5]

$C_{18}H_{16}Cl_2O_5S$ mol.wt. 415.29



Synthesis

- Obtained by treatment of 4-chloro-2-hydroxypropiophenone with thionyl chloride in the presence of aluminium chloride in carbon disulfide (56%) [6478].

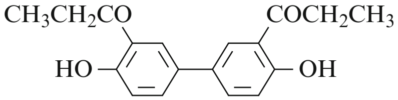
m.p. 219° [6478]; ^{1}H NMR [6478], IR [6478].

USE: Fungicide [6478].

1,1′-(4,4′-Dihydroxy[1,1′-biphenyl]-3,3′-diyl)bis-1-propanone

[36677-69-7]

$C_{18}H_{18}O_4$ mol.wt. 298.34



Syntheses

- Preparation by Fries rearrangement of 4,4′-di-hydroxybiphenyl dipropionate with aluminium chloride without solvent at 120° (55%) [7467] or in refluxing chlorobenzene for 24 h (58%) [8384].
- Preparation by adding 4,4′-dipropionyloxybiphenyl to a melt of sodium chloride-aluminium chloride at 140°. The temperature of the melt was raised rapidly to 200° and maintained there for 2 min (97%) [8385].
- Also refer to: [8386].

m.p. 143–144° [8384,8385], 140–141° [7467]; IR [8384].

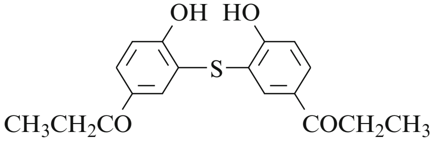
USE: Polymer with 1,3-propanediamine [386704-18-3] [8387]; polymer with 1,2-benzenediamine [401843-27-4] [8388]; polymer with 3-methoxy-1,2-benzenediamine [568600-66-8] [8389].

BIOLOGICAL ACTIVITY: Antibacterial against a cariogenic bacterium [8386].

1,1′-[Thiobis(6-hydroxy-3,1-phenylene)]bis-1-propanone

[95125-20-5]

$C_{18}H_{18}O_4S$ mol.wt. 330.40



Synthesis

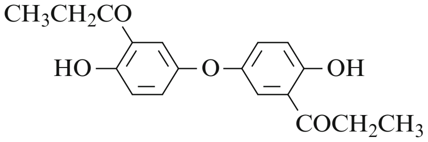
- Obtained by reaction of thionyl chloride or sulfur dichloride with p-hydroxy-propiophenone in the presence of copper powder at r.t. overnight [6550].

m.p. 103° [6550].

1,1′-[Oxybis(4-hydroxy-3,1-phenylene)]bis-1-propanone

[880134-95-2]

$C_{18}H_{18}O_5$ mol.wt. 314.34



Synthesis

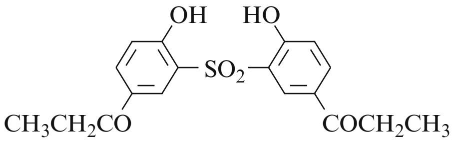
- Preparation by Fries rearrangement of 4,4′-dihydroxydiphenyl oxide dipropionate with aluminium chloride at 150° for 30 min (99%) [8390].

^{1}H NMR [8390], ^{13}C NMR [8390], IR [8390].

1,1′-[Sulfonylbis(6-hydroxy-3,1-phenylene)]bis-1-propanone

[95699-99-3]

$C1_8H_{18}O_6S$ mol.wt. 362.40



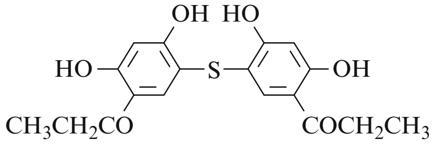
Synthesis

- Obtained by oxidation of 2,2′-dihydroxy-5,5′-dipropionyldiphenyl sulfide with hydrogen peroxide (100 vols.) in acetone at r.t. overnight (73%) [6550].

m.p. 105° (d) [6550].

1,1′-[Thiobis(4,6-dihydroxy-3,1-phenylene)]bis-1-propanone

[103154-02-5] $C_{18}H_{18}O_6S$ mol.wt. 362.40



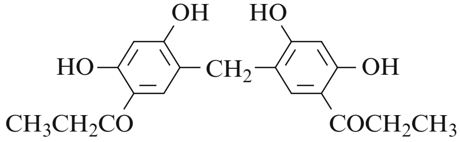
Syntheses

- Obtained by reaction of thionyl chloride with respropiophenone in chloroform in the presence of finely divided copper at r.t. overnight (31%) [6368].
- The same thioether was obtained by interaction of the respropiophenone and sulfur monochloride or sulfur dichloride in ethyl ether, first at 5°, then at r.t. for 1 h (9% and 21% yields, respectively) [6368].

m.p. 161–162° [6368].

1,1′-[Methylenebis(4,6-dihydroxy-3,1-phenylene)]bis-1-propanone

[124300-19-2] $C_{19}H_{20}O_6$ mol.wt. 344.36



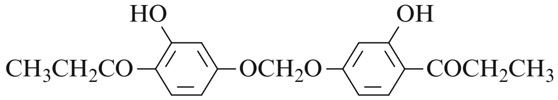
Synthesis

- Obtained (poor yield) by reaction of ethoxymethyl chloride with respropiophenone in the presence of potassium carbonate in acetone at 60° for 3 h (6%) [6757].

m.p. 163° [6757]; TLC [6757].

1,1′-[Methylenebis[oxy(2-hydroxy-4,1-phenylene)]]bis-1-propanone

[66047-39-0] $C_{19}H_{20}O_6$ mol.wt. 344.36



Synthesis

- Obtained by condensation of respropiophenone with methylene dibromide or methylene dichloride (25%) [8391].

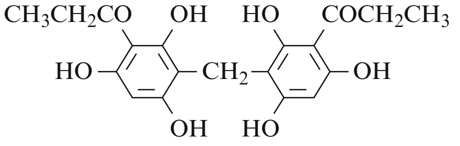
m.p. 142° [8391].

Dioxime [66047-44-7] $C_{19}H_{22}N_2O_6$ mol.wt. 374.39 [8391].

1,1′-[Methylenebis(2,4,6-trihydroxy-3,1-phenylene)]bis-1-propanone

Bis(3-propionyl-2,4,6-trihydroxyphenyl)methane

[2828-37-7]



$C_{19}H_{20}O_8$ mol.wt. 376.36

Synthesis

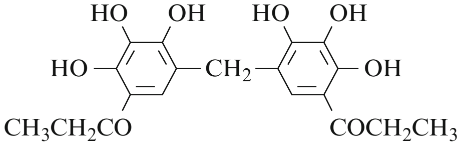
- Obtained by reaction of 35% formaldehyde solution with phloropropiophenone in ethanol in the presence of concentrated sulfuric acid at r.t. for 30 min [8392].

m.p. 300° (d) [8392].

1,1′-[Methylenebis(4,5,6-trihydroxy-3,1-phenylene)]bis-1-propanone

2,2′,3,3′,4,4′-Hexahydroxy-5,5′-dipropionyldiphenylmethane

[124300-24-9]



$C_{19}H_{20}O_8$ mol.wt. 376.36

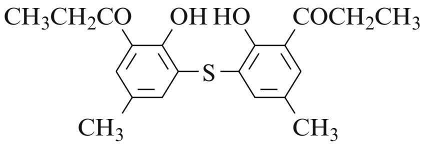
Syntheses

- Obtained by reaction of ethoxymethyl chloride with 2,3,4-trihydroxy-propiophenone in the presence of potassium carbonate in acetone at 60° for 3 h (35%) [6757].
- Preparation by reaction of 35% aqueous formaldehyde with 4-propionylpyrogallol in the presence of hydrochloric acid in refluxing ethanol (90%) [6454].

m.p. 242–243° [6757], 238° [6454];
^{1}H NMR [6757], ^{13}C NMR [6757], IR [6757], UV [6757]; TLC [6757].

1,1′-[Thiobis(2-hydroxy-5-methyl-3,1-phenylene)]bis-1-propanone

[103863-52-1]



$C_{20}H_{22}O_4S$ mol.wt. 358.46

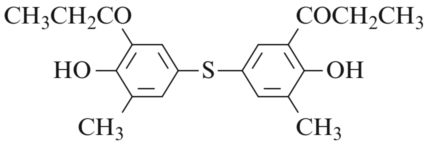
Syntheses

- Preparation from 2-hydroxy-5-methyl-propiophenone,
  - with refluxing thionyl chloride in the presence of copper powder for 3 h (61%) [7049,8393];
  - with refluxing sulfur monochloride or sulfur dichloride in the presence of copper powder for 3 h (15%) [7049,8393].

m.p. 110° [7049,8393].

1,1′-[Thiobis(4-hydroxy-5-methyl-3,1-phenylene)]bis-1-propanone

[103858-21-5] $C_{20}H_{22}O_4S$ mol.wt. 358.46



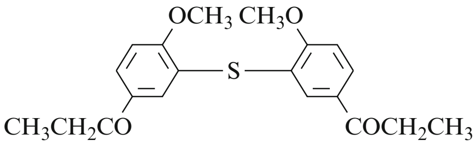
Syntheses

- Preparation, first, gradually adding copper powder to the mixture of 2-hydroxy-3-methylpropiophenone and thionyl chloride, then refluxing the mixture for 3 h [7017].
- Also obtained by allowing 2-hydroxy-3-methylpropiophenone, sulfur dichloride and copper powder to react overnight at r.t. [7017].

m.p. 98° [7017].

1,1′-[Thiobis(6-methoxy-3,1-phenylene)]bis-1-propanone

[97921-47-6] $C_{20}H_{22}O_4S$ mol.wt. 358.46



Synthesis

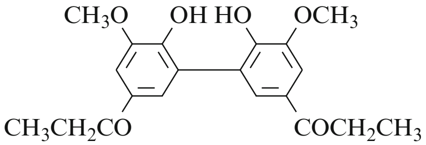
- Obtained by treatment of p-methoxy-propiophenone with thionyl chloride or sulfur dichloride in the presence of copper powder on a boiling water bath for 15 min and at r.t. overnight [7003].

m.p. 175° [7003].

1,1′-(6,6′-Dihydroxy-5,5′-dimethoxy[1,1′-biphenyl]-3,3′-diyl)bis-1-propanone

4,4′-Dipropionyl-6,6′-biguaiacol; Dehydrodipropioguaiacone

[18592-97-7] $C_{20}H_{22}O_6$ mol.wt. 358.39



Syntheses

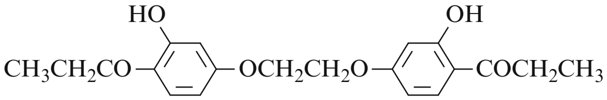
- Obtained by enzymic dehydrogenation of propioguaiacone with horseradish peroxidase in water in the presence of hydrogen peroxide at 30° for 1 h (60%) (compound 10) [7161].
- Also obtained by oxidation of 4-hydroxy-3-methoxypropiophenone in the HSO_3F-PbO_2 system [8394].
- Also refer to: [8395].

m.p. 207–208° [7161]; UV [7161]; photoelectron spectrum [7212]; Raman spectroscopy [8396]; ionization potential [7232].

USE: As UV-absorbers for sunscreens and other products [8397]; as active oxygen scavengers for therapeutic uses [8398].

1,1′-[1,2-Ethanediylbis[oxy(2-hydroxy-4,1-phenylene)]]bis-1-propanone

[66047-41-4] $C_{20}H_{22}O_6$ mol.wt. 358.39



Synthesis

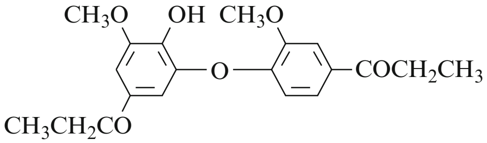
- Obtained by condensation of respropiophenone with ethylene dibromide or ethylene dichloride (25%) [8391].

m.p. 165° [8391].
Dioxime [66047-46-9] $C_{20}H_{24}N_2O_6$ mol.wt. 388.42 [8391].

1-[4-Hydroxy-3-methoxy-5-[2-methoxy-4-(1-oxopropyl)phenoxy] phenyl]-1-propanone

2-Hydroxy-3-methoxy-5-propionyl 2′-methoxy-4′-propionylphenyl ether
4′-Hydroxy-3‴,5′-dimethoxy-3′,4‴-oxydipropiophenone

[16737-81-8] $C_{20}H_{22}O_6$ mol.wt. 358.39



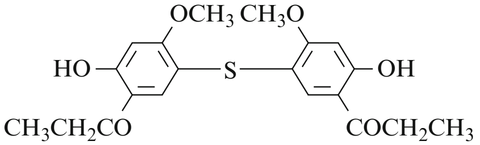
Syntheses

- Isolation from the dehydrogenation resin of propioguaiacone through preparative TLC (6%) (compound 14) [7161].
- Also obtained by dehydrogenation of propioguaiacone in aqueous solution with hydrogen peroxide (small amount) [8399].

^{1}H NMR [7161], IR [7161], UV [7161].

1,1′-[Thiobis(4-hydroxy-6-methoxy-3,1-phenylene)]bis-1-propanone

[103509-21-3] $C_{20}H_{22}O_6S$ mol.wt. 390.46



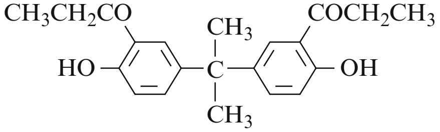
Syntheses

- Obtained by methylating 3, 3′-dipropionyl-4,4′,6,6′-tetra hydroxydiphenyl thioether with dimethyl sulfate (1:1) in the presence of alkali (unspecified) at 100° [6368].
- Also obtained by reaction of thionyl chloride with 2-hydroxy-4-methoxypropiophenone in the presence of copper powder in ethyl ether at r.t. overnight (38%) [6368].
- Also obtained by reaction of sulfur monochloride or sulfur dichloride with 2-hydroxy-4-methoxy-propiophenone in the presence of copper powder in ethyl ether at r.t. overnight (13% and 26% yields, respectively) [6368].

m.p. 202–203° [6368].

1,1′-[(Methylethylidene)bis(4-hydroxy-3,1-phenylene)]bis-1-propanone

[3511-70-4] $C_{21}H_{24}O_4$ mol.wt. 340.42



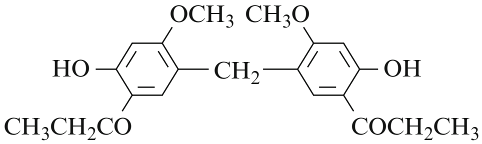
Synthesis

– Obtained by Fries rearrangement of 4,4′-di-(propionoxy)diphenyldimethylmethane also named 2,2-bis[(4-propionyloxy)phenyl]-propane with aluminium chloride in nitrob-enzene at 120–130° for 3 h (23%) [8400].

m.p. 150–151° [8400].

1,1′-[Methylenebis(6-hydroxy-4-methoxy-3,1-phenylene)]bis-1-propanone

[124300-15-8] $C_{21}H_{24}O_6$ mol.wt. 372.42



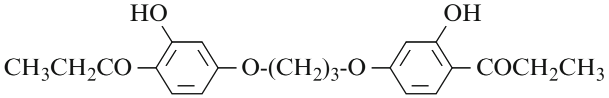
Synthesis

– Obtained by reaction of ethoxymethyl chloride with 2-hydroxy-4-methoxy-propiophenone in the presence of potassium carbonate in acetone at 60° for 24 h (20%) [6757].

m.p. 170° [6757];
^{1}H NMR [6757], IR [6757], UV [6757]; TLC [6757].

1,1′-[1,3-Propanediylbis[oxy(2-hydroxy-4,1-phenylene)]]bis-1-propanone

[66047-42-5] $C_{21}H_{24}O_6$ mol.wt. 372.42

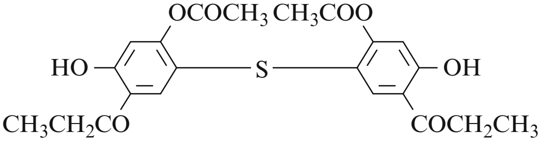


Synthesis

– Obtained by condensation of respropiophenone with 1,3-dibromopropane or 1,3-dichloropropane (18%) [8391].

m.p. 151° [8391].
Dioxime [66047-47-0] $C_{21}H_{26}N_2O_6$ mol.wt. 402.45 [8391].

1,1′-[Thiobis[6-(acetyloxy)-4-hydroxy-3,1-phenylene]]bis-1-propanone



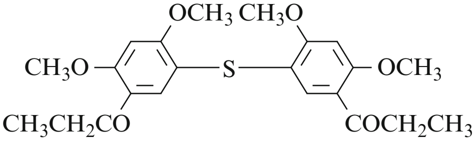
$C_{22}H_{22}O_8S$ mol.wt. 446.48

Synthesis

- Obtained by partial acetylation of 3,3′-dipropionyl-4,4′,6,6′-tetrahydroxydiphenyl thioether with acetic anhydride in the presence of pyridine [6368].

m.p. 216–217° [6368].

1,1′-[Thiobis(4,6-dimethoxy-3,1-phenylene)]bis-1-propanone



$C_{22}H_{26}O_6S$ mol.wt. 418.51

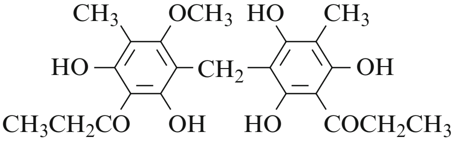
Synthesis

- Obtained by methylating 3,3′-dipropionyl-4,4′,6,6′-tetrahydroxydiphenyl thioe ther with excess of dimethyl sulfate in the presence of alkali (unspecified) at 100° [6368].

m.p. 197–198° [6368].

1-[3-[[2,4-Dihydroxy-6-methoxy-5-methyl-3-(1-oxopropyl)phenyl]methyl]-2,4,6-trihydroxy-5-methylphenyl]-1-propanone *(Margaspidin PP)*

[57765-49-8]



$C_{22}H_{26}O_8$ mol.wt. 418.44

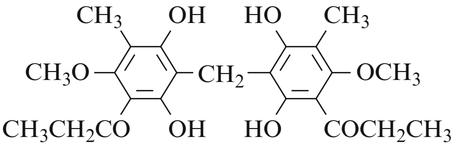
Isolation from natural sources

- From *Dryopteris bissetiana* and Dryopteris sacrosancta of Japan [7262].
- From *Dryopteris marginata* (WALL.) CHRIST and *Dryopteris inaequalis* [8401].

m.p. 176° [8401], 169–173° [7262]; MS [7262,8401]; TLC [8401].

1,1′-[Methylenebis(2,6-dihydroxy-4-methoxy-5-methyl-3,1-phenylene)]bis-1-propanone

[49582-12-9]



$C_{23}H_{28}O_8$ mol.wt. 432.47

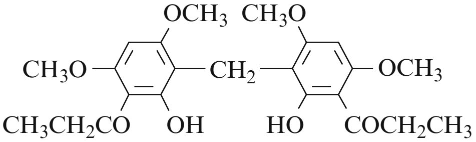
Isolation from natural sources

- From *Dryopteris* species of Japan [7262].

MS [7393]; TLC [7262]; paper chromatography [7262].

1,1′-[Methylenebis(2-hydroxy-4,6-dimethoxy-3,1-phenylene)]bis-1-propanone

[124300-29-4]



$C_{23}H_{28}O_8$ mol.wt. 432.47

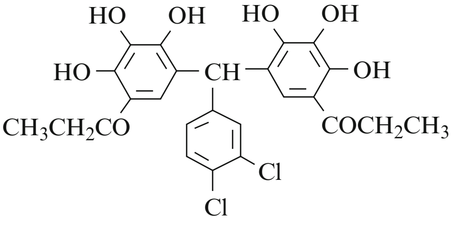
Synthesis

- Preparation by reaction of ethoxymethyl chloride with 2-hydroxy-4,6-dimethoxy-propiophenone in the presence of potassium carbonate in acetone at 60° for 4 h (91%) [6757].

m.p. 211–212° [6757]; ^{1}H NMR [6757], IR [6757], UV [6757]; TLC [6757].

1,1′-[[(3,4-Dichlorophenyl)methylene]bis(4,5,6-trihydroxy-3,1-phenylene)]bis-1-propanone

3″,4″-Dichloro-2,2′3,3′,4,4′-hexahydroxy-5,5′-dipropionyltriphenylmethane



$C_{25}H_{22}Cl_2O_8$ mol.wt. 521.35

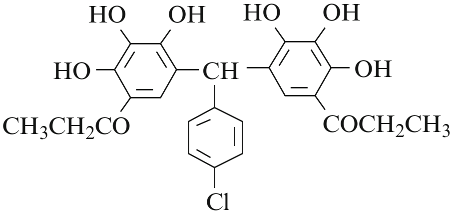
Synthesis

- Obtained by reaction of 3,4-dichloro-benzaldehyde with 4-propionylpyrogallol in the presence of hydrochloric acid in refluxing ethanol for 30 min [6454].

m.p. 209–210° [6454].

1,1′-[[(4-Chlorophenyl)methylene]bis(4,5,6-trihydroxy-3,1-phenylene)]bis-1-propanone

4″-Chloro-2,2′,3,3′,4,4′-hexahydroxy-5,5′-dipropionyltriphenylmethane



$C_{25}H_{23}ClO_8$ mol.wt. 486.91

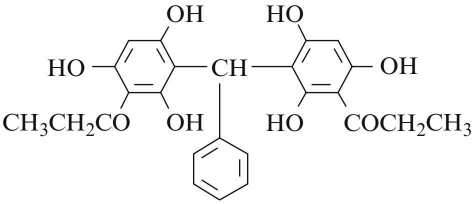
Synthesis

- Obtained by reaction of 4-chlorobenzaldehyde with 4-propionylpyrogallol in the presence of hydrochloric acid in refluxing ethanol for 30 min [6454].

m.p. 189–190° [6454].

1,1′-[(Phenylmethylene)bis(2,4,6-trihydroxy-3,1-phenylene)]bis-1-propanone

[147170-16-9]



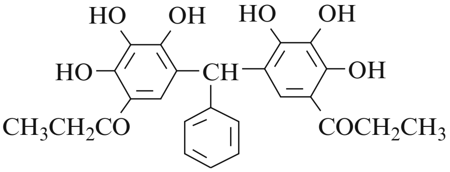
$C_{25}H_{24}O_8$ mol.wt. 452.46

Synthesis

- Refer to: [8402] (Japanese patent).

1,1′-[(Phenylmethylene)bis(4,5,6-trihydroxy-3,1-phenylene)]bis-1-propanone

2,2′,3,3′,4,4′-Hexahydroxy-5,5′-dipropionyltriphenylmethane



$C_{25}H_{24}O_8$ mol.wt. 452.46

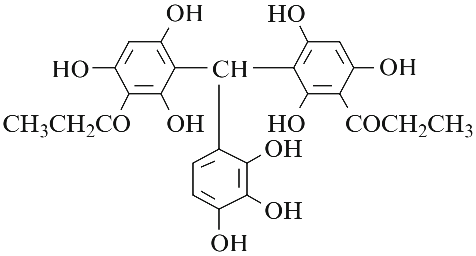
Synthesis

- Obtained by reaction of benzaldehyde with 4-propionylpyrogallol in the presence of hydrochloric acid in refluxing ethanol for 30 min [6454].

m.p. 162° [6454].

1,1′-[[(2,3,4-Trihydroxyphenyl)methylene]bis(2,4,6-trihydroxy-3,1-phenylene)]bis-1-propanone

[143868-75-1]



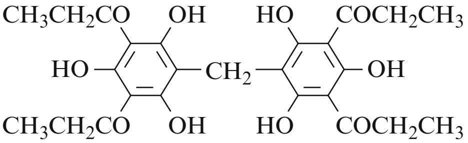
$C_{25}H_{24}O_{11}$ mol.wt. 500.46

Synthesis

- Refer to: [8403] (Japanese patent).

1,1′,1″,1‴-[Methylenebis(2,4,6-trihydroxy-5,1,3-benzenetriyl)]tetrakis-1-propanone

[68223-29-0]



$C_{25}H_{28}O_{10}$ mol.wt. 488.49

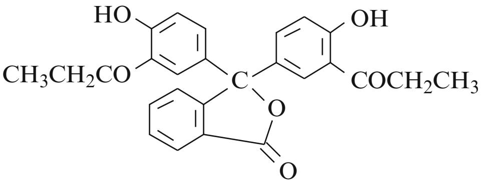
Syntheses

- Obtained by condensation of 2,4-di-propionylphloroglucinol with 40% formaldehyde [8404], (58–65%) [6879] or with methoxymethyl acetate in acetic acid in the presence of few drops of concentrated sulfuric acid [8405].

m.p. 235–237° [6879].

BIOLOGICAL ACTIVITY: Antischistosomal (an analog of agrimophol) [6879].

3,3-Bis[4-hydroxy-3-(1-oxopropyl)phenyl]phthalide



$C_{26}H_{22}O_6$ mol.wt. 430.46

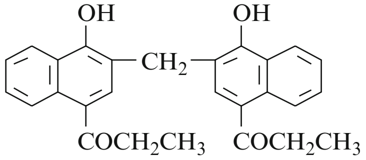
Synthesis

- Obtained by Fries rearrangement of 3,3-bis[4-(propionyloxy)phenyl]phthalide with aluminium chloride in nitrobenzene at 120–130° for 3 h (20%) [8400].

m.p. 199–200° [8400].

1,1′-[Methylenebis(1-hydroxy-4,2-naphthalenediyl)]bis-1-propanone

[76288-10-3] $C_{27}H_{24}O_4$ mol.wt. 412.49



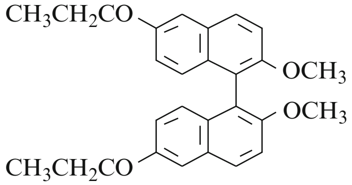
Synthesis

- Refer to: [7789].

Dioxime [76288-07-8] $C_{27}H_{26}N_2O_4$ mol.wt. 442.51 [7789].

1,1′-[(1*R*)-2,2′-Dimethoxy[1,1′-binaphthalene]-6,6′-diyl]bis-1-propanone

[373388-81-9] $C_{28}H_{26}O_4$ mol.wt. 426.51



Synthesis

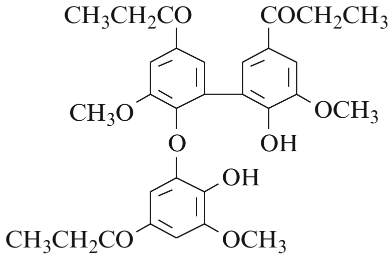
- Refer to: [6445].

1-[2′-Hydroxy-6-(2-hydroxy-3-methoxy-5-propionylphenoxy)-5,3′-dimethoxy-5′-propionyl[1,1′-biphenyl]-3-yl]-1-propanone

4′-Hydroxy-4‴-(2-hydroxy-3-methoxy-5-propionylphenoxy)-5′,5‴-dimethoxy-3′,3‴-bipropiophenone

3,3′-Dimethoxy-2′-hydroxy-5,5′-dipropionylbiphenyl-2-yl 2″-hydroxy-3″-methoxy-5″-propionylphenyl ether

[18593-02-7] $C_{30}H_{32}O_9$ mol.wt. 536.58



Syntheses

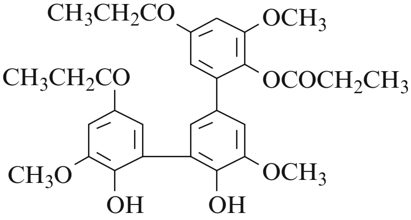
- Isolation from the dehydrogenation resin of propioguaiacone through preparative TLC (trace amounts) (compound 15) [7161].
- Also obtained by dehydrogenation of propioguaiacone in aqueous solution with hydrogen peroxide (small amount) [8399].

m.p. 235–236° [7161];
^{1}H NMR [7161], IR [7161], UV [7161].

Propionic acid 4′,2″-dihydroxy-3,5′,3″-trimethoxy-5,5″-dipropionyl-[1,1′:3′,1″]terphenyl-2-yl ester

2,2″,4′-Triol-3,3″,5′-trimethoxy-5,5″-dipropionyl[m-terphenyl] 2-propionate
4′,2″-Dihydroxy-5,5″-dipropionyl-2-propionoxy-3,5′,3″-trimethoxyterphenyl

[16737-80-7]



$C_{30}H_{32}O_9$ mol.wt. 536.58

Syntheses

- Isolation from the dehydrogenation resin of propioguaiacone through preparative TLC (62%) (compound 11) [7161].
- Also obtained by dehydrogenation of propioguaiacone in aqueous solution with hydrogen peroxide (major compound) [8399].

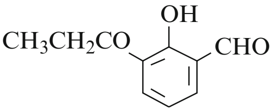
^{1}H NMR [7161], IR [7161], UV [7161].

Chapter 27
Aromatic Polyketones Containing At Least One Propionyl Group

27.1 Carbonyl Groups Located on the Same Ring

2-Hydroxy-3-(1-oxopropyl)benzaldehyde

[35888-90-5] $C_{10}H_{10}O_3$ mol.wt. 178.19



Syntheses

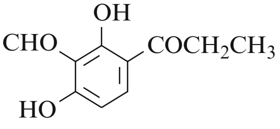
- Preparation by ozonization of 2-hydroxy-3-propenyl-propiophenone in an acetic acid-formic acid mixture at 0° (67%) [6935].

- Also refer to: [6933].

2,6-Dihydroxy-3-(1-oxopropyl)benzaldehyde

2,4-Dihydroxy-3-formylpropiophenone

$C_{10}H_{10}O_4$ mol.wt. 194.19



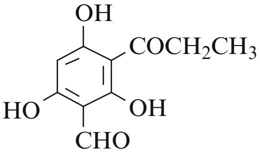
Synthesis

- Obtained by adding zinc cyanide, potassium chloride, followed by aluminium chloride in ethyl ether to a solution of respropiophenone in ethyl acetate, then passing hydrogen chloride into the mixture for 30 min (64%) [6768].

m.p. 140–141° [6768].

2,4,6-Trihydroxy-3-(1-oxopropyl)benzaldehyde

[96573-29-4] $C_{10}H_{10}O_5$ mol.wt. 210.19



Syntheses

- Preparation by reaction of ethyl orthoformate with phloropropiophenone in methylene chloride in the presence of aluminium chloride cooling in an ice bath for 30 min [8378,8406] (70%) [7007].

N.B.: Compound 27 [8406], compound 5 or 9 [8378] and compound 17 [7007].

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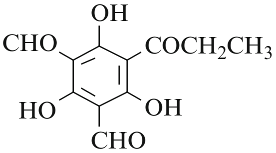
- Also obtained by reaction of phosphorous oxychloride with phloropropiophenone in DMF at r.t. (70%) [6882].

m.p. 158–160° [7007]; ^{1}H NMR [7007], IR [7007], MS [7007].

2,4,6-Trihydroxy-5-(1-oxopropyl)-1,3-benzenedicarboxaldehyde

[96573-42-1] $C_{11}H_{10}O_6$ mol.wt. 238.20



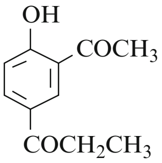
Synthesis

- Refer to: [7007] (compound 34).

m.p. 215° [7007];
^{1}H NMR [7007], IR [7007], MS [7007].

1-(3-Acetyl-4-hydroxyphenyl)-1-propanone

[79010-36-9] $C_{11}H_{12}O_3$ mol.wt. 192.21



Syntheses

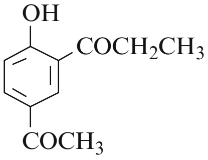
- Preparation by Fries rearrangement of 4-propionylphenyl acetate (1 mol) with aluminium chloride (3.6 mol) at 150° for 3 h (80%) [8407].
- Preparation by reaction of propionyl chloride with 2-hydroxy-acetophenone in carbon disulfide in the presence of aluminium chloride, first at r.t. for 1 h, then at reflux for 1 h (95%) [8408].
- Also refer to: [8409,8410].

m.p. 69° [8407,8411], 68–69° [8408]; ^{1}H NMR [8407], IR [8407].

PHARMACOLOGICAL DATA [8407].

1-(5-Acetyl-2-hydroxyphenyl)-1-propanone

[36039-26-6] $C_{11}H_{12}O_3$ mol.wt. 192.21



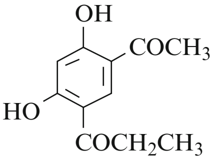
Syntheses

- Preparation by Fries rearrangement of p-(propionyloxy)-acetophenone with aluminium chloride (4 mol) without solvent at 150° for 3 h (62%) [8412].
- Also obtained by Friedel–Crafts acylation of p-hydroxyacetophenone with propionyl chloride in the presence of aluminium chloride (4 mol) in tetrachloroethane at 130° for 4 h (47%) [8412].
- Also obtained by Friedel–Crafts acylation of o-hydroxypropiophenone with acetyl chloride in the presence of aluminium chloride in refluxing carbon disulfide (90–95%) [8413], (90%) [8414], (72%) [8415].

- Also obtained by deacylation of 2-(DL-2′-acetoxypropionyloxy)-5-acetylpropiophenone (24%) [8416].

b.p.$_{4\text{-}5}$ 175–180° [8415];
m.p. 67–69° [8412], 64–65° [8415], 64° [8413];
^{1}H NMR [8412], IR [8412].

1-(5-Acetyl-2,4-dihydroxyphenyl)-1-propanone



$C_{11}H_{12}O_4$ mol.wt. 208.21

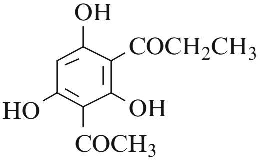
Synthesis

- Obtained by reaction of propionic acid with resacetophenone (m.p. 142°) in the presence of polyphosphoric acid [8383] for 10 min in a boiling water bath (32%) [6738].

m.p. 122° [6738].

1-(3-Acetyl-2,4,6-trihydroxyphenyl)-1-propanone

[3118-35-2]



$C_{11}H_{12}O_5$ mol.wt. 224.21

Syntheses

- Preparation by Friedel–Crafts acylations of phloroglucinol [8375,8406] according to the method (i) [7007].
- Preparation by reaction of propionic anhydride with 2-acetylphloroglucinol in the presence of boron trifluoride in ethyl ether at −2° to 5° for 24–30 h [8417].
- Also refer to: [7007,8406].

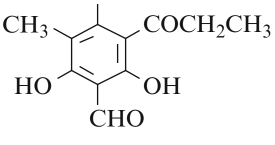
m.p. 161° [7007], 149–150° [8375]; ^{1}H NMR [7007], IR [7007], MS [7007].

USE: Fungicide [8417].

BIOLOGICAL ACTIVITY: Anthelmintic and antibacterial [8418,8419]; antitumor promoting agent [8406].

2,4,6-Trihydroxy-3-methyl-5-(1-oxopropyl)benzaldehyde

[96573-37-4]



$C_{11}H_{12}O_5$ mol.wt. 224.21

Syntheses

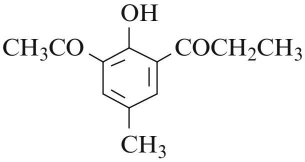
- Preparation by reaction of methyl iodide with formyl-phlorophenone (3-formyl-2,4,6-trihydroxypropiophenone) in the presence of potassium hydroxide in methanol at 70° (50%) [6882] or in dilute methanol at 65° for 24 h (30%) [7007].
- Also refer to: [8378].

N.B.: Compound 1 [8378] and compound 25 [7007].

m.p. 123–125° [7007]; ^{1}H NMR [7007], IR [7007], MS [7007].

1-(3-Acetyl-2-hydroxy-5-methylphenyl)-1-propanone

[91496-99-0] $C_{12}H_{14}O_3$ mol.wt. 206.24



Syntheses

- Preparation by reaction of propionyl chloride with 2-hydroxy-5-methylacetophenone in the presence of aluminium chloride in carbon disulfide at r.t. for 1 h (95%) [8420].
- Also refer to: [8421].

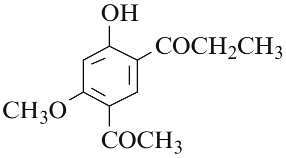
m.p. 51–52° [8420];

^{1}H NMR [8420,8422], ^{7}Li NMR [8422], ^{13}C NMR [8422], IR [8420], X-ray data [8422].

Lithium salt [199329-92-5] [8422] (compound 4).

1-(5-Acetyl-2-hydroxy-4-methoxyphenyl)-1-propanone

$C_{12}H_{14}O_4$ mol.wt. 222.24



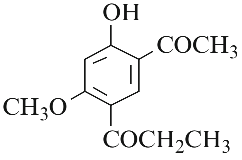
Synthesis

- Obtained by reaction of acetic acid with 2-hydroxy-4-methoxypropiophenone in the presence of polyphosphoric acid for 10 min in a boiling water bath (38%) [6738].

m.p. 129° [6738].

1-(5-Acetyl-4-hydroxy-2-methoxyphenyl)-1-propanone

$C_{12}H_{14}O_4$ mol.wt. 222.24



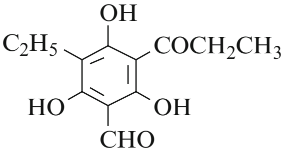
Synthesis

- Obtained by reaction of propionic acid with 2-hydroxy-4-methoxyacetophenone (m.p. 51°) in the presence of polyphosphoric acid [8383] for 10 min in a boiling water bath (26%) [6738].

m.p. 92° [6738].

3-Ethyl-2,4,6-trihydroxy-5-(1-oxopropyl)benzaldehyde

[96573-38-5] $C_{12}H_{14}O_5$ mol.wt. 238.24



Synthesis

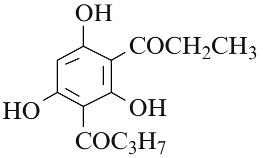
- Preparation by reaction of ethyl iodide with formyl-phlorophenone (3-formyl-2,4,6-trihydroxypropiophenone) in the presence of potassium hydroxide in dilute methanol at 65° for 24 h (30%) [7007].

N.B.: Compound 14 [8378] and compound 26 [7007].

m.p. 146–149° [7007]; ^{1}H NMR [7007], IR [7007], MS [7007].

1-[2,4,6-Trihydroxy-3-(1-oxopropyl)phenyl]-1-butanone

[96573-40-9] $C_{13}H_{16}O_5$ mol.wt. 252.27



Synthesis

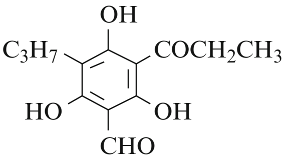
– Preparation by Friedel–Crafts acylation of phloroglucinol [8406] according to the method (i) [7007].

N.B.: Compound 34 [8406] and compound 29 [7007].

m.p. 135° [7007];
^{1}H NMR [7007], IR [7007], MS [7007].

2,4,6-Trihydroxy-3-(1-oxopropyl)-5-propylbenzaldehyde

[96573-39-6] $C_{13}H_{16}O_5$ mol.wt. 252.27



Synthesis

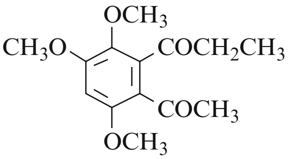
– Preparation by reaction of propyl iodide with formyl-phlorophenone (3-formyl-2,4,6-trihydroxypropiophenone) in the presence of potassium hydroxide in dilute methanol at 65° for 24 h (30%) [7007].

N.B.: Compound 15 [8378] and compound 27 [7007].

m.p. 133–135° [7007]; ^{1}H NMR [7007], IR [7007], MS [7007].

1-(2-Acetyl-3,5,6-trimethoxyphenyl)-1-propanone

[100972-94-9] $C_{14}H_{18}O_5$ mol.wt. 266.29



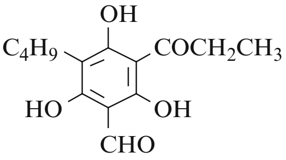
Synthesis

– Preparation: 3,4,6-trimethoxy-2-propenylacetophenone (m.p. 80°) formed with osmium tetroxide an adduct which, when decomposed by sulfur dioxide in aqueous methanol, give the titled diketone [8423].

m.p. 94–97° [8423]; UV [8423].

3-Butyl-2,4,6-trihydroxy-5-(1-oxopropyl)benzaldehyde

[120716-99-6] $C_{14}H_{18}O_5$ mol.wt. 266.29

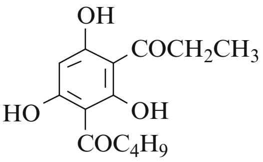


Synthesis

– Preparation by reaction of n-butyl iodide with formyl-phlorophenone (3-formyl-2,4,6-trihydroxypropiophenone) in the presence of potassium hydroxide in dilute methanol at 65° for 24 h (30%) [7007,8378].

N.B.: Compound 16 [8378].

1-[2,4,6-Trihydroxy-3-(1-oxopropyl)phenyl]-1-pentanone



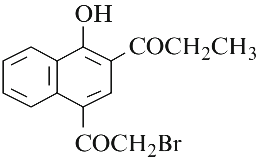
$C_{14}H_{18}O_5$ mol.wt. 266.29

Synthesis

- Preparation [8378] according to the method [7007].

PHARMACOLOGICAL DATA: The compound (n° 18) was assayed for its inhibition of the Hill reaction using chloroplasts isolated from the leaves of *Spinacia oleracea*; pl 50 = 4.9 [8378].

1-[4-Bromoacetyl-1-hydroxy-2-naphthalenyl]-1-propanone



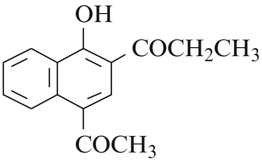
$C_{15}H_{13}BrO_3$ mol.wt. 321.17

Synthesis

- Obtained by reaction of bromine with 4-acetyl-2-propionyl-1-naphthol in chloroform [7767].

m.p. 158° [7767].

1-[1-Hydroxy-4-(1-oxoethyl)-2-naphthalenyl]-1-propanone



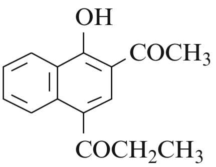
$C_{15}H_{14}O_3$ mol.wt. 242.27

Syntheses

- Preparation by reaction of acetyl chloride with 2-propionyl-1-naphthol in the presence of zinc chloride in nitrobenzene (80%) [7767].
- Preparation by reaction of propionyl chloride with 4-acetyl-1-naphthol in the presence of aluminium chloride (75%) [7767].
- Also obtained by reaction of propionic acid with 4-acetyl-α-naphthol in the presence of polyphosphoric acid at 100° for 15 min (44%) [7777].

m.p. 142° [7767], 134° [7777].

1-[4-Hydroxy-3-(1-oxoethyl)-1-naphthalenyl]-1-propanone



$C_{15}H_{14}O_3$ mol.wt. 242.27

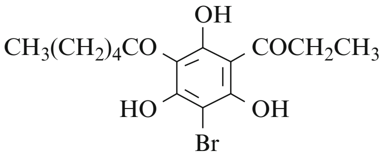
Syntheses

- Obtained by reaction of propionic acid with 2-acetyl-α-naphthol in the presence of polyphosphoric acid at 100° for 15 min (44%) [7777].
- Also obtained by heating 4-propionyl-1-naphthol with acetic acid in the presence of zinc chloride for 3 h [7756].

m.p. 119° [7777].

1-[3-Bromo-2,4,6-trihydroxy-5-(1-oxopropyl)phenyl]-1-hexanone

[98149-39-4]



$C_{15}H_{19}BrO_5$ mol.wt. 359.22

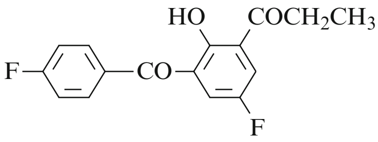
Synthesis

- Refer to: [6879] (Chinese paper), (compound 8) (72–78%).

m.p. 183° [6879].

1-[3-(4-Fluorobenzoyl)-5-fluoro-2-hydroxyphenyl]-1-propanone

[2247-76-9]



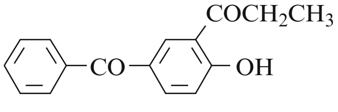
$C_{16}H_{12}F_2O_3$ mol.wt. 290.27

Synthesis

- Obtained by Fries rearrangement of 4-fluoro-2-propionylphenyl 4-fluorobenzoate (b.p.$_{0.5}$ 110°) with aluminium chloride (4–5 mol) at 150° for 5–6 h [8424].

m.p. 160–161° [8424].

1-(5-Benzoyl-2-hydroxyphenyl)-1-propanone

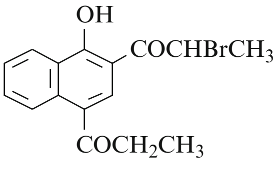


$C_{16}H_{14}O_3$ mol.wt. 254.29

Synthesis

- Obtained by Friedel–Crafts acylation of o-hydroxy-propiophenone with benzoyl chloride [8425].

1-[3-(2-Bromo-1-oxopropyl)-4-hydroxy-1-naphthalenyl]-1-propanone



$C_{16}H_{15}BrO_3$ mol.wt. 335.20

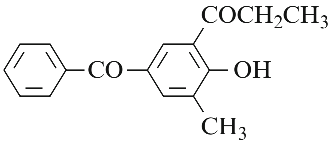
Synthesis

- Obtained by reaction of bromine with 2,4-dipropionyl-1-naphthol in acetic acid [7767].

m.p. 100° [7767].

1-(5-Benzoyl-2-hydroxy-3-methylphenyl)-1-propanone

[101597-03-9]



$C_{17}H_{16}O_3$ mol.wt. 268.31

Syntheses

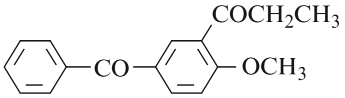
- Preparation by Fries rearrangement of 4-benzoyl-2-methylphenyl propionate with aluminium chloride (3.3 mol) at 160° for 2 h (70%) [7015].

- Also obtained by Friedel–Crafts acylation of 2-hydroxy-3-methylpropiophenone with benzoyl chloride in the presence of aluminium chloride at 160° for 2 h [7015].

m.p. 86° [7015].

1-(5-Benzoyl-2-methoxyphenyl)-1-propanone

[502924-43-8]



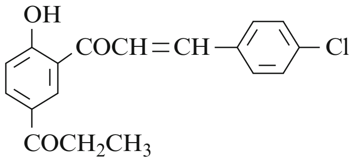
$C_{17}H_{16}O_3$ mol.wt. 268.31

Synthesis

- Refer to: [6958].

3-(4-Chlorophenyl)-1-[2-hydroxy-5-(1-oxopropyl)phenyl]-2-propen-1-one

[141070-42-0]



$C_{18}H_{15}ClO_3$ mol.wt. 314.77

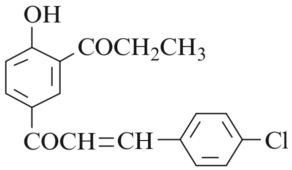
Synthesis

- Preparation by Claisen–Schmidt condensation of p-chlorobenzaldehyde with 2-hydroxy-5-propionyl-acetophenone in the presence of potassium hydroxide in methanol at r.t. for 24 h (96%) [8408].

m.p. 103–104° [8408].

3-(4-Chlorophenyl)-1-[4-hydroxy-3-(1-oxopropyl)phenyl]-2-propen-1-one

[154185-30-5]



$C_{18}H_{15}ClO_3$ mol.wt. 314.77

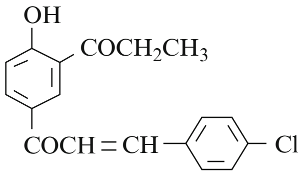
Syntheses

- Preparation by reaction of p-chlorobenzaldehyde with 5-acetyl-2-hydroxypropiophenone in the presence of potassium hydroxide in methanol at r.t. for 24 h (90%) [8413,8414].

m.p. 121–122° [8414], 120–121° [8413]; IR [8414].

3-(4-Chlorophenyl)-1-[4-hydroxy-3-(1-oxopropyl)phenyl]-2-propen-1-one *(E)*

[144728-36-9]



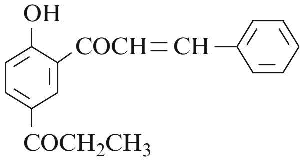
$C_{18}H_{15}ClO_3$ mol.wt. 314.77

Synthesis

- Refer to: [8414].

1-[2-Hydroxy-5-(1-oxopropyl)phenyl]-3-phenyl-2-propen-1-one

[87544-99-8] $C_{18}H_{16}O_3$ mol.wt. 280.32



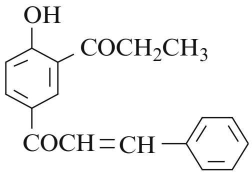
Synthesis

– Preparation by Claisen–Schmidt condensation of benzaldehyde with 2-hydroxy-5-propionyl-acetophenone in the presence of potassium hydroxide in methanol at r.t. for 24 h (96%) [8408].

m.p. 95–96° [8408].

1-[4-Hydroxy-3-(1-oxopropyl)phenyl]-3-phenyl-2-propen-1-one

[154185-29-2] $C_{18}H_{16}O_3$ mol.wt. 280.32



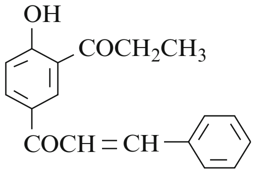
Syntheses

– Preparation by reaction of benzaldehyde with 5-acetyl-2-hydroxypropiophenone in the presence of potassium hydroxide in methanol at r.t. for 24 h (90%) [8413,8414].

m.p. 120–121° [8414], 120° [8413];
^{1}H NMR [8414], IR [8414].

1-[4-Hydroxy-3-(1-oxopropyl)phenyl]-3-phenyl-2-propen-1-one *(E)*

[144728-32-5] $C_{18}H_{16}O_3$ mol.wt. 280.32

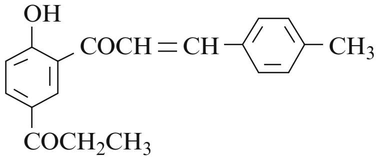


Synthesis

– Refer to: [8414].

1-[2-Hydroxy-5-(1-oxopropyl)phenyl]-3-(4-methylphenyl)-2-propen-1-one

[141070-41-9] $C_{19}H_{18}O_3$ mol.wt. 294.35



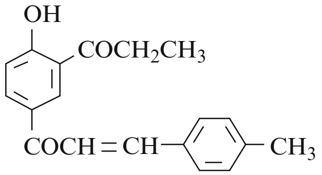
Synthesis

– Preparation by Claisen-Schmidt conensation of p-methylbenzaldehyde with 2-hydroxy-5-propionyl-acetophenone in the presence of potassium hydroxide in methanol at r.t. for 24 h (95%) [8408].

m.p. 101–102° [8408].

1-[4-Hydroxy-3-(1-oxopropyl)phenyl]-3-(4-methylphenyl)-2-propen-1-one

[154185-31-6]



$C_{19}H_{18}O_3$ mol.wt. 294.35

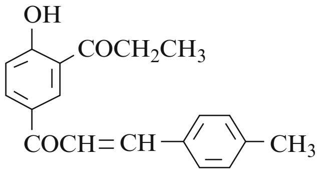
Syntheses

- Preparation by reaction of p-tolualdehyde with 5-acetyl-2-hydroxypropiophenone in the presence of potassium hydroxide in methanol at r.t. for 24 h (90%) [8413,8414].

m.p. 142–143° [8414], 140–142° [8413]; IR [8414].

1-[4-Hydroxy-3-(1-oxopropyl)phenyl]-3-(4-methylphenyl)-2-propen-1-one *(E)*

[144728-37-0]



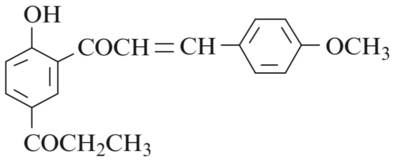
$C_{19}H_{18}O_3$ mol.wt. 294.35

Synthesis

- Refer to: [8414].

1-[2-Hydroxy-5-(1-oxopropyl)phenyl]-3-(4-methoxyphenyl)-2-propen-1-one

[87545-01-5]



$C_{19}H_{18}O_4$ mol.wt. 310.35

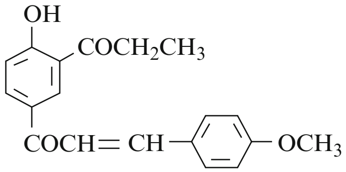
Synthesis

- Preparation by Claisen–Schmidt condensation of p-anisaldehyde with 2-hydroxy-5-propionyl-acetophenone in the presence of potassium hydroxide in methanol at r.t. for 24 h (94%) [8408].

m.p. 74–75° [8408]; ^{1}H NMR [8408], IR [8408].

1-[4-Hydroxy-3-(1-oxopropyl)phenyl]-3-(4-methoxyphenyl)-2-propen-1-one

[154185-32-7]



$C_{19}H_{18}O_4$ mol.wt. 310.35

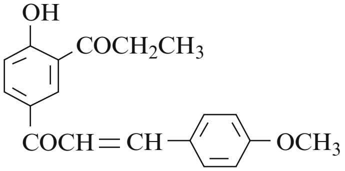
Syntheses

- Preparation by reaction of p-methoxybenzaldehyde with 5-acetyl-2-hydroxypropiophenone in the presence of potassium hydroxide in methanol at r.t. for 24 h (88%) [8413,8414].

m.p. 129–130° [8414], 128–130° [8413], IR [8414].

1-[4-Hydroxy-3-(1-oxopropyl)phenyl]-3-(4-methoxyphenyl)-2-propen-1-one *(E)*

[144728-38-1] $C_{19}H_{18}O_4$ mol.wt. 310.35

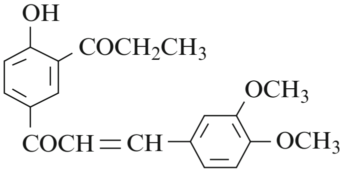


Synthesis

– Refer to: [8414].

3-(3,4-Dimethoxyphenyl)-1-[4-hydroxy-3-(1-oxopropyl)phenyl]-2-propen-1-one

[154185-33-8] $C_{20}H_{20}O_5$ mol.wt. 340.38



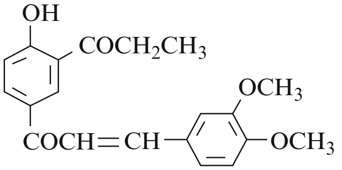
Syntheses

– Preparation by reaction of 3,4-dimethoxybenzaldehyde with 5-acetyl-2-hydroxypropiophenone in the presence of potassium hydroxide in methanol at r.t. for 24 h (85%) [8413,8414].

m.p. 125–126° [8414], 124° [8413]; IR [8414].

3-(3,4-Dimethoxyphenyl)-1-[4-hydroxy-3-(1-oxopropyl)phenyl]-2-propen-1-one *(E)*

[144728-39-2] $C_{20}H_{20}O_5$ mol.wt. 340.38

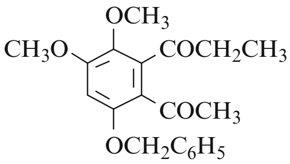


Synthesis

– Refer to: [8414].

1-[2-Acetyl-5,6-dimethoxy-3-(phenylmethoxy)phenyl]-1-propanone

[102553-89-9] $C_{20}H_{22}O_5$ mol.wt. 342.39



Synthesis

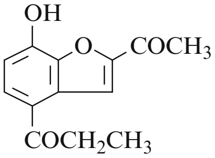
– The 6-benzyloxy-3,4-dimethoxy-2-propenylacetophenone (m.p. 80°; IR, UV) formed with osmium tetroxide an adduct which, when decomposed by sulfur dioxide in 80% ethanol give the titled diketone [8423].

m.p. 111° [8423].

27.2 Carbonyl Groups Located on Different Rings

1-(2-Acetyl-7-hydroxy-4-benzofuranyl)-1-propanone

[59445-80-6] $C_{13}H_{12}O_4$ mol.wt. 232.24



Synthesis

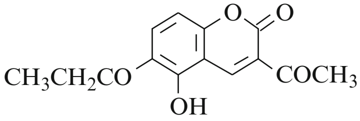
- Obtained by treatment of its methyl ether with aluminium chloride (4 equiv) in methylene chloride at r.t. (89%) [8426].

m.p. 213° [8426].

3-Acetyl-5-hydroxy-6-(1-oxopropyl)-2*H*-1-benzopyran-2-one

3-Acetyl-5-hydroxy-6-propionylcoumarin

$C_{14}H_{12}O_5$ mol.wt. 260.25



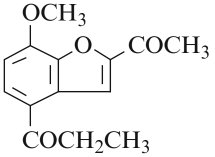
Synthesis

- Preparation from 2,4-dihydroxy-3-formylpropiophenone, ethyl acetoacetate and piperidine (45%) [6768].

m.p. 188–190° [6768].

1-(2-Acetyl-7-methoxy-4-benzofuranyl)-1-propanone

$C_{14}H_{14}O_4$ mol.wt. 246.26



Syntheses

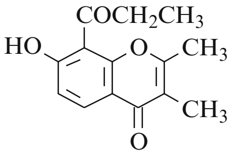
- Obtained by reaction of dimethyl sulfate with 7-hydroxy-4-methyl-6-propionylcoumarin in aqueous alkaline solution [6800].
- Also obtained by oxidation of 7-methoxy-4-methyl-6-propylcoumarin in diethyl ether with ceric ammonium nitrate in aqueous acetic acid (94%) [7960].

m.p. 180° [6800], 172° [7960]; ^{1}H NMR [7960], IR [7960].

7-Hydroxy-2,3-dimethyl-8-(1-oxopropyl)-4*H*-1-benzopyran-4-one

7-Hydroxy-2,3-dimethyl-8-(1-oxopropyl)chromone

[100886-53-1] $C_{14}H_{14}O_4$ mol.wt. 246.26



Syntheses

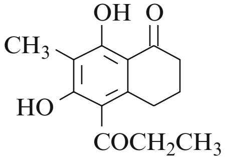
- Preparation by Fries rearrangement of 7-propionoxy-2,3-dimethylchromone with aluminium chloride for 30 min at 120–130° (90%) [8367].

- Preparation by Friedel–Crafts acylation of 7-hydroxy-2,3-dimethylchromone with propionic anhydride in the presence of aluminium chloride at 155–160° for 90 min (55%) [8427].

m.p. 163° [8427], 161.5–162° [8367].

3,4-Dihydro-6,8-dihydroxy-7-methyl-5-(1-oxopropyl)-1(2*H*)-naphthalenone

[78377-69-2] $C_{14}H_{16}O_4$ mol.wt. 248.28



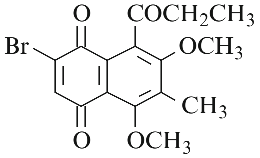
Synthesis

- Obtained by reaction of propionic acid with 3,4-dihydro-6,8-dihydroxy-7-methyl-1(2*H*)-naphthalenone (m.p. 200–201°) [78377-68-1] in the presence of boron trifluoride at 90° (92%) [7876].

m.p. 89–90° [7876].

2-Bromo-5,7-dimethoxy-6-methyl-8-(1-oxopropyl)-1,4-naphthalenedione

[90363-47-6] $C_{16}H_{15}BrO_5$ mol.wt. 367.20



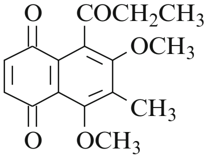
Synthesis

- Obtained by oxidation of 8-hydroxy-2,4-dimethoxy-3-methyl-1-propionaphthone with 3 equiv N-bromo -succinimide in aqueous acetic acid at 25° for 1 h (61%) [7878].

m.p. 144–145° [7878]; ^{1}H NMR [7878].

5,7-Dimethoxy-6-methyl-8-(1-oxopropyl)-1,4-naphthalenedione

[90363-48-7] $C_{16}H_{16}O_5$ mol.wt. 288.30



Synthesis

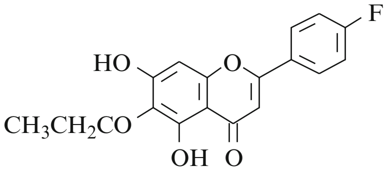
- Obtained (by-product) by oxidation of 8-hydroxy-2,4-dimethoxy-3-methyl-1-propionaphthone with 3 equiv N-bromosuccinimide in aqueous acetic acid at 25° for 1 h (17%) [7878].

m.p. 145–150° [7878]; ^{1}H NMR [7878].

2-(4-Fluorophenyl)-5,7-dihydroxy-6-(1-oxopropyl)-4*H*-1-benzopyran-4-one

4’-Fluoro-5,7-dihydroxy-6-propionylflavone

[848734-12-3] $C_{18}H_{13}FO_5$ mol.wt. 328.30



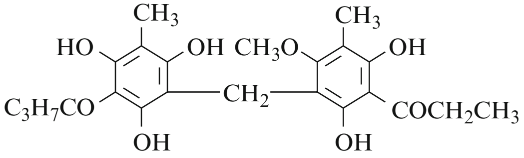
Synthesis

- Obtained from 4-fluorobenzoylacetic acid ethyl ester and phloropropiophenone in boiling diphenyl ether for 3 h (56%) [8428].

m.p. 201–202° [8428];
^{1}H NMR [8428], MS [8428].

1-[3-[[2,4-Dihydroxy-6-methoxy-5-methyl-3-(1-oxopropyl)phenyl]methyl]-2,4,6-trihydroxy-5-methylphenyl]-1-butanone *(Margaspidin BP)*

[57765-48-7]



$C_{23}H_{28}O_8$ mol.wt. 432.47

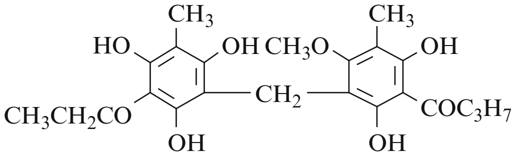
Isolation from natural sources

- From *Dryopteris marginata* (WALL.) CHRIST [8401].

m.p. 176° [8401];
MS [8401]; TLC [8401].

1-[2,6-Dihydroxy-4-methoxy-3-methyl-5-[[2,4,6-trihydroxy-3-methyl-5-(1-oxopropyl)-phenyl]methyl]phenyl]-1-butanone *(Margaspidin PB)*

[66655-97-8]



$C_{23}H_{28}O_8$ mol.wt. 432.47

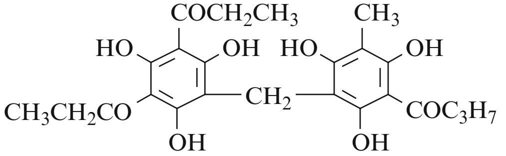
Isolation from natural sources

- From *Dryopteris marginata* (WALL.) CHRIST [8401].

m.p. 176° [8401];
MS [8401]; TLC [8401].

1-[2,4,6-Trihydroxy-3-methyl-5-[[2,4,6-trihydroxy-3,5-bis (1-oxopropyl)phenyl]methyl]-phenyl]-1-butanone

[68223-39-2]



$C_{24}H_{28}O_9$ mol.wt. 460.48

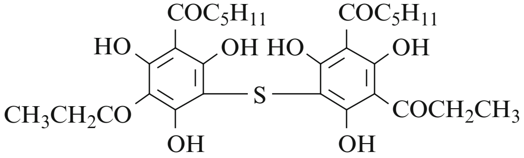
Synthesis

- Refer to: [8404].

BIOLOGICAL ACTIVITY: Antimalarial comparable to that of agrimol [8404].

1,1′-[Thiobis[2,4,6-trihydroxy-5-(1-oxopropyl)-3,1-phenylene]]bis-1-hexanone

[98149-26-9]



$C_{30}H_{38}O_{10}S$ mol.wt. 590.69

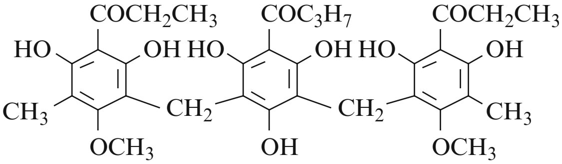
Synthesis

- Refer to: [6879] (Chinese paper), (compound 23) (44–52%).

m.p. 124° [6879].

1-[3,5-Bis[[2,4-dihydroxy-6-methoxy-5-methyl-3-(1-oxopropyl)phenyl]methyl]-2,4,6-trihydroxyphenyl]-1-butanone

[49582-15-2]



$C_{34}H_{40}O_{12}$ mol.wt. 640.68

Isolation from natural sources

- From African *Dryopteris* series [6890, 7262,7393].

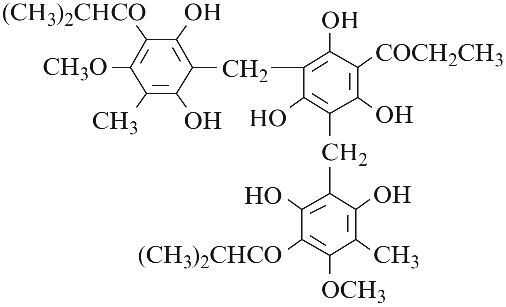
MS [7393].

1-[3,5-Bis[[2,6-dihydroxy-4-methoxy-3-(2-methyl-1-oxopropyl)-5-methylphenyl]methyl]-2,4,6-trihydroxyphenyl]-1-propanone *(Protokosin)*

$C_{35}H_{42}O_{12}$ mol.wt. 654.71

Isolation from natural sources

- From *Hagenia abyssini*ca (Bruce) Gmel. [6932].

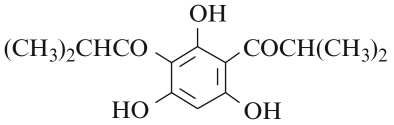


Chapter 28
Aromatic Polyketones Containing Only Isobutyryl Groups

28.1 Isobutyryl Groups Located on the Same Ring

1,1′-(2,4,6-Trihydroxy-1,3-phenylene)bis[2-methyl-1-propanone

[3133-29-7]



$C_{14}H_{18}O_5$ mol.wt. 266.29

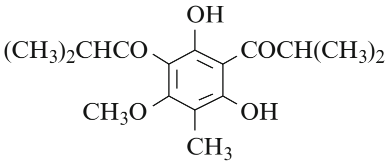
Syntheses

- Preparation by reaction of isobutyric acid with phloroglucinol in the presence of boron trifluoride (compound **20**) (53–78%) [8376], (compound **15**) (62%) [8060], (compound **12**) [8088], (compound **10**) [8059].
- Also refer to: [8382,8429–8431].

m.p. 133–135° [8060], 124–127° [8429,8430];
^{1}H NMR [8059,8060,8088], ^{13}C NMR [8059,8088],
IR [8059,8060,8088], UV [8059,8088].

BIOLOGICAL ACTIVITY: Antimicrobial against *Bacillus subtilis* [8376]; vesicular stomatitis virus inhibition [8059]; as allergy inhibitor [8089]; for herpes virus control [8379]; as herpes virus inhibitor [8380].

1,1′-(2,6-Dihydroxy-4-methoxy-5-methyl-1,3-phenylene)bis[2-methyl-1-propanone



$C_{16}H_{22}O_5$ mol.wt. 294.35

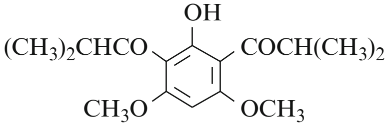
Synthesis

- Refer to: [8172] (compound **8a**).

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_28, © Springer Science+Business Media B.V. 2011

1,1′-(2-Hydroxy-4,6-dimethoxy-1,3-phenylene)bis[2-methyl-1-propanone

[60831-51-8]



$C_{16}H_{22}O_5$ mol.wt. 294.35

Synthesis

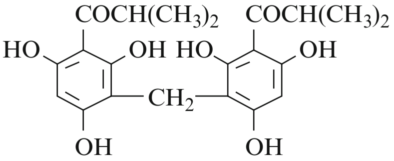
- Preparation in two steps: First, reaction of isobutyric acid with 1,3,5-trimethoxybenzene in the presence of boron trifluoride. Then, the complex obtained* (27%, m.p. 150–151°) was hydrolyzed in dilute methanol at 50° (87%) [8139].

* 2,2-Difluor-1,2-dihydro-8-isobutyryl-4-isopropyl-5,7-dimethoxy-1-oxa-3-oxonia-2-borata-naphthalin (compound **6**).

m.p. 92–94° [8139]; ^{1}H NMR [8139].

28.2 Isobutyryl Groups Located on Different Rings

1,1′-[Methylenebis(2,4,6-trihydroxy-3,1-phenylene)]bis[2-methyl-1-propanone



$C_{21}H_{24}O_8$ mol.wt. 404.42

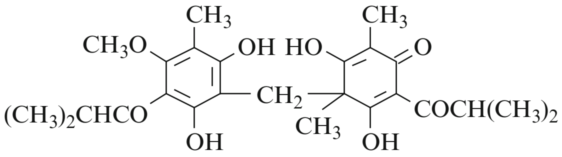
Synthesis

- Obtained by reaction of phloroisobutyrophenone with 35% formaldehyde in the presence of aqueous 2 N sodium hydroxide [8050].

4-[[2,6-Dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl) phenyl]methyl]-3,5-dihydroxy-2,4-dimethyl-6-(2-methyl-1-oxopropyl)-2,5-cyclohexadien-1-one *(Kosotoxin)*

N.B.: Deleted Registry Number [55838-74-9]; other CAS registry [55382-25-7].

[1400-16-4]



$C_{25}H_{32}O_8$ mol.wt. 460.52

Isolation from natural sources

- From *Hagenia abyssinica* (Rosaceae) [8068,8175,8432–8440].

The Flowers of *Hagenia abyssinica* (Bruce) Gmel are known under their names (Kousso, Kosso, Kusso or Koso) [6932].

m.p. 119–122° [6932], 80° [8434,8435] (old papers);

$(\alpha)_D^{25}$ = + 11.7°–12.1° (chloroform) [6932];

^{1}H NMR [6932,8437–8439], ^{13}C NMR [8175],
IR [6932,8437–8439], UV [6932,8437–8439],
MS [6932,8436–8439];
GC [8438]; TLC [8437–8439]; HPLC [8433,8437–8439].

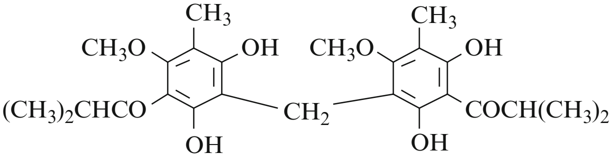
Note: Acute toxicity [8439].

BIOLOGICAL ACTIVITY: Spasmolytic [8432]; antitumor action [8439]; anthelmintic against tapeworm [8441].

PHARMACOLOGICAL DATA [8442].

1-[3-[[2,6-Dihydroxy-4-methoxy-5-methyl-3-(2-methyl-1-oxopropyl)phenyl]methyl]-2,6-dihydroxy-4-methoxy-5-methylphenyl]-2-methyl-1-propanone

[99174-41-1] $C_{25}H_{32}O_8$ mol.wt. 460.52



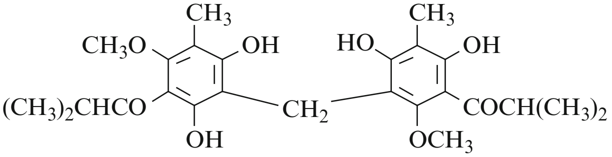
Synthesis

- Obtained by reaction of a 2,6-dihydroxy-4-methoxy-3-methylisobutyrophenone and 4,6-dihydroxy-2-methoxy-3-methyl-isobutyrophenone mixture with formaldehyde in the presence of 1% aqueous potassium hydroxide for 45 min at 0° (7%) [7255].

m.p. 170–171° [7255]; UV [7255].

1-[3-[[2,6-Dihydroxy-4-methoxy-5-methyl-3-(2-methyl-1-oxopropyl)phenyl]methyl]-4,6-dihydroxy-2-methoxy-5-methylphenyl]-2-methyl-1-propanone

[489-48-5] $C_{25}H_{32}O_8$ mol.wt. 460.52



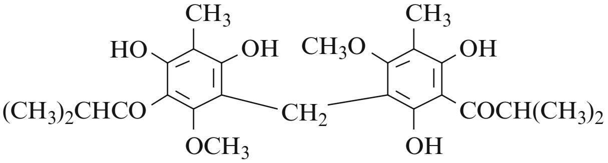
Synthesis

- Obtained by reaction of a 2,4-dihydroxy-6-methoxy-3-methylisobutyrophenone and 4,6-dihydroxy-2-methoxy-3-methyl-isobutyrophenone mixture with formaldehyde in the presence of 1% aqueous potassium hydroxide for 45 min at 0° (14%) [7255].

m.p. 164.5° [7255]; UV [7255].

1-[3-[[4,6-Dihydroxy-2-methoxy-5-methyl-3-(2-methyl-1-oxopropyl)phenyl] methyl]-2,6-dihydroxy-4-methoxy-5-methylphenyl]-2-methyl-1-propanone

[99174-42-2] $C_{25}H_{32}O_8$ mol.wt. 460.52



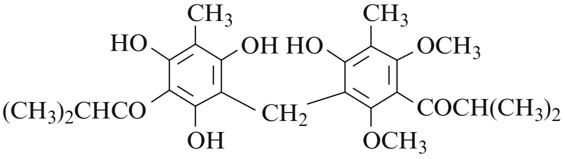
Synthesis

- Obtained by reaction of 2,4-dihydroxy-6-methoxy-3-methylisobutyrophenone and 2,6-dihydroxy-4-methoxy-3-methyl-isobutyrophenone mixture with formaldehyde in the presence of 1% aqueous potassium hydroxide for 45 min at 0° (15%) [7255].

m.p. 183° [7255]; UV [7255].

1-[3-[[2-Hydroxy-4,6-dimethoxy-3-methyl-5-(2-methyl-1-oxopropyl)phenyl] methyl]-2,4,6-trihydroxy-5-methylphenyl]-2-methyl-1-propanone *(β-Kosin)*

[1400-15-3] $C_{25}H_{32}O_8$ mol.wt. 460.52



Syntheses

- Obtained by alkali treatment of protokosin isolated from *Hagenia abyssinica* [7255,8085,8198,8435].

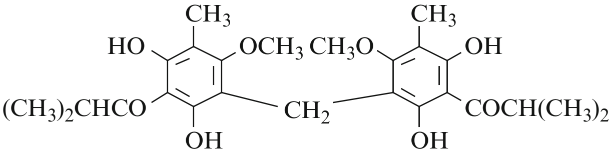
m.p. 120° [8198,8435]; IR [8085], UV [8085].

BIOLOGICAL ACTIVITY: Anthelmintic [8085].

Triacetate $C_{31}H_{38}O_{11}$ mol.wt. 586.64 (m.p. 155°) [8198].

1,1′-[Methylenebis(2,4-dihydroxy-6-methoxy-5-methyl-3,1-phenylene)] bis[2-methyl-1-propanone

[568-50-3] $C_{25}H_{32}O_8$ mol.wt. 460.52



Syntheses

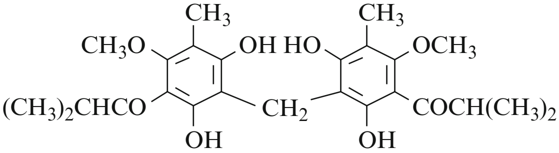
- Obtained by reaction of 2,6-dihydroxy-4-methoxy-3-methylisobutyrophenone with formaldehyde in the presence of 1% aqueous potassium hydroxide for 45 min at 0° (49%) [7255,8170].

m.p. 192–193° [7255,8170]; UV [7255].

Tetraacetate $C_{33}H_{40}O_{12}$ mol.wt. 628.67 (m.p. 141°) [7255].

1,1′-[Methylenebis(2,6-dihydroxy-4-methoxy-5-methyl-3,1-phenylene)]bis[2-methyl-1-propanone *(α-Kosin)*

[99174-40-0] $C_{25}H_{32}O_8$ mol.wt. 460.52



Syntheses

- Obtained by reaction of 4,6-dihydroxy-2-methoxy-3-methylisobutyrophenone with formaldehyde in the presence of 1% aqueous potassium hydroxide for 45 min at 0° (36%) [7255,8170].
- Also obtained by alkaline cleavage of Kosotoxin [8068].
- Also obtained by treatment of Protokosin with zinc dust in boiling aqueous sodium hydroxide for 5 min [8085,8198].
- Also refer to: [8436].

Isolation from natural sources

- From flowers of *Hagenia abyssinica* also named *Brayera anthelmintica* [8175,8437,8439].

m.p. 160–160.5° [7255], 160° [8435], 158° [8170,8198].
^{13}C NMR [8175], IR [8085], UV [7255,8085];
HPLC [8433,8437].

Note: Acute toxicity [8439].

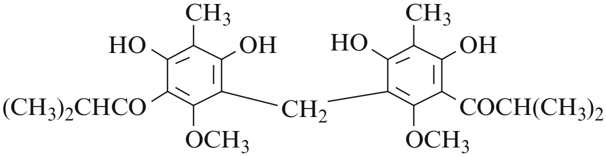
BIOLOGICAL ACTIVITY: Anthelmintic [8085]; antitumor [8439].

Triacetate $C_{31}H_{38}O_{11}$ mol.wt. 586.64 (m.p. 123°) [8198].

Tetraacetate $C_{33}H_{40}O_{12}$ mol.wt. 628.67 (m.p. 124°) [7255].

1,1′-[Methylenebis(4,6-dihydroxy-2-methoxy-5-methyl-3,1-phenylene)] bis[2-methyl-1-propanone

$C_{25}H_{32}O_8$ mol.wt. 460.52



Synthesis

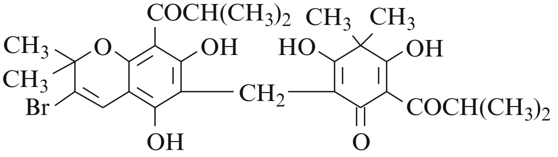
- Obtained by reaction of 2,4-dihydroxy-6-methoxy-3-methylisobutyrophenone with formaldehyde in the presence of 1% aqueous potassium hydroxide for 45 min at 0° (33%) [1484].

m.p. 205–207° [7255]; UV [7255].

Tetraacetate $C_{33}H_{40}O_{12}$ mol.wt. 628.67 (m.p. 172–174°) [7255].

2-[[3-Bromo-5,7-dihydroxy-2,2-dimethyl-8-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-6-yl]-methyl]-3,5-dihydroxy-4,4-dimethyl-6-(2-methyl-1-oxopropyl)-2,5-cyclohexadien-1-one *(Bromouliginosin B)*

[19647-29-1]



$C_{28}H_{33}BrO_8$ mol.wt. 577.47

Synthesis

- Preparation by treatment of *Uliginosin B* with bromine followed by dehydrohalogenation in pyridine [8443].

Isolation from natural sources

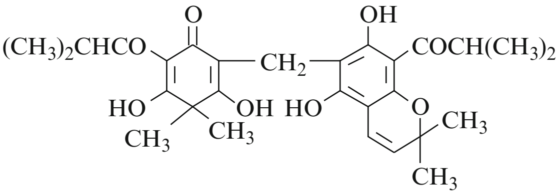
- From *Hypericum uliginosum*, a woody herb found in Mexico and Central America [8443].

X-ray diffraction study [8443].

BIOLOGICAL ACTIVITY: Antibiotic [8443].

2-[[5,7-Dihydroxy-2,2-dimethyl-8-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-6-yl] methyl]-3,5-dihydroxy-4,4-dimethyl-6-(2-methyl-1-oxopropyl)-2,5-cyclohexadien-1-one *(Uliginosin B-iBiB)*

[19809-79-1]



$C_{28}H_{34}O_8$ mol.wt. 498.57

Synthesis

- Obtained (poor yield) by adding a solution of DDQ in benzene over 30 min to a stirred solution of Uliginosin A-iBiB (8%) [8138].

Isolation from natural sources

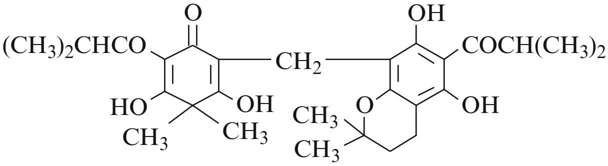
- From the Mexican plant *Hypericum uliginosum* HBK (Tzotzil) [8060, 8138,8207,8444,8445].

m.p. 142.5–143.5° [8138], 139.5–142° [8444,8445];
^{1}H NMR [8138,8444], IR [8138,8444], UV [8138,8444], MS [8444];
X-ray diffraction study [8443]; TLC [8138].

BIOLOGICAL ACTIVITY: Antibiotic [8060,8138,8207,8444,8445].

2-[[3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-6-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-8-yl]methyl]-3,5-dihydroxy-4,4-dimethyl-6-(2-methyl-1-oxopropyl)-2,5-cyclohexadien-1-one *(Isodihydrouliginosin B-iBiB)*

[19809-81-5] $C_{28}H_{36}O_8$ mol.wt. 500.59



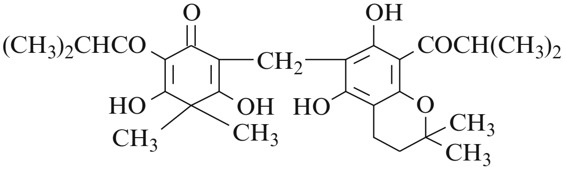
Syntheses

- Obtained by reaction of 5,7-dihydroxy-6-isobutyryl-2,2-dimethylchroman with albaspidin-iBiB in the presence of sodium hydride in refluxing ethanol for 1 h (23%) [8060,8207].
- Also obtained by reaction of p-toluenesulfonic acid with Uliginosin A in refluxing benzene for 30 min [8444].
- Also refer to: [8138].

m.p. 163° [8060], 159° [8207], 156–158.5° [8444];
^{1}H NMR [8060,8444], IR [8060,8444], UV [8060,8444]; TLC [8444].

2-[[3,4-Dihydro-5,7-Dihydroxy-2,2-dimethyl-8-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-6-yl]methyl]-3,5-dihydroxy-4,4-dimethyl-6-(2-methyl-1-oxopropyl)-2,5-cyclohexadien-1-one *(Dihydrouliginosin B-iBiB)*

[19809-80-4] $C_{28}H_{36}O_8$ mol.wt. 500.59



Syntheses

- Obtained by reaction of albaspidin-iBiB with 5,7-dihydroxy-8-isobutyryl-2,2-dimethylchroman (m.p. 145–146°) in the presence of sodium hydride in refluxing ethanol for 45 min (83%) [8060], (ca. 80%) [8207].
- Also obtained by hydrogenation of Uliginosin B in ethyl acetate with hydrogen in the presence of 10% Pd/C [8444].

Isolation from natural sources

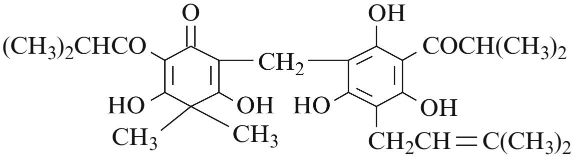
- From the Mexican plant *Hypericum uliginosum* HBK [8060,8138,8444].

pale yellow solid [8138]; m.p. 149° [8207], 141–142° [8060], 138–141° [8444];
^{1}H NMR [8060,8138,8444], IR [8060,8444], UV [8060,8444],
MS [8444].

3,5-Dihydroxy-4,4-dimethyl-2-(2-methyl-1-oxopropyl)-6-[[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)-5-(2-methyl-1-oxopropyl)phenyl]methyl]-2,5-cyclohexadien-1-one

(Uliginosin A-iBiB)

[19809-78-0] $C_{28}H_{36}O_8$ mol.wt. 500.59



Syntheses

- Obtained by reaction of albaspidin-iBiB with 2,4,6-trihydroxy-3-(3-methyl-2-butenyl)isobutyrophenone in the presence of sodium hydride in refluxing methanol for 1.5 h (50%) [8060,8207].

Isolation from natural sources

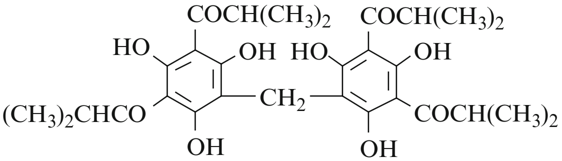
- From the Mexican plant *Hypericum uliginosum* HBK (Tzotzil) [8060, 8138,8207,8444,8445].

m.p. 164° [8207], 161–162° [8060], 160.5–161.5° [8444,8445];
^{1}H NMR [8060,8444], IR [8060,8444], UV [8060,8444], MS [8444];
X-ray diffraction study [8443].

BIOLOGICAL ACTIVITY: Antibiotic [8060,8138,8207,8444,8445].

1,1′,1″,1‴-[Methylenebis(2,4,6-trihydroxy-5,1,3-benzenetriyl)]tetrakis[2-methyl-1-propanone

[68223-31-4] $C_{29}H_{36}O_{10}$ mol.wt. 544.60



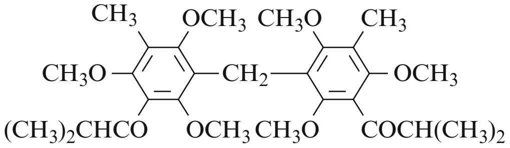
Syntheses

- Preparation by bimolecular condensation of 2,4-diisobutyryl-phloroglucinol with 40% formaldehyde [6879,8404] or with methoxymethyl acetate in the presence of a few drops of concentrated sulfuric acid [8405].

BIOLOGICAL ACTIVITY: Antischistosomal (an analog of agrimophol) [6879].

1,1′-[Methylenebis(2,4,6-trimethoxy-5-methyl-3,1-phenylene)] bis[2-methyl-1-propanone

$C_{29}H_{40}O_8$ mol.wt. 516.63



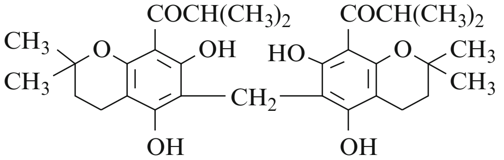
Synthesis

- Obtained by treatment of α or β-Kosins with dimethyl sufate in the presence of N sodium hydroxide on a steam bath under nitrogen [8085].

gum [8085]; IR [8085], UV [8085].

1,1′-[Methylenebis(3,4-dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-6,8-diyl)]bis[2-methyl-1-propanone

[72935-02-5] $C_{31}H_{40}O_8$ mol.wt. 540.65



Syntheses

- Obtained by heating a solution of 8-isobutyryl-7-hydroxy-5-methoxy-methyleneoxy-2,2-dimethylchroman in 80% acetic acid containing 2 drops of concentrated sulfuric acid on a steam bath for 45 min (91%) [8138].
- Also obtained by hydrolysis of 8-isobutyryl-5-methoxymethyleneoxy-2,2-dimethylchroman-7-ol in the presence of an excess of isobutyrylfilicinic acid [8060].

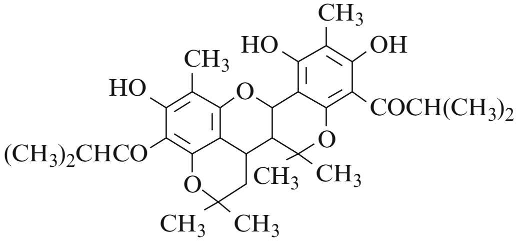
^{1}H NMR [8060], IR [8060].

1,1′-(1,7a,13a,13b-Tetrahydro-5,8,10-trihydroxy-2,2,6,9,13,13-hexamethyl-2*H*,13*H*-bis[1]benzopyrano[5,4-*bc*:3′,4′-*e*]pyran-4,11-diyl)bis[2-methyl-1-propanone

(7aα, 13aα, 13bα) [111983-98-3] [8275]

(7aα, 13aα, 13bβ) [112709-78-1] [8277] *(Hyperevoline)*

$C_{32}H_{40}O_8$ mol.wt. 552.66



Isolation from natural sources

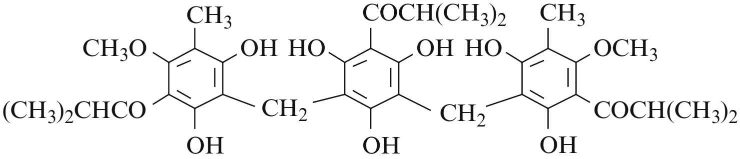
- From *Hypericum revolutum* VAHL (Guttiferae) [8275–8277].

m.p. 206–210° [8276];
TLC [8276,8277];
HPLC [8275,8277];
^{1}H NMR [8276],
^{13}C NMR [8276], IR [8276], UV [8276], MS [8276,8277], X-Ray Analysis [8276,8277].
Note: Fungicidal [8276,8277].

BIOLOGICAL ACTIVITY: Antibiotic [8277].

1,1′-[[2,4,6-Trihydroxy-5-(2-methyl-1-oxopropyl)-1,3-phenylene]bis[methylene-(2,4-dihydroxy-6-methoxy-5-methyl-3,1-phenylene)]]bis[2-methyl-1-propanone *(Agrimol G) (Tripseudo-aspidinol iB, iB, iB)*

[121693-17-2] $C_{36}H_{44}O_{12}$ mol.wt. 668.74



Isolation from natural sources

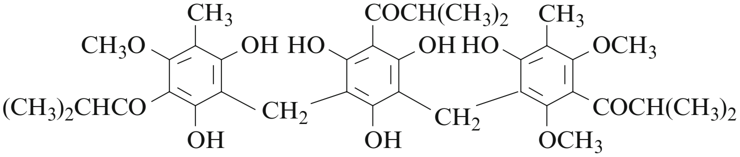
- From Xian he cao (*Agrimonia pilosa* Ledb.) [8446].
- From *Hagenia abyssinica* [6932].
- Also refer to: [8447].

m.p. 167–170° [8446];
^{1}H NMR [8446], ^{13}C NMR [8446], IR [8446], MS [8446].

BIOLOGICAL ACTIVITY: Antimicrobial [8446]; antimicrobial for *Staphylococcus aureus* [6891].

1-[3-[[2,6-Dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl) phenyl]methyl]-2,4,6-trihydroxy-5-[[2-hydroxy-4,6-dimethoxy-3-methyl-5-(2-methyl-1-oxopropyl)-phenyl]methyl]phenyl]-2-methyl-1-propanone

$C_{37}H_{46}O_{12}$ mol.wt. 682.77



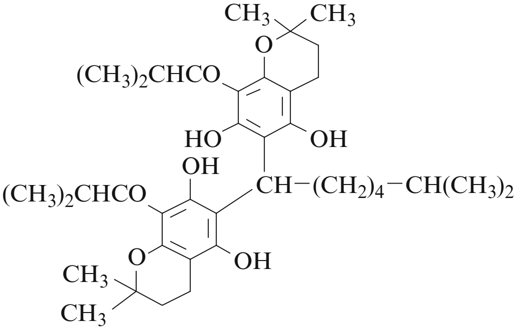
Isolation from natural sources

- Of *Hagenia abyssinica*, Gmel female flowers [8440].
- Also refer to: [7255,8434].

m.p. 176° [8434].

1,1′-[(6-Methylheptylidene)bis(3,4-dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-6,8-diyl)]bis[2-methyl-1-propanone

[99814-61-6]



$C_{38}H_{54}O_8$ mol.wt. 638.84

Isolation from natural sources

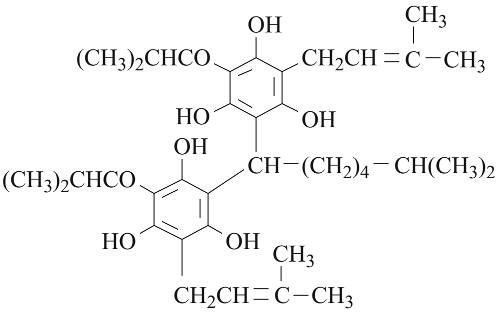
- From the aerial parts of *Helichrysum platypterum* DC (Compositae) (compound **5**) [8080].

yellow crystals [8080];

m.p. 189° [8080];
^{1}H NMR [8080], IR [8080],
MS [8080].

1,1′-[(6-Methylheptylidene)bis[2,4,6-trihydroxy-5-(3-methyl-2-butenyl)-3,1-phenylene]]bis[2-methyl-1-propanone

[103771-72-8]



$C_{38}H_{54}O_8$ mol.wt. 638.84

Isolation from natural sources

- From the aerial parts of *Helichrysum platypterum* DC (Compositae) (compound **28**) [8080].

yellow crystals [8080];

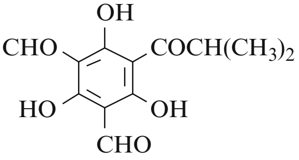
m.p. 120–121° [8080];
^{1}H NMR [8080], IR [8080],
MS [8080].

Chapter 29
Aromatic Polyketones Containing At Least One Isobutyryl Group

29.1 Carbonyl Groups Located on the Same Ring

2,4,6-Trihydroxy-5-(2-methyl-1-oxopropyl)-1,3-benzenedicarboxaldehyde

[245052-19-1] $C_{12}H_{12}O_6$ mol.wt. 252.22



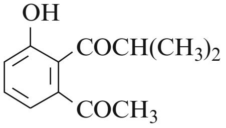
Isolation from natural sources

- From eucalyptus essential oil, *Eucalyptus apodophylla* (Myrtaceae) (compound **28**) (component C) [8221].

MS [8221]; GC [8221], GC-MS [8221].

1-(2-Acetyl-6-hydroxyphenyl)-2-methyl-1-propanone

[171609-28-2] $C_{12}H_{14}O_3$ mol.wt. 206.24

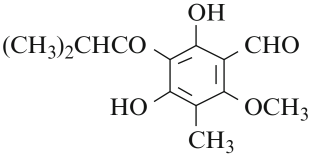


Synthesis

- Refer to: [8336] (Japanese patent).

2,4-Dihydroxy-6-methoxy-5-methyl-3-(2-methyl-1-oxopropyl)benzaldehyde

$C_{13}H_{16}O_5$ mol.wt. 252.27



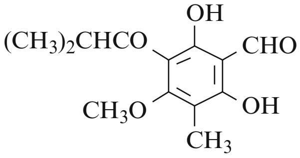
Synthesis

- Obtained (poor yield) by reaction of zinc cyanide with 2,6-dihydroxy-4-methoxy-3-methylisobutyrophenone in the presence of aluminium chloride according to the Gattermann–Adams reaction (10%) [8171].

m.p. 70° [8171];
^{1}H NMR [8171], ^{13}C NMR [8171], IR [8171], MS [8171].

R. Martin, *Aromatic Hydroxyketones: Preparation and Physical Properties*,
DOI 10.1007/978-1-4020-9787-4_29, © Springer Science+Business Media B.V. 2011

2,6-Dihydroxy-4-methoxy-5-methyl-3-(2-methyl-1-oxopropyl)benzaldehyde



$C_{13}H_{16}O_5$ mol.wt. 252.27

Synthesis

- Preparation by reaction of zinc cyanide with 4,6-dihydroxy-2-methoxy-3-methylisobutyrophenone in the presence of aluminium chloride according to the Gattermann–Adams reaction (54%) [8171].

Isolation from natural sources

- From *Hagenia abyssinica* (Rosaceae) [8178].

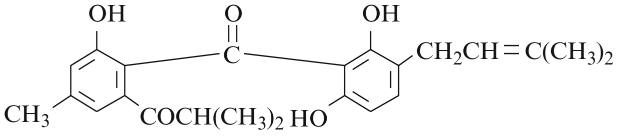
m.p. 65–66° [8171], 54–56° [8178]. One of the reported melting points is obviously wrong.

^{1}H NMR [8171,8178], ^{13}C NMR [8178], IR [8171,8178], UV [8178], MS [8178].

29.2 Carbonyl Groups Located on Different Rings

1-[2-[2,6-Dihydroxy-3-(3-methyl-2-butenyl)benzoyl]-3-hydroxy-5-methylphenyl]-2-methyl-1-propanone *(FD-549)*

[175413-69-1] $C_{23}H_{26}O_5$ mol.wt. 382.46



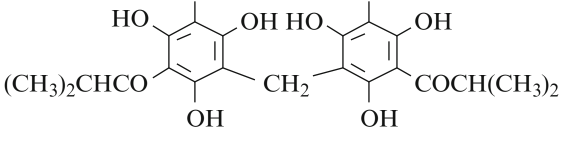
Biosynthesis

- From Penicillium sp. TF-0379 (FERMP-13534) [8448].

BIOLOGICAL ACTIVITY: Antitumor [8448].

1,1’-[Methylenebis(5-acetyl-2,4,6-trihydroxy-3,1-phenylene)]bis [2-methyl-1-propanone

[98149-24-7] $C_{25}H_{28}O_{10}$ mol. t. 488.49



Synthesis

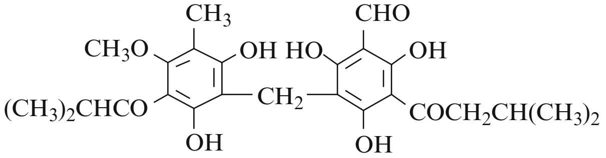
- Obtained by condensation of 3-acetyl-2,4,6-trihydroxyisobutyrophenone (2 mol) in the presence of 40% formaldehyde (58–65%) [6879].

m.p. 180–182° [6879].

BIOLOGICAL ACTIVITY: Antischistosomal (an analog of agrimophol) [6879].

3-[[2,6-Dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl)phenyl] methyl]-2,4,6-trihydroxy-5-(3-methyl-1-oxobutyl)benzaldehyde *(Robustaol A)*

[78411-76-4] $C_{25}H_{30}O_9$ mol.wt. 474.51



Synthesis

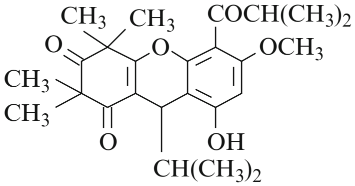
- Preparation from 4,6-dihydroxy-2-methoxy-3-methylisobutyrophenone and 3-isovaleryl-2,4,6-trihydroxybenzaldehyde [8176].

Isolation from natural sources

- From *Eucalyptus robusta* [8176].

4,9-Dihydro-8-hydroxy-6-methoxy-2,2,4,4-tetramethyl-5-(2-methyl-1-oxopropyl)-9-(2-methylethyl)-1*H*-xanthene-1,3(2*H*)-dione *(Myrtucommulone B)*

$C_{25}H_{32}O_6$ mol.wt. 428.53

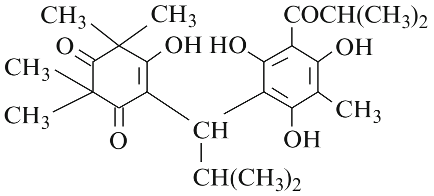


Synthesis

- Refer to: [8135].

4-[1-[2,4,6-Trihydroxy-5-methyl-3-(2-methyl-1-oxopropyl)phenyl]-2-methylpropyl]-5-hydroxy-2,2,6,6-tetramethyl-4-cyclohexene-1,3-dione *(Semimyrtucommulone)*

$C_{25}H_{34}O_7$ mol.wt. 446.97



Isolation from natural sources

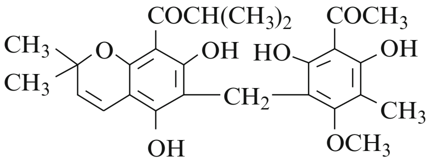
- From plants of family Myrtaceae [6882,8449].

USE: Antioxidizing agent [6882,8449].

1-[6-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone

(Isobutyrylmallotochromene)

[116964-16-0]



$C_{26}H_{30}O_8$ mol.wt. 470.52

Isolation from natural sources

- From the pericarps of *Mallotus japonicus* MUELL (Euphorbiaceae) [8450], (compound **11**) [8451], (compound **9**) [8452,8453].

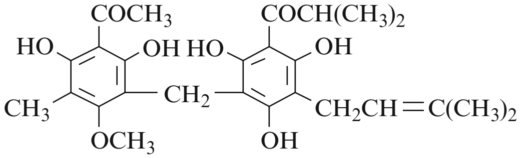
m.p. 180–181° [8451]; ^{1}H NMR [8451], ^{13}C NMR [8451], IR [8451], UV [8451], MS [8451].

Note: Cytotoxicity [8453].

1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-2,4,6-trihydroxy-5-(3-methyl-2-butenyl)phenyl]-2-methyl-1-propanone

(Isobutyrylmallotojaponin)

[96853-74-6]



$C_{26}H_{32}O_8$ mol.wt. 472.54

Isolation from natural sources

- From *Mallotus japonicus* MUELL ARG. (Euphorbiaceae) (compound D) [8454] (compound **5**) [8451,8455], (compound **6**) [8456] (compound **3**) [8452,8453], (compound **9**) [8457].

N.B.: The compound D was a mixture of butyrophenone and isobutyrophenone (2:1) [8454,8456].

m.p. 157–158° [8454]; ^{1}H NMR [8454], ^{13}C NMR [8454], IR [8454], MS [8454].

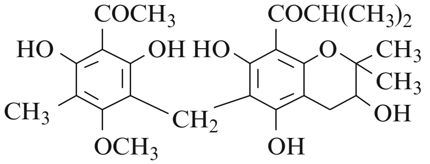
Note: Cytotoxicity [8453,8455,8458].

BIOLOGICAL ACTIVITY: Antiviral and antitumor [8452].

1-[6-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-3,4-dihydro-3,5,7-trihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone

(Isobutyrylmallotochromanol)

[129399-53-7]



$C_{26}H_{32}O_9$ mol.wt. 488.53

Isolation from natural sources

- From the pericarps of *Mallotus japonicus* MUELL. ARG. (Euphorbiaceae) (compound **13**) [8452], (compound **2**) [8457], (compounds 5) [8459,8460].
- Also refer to: [8461–8466].

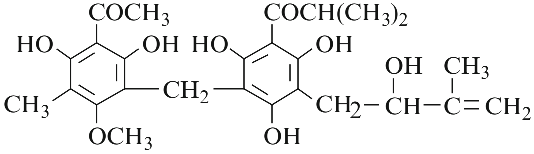
m.p. 211–212° [8457];
^{1}H NMR [8457], ^{13}C NMR [8457], IR [8457], UV [8457], MS [8457].
Note: Cytotoxicity [8452,8457].

BIOLOGICAL ACTIVITY: Antiviral and antitumor [8452]; antiherpetic [8452]; drug-coated coronary stent system [8464]; catheter with balloon for the expansion of blood vessels carrying contrast agents and drugs [8466]; method and device for coating medical goods using ultrasound spraying [8465]; inhibition of lipopolysaccharide-induced pro-inflammatory cytokine expression *via* suppression of nuclear factor-kB activation [8460]; prostaglandin E_2 production and induction of prostaglandin endoperoxide synthase-2 is inhibited in a murine macrophage-like cell line RAW 264.7 [8459]; for treatment of carcinoid syndrome [8467]; expandable medical devices with Parylene C and paclitaxel coating [8468]; method for targeted delivery of therapeutic substances into cells using nanoparticles [8469].

1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-2,4,6-trihydroxy-5-(2-hydroxy-3-methyl-3-butenyl)phenyl]-2-methyl-1-propanone

(Isobutyrylmallotolerin), old name *(isomallotolerin)*

[126026-30-0] $C_{26}H_{32}O_9$ mol.wt. 488.53



Isolation from natural sources

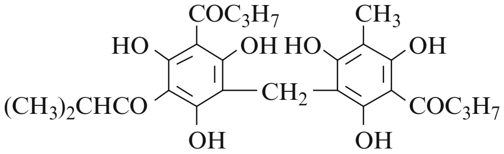
- From the pericarps of *Mallotus japonicus* MUELL. ARG. (Euphorbiaceae) [8450], (compound **1**) [8470], (compound **6**) [8452,8453], (compound **11**) [8457].

m.p. 216–217° [8470]; ^{1}H NMR [8470], ^{13}C NMR [8470], IR [8470], UV [8470], MS [8470]; HPLC [8470].
Note: Cytotoxicity [8453].

BIOLOGICAL ACTIVITY: Antiviral and antitumor [8452].

1-[2,4,6-Trihydroxy-3-(2-methyl-1-oxopropyl)-5-[[2,4,6-trihydroxy-3-methyl-5-(1-oxobutyl)phenyl]methyl]phenyl]-1-butanone

[68223-53-0]



$C_{26}H_{32}O_9$ mol.wt. 488.53

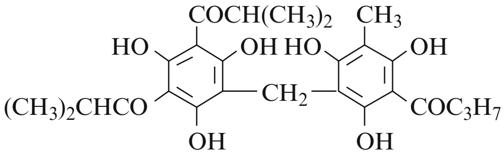
Synthesis

- Preparation by bimolecular condensation of acylphloroglucinol derivatives [8404] (Chinese paper).

BIOLOGICAL ACTIVITY: Antimalarial [8404].

1-[2,4,6-Trihydroxy-3-methyl-5-[[2,4,6-trihydroxy-3,5-bis(2-methyl-1-oxopropyl)-phenyl]methyl]phenyl]-1-butanone

[68223-40-5]



$C_{26}H_{32}O_9$ mol.wt. 488.53

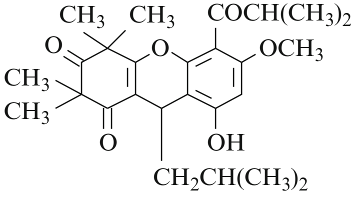
Synthesis

- Preparation by bimolecular condensation of acylphloroglucinol derivatives [8404] (Chinese paper).

BIOLOGICAL ACTIVITY: Antimalarial [8404].

4,9-Dihydro-8-hydroxy-6-methoxy-2,2,4,4-tetramethyl-5-(2-methyl-1-oxopropyl)-9-(2-methylpropyl)-1*H*-xanthene-1,3(2*H*)-dione

[139979-87-6]



$C_{26}H_{34}O_6$ mol.wt. 442.55

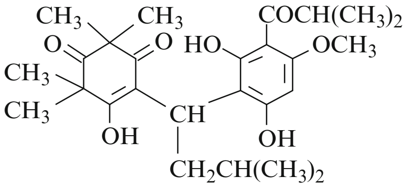
Synthesis

- Obtained by treatment of 4-[1-[2,6-Dihydroxy-4-methoxy-3-(2-methyl-1-oxopropyl) phenyl]-3-methylbutyl]-5-hydroxy-2,2,6,6-tetramethyl-4-cyclohexene-1,3-dione (compound **4**) with p-toluenesulfonic acid in refluxing benzene for 2.5 h [8135].

^{1}H NMR [8135], IR [8135], UV [8135], MS [8135].

4-[1-[2,6-Dihydroxy-4-methoxy-3-(2-methyl-1-oxopropyl)phenyl]-3-methylbutyl]-5-hydroxy-2,2,6,6-tetramethyl-4-cyclohexene-1,3-dione

[139955-98-9]



$C_{26}H_{36}O_7$ mol.wt. 460.57

Isolation from natural sources

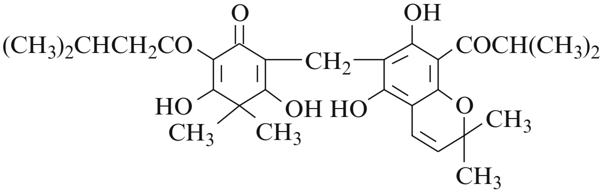
- From *Kunzea ericoides* (A. Rich) leaves and twigs (Myrtaceae) [8135].

m.p. 129–132° [8135]; TLC [8135]; ^{1}H NMR [8135], ^{13}C NMR [8135], IR [8135], UV [8135], MS [8135].

BIOLOGICAL ACTIVITY: Antiviral [8135].

2-[[5,7-Dihydroxy-2,2-dimethyl-8-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-6-yl]-methyl]-3,5-dihydroxy-4,4-dimethyl-6-(3-methyl-1-oxobutyl)-2,5-cyclohexadien-1-one *(Uliginosin B-iViB)*

[72934-91-9] $C_{29}H_{36}O_8$ mol.wt. 512.60

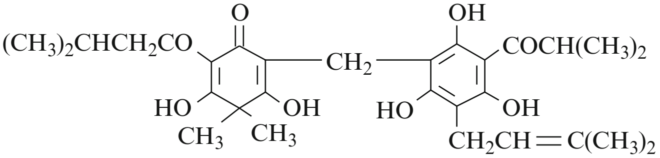


Isolation from natural sources

- From the Mexican plant *Hypericum uliginosum* HBK (natural impurity) [8138].

3,5-Dihydroxy-4,4-dimethyl-2-(3-methyl-1-oxobutyl)-6-[[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)-5-(2-methyl-1-oxopropyl)phenyl]methyl]-2,5-cyclohexadien-1-one *(Uliginosin A-iViB)*

[69299-75-8] $C_{29}H_{38}O_8$ mol.wt. 514.62



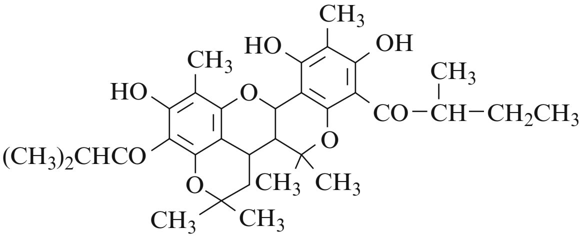
Syntheses

- Obtained by reaction of albaspidin-iViV (m.p. 133–134°) and 2,4,6-trihydroxy-3-(3-methyl-2-butenyl)-isobutyrophenone in the presence of sodium hydride in refluxing methanol for 1.5 h (74%) [8060].
- Also refer to: [8138].

m.p. 157–158° [8060]; ^{1}H NMR [8060], IR [8060].

2-Methyl-1-[1,7a,13a,13b-tetrahydro-5,8,10-trihydroxy-2,2,6,9,13,13-hexamethyl-4-(2-methyl-1-oxopropyl)-2*H*,13*H*-bis[1]benzopyrano[5,4-*bc*:3′,4′-*e*]pyran-11-yl]-1-butanone

[112613-99-7] $C_{33}H_{42}O_8$ mol.wt. 566.69



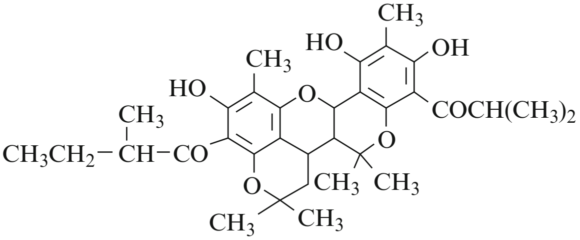
Isolation from natural sources

- From *Hypericum revolutum* VAHL (Guttiferae) (compound **4a**) [8275,8276].

m.p. 193–197° [8276]; ^{1}H NMR [8276], ^{13}C NMR [8276],
IR [8276], UV [8276],
MS [8276]; HPLC [8275]; TLC [8276].

2-Methyl-1-[1,7a,13a,13b-tetrahydro-5,8,10-trihydroxy-2,2,6,9,13,13-hexamethyl-11-(2-methyl-1-oxopropyl)-2*H*,13*H*-bis[1]benzopyrano[5,4-*bc*:3′,4′-*e*]pyran-4-yl]-1-butanone

[112614-00-3] $C_{33}H_{42}O_8$ mol.wt. 566.69



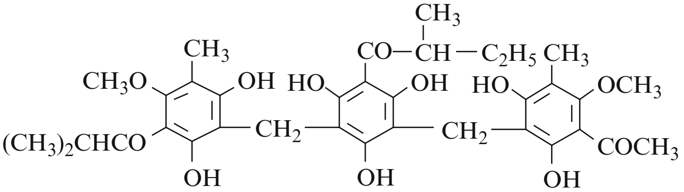
Isolation from natural sources

- From *Hypericum revolutum* VAHL (Guttiferae) (compound **4b**) [8275,8276].

m.p. 193–197° [8276]; ^{1}H NMR [8276], ^{13}C NMR [8276], IR [8276], UV [8276], MS [8276]; HPLC [8275]; TLC [8276].

1-[3-[(3-Acetyl-2,6-dihydroxy-4-methoxy-5-methylphenyl)methyl]-5-[[2,6-dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl)phenyl]methyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-butanone *(Agrimol D)*

[55576-64-2] $C_{35}H_{42}O_{12}$ mol.wt. 654.71



Synthesis

- Refer to: [8174] (Chinese paper).

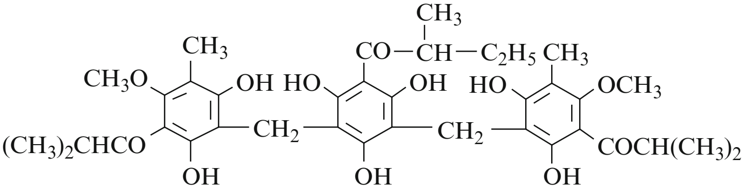
Isolation from natural sources

- From the whole plant *Agrimol pilosa*, ledeb [8471] (Chinese paper).
- From the Chinese herb medicine *Agrimonia pilosa* [8472] (Chinese paper).

m.p. 147–149° [8471]; ^{1}H NMR [8472], IR [8472], UV [8472], MS [8472].

1-[3,5-Bis[[2,6-dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl)phenyl]methyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-butanone *(Agrimol A)*

[55576-65-3] $C_{37}H_{46}O_{12}$ mol.wt. 682.77



Synthesis

- Refer to: [8174] (Chinese paper).

Isolation from natural sources

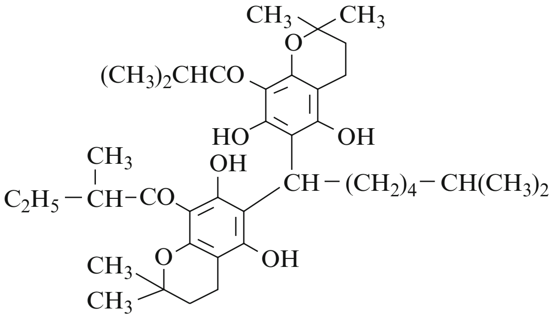
- From the whole plant *Agrimol pilosa*, ledeb [8471] (Chinese paper).
- From the Chinese herb medicine *Agrimonia pilosa* [8472] (Chinese paper).

m.p. 176–178° [8471]; ^{1}H NMR [8472], IR [8472], UV [8472], MS [8472].

1-[6-[1-[3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-8-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-6-yl]-6-methylheptyl]-3,4-dihydro-5,7-dihydroxy-2,2-dimethyl- 2*H*-1-benzopyran-8-yl]-2-methyl-1-butanone

[103771-65-9]

$C_{39}H_{56}O_8$ mol.wt. 652.87



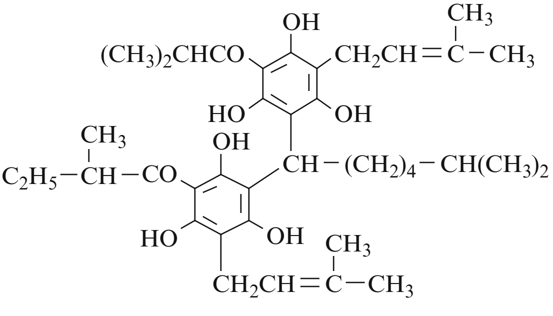
Isolation from natural sources

- From the aerial parts of *Helichrysum platypterum* DC (Compositae) (compound **6**) [8080].

2-Methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)-5-[6-methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)-5-(2-methyl-1-oxopropyl)phenyl] heptyl]phenyl]-1-butanone

[103771-73-9]

$C_{39}H_{56}O_8$ mol.wt. 652.87



Isolation from natural sources

- From the aerial parts of *Helichrysum platypterum* DC (Compositae) (compound **29**) [8080].

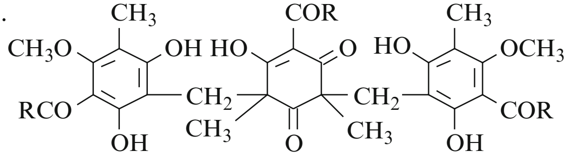
Protokosin

[1392-97-8]

Protokosin proved to be a mixture of isobutyryl, isovaleryl and 2-methylbutyryl side-chain homologues [6932]; (compound **7987**) [6640]. Protokosin was therefore represented by the general formula below:

$C_{41}H_{54}O_{12}$, $C_{40}H_{52}O_{12}$, $C_{39}H_{50}O_{12}$ and $C_{38}H_{48}O_{12}$ [6932]

.



Isolation from natural sources

- From kousso flowers *Hagenia abyssinica* (Bruce) Gmel (*Brayera anthelmintica*) (Rosaceae) [6932,8085,8178, 8198,8434,8435].

R = $-CH(CH_3)_2$ $-CH_2CH(CH_3)_2$ $-CH(CH_3)CH_2CH_3$

m.p. 182° [8198,8435], 181–183° [6932], 176° [8434], 174–176° [8473];

$(\alpha)_D^{25}$ = + 13.4°–14.4° (chloroform) [6932];

^{1}H NMR [6932], IR [6932,8085], UV [6932,8085], MS [6932].

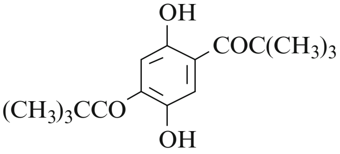
BIOLOGICAL ACTIVITY: Anthelmintic [8085].

Chapter 30
Aromatic Polyketones Containing Only Pivaloyl Groups

30.1 Pivaloyl Groups Located on the Same Ring

1,1′-(2,5-Dihydroxy-1,4-phenylene)bis[2,2-dimethyl-1-propanone

[39868-19-4]



$C_{16}H_{22}O_4$ mol.wt. 278.35

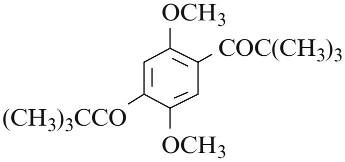
Syntheses

- Obtained by total demethylation of its dimethyl ether with 48% hydrobromic acid in refluxing acetic acid for 24 h (32%) [8310].
- Also obtained by visible light irradiation of 1,4-benzoquinone and trimethylacetaldehyde mixture for 18 days (4%) [8312].

m.p. 184–185° [8312], 180–181° [8310];
^{1}H NMR [8310,8312], IR [8310,8312], MS [8310,8312].

1,1′-(2,5-Dimethoxy-1,4-phenylene)bis[2,2-dimethyl-1-propanone

[39868-16-1]



$C_{18}H_{26}O_4$ mol.wt. 306.40

Syntheses

- Refer to: [8310,8315].

m.p. 195–199° [8310].

Note: Antioxidant [8315].

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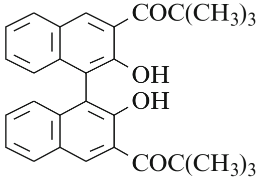
30.2 Pivaloyl Groups Located on Different Rings

1,1′-[(1*R*)-2,2′-Dihydroxy[1,1′-binaphthalene]-3,3′-diyl]bis[2,2-dimethyl-1-propanone

[574004-35-6] $C_{30}H_{30}O_4$ mol.wt. 454.57



Synthesis

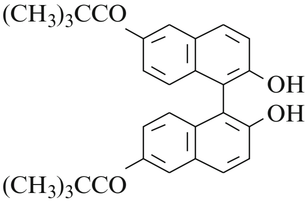
– Preparation from 3-hydroxy-2-pivaloylnaphthalene by oxidative biaryl coupling using cuprous iodide in the presence of (*S,S*)-1,5-diazadecalin in a methylene chloride/acetonitrile mixture under oxygen for 48 h at 40° (58%) (compound 9u) [7694].

^{1}H NMR [7694], ^{13}C NMR [7694], IR [7694], MS [7694];

$(\alpha)_D^{25}$ = +81° (chloroform) [7694]; TLC [7694]; HPLC [7694].

1,1′-(2,2′-Dihydroxy[1,1′-binaphthalene]-6,6′-diyl)bis[2,2-dimethyl-1-propanone

[874183-64-9] (racemic) $C_{30}H_{30}O_4$ mol.wt. 454.57
[874187-24-3] (1*R*)
[874187-25-4] (1*S*)



Synthesis

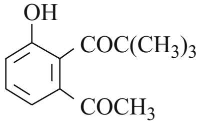
Note: Highly efficient chromatographic resolution of α,α′-dihydroxybiaryls [8474].

Chapter 31
Aromatic Polyketones Containing At Least One Pivaloyl Group

31.1 Carbonyl Groups Located on the Same Ring

1-(2-Acetyl-6-hydroxyphenyl)-2,2-dimethyl-1-propanone

[171609-27-1] $C_{13}H_{16}O_3$ mol.wt. 220.27



Synthesis

- Refer to: [8336] (Japanese patent).

N.B.: No polyketone located on different rings is described in the literature till Dec., 31, 2007.

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[18619-94-8]	(2-Hydroxy-3,6-dimethyl-4-nitrophenyl)phenylmethanone, 89
[18619-95-9]	(2-Hydroxy-3,6-dimethyl-5-nitrophenyl)phenylmethanone, 89
[18733-07-8]	(2-Hydroxyphenyl)(4-methoxyphenyl)methanone, 171
[18738-74-4]	Cyclohexyl[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone, 518
[18803-19-5]	(2-Hydroxy-5-nitrophenyl)phenylmethanone, 57
[18803-24-2]	[4-(Acetyloxy)-2-hydroxyphenyl]phenylmethanone, 86
[18803-25-3]	[4-(Benzoyloxy)-2-hydroxyphenyl]phenylmethanone, 129
[18902-63-1]	[4-(2-Bromoethoxy)-2-hydroxyphenyl]phenylmethanone, 88
[18920-70-2]	(4-Hydroxyphenyl)(4-nitrophenyl)methanone, 159
[19389-82-3]	[2-Hydroxy-4-(oxiranylmethoxy)phenyl]phenylmethanone, 104
[19390-38-6]	(2,4-Dihydroxyphenyl)(2-fluorophenyl)methanone, 397
[19434-30-1]	(2-Hydroxyphenyl)(4-methylphenyl)methanone, 167
[20010-69-9]	(3-Hydroxy-4,6-dimethyl-2-nitrophenyl)phenylmethanone, 89

[24018-76-6]	Bis(2-hydroxy-4-methylphenyl)methanone, 446
[24242-58-8]	[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]phenylmethanone, 132
[24248-99-5]	[3-(1,1-Dimethylethyl)-2-hydroxyphenyl]phenylmethanone, 114
[25138-53-8]	(4-Hydroxy-3,5-dimethoxyphenyl)(2-hydroxy-3-methoxy-5-methylphenyl)-methanone, 460
[25148-21-4]	(2-Hydroxy-5-methylphenyl)(4-hydroxyphenyl)methanone, 430
[25446-98-4]	Bis[5-(1,1-dimethylethyl)-2-hydroxyphenyl]methanone, 450
[25576-99-2]	(2,6-Dihydroxyphenyl)(3,4-dihydroxyphenyl)methanone, 32
[25577-00-8]	(2,3-Dihydroxyphenyl)(2-hydroxy-6-methoxyphenyl)methanone, 476
[25577-01-9]	(2,3-Dihydroxyphenyl)(2,6-dihydroxyphenyl)methanone, 30
[25577-03-1]	(3,4-Dihydroxyphenyl)(2,3,6-trihydroxyphenyl)methanone, 37
[25913-05-7]	(4-Fluorophenyl)(4-hydroxyphenyl)methanone, 156
[26271-33-0]	(3-Hydroxyphenyl)(2,4,6-trihydroxyphenyl)methanone, 33
[26733-16-4]	(3,5-Dibromo-4-hydroxyphenyl)phenylmethanone, 44
[26880-95-5]	(2-Hydroxy-5-methylphenyl)(4-methylphenyl)methanone, 280
[26880-96-6]	(2-Hydroxy-5-methylphenyl)(4-methoxyphenyl)methanone, 284
[26880-98-8]	(2-Hydroxy-5-methylphenyl)(3-methylphenyl)methanone, 280
[26880-99-9]	(2-Hydroxy-5-methylphenyl)(3-methoxyphenyl)methanone, 284
[26881-03-8]	(2-Hydroxy-5,6-dimethyl-3-pentylphenyl)phenylmethanone, 131
[26940-71-6]	(2-Hydroxy-6-methyl-3-pentylphenyl)phenylmethanone, 127
[26955-00-0]	(3,4-Dimethoxyphenyl)(4-hydroxyphenyl)methanone, 179
[27065-46-9]	(2,4-Dibromo-3,6-dihydroxyphenyl)phenylmethanone, 365
[27065-50-5]	(2,4-Dihydroxy-3,5-dinitrophenyl)phenylmethanone, 368
[27404-61-1]	(2-Hydroxy-5-methyl-1,3-phenylene)bis[(2-hydroxy-5-methylphenyl)-methanone, 539
[27404-62-2]	Bis(2-hydroxy-5-methylphenyl)methanone, 446
[27404-63-3]	(3-Chloro-2-hydroxy-5-methylphenyl)(2-hydroxy-5-methylphenyl)-methanone, 455
[27847-83-2]	(2-Hydroxy-4-methoxyphenyl)(2-methylphenyl)methanone, 282
[27992-95-6]	[2-Hydroxy-4-(2-methoxyethoxy)phenyl]phenylmethanone, 109
[28178-94-1]	[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]phenylmethanone, 117
[28363-58-8]	(2-Amino-5-chloro-3-hydroxyphenyl)phenylmethanone, 59
[28440-98-4]	[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl] cyclohexylmethanone, 519
[28440-99-5]	[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](4-methoxyphenyl) methanone, 356
[28441-13-6]	[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](2-methoxyphenyl) methanone, 354
[28818-29-3]	Bis(3,5-dibromo-4-hydroxyphenyl)methanone, 442
[29372-72-3]	Bis[3,5-bis(1,1-dimethylethyl)-4-hydroxyphenyl]methanone, 451
[29627-01-8]	(4-Chlorophenyl)(2,6-dihydroxyphenyl)methanone, 396
[30381-72-7]	Bis[3,5-bis(1,1-dimethylethyl)-2-hydroxyphenyl]methanone, 451
[31127-54-5]	(4-Hydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone, 34
[31188-65-5]	1-(3-Benzoyl-2,4,6-trihydroxyphenyl)ethanone, 528

[34171-64-7]	(2-Methylphenyl)(2,3,5-trichloro-6-hydroxyphenyl)methanone, 217
[34174-00-0]	(3,4-Dimethylphenyl)(2,3,5-trichloro-6-hydroxyphenyl) methanone, 259
[34174-01-1]	(2,4-Dimethylphenyl)(2,3,5-trichloro-6-hydroxyphenyl) methanone, 258
[34174-02-2]	(3-Chloro-6-hydroxy-2,4-dimethylphenyl)phenylmethanone, 88
[34174-03-3]	(3-Chloro-6-hydroxy-2,4-dimethylphenyl)(2-chlorophenyl) methanone, 262
[34174-04-4]	(4-Bromophenyl)(2-hydroxy-4,6-dimethylphenyl)methanone, 267
[34174-05-5]	(2,4-Dichloro-6-hydroxyphenyl)(2,4-dichlorophenyl) methanone, 192
[34174-06-6]	(4-Bromophenyl)(2,4-dichloro-6-hydroxyphenyl)methanone, 194
[34174-07-7]	(2,4-Dichloro-6-hydroxyphenyl)(4-methylphenyl)methanone, 223
[34174-08-8]	(2,4-Dichloro-6-hydroxyphenyl)(2-methylphenyl)methanone, 223
[34174-09-9]	(2,4-Dichloro-6-hydroxyphenyl)(2,5-dichlorophenyl)methanone, 192
[34174-11-3]	(2-Chlorophenyl)(2,4-dichloro-6-hydroxyphenyl)methanone, 198
[34174-12-4]	(2-Chlorophenyl)(2,3,5-trichloro-6-hydroxyphenyl)methanone, 191
[34174-13-5]	(3-Chloro-6-hydroxy-2-methylphenyl)(2,5-dichlorophenyl) methanone, 217
[34174-14-6]	(3,5-Dichloro-2-hydroxy-4,6-dimethylphenyl)(3,4-dichlorophenyl) methanone, 257
[34174-15-7]	(3,5-Dichloro-2-hydroxy-4,6-dimethylphenyl)(2,4-dichlorophenyl) methanone, 257
[34174-16-8]	(2-Chlorophenyl)(3,5-dichloro-2-hydroxy-4,6-dimethylphenyl) methanone, 258
[34182-96-2]	(3-Chloro-4-hydroxyphenyl)(2,4-dichlorophenyl)methanone, 197
[34182-97-3]	(4-Chlorophenyl)(3,5-dichloro-4-hydroxyphenyl)methanone, 200
[34182-98-4]	(3,5-Dichloro-4-hydroxyphenyl)(2,4-dichlorophenyl)methanone, 194
[34182-99-5]	(3-Chloro-4-hydroxy-5-methylphenyl)(2,4-dichlorophenyl) methanone, 217
[34183-00-1]	(2,6-Dichloro-4-hydroxyphenyl)(3,4-dichlorophenyl)methanone, 193
[34183-01-2]	(2,4-Dichlorophenyl)(4-hydroxyphenyl)methanone, 145
[34183-02-3]	(3,4-Dichlorophenyl)(4-hydroxy-2,6-dimethylphenyl)methanone, 264
[34183-03-4]	(2,5-Dichloro-4-hydroxyphenyl)(3,4-dichlorophenyl)methanone, 193
[34183-04-5]	(2,5-Dichloro-4-hydroxyphenyl)(2,4-dichlorophenyl)methanone, 193
[34183-05-6]	(2,3-Dichloro-4-hydroxyphenyl)(2,4-dichlorophenyl)methanone, 191
[34183-06-7]	(3,5-Dichloro-4-hydroxyphenyl)phenylmethanone, 47
[34183-07-8]	(4-Bromophenyl)(3,5-dichloro-4-hydroxyphenyl)methanone, 195
[34183-08-9]	(3-Chloro-4-hydroxy-5-methylphenyl)phenylmethanone, 66
[34183-09-0]	(3-Chloro-4-hydroxy-5-methylphenyl)(4-chlorophenyl) methanone, 221
[34183-10-3]	(3,5-Dichloro-4-hydroxyphenyl)(4-methylphenyl)methanone, 224
[34183-11-4]	(4-Chlorophenyl)(2,6-dichloro-4-hydroxyphenyl)methanone, 200
[34183-12-5]	(4-Bromophenyl)(2,6-dichloro-4-hydroxyphenyl)methanone, 195
[34183-13-6]	(2,6-Dichloro-4-hydroxyphenyl)phenylmethanone, 46

[35698-58-9]	[4-(4-Butylphenoxy)-2-hydroxyphenyl](3-methylphenyl) methanone, 357
[35698-59-0]	(4-Butoxyphenyl)[2-hydroxy-4-(4-methylphenoxy)phenyl] methanone, 358
[35698-60-3]	[2-Hydroxy-4-(4-methylphenoxy)phenyl][4-(octyloxy)phenyl] methanone, 361
[35698-61-4]	[4-(Dodecyloxy)phenyl][2-hydroxy-4-(4-methylphenoxy)phenyl] methanone, 363
[35698-62-5]	(4-Butoxyphenyl)[2-hydroxy-4-(3-methylphenoxy)phenyl] methanone, 357
[35836-41-0]	(2,3-Dihydroxy-4-methoxyphenyl)phenylmethanone, 378
[35839-45-3]	(2-Hydroxyphenyl)[4-(1-methylethyl)phenyl]methanone, 181
[35839-46-4]	[Sulfinylbis(6-hydroxy-4-methoxy-3,1-phenylene)] bis[phenylmethanone, 556
[35839-47-5]	[Sulfinylbis[6-hydroxy-4-(octyloxy)-3,1-phenylene]] bis[phenylmethanone, 557
[35839-48-6]	[Sulfonylbis(4,6-dihydroxy-3,1-phenylene)]bis [phenylmethanone, 557
[36118-66-8]	[2-Hydroxy-4-(2,4,6-trimethylphenoxy)phenyl][4-(isononyloxy) phenyl]-methanone, 362
[36130-57-1]	[1,1′-Biphenyl]-4-yl(2,4-dihydroxyphenyl)methanone, 508
[36130-58-2]	[1,1′-Biphenyl]-4-yl[2-hydroxy-4-(octyloxy)phenyl]methanone, 507
[36130-59-3]	(2,4-Dihydroxyphenyl)(2,4-dimethylphenyl)methanone, 407
[36130-60-6]	(2,4-Dimethylphenyl)[2-hydroxy-4-(pentyloxy)phenyl] methanone, 350
[36130-62-8]	[2-Hydroxy-4-(3-methylbutoxy)phenyl]phenylmethanone, 124
[36130-66-2]	[2-Hydroxy-4-[2-(octylthio)ethoxy]phenyl]phenylmethanone, 138
[36130-67-3]	[4-(Dodecyloxy)-2-hydroxyphenyl](4-methylphenyl)methanone, 359
[36130-68-4]	[2-Hydroxy-4-(octadecyloxy)phenyl](4-methoxyphenyl) methanone, 363
[36412-61-0]	(2-Hydroxyphenyl)(3-nitrophenyl)methanone, 157
[36414-88-7]	(3,4-Dichlorophenyl)[2-hydroxy-4-(octyloxy)phenyl] methanone, 351
[36414-89-8]	[1,1′-Biphenyl]-4-yl[2-hydroxy-4-(1-propylbutoxy)phenyl] methanone, 507
[36414-90-1]	[1,1′-Biphenyl]-4-yl[4-(2-butenyloxy)-2-hydroxyphenyl] methanone, 506
[36414-91-2]	[4-(4-Aminophenoxy)-2-hydroxyphenyl][1,1′-biphenyl]-4-ylmethanone, 507
[36414-93-4]	1-[2-(2,4-Dihydroxybenzoyl)phenyl]ethanone, 526
[36414-94-5]	(2,4-Dihydroxyphenyl)(4′-nitro[1,1′-biphenyl]-4-yl)methanone, 508
[36414-95-6]	[1,1′-Biphenyl]-4-yl[4-(cyclohexyloxy)-2-hydroxyphenyl] methanone, 507
[36415-12-0]	[4-(Acetyloxy)-2-hydroxyphenyl][1,1′-biphenyl]-4-yl methanone, 505

[39803-63-9]	(3,5-Dichloro-2-hydroxy-4-methoxy-6-methylphenyl)(2,4-dihydroxy-6-methyl-phenyl)methanone, 483
[39803-81-1]	(3,5-Dibromo-2-hydroxy-6-methoxy-4-methylphenyl)(2,4-dihydroxy-6-methyl-phenyl)methanone, 482
[40444-43-7]	(2,4-Dihydroxyphenyl)(4-methylphenyl)methanone, 402
[40990-66-7]	(2,4-Dibromo-6-hydroxy-3-methoxyphenyl)phenylmethanone, 64
[40990-70-3]	(2,5-Dihydroxy-4-nitrophenyl)phenylmethanone, 372
[40990-72-5]	(2-Hydroxy-5-methoxy-4-nitrophenyl)phenylmethanone, 71
[40990-74-7]	(2-Bromo-6-hydroxy-3-methoxy-4-nitrophenyl)phenylmethanone, 63
[40990-79-2]	(2,4-Dihydroxy-5-nitrophenyl)phenylmethanone, 372
[41123-21-1]	(2-Hydroxy-4-methoxy-5-nitrophenyl)phenylmethanone, 71
[41351-30-8]	(2,4-Dimethoxyphenyl)(4-hydroxyphenyl)methanone, 178
[41351-32-0]	(4-Hydroxyphenyl)(2,4,6-trimethoxyphenyl)methanone, 184
[41796-26-3]	(3-Chloro-2-hydroxyphenyl)(3-chlorophenyl)methanone, 207
[42019-78-3]	(4-Chlorophenyl)(4-hydroxyphenyl)methanone, 153
[42045-60-3]	(3,4-Dimethoxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone, 306
[42045-61-4]	(4,4′-Dihydroxy-6,6′-dimethoxy[1,1′-biphenyl]-3,3′-diyl)bis[(4-methoxyphenyl)-methanone, 551
[42045-62-5]	(4,4′-Dihydroxy-6,6′-dimethoxy[1,1′-biphenyl]-3,3′-diyl)bis[phenylmethanone, 550
[42045-63-6]	(2,5-Dihydroxy-4-methoxyphenyl)(4-methoxyphenyl)methanone, 420
[42204-63-7]	(2-Hydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone, 32
[42404-41-1]	(3-Amino-4-hydroxyphenyl)phenylmethanone, 61
[42470-88-2]	(2,4-Dihydroxyphenyl)(5-hydroxy-2-methylphenyl)methanone, 475
[42470-91-7]	(3,6-Dihydroxy-2,4-dimethylphenyl)(2,4-dihydroxyphenyl)methanone, 492
[42594-58-1]	(2,6-Dimethoxyphenyl)(3-hydroxy-6-methoxy-2,4-dimethylphenyl) methanone, 336
[42594-59-2]	(3,6-Dihydroxy-2,4-dimethylphenyl)(2-hydroxy-6-methoxyphenyl) methanone, 485
[42594-60-5]	(3,6-Dihydroxy-2,4-dimethylphenyl)(2,6-dihydroxyphenyl)methanone, 492
[42832-64-4]	(2-Hydroxy-5-methoxyphenyl)(2,4,6-trimethoxyphenyl)methanone, 328
[42833-48-7]	(2-Hydroxy-4,5-dimethoxyphenyl)(2-methoxyphenyl)methanone, 307
[42833-51-2]	(2-Hydroxy-5-methoxyphenyl)(2-methoxyphenyl)methanone, 288
[42833-53-4]	(2,6-Dimethoxyphenyl)(2-hydroxy-4,5-dimethoxyphenyl)methanone, 326
[42833-55-6]	(2,6-Dimethoxyphenyl)(2-hydroxy-3,4-dimethoxyphenyl)methanone, 326

[52196-47-1]	[5-Chloro-3-(1,1-dimethylethyl)-2-hydroxyphenyl] phenylmethanone, 113
[52220-71-0]	(2,4-Dihydroxy-5-methylphenyl)phenylmethanone, 376
[52220-72-1]	[2-Hydroxy-3-methyl-4-(octyloxy)phenyl]phenylmethanone, 137
[52220-73-2]	[2-Hydroxy-5-methyl-4-(octyloxy)phenyl]phenylmethanone, 137
[52479-85-3]	(2,3,4-Trihydroxyphenyl)(3,4,5-trihydroxyphenyl)methanone, 40
[52591-10-3]	(4-Hydroxyphenyl)(2,4,6-trihydroxyphenyl)methanone, 34
[52811-37-7]	(2,5-Dihydroxy-4-methoxyphenyl)phenylmethanone, 379
[52811-38-8]	(5-Ethoxy-2-hydroxy-4-methoxyphenyl)phenylmethanone, 108
[52870-68-5]	(2,3-Dihydroxyphenyl)phenylmethanone, 11
[52980-94-6]	(5-Chloro-2-hydroxyphenyl)(2-methylphenyl)methanone, 234
[52980-95-7]	(5-Chloro-2-hydroxyphenyl)(3-methylphenyl)methanone, 234
[52981-01-8]	(4-Hydroxyphenyl)(2-methylphenyl)methanone, 168
[53250-52-5]	(3,5-Dihydroxyphenyl)(2,4,6-trihydroxyphenyl)methanone, 38
[53250-54-7]	(3,5-Dimethoxyphenyl)(2-hydroxy-4,6-dimethoxyphenyl) methanone, 327
[53271-51-5]	(2-Hydroxy-5-methylphenyl)(2-methoxyphenyl)methanone, 284
[53347-30-1]	(5-Chloro-2-hydroxy-3-methylphenyl)phenylmethanone, 67
[53669-31-1]	(2-Hydroxy-5-methylphenyl)(3-nitrophenyl)methanone, 248
[53669-32-2]	(2-Hydroxy-5-methylphenyl)(4-nitrophenyl)methanone, 248
[54439-82-6]	(3,5-Dihydroxy[1,1′-biphenyl]-2-yl)phenylmethanone, 509
[54439-89-3]	(2-Amino-4-hydroxy-6-methylphenyl)phenylmethanone, 84
[54439-92-8]	(4-Amino-2-hydroxy-6-methylphenyl)phenylmethanone, 85
[54468-79-0]	(2-Hydroxy-4-methylphenyl)(2-methoxy-4-methylphenyl) methanone, 300
[54468-80-3]	(2-Hydroxy-3,4-dimethylphenyl)(2-methoxy-3,4-dimethylphenyl) methanone, 335
[54468-82-5]	(2-Hydroxy-4,5-dimethylphenyl)(2-methoxy-4,5-dimethylphenyl) methanone, 335
[54808-42-3]	[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl] (3,4,5-trimethoxyphenyl) methanone, 358
[54808-43-4]	[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](4-hydroxy-3,5-dimethoxyphenyl)-methanone, 463
[54808-44-5]	(4-Hydroxy-3,5-dimethoxyphenyl)(4-methoxyphenyl) methanone, 309
[54903-59-2]	[3-Hydroxy-4-(methylamino)phenyl]phenylmethanone, 85
[54921-19-6]	(2-Hydroxy-4,5-dimethylphenyl)(4-methoxyphenyl)methanone, 300
[54923-64-7]	(3,4-Dichloro-2-hydroxyphenyl)phenylmethanone, 46
[55018-96-7]	(2,4-Dihydroxy-6-methylphenyl)(2,4,6-trihydroxyphenyl) methanone, 497
[55044-96-7]	[4-(1,1-Dimethylethyl)phenyl](4-hydroxyphenyl)methanone, 185
[55051-85-9]	(2,6-Dihydroxy-4-methoxyphenyl)(4-hydroxyphenyl) methanone, 476
[55051-89-3]	(2,6-Dihydroxy-4-methoxyphenyl)(4-methoxyphenyl) methanone, 420

[59746-94-0]	(2,4-Dichlorophenyl)[2-hydroxy-5-(1-methylpropyl)phenyl] methanone, 312
[59746-95-1]	(2-Chlorophenyl)[2-hydroxy-5-(1-methylpropyl)phenyl] methanone, 317
[59746-96-2]	(4-Chlorophenyl)[2-hydroxy-5-(1-methylpropyl)phenyl] methanone, 318
[59746-97-3]	[2-Hydroxy-5-(1-methylpropyl)phenyl]phenylmethanone, 118
[59802-03-8]	(2-Hydroxy-5-isononylphenyl)phenylmethanone, 136
[59954-92-6]	(2-Hydroxy-5-methoxy-4-methylphenyl)phenylmethanone, 98
[59954-93-7]	(2,5-Dihydroxy-4-methylphenyl)phenylmethanone, 377
[59954-97-1]	(2-Hydroxy-4-methoxy-5-methylphenyl)phenylmethanone, 98
[60013-02-7]	(3,4-Dichlorophenyl)(4-hydroxyphenyl)methanone, 146
[60014-09-7]	(4-Amino-3-nitrophenyl)(4-hydroxyphenyl)methanone, 161
[60044-21-5]	(4-Chlorophenyl)(4-hydroxyphenyl)methanone-^{14}C, 154
[60138-98-9]	(3-Chloro-6-hydroxy-4-methoxy-2,5-dimethylphenyl) (2,4-dihydroxy-6-methyl-phenyl)methanone, 487
[60302-91-2]	(2-Amino-3-hydroxy-5-nitrophenyl)phenylmethanone, 60
[60487-86-7]	Phenyl(3,4,5-trihydroxyphenyl)methanone, 24
[60556-46-9]	(2,6-Dihydroxy-4-methoxyphenyl)(2,4-dihydroxy-6-methylphenyl)-methanone, 495
[60556-49-2]	(4-Hydroxy-2-methoxy-6-methylphenyl)(2,4,6-trihydroxyphenyl) methanone, 493
[60805-30-3]	(4-Chloro-3-hydroxyphenyl)(4-chlorophenyl)methanone, 208
[60805-31-4]	(4-Chloro-2-hydroxyphenyl)(4-chlorophenyl)methanone, 208
[61002-51-5]	(4-Chlorophenyl)(4-hydroxy-2-methylphenyl)methanone, 239
[61002-52-6]	(3-Chlorophenyl)(4-hydroxyphenyl)methanone, 152
[61002-53-7]	(2,6-Dichlorophenyl)(4-hydroxyphenyl)methanone, 146
[61002-54-8]	(4-Hydroxyphenyl)(4-methoxyphenyl)methanone, 172
[61002-55-9]	(2,6-Dimethylphenyl)(4-hydroxyphenyl)methanone, 175
[61002-59-3]	(4-Chlorophenyl)(4-hydroxy-3,5-dimethylphenyl)methanone, 271
[61101-84-6]	(2-Chlorophenyl)(2,4,6-trihydroxyphenyl)methanone, 466
[61101-86-8]	(2,6-Dimethoxyphenyl)(2,4,6-trihydroxyphenyl)methanone, 471
[61101-87-9]	(2,4-Dichlorophenyl)(2,4,6-trihydroxyphenyl)methanone, 466
[61101-88-0]	(4-Hydroxyphenyl)(2-nitrophenyl)methanone, 159
[61227-12-1]	(2,4-Dihydroxy-6-methoxyphenyl)(3-hydroxyphenyl) methanone, 475
[61227-13-2]	(2,4-Dihydroxy-3-methylphenyl)(3-hydroxyphenyl) methanone, 473
[61227-14-3]	(2,4-Dihydroxy-5-methylphenyl)(3-hydroxyphenyl) methanone, 473
[61227-15-4]	(2,4-Dihydroxyphenyl)(5-hydroxy-2-methoxyphenyl) methanone, 477
[61227-16-5]	(2,4-Dihydroxy-6-methylphenyl)(5-hydroxy-2-methoxyphenyl) methanone, 480
[61227-17-6]	(2,4-Dihydroxy-3-methylphenyl)(5-hydroxy-2-methoxyphenyl) methanone, 480

[61234-44-4]	(2,4-Dihydroxyphenyl)(2,5-dihydroxyphenyl)methanone, 31
[61234-45-5]	(2,4-Dihydroxy-5-methylphenyl)(2,5-dihydroxyphenyl) methanone, 490
[61234-46-6]	(2,4-Dihydroxy-3-methylphenyl)(2,5-dihydroxyphenyl) methanone, 490
[61234-68-2]	(2,4-Dihydroxy-5-methylphenyl)(3,5-dihydroxyphenyl) methanone, 491
[61445-49-6]	Bis(3,4-dihydroxyphenyl)methanone, 30
[61445-50-9]	(2,4-Dihydroxyphenyl)(3,4-dihydroxyphenyl)methanone, 31
[61445-51-0]	(3,4-Dihydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone, 37
[61445-52-1]	(3,4-Dihydroxyphenyl)(2,4,5-trihydroxyphenyl)methanone, 37
[61466-73-7]	(4-Chlorophenyl)(2-ethyl-4-hydroxyphenyl)methanone, 269
[61466-78-2]	(2,4-Dichlorophenyl)(3-ethyl-2-hydroxyphenyl)methanone, 263
[61466-80-6]	(4-Chlorophenyl)(3-ethyl-2-hydroxyphenyl)methanone, 269
[61466-81-7]	(4-Chlorophenyl)(2-hydroxy-5-propylphenyl)methanone, 293
[61466-83-9]	(2,4-Dichlorophenyl)(5-ethyl-2-hydroxyphenyl)methanone, 263
[61466-85-1]	(3-Chlorophenyl)(5-ethyl-2-hydroxyphenyl)methanone, 269
[61466-87-3]	(3,4-Dichlorophenyl)(5-ethyl-2-hydroxyphenyl)methanone, 264
[61466-88-4]	(4-Ethyl-2-hydroxyphenyl)(3-fluorophenyl)methanone, 272
[61709-37-3]	(2,4-Dichlorophenyl)[5-(1,1-dimethylethyl)-2-hydroxyphenyl] methanone, 311
[61736-69-4]	(2-Nitrophenyl)(2,4,6-trihydroxyphenyl)methanone, 468
[61736-72-9]	(2-Aminophenyl)(2-hydroxy-4,6-dimethoxyphenyl) methanone, 298
[61736-75-2]	(2-Hydroxy-4,6-dimethoxyphenyl)(2-nitrophenyl)methanone, 277
[61750-25-2]	(5-Ethyl-2-hydroxyphenyl)(4-methylphenyl)methanone, 297
[61750-26-3]	(4-Chlorophenyl)(4,5-diethyl-2-hydroxyphenyl)methanone, 316
[61750-29-6]	(5-Ethyl-2-hydroxyphenyl)[4-(trifluoromethyl)phenyl] methanone, 290
[61785-35-1]	(5-Chloro-2-hydroxyphenyl)(2-chlorophenyl)methanone, 208
[61785-36-2]	(5-Chloro-2-hydroxyphenyl)(3-chlorophenyl)methanone, 208
[61785-37-3]	(5-Chloro-2-hydroxyphenyl)(4-chlorophenyl)methanone, 209
[61852-15-1]	(3-Chloro-6-hydroxy-4-methoxy-2,5-dimethylphenyl) (2,4-dihydroxy3,6-dimethyl-phenyl)methanone, 487
[61871-78-1]	(2-Amino-5-hydroxyphenyl)(2-chlorophenyl)methanone, 213
[62064-85-1]	(4-Hydroxy-3-methylphenyl)(3-methylphenyl)methanone, 282
[62064-88-4]	[3-(Hydroxymethyl)phenyl](4-Hydroxy-3-methylphenyl) methanone, 285
[62064-89-5]	[4-Hydroxy-3-(hydroxymethyl)phenyl](3-methylphenyl) methanone, 282
[62261-95-4]	(2-Hydroxy-4,6-dimethylphenyl)(4-methylphenyl)methanone, 298
[62261-96-5]	(2-Hydroxy-4,6-dimethylphenyl)(2-methylphenyl)methanone, 298
[62262-03-7]	(4-Hydroxy-2,5-dimethylphenyl)phenylmethanone, 94
[62433-26-5]	(4-Chlorophenyl)(5-fluoro-2-hydroxyphenyl)methanone, 204
[62433-27-6]	(5-Fluoro-2-hydroxyphenyl)(2-nitrophenyl)methanone, 209

[63565-02-6]	[4-Hydroxy-3-(3-methyl-2-butenyl)phenyl]phenylmethanone, 121
[63565-03-7]	[2-Hydroxy-3,5-bis(3-methyl-2-butenyl)-4-[(3-methyl-2-butenyl) oxy]-phenyl]phenylmethanone, 140
[63565-04-8]	[2,4-Dihydroxy-3-(3-methyl-2-butenyl)phenyl] phenylmethanone, 385
[63565-06-0]	[2-Hydroxy-3-(3-methyl-2-butenyl)-4-[(3-methyl-2-butenyl)oxy] phenyl]-(4-hydroxyphenyl)methanone, 440
[63565-08-2]	[2-Hydroxy-3-(3-methyl-2-butenyl)-4,6-bis[(3-methyl-2-butenyl) oxy]-phenyl]phenylmethanone, 140
[64357-90-0]	(4-Hydroxyphenyl)(4-propylphenyl)methanone, 182
[64357-91-1]	(4-Hydroxyphenyl)(4-pentylphenyl)methanone, 185
[64857-83-6]	1-(5-Benzoyl-2,4-dihydroxyphenyl)ethanone, 526
[64857-84-7]	1-(3-Benzoyl-2-hydroxy-4-methoxyphenyl)ethanone, 524
[65185-33-3]	(5-Chloro-2-hydroxyphenyl)(2-fluorophenyl)methanone, 202
[65202-31-5]	(2-Hydroxy-3-methoxyphenyl)phenylmethanone, 79
[65202-37-1]	(2,3-Dihydroxy-4,6-dinitrophenyl)phenylmethanone, 368
[65202-42-8]	(4,6-Dibromo-2,3-dihydroxyphenyl)phenylmethanone, 366
[65202-46-2]	(2-Hydroxy-3-methoxy-6-nitrophenyl)phenylmethanone, 70
[65202-49-5]	(4-Bromo-2-hydroxy-3-methoxyphenyl)phenylmethanone, 65
[65221-06-9]	[4-(Hexyloxy)-2-hydroxyphenyl](2-hydroxyphenyl) methanone, 438
[65221-07-0]	[2,4-Dihydroxy-3-(1-phenylethyl)phenyl]phenylmethanone, 389
[65611-78-1]	(2-Hydroxy-3-methylphenyl)(4-nitrophenyl)methanone, 247
[65611-79-2]	(2-Hydroxy-3-methylphenyl)(4-methoxyphenyl)methanone, 283
[65953-50-6]	[2-Hydroxy-4-(octadecanoyloxy)phenyl]phenylmethanone, 142
[66306-91-0]	(5-Chloro-2-hydroxy-4-nitrophenyl)phenylmethanone, 45
[66476-03-7]	(3-Hydroxy-4-methoxyphenyl)phenylmethanone, 83
[66625-08-9]	[2,4-Dihydroxy-3,5-bis[(trifluoromethyl)thio]phenyl] phenylmethanone, 381
[66666-07-7]	(2-Hydroxy-4-methoxy-5-nitrophenyl)(4-methoxyphenyl) methanone, 278
[66666-08-8]	(2-Hydroxy-4-methoxy-3,5-dinitrophenyl)(4-methoxyphenyl) methanone, 266
[66666-17-9]	(5-Ethyl-2-hydroxy-4-methoxyphenyl)(4-methoxyphenyl) methanone, 324
[66666-25-9]	(3,5-Dibromo-2-hydroxy-4-methoxyphenyl)(4-methoxyphenyl) methanone, 261
[66787-22-2]	(2,4-Dihydroxyphenyl)(4-ethenylphenyl)methanone, 406
[66802-91-3]	(2,4-Dihydroxyphenyl)(4-ethylphenyl)methanone, 407
[66832-95-9]	1,4-Phenylenebis[(2-hydroxyphenyl)methanone, 537
[67097-17-0]	[3-Bromo-6-hydroxy-4-methoxy-5-methyl-2-(1-methylpropyl) phenyl] (2,4-dihydroxy-6-methylphenyl)methanone, 488
[67217-94-1]	(3-Ethyl-4-hydroxyphenyl)phenylmethanone, 90
[67246-02-0]	(2,4-Dimethoxyphenyl)(4-hydroxy-3,5-dinitrophenyl) methanone, 265

[67246-03-1]	(2,4-Dihydroxy-3,5,6-trinitrophenyl)(4-hydroxy-3-nitrophenyl) methanone, 480
[67246-05-3]	(2-Hydroxy-4-methoxy-5-nitrophenyl)(4-hydroxy-3-nitrophenyl) methanone, 454
[67246-06-4]	(2-Hydroxy-5-methyl-3-nitrophenyl)(3-nitrophenyl)methanone, 230
[67246-07-5]	(2,4-Dihydroxy-3,5,6-trinitrophenyl)(4-methoxy-3-nitrophenyl) methanone, 416
[67286-44-6]	(2,4-Dimethoxyphenyl)(4-hydroxy-3-nitrophenyl)methanone, 277
[67548-59-8]	(3-Chlorophenyl)(2-hydroxy-4-methylphenyl)methanone, 236
[68048-15-7]	(3-Chloro-6-hydroxy-2,4-dimethoxyphenyl)(2,4-dimethoxy-6-methylphenyl)methanone, 331
[68048-17-9]	(3-Chloro-6-hydroxy-2,4-dimethoxyphenyl)(2,4-dihydroxy-6-methylphenyl)methanone, 484
[68048-19-1]	(3-Chloro-4,6-dihydroxy-2-methylphenyl) (2,4,6-trimethoxyphenyl)methanone, 421
[68048-21-5]	(3-Chloro-4,6-dimethoxy-2-methylphenyl)(3-chloro-6-hydroxy-2,4-dimethoxyphenyl)methanone, 330
[68048-23-7]	(3-Chloro-4,6-dihydroxy-2-methylphenyl)(3-chloro-6-hydroxy-2,4-dimethoxy-phenyl)methanone, 484
[68048-30-6]	(3-Chloro-4,6-dihydroxy-2-methylphenyl) (2,4,6-trihydroxyphenyl)methanone, 496
[68048-31-7]	(3-Chloro-2,4,6-trihydroxyphenyl)(2,4-dihydroxy-6-methylphenyl)methanone, 499
[68048-32-8]	(3-Chloro-4,6-dihydroxy-2-methylphenyl)(3-chloro-2,4,6-trihydroxyphenyl)methanone, 499
[68223-20-1]	(2-Hydroxyphenyl)(4-nitrophenyl)methanone, 158
[68223-56-3]	Phenyl(2,4,6-trihydroxy-3-methylphenyl)methanone, 464
[68430-99-9]	(2-Hydroxy-4-methyl-5-nitrophenyl)phenylmethanone, 69
[68436-77-1]	(2,6-Dihydroxy-4-methylphenyl)phenylmethanone, 377
[68751-90-6]	(5-Chloro-2-hydroxy-4-methylphenyl)phenylmethanone, 67
[69169-87-5]	(2,4-Dihydroxy-3-nitrophenyl)(2-hydroxyphenyl)methanone, 472
[69210-88-4]	Cyclohexyl(2-hydroxy-4-methoxyphenyl)methanone, 517
[69218-66-2]	(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)(4-hydroxy-2-methyl-6-propoxy-phenyl)methanone, 488
[69443-76-1]	[2,4-Dihydroxy-3,5-bis(3-methyl-2-butenyl)phenyl] phenylmethanone, 390
[69451-08-7]	[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl](4-fluorophenyl) methanone, 353
[69471-29-0]	(4-Ethoxyphenyl)(2,3,4-trihydroxyphenyl)methanone, 470
[69471-31-4]	(4-Ethoxyphenyl)(2-hydroxy-3,4-dimethoxyphenyl)methanone, 324
[69471-32-5]	(2,5-Dihydroxy-3,4-dimethoxyphenyl)(4-ethoxyphenyl) methanone, 422
[69471-33-6]	(5-Ethoxy-2-hydroxy-3,4-dimethoxyphenyl)(4-ethoxyphenyl) methanone, 346
[69709-89-3]	(3-Chloro-4,6-dihydroxy-2-methylphenyl)(3,5-dichloro-2-hydroxy-4-methoxy-6-methylphenyl)methanone, 481

[72324-24-4]	(4-Hydroxy-3-methylphenyl)(2-methoxyphenyl)methanone, 285
[72482-00-9]	(3-Chloro-2-hydroxy-4-methoxyphenyl)(2,3-difluorophenyl) methanone, 216
[72482-07-6]	(3-Chloro-2-hydroxy-4-methoxyphenyl)(2-fluorophenyl) methanone, 219
[72482-10-1]	(3-Chloro-2-hydroxy-4-methoxyphenyl)(2,5-difluorophenyl) methanone, 216
[72482-16-7]	(3-Chloro-2-hydroxy-4-methoxyphenyl)[2-(trifluoromethyl) phenyl]-methanone, 257
[72482-27-0]	(2-Chloro-6-hydroxy-4-methoxyphenyl)(2-fluorophenyl) methanone, 219
[72482-30-5]	(2,3-Dichloro-4-hydroxyphenyl)(2-hydroxyphenyl)methanone, 423
[72482-40-7]	(2,3-Dichloro-4-hydroxyphenyl)(3-fluorophenyl)methanone, 196
[72482-75-8]	(2,3-Dichloro-4-hydroxyphenyl)(3,4-dichlorophenyl) methanone, 192
[72482-80-5]	(2-Chlorophenyl)(2,3-dichloro-4-hydroxyphenyl)methanone, 198
[72482-84-9]	(2,3-Dichloro-4-hydroxyphenyl)(2-methylphenyl)methanone, 222
[72482-89-4]	(2,3-Dichloro-4-hydroxyphenyl)(2,3-dimethylphenyl) methanone, 263
[72483-03-5]	(2-Fluorophenyl)(2-hydroxy-4-methoxy-3-methylphenyl) methanone, 274
[72498-54-5]	(2,3-Dichloro-4-hydroxyphenyl)(2-fluorophenyl)methanone, 196
[72498-72-7]	(2,3-Dichloro-4-hydroxyphenyl)(4-methylphenyl)methanone, 223
[72498-76-1]	(4-Chlorophenyl)(2,3-dichloro-4-hydroxyphenyl)methanone, 199
[72614-88-1]	(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)(4-hydroxy-2-methyl-6-propoxy-phenyl)methanone, 487
[73720-57-7]	[4-Hydroxy-3-(1-methyl-2-propenyl)phenyl]phenylmethanone, 110
[73720-75-9]	[4-Hydroxy-3-(2-propenyl)phenyl]phenylmethanone, 104
[74079-07-5]	[2-Hydroxy-4-methoxy-3-(2-propenyl)phenyl](4-methoxyphenyl) methanone, 330
[74167-86-5]	(4-Chlorophenyl)(3-hydroxy-2-methylphenyl)methanone, 239
[74167-87-6]	(3-Hydroxy-2-methylphenyl)phenylmethanone, 76
[74167-88-7]	(2,4-Dichlorophenyl)(3-hydroxy-2-methylphenyl)methanone, 225
[74167-89-8]	(3-Hydroxy-2-methylphenyl)(4-methylphenyl)methanone, 281
[74167-90-1]	(2,4-Dimethylphenyl)(3-hydroxyphenyl)methanone, 175
[74177-55-2]	(4-Chlorophenyl)(3-hydroxy-4-methylphenyl)methanone, 239
[74627-90-0]	[2-Hydroxy-4,6-bis(methoxymethoxy)-3-methylphenyl] phenylmethanone, 124
[74627-92-2]	[4,6-Dihydroxy-3-methyl-2-(phenylmethoxy)phenyl] phenylmethanone, 389
[74627-93-3]	[6-Hydroxy-4-methoxy-3-methyl-2-(phenylmethoxy)phenyl] phenyl-methanone, 135
[74628-36-7]	(4-Hydroxy-2,6-dimethoxyphenyl)(2-hydroxy-4-methoxy-6-methylphenyl)-methanone, 459
[74628-37-8]	(2-Hydroxy-4-methoxy-6-methylphenyl)(2,4,6-trimethoxyphenyl) methanone, 338

[77151-84-9]	(2,3-Dichlorophenyl)(2-hydroxy-5-methylphenyl)methanone, 224
[77156-44-6]	(2,6-Dichlorophenyl)(2-hydroxy-4-methoxyphenyl) methanone, 229
[77347-19-4]	(2-Hydroxy-5-methyl-1,3-phenylene)bis[phenylmethanone, 529
[78023-64-0]	[3-Chloro-6-hydroxy-4-methoxy-5-methyl-2-(1-methylpropyl) phenyl]-(2,4-dihydroxy-6-methylphenyl)methanone, 488
[78044-92-5]	(2,4-Dimethoxy-6-methylphenyl)(2-hydroxy-5-methoxyphenyl) methanone, 324
[78044-94-7]	(2,5-Dihydroxyphenyl)(2,4-dimethoxy-6-methylphenyl) methanone, 408
[78044-96-9]	(2,5-Dihydroxyphenyl)(2-hydroxy-4-methoxy-6-methylphenyl) methanone, 477
[78135-54-3]	(3-Chloro-4,6-dihydroxy-2-methylphenyl)(3-chloro-6-hydroxy-4-methoxy-2-methylphenyl)methanone, 483
[78135-60-1]	Bis(2-hydroxy-4-methoxy-6-methylphenyl)methanone, 449
[78235-18-4]	(2,3-Dichloro-4-hydroxyphenyl)(4-methoxyphenyl) methanone, 228
[78473-49-1]	(4-Ethyl-2-hydroxyphenyl)(4-nitrophenyl)methanone, 275
[78473-50-4]	(4-Ethyl-2-hydroxyphenyl)phenylmethanone, 90
[78563-16-3]	(5-Fluoro-2-hydroxy-1,3-phenylene)bis[(5-fluoro-2-hydroxyphenyl)-methanone, 534
[78563-18-5]	(2,5-Dihydroxy-1,3-phenylene)bis[(2,5-dihydroxyphenyl) methanone, 537
[78563-33-4]	Bis[5-chloro-3-(5-chloro-2-hydroxybenzoyl)-2-hydroxyphenyl] methanone, 540
[78563-35-6]	Bis[5-fluoro-3-(5-fluoro-2-hydroxybenzoyl)-2-hydroxyphenyl] methanone, 540
[78697-41-3]	(2,3-Dichloro-4-hydroxyphenyl)(4-hydroxyphenyl) methanone, 424
[78930-16-2]	(4-Hydroxyphenyl)(4-phenoxyphenyl)methanone, 187
[78930-23-1]	(3-Bromo-4-chlorophenyl)(4-hydroxyphenyl)methanone, 143
[79002-05-4]	(4-Hydroxy-3-methylphenyl)(4-methoxy-3-methylphenyl) methanone, 301
[79204-64-1]	(2-Hydroxy-4-methoxy-5-nitrophenyl)(2-methoxyphenyl) methanone, 278
[79204-68-5]	(2,4-Dihydroxy-3,5-dinitrophenyl)(2-methoxyphenyl) methanone, 417
[79204-71-0]	(2-Hydroxy-4-methoxy-3,5-dinitrophenyl)(2-methoxyphenyl) methanone, 266
[79215-32-0]	(2,4-Dihydroxyphenyl)(2-methoxyphenyl)methanone, 404
[79557-81-6]	(2,4-Dihydroxy-3-propylphenyl)phenylmethanone, 384
[79578-62-4]	[4-(2-Bromoethoxy)phenyl](4-hydroxyphenyl)methanone, 174
[79578-67-9]	[4-(2-Bromoethoxy)phenyl](4-hydroxy-3-iodophenyl) methanone, 260
[79616-16-3]	Bis(3-chloro-4-hydroxyphenyl)methanone, 444

[86432-12-4]	(Oxydi-4,1-phenylene)bis(4-hydroxyphenyl)methanone, 555
[86432-13-5]	(4′-Hydroxy[1,1′-biphenyl]-4-yl)(4-hydroxyphenyl)methanone, 510
[86914-72-9]	(5-Chloro-2-hydroxy-3-methylphenyl)(4-chlorophenyl) methanone, 222
[86914-74-1]	(5-Chloro-2-hydroxy-3-methylphenyl)(4-ethylphenyl) methanone, 293
[86914-75-2]	(5-Chloro-2-hydroxy-3-methylphenyl)[4-(1-methylethyl)phenyl] methanone, 315
[86914-77-4]	(5-Chloro-2-hydroxy-3-methylphenyl)(2-methylphenyl) methanone, 267
[86914-78-5]	(2,5-Dimethylphenyl)(2-hydroxy-3,5-dimethylphenyl) methanone, 322
[86914-79-6]	(2-Hydroxy-3,5-dimethylphenyl)(2-methylphenyl)methanone, 297
[86914-80-9]	(2,4-Dimethylphenyl)(2-hydroxy-3,5-dimethylphenyl) methanone, 322
[86914-81-0]	(2-Bromophenyl)(2-hydroxy-3,5-dimethylphenyl)methanone, 266
[86914-82-1]	(5-Chloro-2-hydroxy-3-methylphenyl)(2-chlorophenyl) methanone, 221
[86914-83-2]	(5-Chloro-2-hydroxy-3-methylphenyl)(4-chloro-2-methylphenyl) methanone, 263
[86914-84-3]	(4-Chlorophenyl)(2-hydroxy-3,5-dimethylphenyl)methanone, 270
[86914-85-4]	(2-Hydroxy-3,5-dimethylphenyl)(4-methylphenyl)methanone, 298
[86914-86-5]	(5-Chloro-2-hydroxy-3-methylphenyl)(4-methylphenyl) methanone, 268
[86914-87-6]	(5-Chloro-2-hydroxy-3-methylphenyl)(3-chlorophenyl) methanone, 221
[86914-88-7]	(3,4-Dimethylphenyl)(2-hydroxy-3,5-dimethylphenyl) methanone, 322
[86914-89-8]	(5-Chloro-2-hydroxy-3-methylphenyl)(3,4-dimethylphenyl) methanone, 293
[86914-90-1]	(5-Chloro-2-hydroxy-3-methylphenyl)(3-methylphenyl) methanone, 268
[87118-99-8]	(5-Chloro-2-hydroxy-4-methoxyphenyl)(4-chlorophenyl) methanone, 228
[87119-01-5]	(5-Amino-2,4-dihydroxyphenyl)phenylmethanone, 374
[87119-02-6]	(5-Amino-2,4-dihydroxyphenyl)phenylmethanone *(Hydrochloride)*, 374
[87119-03-7]	(3-Amino-2,4-dihydroxyphenyl)phenylmethanone, 373
[87119-04-8]	(3-Amino-2,4-dihydroxyphenyl)phenylmethanone *(Hydrochloride)*, 374
[87119-05-9]	(3-Amino-5-chloro-2,4-dihydroxyphenyl)(4-chlorophenyl) methanone, 415
[87119-06-0]	(3-Amino-5-chloro-2,4-dihydroxyphenyl)(4-chlorophenyl) methanone *(Hydrochloride)*, 415
[87750-63-8]	(2-Chlorophenyl)(4-fluoro-2-hydroxyphenyl)methanone, 203

[87750-64-9]	(2-Chlorophenyl)(2-fluoro-4-hydroxyphenyl)methanone, 203
[87855-75-2]	(3-Amino-4-hydroxyphenyl)phenylmethanone *(Hydrochloride)*, 61
[88133-95-3]	(2,5-Dimethoxyphenyl)(2-hydroxy-4,5-dimethoxyphenyl) methanone, 325
[88331-62-8]	(2,5-Dihydroxyphenyl)(2,6-dihydroxyphenyl)methanone, 32
[89899-44-5]	(3-Bromo-4-hydroxyphenyl)phenylmethanone, 50
[90986-69-9]	[1,1′-Biphenyl]-4-yl(2-hydroxy-4-methoxyphenyl)methanone, 505
[91197-04-5]	(2-Bromophenyl)(4,5-dihydroxy-2-methylphenyl)methanone, 417
[91197-05-6]	(2,6-Dichlorophenyl)(4,5-dihydroxy-2-methylphenyl) methanone, 417
[91197-06-7]	(4,5-Dihydroxy-2-methylphenyl)(2,6-dimethylphenyl) methanone, 421
[91197-07-8]	(4,5-Dihydroxy-2-methylphenyl)(2-methylphenyl)methanone, 419
[91197-10-3]	(2-Bromo-4,5-dihydroxyphenyl)phenylmethanone, 368
[91197-11-4]	(2-Chloro-4,5-dihydroxyphenyl)(3-chlorophenyl)methanone, 412
[91197-12-5]	(2-Ethyl-4,5-dihydroxyphenyl)phenylmethanone, 381
[91290-75-4]	(4-Chlorophenyl)(2,5-dihydroxyphenyl)methanone, 396
[91692-34-1]	(3-Hydroxy-2,4,6-triiodophenyl)phenylmethanone, 43
[92005-08-8]	(3-Chloro-4-hydroxyphenyl)(3-hydroxyphenyl)methanone, 426
[92005-11-3]	(4-Hydroxy-3-methylphenyl)(4-hydroxyphenyl)methanone, 432
[92005-13-5]	(4-Hydroxy-3,5-dimethylphenyl)(4-hydroxyphenyl)methanone, 436
[92005-17-9]	(3-Chloro-4-hydroxyphenyl)(4-hydroxyphenyl)methanone, 426
[92005-19-1]	(3,5-Dichloro-4-hydroxyphenyl)(4-hydroxyphenyl)methanone, 424
[92005-26-0]	(4-Hydroxy-3,5-dimethylphenyl)(3-hydroxyphenyl)methanone, 435
[92005-28-2]	(3,5-Dichloro-4-hydroxyphenyl)(3-hydroxyphenyl)methanone, 424
[92005-62-4]	(4-Hydroxy-3-methylphenyl)(3-hydroxyphenyl)methanone, 431
[92103-15-6]	(2-Chlorophenyl)(4-hydroxy-2-methylphenyl)methanone, 235
[92254-59-6]	(2,4-Dihydroxyphenyl)(2-hydroxy-4-methylphenyl)methanone, 474
[92285-27-3]	(2,3-Dichloro-4-methoxyphenyl)(4-hydroxyphenyl)methanone, 164
[92285-28-4]	(2,3-Dichloro-4-hydroxyphenyl)(4-nitrophenyl)methanone, 197
[92379-42-5]	(2,4-Dihydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone, 35
[92735-01-8]	(5-Chloro-2,3-dihydroxyphenyl)(4-chlorophenyl)methanone, 413
[92735-05-2]	(4-Chlorophenyl)(5-fluoro-2,3-dihydroxyphenyl)methanone, 411
[92739-90-7]	(2-Bromophenyl)(5-chloro-2-hydroxyphenyl)methanone, 201
[92739-91-8]	(5-Chloro-3-hexyl-2-hydroxyphenyl)(4-chlorophenyl) methanone, 343
[92739-93-0]	(3-Butyl-5-chloro-2-hydroxyphenyl)(4-methylphenyl) methanone, 331
[92739-94-1]	(5-Chloro-2-hydroxy-3-propylphenyl)(4-methylphenyl) methanone, 315
[92739-95-2]	(3-Ethyl-2-hydroxy-5-methylphenyl)(4-methylphenyl) methanone, 322
[93097-75-7]	(2-Hydroxy-5-methylphenyl)(2-hydroxyphenyl)methanone, 429
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[84795-00-6]	(4-Fluorophenyl)(2,3,4-trihydroxyphenyl)methanone, 639
[85602-45-5]	Cyclohexyl(2,4,6-trihydroxyphenyl)methanone, 644
[87119-03-7]	(3-Amino-2,4-dihydroxyphenyl)phenylmethanone, 625
[89899-44-5]	(3-Bromo-4-hydroxyphenyl)phenylmethanone, 577
[91387-68-7]	[2,6-Dihydroxy-4-methoxy-3-(3-methyl-2-butenyl)-5-[5-methyl-2-(1-methylethenyl)-5-hexenyl]phenyl](3-hydroxyphenyl)methanone (-), 641
[92005-11-3]	(4-Hydroxy-3-methylphenyl)(4-hydroxyphenyl)methanone, 634
[92005-17-9]	(3-Chloro-4-hydroxyphenyl)(4-hydroxyphenyl)methanone, 633
[92103-15-6]	(2-Chlorophenyl)(4-hydroxy-2-methylphenyl)methanone, 610
[92379-42-5]	(2,4-Dihydroxyphenyl)(2,3,4-trihydroxyphenyl)methanone, 574
[92548-90-8]	(2-Hydroxy-4-methylphenyl)(3-methylphenyl)methanone, 616
[92739-90-7]	(2-Bromophenyl)(5-chloro-2-hydroxyphenyl)methanone, 605
[93796-20-4]	Phenyl[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl] methanone, 637
[93796-23-7]	Phenyl[2,4,6-trihydroxy-3-(3-methylbutyl)phenyl]methanone, 638
[97732-63-3]	[4-(Acetylamino)phenyl](4-methoxyphenyl)methanone, 651
[98085-85-9]	Bis[5-(1,1-dimethylethyl)-2-methoxyphenyl]methanone, 636
[98155-72-7]	(4-Hydroxy-2-methylphenyl)(4-hydroxyphenyl)methanone, 634
[98155-74-9]	Bis(4-hydroxy-2-methylphenyl)methanone, 636
[98155-77-2]	(2-Chloro-4-hydroxyphenyl)(4-hydroxyphenyl)methanone, 632
[99768-27-1]	(3-Chloro-4-hydroxyphenyl)(4-nitrophenyl)methanone, 605
[107622-28-6]	(4-Chlorophenyl)(2-hydroxy-4-methylphenyl)methanone, 610
[107623-97-2]	(2-Chlorophenyl)(2-hydroxy-4-methylphenyl)methanone, 609
[108294-71-9]	(4-Fluorophenyl)(2-hydroxy-4-methylphenyl)methanone, 611

[445-38-5]	1-(3-Chloro-5-fluoro-2-hydroxyphenyl)ethanone, 668
[480-66-0]	1-(2,4,6-Trihydroxyphenyl)ethanone *(Phloroacetophenone)*, 722
[490-78-8]	1-(2,5-Dihydroxyphenyl)ethanone *(Quinacetophenone)*, 716
[493-33-4]	1-(4-Hydroxy-2-methoxyphenyl)ethanone *(Isopaeonol)*, 780
[498-02-2]	1-(4-Hydroxy-3-methoxyphenyl)ethanone *(Apocynin; Acetovanillone; Acetoguaiacone)*, 781
[528-21-2]	1-(2,3,4-Trihydroxyphenyl)ethanone *(Gallacetophenone)*, 720
[552-41-0]	1-(2-Hydroxy-4-methoxyphenyl)ethanone *(Paeonol)*, 775
[577-45-7]	1-(2,4-Dihydroxy-3,5-dimethylphenyl)ethanone *(Clavatol)*, 820
[699-83-2]	1-(2,6-Dihydroxyphenyl)ethanone *(γ-Resacetophenone)*, 718
[699-91-2]	1-(2-Hydroxy-3-methylphenyl)ethanone, 757
[699-92-3]	1-(3-Fluoro-2-hydroxyphenyl)ethanone, 691
[703-23-1]	1-(2-Hydroxy-6-methoxyphenyl)ethanone, 778
[703-29-7]	1-(2,4-Dihydroxy-6-methylphenyl)ethanone *(Orcacetophenone; Orsacetophenone; β-Orcacetophenone)*, 769
[703-97-9]	1-(3-Chloro-4-fluoro-2-hydroxyphenyl)ethanone, 667
[703-98-0]	1-(2-Hydroxy-3-methoxyphenyl)ethanone *(o-Acetovanillone)*, 775
[705-15-7]	1-(2-Hydroxy-5-methoxyphenyl)ethanone, 777
[708-53-2]	1-(2,3-Dihydroxy-4-methoxyphenyl)ethanone, 783
[713-23-5]	1-[4-Hydroxy-3-methyl-5-(1-methylethyl)phenyl]ethanone, 918
[875-59-2]	1-(4-Hydroxy-2-methylphenyl)ethanone, 765
[876-02-8]	1-(4-Hydroxy-3-methylphenyl)ethanone, 766
[1132-05-4]	1-[4-Hydroxy-3-(2-propenyl)phenyl]ethanone, 851
[1197-09-7]	1-(3,4-Dihydroxyphenyl)ethanone, 718
[1198-66-9]	1-(2-Hydroxy-3,5-dimethylphenyl)ethanone, 814
[1450-72-2]	1-(2-Hydroxy-5-methylphenyl)ethanone, 760
[1450-74-4]	1-(5-Chloro-2-hydroxyphenyl)ethanone, 687
[1450-75-5]	1-(5-Bromo-2-hydroxyphenyl)ethanone, 681
[1450-76-6]	1-(2-Hydroxy-5-nitrophenyl)ethanone, 698
[1481-27-2]	1-(4-Fluoro-2-hydroxyphenyl)ethanone, 692
[1632-59-3]	1-[4-Hydroxy-3-(1-methylethyl)phenyl]ethanone, 867
[1634-34-0]	1-(2,6-Dihydroxy-4-methylphenyl)ethanone *(γ- or p-orcacetophenone)*, 772
[1634-36-2]	1-[2-Hydroxy-5-(1-methylethyl)phenyl]ethanone, 867
[1634-62-4]	1-[3-Hydroxy-4-(1-methylethyl)phenyl]ethanone, 867
[1818-27-5]	1-(2,4,5-Trihydroxyphenyl)ethanone, 721
[1834-91-9]	1-(2-Hydroxy-4-nitrophenyl)ethanone, 697
[1836-05-1]	1-(3-Bromo-2-hydroxyphenyl)ethanone, 679
[1836-06-2]	1-(3-Bromo-4-hydroxyphenyl)ethanone, 680
[1990-24-5]	1-(2-Hydroxy-5-propylphenyl)ethanone, 868
[2476-29-1]	1-(4-Amino-2-hydroxyphenyl)ethanone, 728
[2478-38-8]	1-(4-Hydroxy-3,5-dimethoxyphenyl)ethanone *(Acetosyringone)*, 840
[2652-27-9]	1-[2-Hydroxy-3,4-bis(phenylmethoxy)phenyl]ethanone, 1070
[2657-28-5]	1-(2,4,6-Trihydroxy-3-methylphenyl)ethanone, 790

[7191-41-5]	1-(2-Hydroxy-5-iodophenyl)ethanone, 694
[7191-46-0]	1-(2-Hydroxy-3,5-diiodophenyl)ethanone, 676
[7191-55-1]	1-(4-Hydroxy-3,5-diiodophenyl)ethanone, 676
[7253-20-5]	1-(3-Bromo-6-hydroxy-2-methoxy-5-nitrophenyl)ethanone, 732
[7298-21-7]	1-(2,4-Dihydroxy-5-methoxyphenyl)ethanone, 785
[7298-39-7]	1-[2-Hydroxy-4,5-bis(phenylmethoxy)phenyl]ethanone, 1070
[7452-85-9]	1-(2-Hydroxy-3-methoxy-5-methylphenyl)ethanone, 826
[7499-99-2]	1-(2,4-Dihydroxy-3,6-dimethoxyphenyl)ethanone, 844
[7507-88-2]	1-(3-Chloro-2-hydroxy-5-methylphenyl)ethanone, 741
[7507-89-3]	1-(2,6-Dihydroxy-4-methoxyphenyl)ethanone, 787
[7507-98-4]	1-(2-Hydroxy-3,4,6-trimethoxyphenyl)ethanone *(Xanthoxylone)*, 888
[7714-14-9]	1-(2,4,5-Trichloro-3,6-dihydroxyphenyl)ethanone, 661
[7743-16-0]	1-(2,6-Dihydroxy-3,4-dimethylphenyl)ethanone, 821
[10139-84-1]	1-(2,4-Dihydroxy-3-methylphenyl)ethanone, 768
[10299-59-9]	1-[2-Hydroxy-4-methoxy-6-(phenylmethoxy)phenyl] ethanone, 1020
[13246-14-5]	1-(4-Hydroxy-2,6-dimethoxyphenyl)ethanone, 840
[13383-63-6]	1-(2,4,6-Trihydroxy-3,5-dimethylphenyl)ethanone, 843
[13494-10-5]	1-(2,3-Dihydroxyphenyl)ethanone, 713
[13667-21-5]	1-(3-Hydroxy-4,5,6-trimethyl-2-nitrophenyl)ethanone, 862
[13667-28-2]	1-(5-Hydroxy-2,3,4-trimethylphenyl)ethanone, 871
[13684-24-7]	1-(2-Hydroxy-4,6-dinitrophenyl)ethanone, 677
[13909-71-2]	1-(4-Hydroxy-2,5-dimethoxyphenyl)ethanone, 839
[14031-80-2]	1-(4-Hydroxy[1,1′-biphenyl]-3-yl)ethanone, 971
[14035-33-7]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]ethanone, 1026
[14347-14-9]	1-(3-Amino-4-hydroxyphenyl)ethanone *(Hydrochloride)*, 727
[14718-38-8]	1-(2-Hydroxy-6-propoxyphenyl)ethanone, 878
[14764-76-2]	1-[4,6-Dihydroxy-2-methoxy-3-(3-methylbutyl)phenyl] ethanone, 993
[14813-18-4]	1-[3-(1,1-Dimethylethyl)-2-hydroxy-5-methylphenyl]ethanone, 953
[14964-98-8]	1-(6-Hydroxy-2,4-dimethoxy-3-methylphenyl)ethanone (*Bancroftinone)*, 883
[15516-61-7]	1-(2-Hydroxy-5-nitrosophenyl)ethanone, 696
[15994-32-8]	1-(2,4,5-Trihydroxy-3,6-dimethoxyphenyl)ethanone, 846
[16108-50-2]	1-(2-Hydroxy-4,6-dimethylphenyl)ethanone, 816
[16130-28-2]	1-[2-Hydroxy-6-(oxiranylmethoxy)phenyl]ethanone, 856
[16139-53-0]	1-[2-Hydroxy-5-(oxiranylmethoxy)phenyl]ethanone, 856
[16290-04-3]	1-(3,5-Dibromo-2-hydroxy-6-methoxyphenyl)ethanone, 733
[16297-01-1]	1-(2,4,6-Trihydroxy-3-methoxyphenyl)ethanone, 791
[16928-01-1]	1-[3-(1,1-Dimethylethyl)-4-hydroxyphenyl]ethanone, 912
[17044-70-1]	1-(3,5-Dichloro-4-hydroxyphenyl)ethanone, 672
[17063-43-3]	1-[4-(β-D-Glucopyranosyloxy)-3-hydroxyphenyl]ethanone *(Cynanoneside A)*, 988
[17488-68-5]	1-[2-Hydroxy-6-methoxy-3-(2-propenyl)phenyl]ethanone, 900

[17488-71-0]	1-[2,6-Dihydroxy-3-(2-propenyl)phenyl]ethanone, 852
[17605-00-4]	1-(2-Hydroxy-3,5-dimethoxyphenyl)ethanone, 835
[17820-32-5]	1-[2,6-Bis(acetyloxy)-4-hydroxyphenyl]ethanone, 895
[17820-33-6]	1-[2,4-Bis(acetyloxy)-6-hydroxyphenyl]ethanone, 894
[18064-89-6]	1-(3-Bromo-2-hydroxy-4,6-dimethoxyphenyl)ethanone, 804
[18065-05-9]	1-[2-Hydroxy-4,6-bis(phenylmethoxy)phenyl]ethanone, 1070
[18065-06-0]	1-[2-Hydroxy-4,6-bis(phenylmethoxy)-3-(phenylmethyl)phenyl] ethanone, 1086
[18086-01-6]	1-(2,4-Diethoxy-6-hydroxy-3-methoxyphenyl)ethanone, 962
[18087-17-7]	1-(4,5-Dihydroxy-2-methylphenyl)ethanone, 774
[18296-18-9]	1-[3-(3,7-Dimethyl-2,6-octadienyl)-2,4,5-trihydroxyphenyl] ethanone (*Z*), 1051
[18296-19-0]	1-[3-(3,7-Dimethyl-2,6-octadienyl)-2,4-dihydroxyphenyl]ethanone (*Z*), 1048
[18606-50-3]	1-[3-(1,1-Dimethylethyl)-4-hydroxy-5-methylphenyl]ethanone, 953
[18606-87-6]	1-[4-(1,1-Dimethylethyl)-3-hydroxyphenyl]ethanone, 912
[18780-96-6]	1-[4-Hydroxy-2,6-dimethoxy-3-(3-methyl-2-butenyl)phenyl] ethanone, 1011
[19687-48-0]	1-(3,5-Diethyl-2,4,6-trihydroxyphenyl)ethanone, 928
[19825-40-2]	1-[2,4-Dihydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone, 941
[20180-88-5]	1-[2,5-Dihydroxy-4-methoxy-3-(3,7-dimethyl-2,6-octadienyl) phenyl]ethanone (*E*), 1060
[20212-64-0]	1-[2,4-Dihydroxy-5-methoxy-3-(3,7-dimethyl-2,6-octadienyl) phenyl]ethanone (*E*), 1059
[20212-65-1]	1-[2-Hydroxy-4,5-dimethoxy-3-(3,7-dimethyl-2,6-octadienyl) phenyl]ethanone (*E*), 1062
[20212-66-2]	1-[3-(3,7-Dimethyl-2,6-octadienyl)-2,4,5-trihydroxyphenyl] ethanone (*E*), 1051
[20212-67-3]	1-[3-(3,7-Dimethyl-2,6-octadienyl)-2,4-dihydroxyphenyl]ethanone (*E*), 1048
[20212-68-4]	1-[5-(3,7-Dimethyl-2,6-octadienyl)-2,4-dihydroxyphenyl]ethanone (*E*), 1049
[20281-51-0]	1-(6-Hydroxy[1,1′-biphenyl]-3-yl)ethanone, 972
[20628-06-2]	1-(2-Hydroxy-4,5-dimethoxyphenyl)ethanone, 836
[20716-41-0]	1-(4-Hydroxy-3-methoxy-5-nitrophenyl)ethanone, 756
[20951-24-0]	1-[2-Hydroxy-5-(methylsulfonyl)phenyl]ethanone, 790
[21092-94-4]	1-[3-Hydroxy-4-(phenylmethoxy)phenyl]ethanone, 1005
[21222-04-8]	1-[5-(Acetyloxy)-2-hydroxyphenyl]ethanone, 801
[21312-85-6]	1-(3-Amino-5-chloro-2-hydroxyphenyl)ethanone, 704
[21424-82-8]	1-(2-Hydroxy[1,1′-biphenyl]-3-yl)ethanone, 970
[21472-87-7]	1-(2,3-Dichloro-4-hydroxy-6-methylphenyl)ethanone, 734
[21722-31-6]	1-(2-Hydroxy-4,6-dimethoxy-3,5-dimethylphenyl)ethanone, 929
[21919-63-1]	1-[3-(Acetyloxy)-6-hydroxy-2,4-dimethoxyphenyl]ethanone, 906
[21919-65-3]	1-[3-(Acetyloxy)-2-hydroxy-4,6-dimethoxyphenyl]ethanone, 906
[21919-66-4]	1-(2,3-Dihydroxy-4,6-dimethoxyphenyl)ethanone, 843

[22089-12-9]	1-(2,5-Dihydroxy-4-methoxyphenyl)ethanone, 786
[22248-13-1]	1-(6-Hydroxy-2,3-dimethoxyphenyl)ethanone, 841
[22248-14-2]	1-(6-Hydroxy-2,3,4-trimethoxyphenyl)ethanone, 891
[22362-66-9]	1-(3,5-Dibromo-2-hydroxyphenyl)ethanone, 665
[22518-00-9]	1-[2-Hydroxy-4-(hydroxymethyl)phenyl]ethanone, 774
[22526-30-3]	1-(4,5-Dichloro-2-hydroxyphenyl)ethanone, 673
[22633-36-9]	1-(5-Hydroxy-2,4-dinitrophenyl)ethanone, 678
[22934-47-0]	1-(3-Ethyl-4-hydroxyphenyl)ethanone, 813
[23053-45-4]	1-(3-Chloro-6-hydroxy-2,4-dimethoxy-5-methylphenyl) ethanone, 861
[23053-47-6]	1-(3-Chloro-2,4,6-trihydroxy-5-methylphenyl)ethanone, 748
[23121-32-6]	1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)ethanone, 882
[23133-83-7]	1-(3-Hydroxy-2,4-dimethoxyphenyl)ethanone, 839
[23141-00-6]	1-[2-Hydroxy-6-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy] phenyl]ethanone, 1073
[23164-97-8]	1-(3-Chloro-5-hydroxyphenyl)ethanone, 686
[23226-84-8]	1-[2-Hydroxy-6-(2-propenyloxy)phenyl]ethanone, 853
[23299-00-5]	1-(3-Cyclohexyl-4-hydroxyphenyl)ethanone, 979
[23343-03-5]	1-(2-Hydroxy-3-methoxy-5-propylphenyl)ethanone, 925
[23343-04-6]	1-[2-Hydroxy-3-methoxy-5-(2-propenyl)phenyl]ethanone, 899
[23343-08-0]	1-(5-Hydroxy-4-methoxy-2-propylphenyl)ethanone, 927
[23780-60-1]	1-(4-Hydroxy-1,3-benzodioxol-5-yl)ethanone, 736
[24046-00-2]	1-(2,2′-Dihydroxy-5,5′-dimethyl[1,1′-biphenyl]-3-yl)ethanone, 1017
[24046-01-3]	1-(2′-Acetoxy-2-hydroxy-5,5′-dimethyl[1,1′-biphenyl]-3-yl) ethanone, 1044
[24085-05-0]	1-[3-(Chloromethyl)-4-hydroxyphenyl]ethanone, 744
[24126-73-6]	1-[2-Hydroxy-3-methoxy-4,6-bis(phenylmethoxy)phenyl] ethanone, 1078
[24242-55-5]	1-[3-(1,1-Dimethylethyl)-2-hydroxyphenyl]ethanone, 911
[24490-25-3]	1-(2-Chloro-6-hydroxy-4-methylphenyl)ethanone, 741
[24539-92-2]	1-(5-Ethyl-2-hydroxyphenyl)ethanone, 814
[24587-97-1]	1-[2-(β-D-Glucopyranosyloxy)-6-hydroxy-4-methoxyphenyl] ethanone, 1012
[24672-82-0]	1-[2,4-Dihydroxy-3,5-bis-(3-methyl-2-butenyl)phenyl] ethanone, 1048
[24672-83-1]	1-[2-Hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]ethanone, 944
[25205-34-9]	1-(2,6-Dihydroxy-3-nitrophenyl)ethanone, 702
[25892-94-8]	1-[3,6-Dihydroxy-2-methoxy-4-(phenylmethoxy)phenyl] ethanone, 1022
[25892-95-9]	1-[6-Hydroxy-2,3-dimethoxy-4-(phenylmethoxy)phenyl] ethanone, 1036
[26089-54-3]	1-[2-(β-D-Glucopyranosyloxy)-4,6-dihydroxyphenyl]ethanone, 988
[26207-59-0]	1-(2,4-Diethoxy-6-hydroxyphenyl)ethanone, 927
[26216-10-4]	1-(4-Hydroxy-2,5-dimethylphenyl)ethanone, 818
[26674-05-5]	1-[2-(Acetyloxy)-6-hydroxyphenyl]ethanone, 800

[26931-60-2]	1-[4-Hydroxy-3-(3-methoxy-3-methyl-1-butenyl)phenyl]ethanone *(E)*, 981
[26931-61-3]	1-[4-Hydroxy-3-(3-hydroxy-3-methyl-1-butenyl)phenyl]ethanone *(E)*, 942
[26932-05-8]	1-[4-Hydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone, 939
[27192-99-0]	1-(6-Hydroxy-2,3,4-trimethylphenyl)ethanone, 871
[27193-00-6]	1-(2,3-Diethyl-6-hydroxy-4-methylphenyl)ethanone, 951
[27364-64-3]	1-[4,6-Dihydroxy-2-methoxy-3-(3-methyl-2-butenyl)phenyl] ethanone *(Acronylin)*, 983
[27364-71-2]	1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone, 947
[27513-07-1]	1-(5-Ethyl-2-hydroxy-4-methylphenyl)ethanone, 866
[27829-93-2]	1-(3,4-Dihydroxy-2-methoxyphenyl)ethanone, 788
[27865-58-3]	1-[3,4-Bis(acetyloxy)-2-hydroxyphenyl]ethanone, 895
[27865-59-4]	1-[3,4-Bis(benzoyloxy)-2-hydroxyphenyl]ethanone, 1066
[28177-69-7]	1-(2-Hydroxy-3-nitrophenyl)ethanone, 697
[28437-37-8]	1-[2,4-Dihydroxy-5-(3-methyl-2-butenyl)phenyl]ethanone, 942
[28448-83-1]	1-[2-Hydroxy-4-methoxy-5-(3-methyl-2-butenyl)phenyl] ethanone, 981
[28467-11-0]	1-(5-Bromo-2-hydroxy-3-iodophenyl)ethanone, 663
[28480-70-8]	1-(5-Chloro-2-hydroxy-4-methylphenyl)ethanone, 743
[28862-10-4]	1-[2-[(5-Bromopentyl)oxy]-6-hydroxyphenyl]ethanone, 949
[29183-78-6]	1-(2,6-Dihydroxy-3-methylphenyl)ethanone, 771
[29376-65-6]	1-[4-(Acetyloxy)-2,6-dihydroxyphenyl]ethanone, 802
[29376-66-7]	1-[4-(Acetyloxy)-2-hydroxy-6-methoxyphenyl]ethanone, 858
[29682-12-0]	1-[2-Hydroxy-4-(phenylmethoxy)phenyl]ethanone, 1003
[30095-73-9]	1-[5-(Acetyloxy)-2-hydroxy-3-nitrophenyl]ethanone, 797
[30095-74-0]	1-(2,5-Dihydroxy-3-nitrophenyl)ethanone, 702
[30095-76-2]	1-(2-Bromo-3,6-dihydroxyphenyl)ethanone, 681
[30186-15-3]	1-(4,5-Dibromo-2-hydroxyphenyl)ethanone, 666
[30186-18-6]	1-(4-Bromo-2-hydroxyphenyl)ethanone, 680
[30186-22-2]	1-(5-Amino-3-bromo-2-hydroxyphenyl)ethanone *(Hydrochloride)*, 703
[30225-96-8]	1-(2-Hydroxy-3,4,5-trimethoxyphenyl)ethanone, 888
[30299-53-7]	2′-Hydroxy-5′-(1,1,3,3-tetramethylbutyl)acetophenone, 1028
[30299-56-0]	1-[2-Hydroxy-3-nitro-5-(1,1,3,3-tetramethylbutyl)phenyl] ethanone, 1026
[30403-01-1]	1-[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl] ethanone, 983
[30787-43-0]	1-[5-(Chloromethyl)-2-hydroxyphenyl]ethanone, 744
[30879-49-3]	1-(5-Hydroxy-2-nitrophenyl)ethanone, 700
[30954-71-3]	1-(2-Amino-5-hydroxyphenyl)ethanone, 726
[30992-63-3]	1-[2-Hydroxy-5-(phenylmethoxy)phenyl]ethanone, 1004
[30992-64-4]	1-[2-Hydroxy-3-(phenylmethoxy)phenyl]ethanone, 1003
[31273-60-6]	1-[4-[(1,1-Dimethyl-2-propynyl)oxy]-2-hydroxy-6-methoxyphenyl]ethanone, 977

[53771-29-2]	1-[2,4,6-Trihydroxy-3,5-bis(2-propenyl)phenyl]ethanone, 978
[53889-99-9]	1-[4-Hydroxy-3-(1-propenyl)phenyl]ethanone, 851
[53967-72-9]	1-(3-Hydroxy-2-nitrophenyl)ethanone, 698
[54255-50-4]	1-(3-Amino-4-hydroxyphenyl)ethanone, 727
[54299-56-8]	1-[6-Hydroxy-4-methoxy-2,3-bis(phenylmethoxy)phenyl] ethanone, 1079
[54299-57-9]	1-[2-Hydroxy-4,6-dimethoxy-3-(phenylmethoxy)phenyl] ethanone, 1035
[54337-59-6]	1-(3-Ethyl-2,6-dihydroxyphenyl)ethanone, 824
[54439-83-7]	1-(3,5-Dihydroxy[1,1′-biphenyl]-2-yl)ethanone, 972
[54439-90-6]	1-(5-Amino-3-hydroxy[1,1′-biphenyl]-2-yl)ethanone, 974
[54439-91-7]	1-(3-Amino-5-hydroxy[1,1′-biphenyl]-2-yl)ethanone, 974
[54514-40-8]	1-(4-Hydroxy-3-methoxy-5-propylphenyl)ethanone, 927
[54556-95-5]	1-(3-Chloro-4-hydroxy-5-methylphenyl)ethanone, 742
[54698-17-8]	1-(2,5-Dihydroxy-4-methylphenyl)ethanone, 771
[54903-54-7]	1-(4-Amino-3-hydroxyphenyl)ethanone, 728
[54903-57-0]	1-[3-Hydroxy-4-(methylamino)phenyl]ethanone, 794
[54917-82-7]	1-(2,4-Dihydroxy-3,5-dinitrophenyl)ethanone, 678
[54918-24-0]	1-[4-(β-D-Glucopyranosyloxy)-2-hydroxyphenyl]ethanone, 987
[54918-26-2]	1-[4-(β-D-Galactopyranosyloxy)-2-hydroxyphenyl]ethanone, 986
[54918-27-3]	1-[2-Hydroxy-4-[(2,3,4,6-tetra-O-acetyl-β-D-galactopyranosyl) oxy]-phenyl]ethanone, 1072
[54918-29-5]	1-[2-Hydroxy-4-(β-D-xylopyranosyloxy)phenyl]ethanone, 949
[54918-30-8]	1-[4-[(4-O-β-D-Glucopyranosyl-β-D-glucopyranosyl)oxy]-2-hydroxy-phenyl]ethanone, 1064
[55008-15-6]	1-(3-Amino-2-hydroxy-5-methoxyphenyl)ethanone, 795
[55168-29-1]	1-[5-(Acetyloxy)-2,4-dihydroxyphenyl]ethanone, 802
[55168-32-6]	1-(5-Hexyl-2-hydroxyphenyl)ethanone, 991
[55168-33-7]	1-(5-Cyclohexyl-2-hydroxyphenyl)ethanone, 979
[55329-63-0]	1-(2-Hydroxy-4-propoxyphenyl)ethanone, 878
[55380-57-9]	1-[2,4,6-Trihydroxy-3,5-bis(3-methylbutyl)phenyl]ethanone, 1056
[55380-58-0]	1-[2,4,6-Trihydroxy-3-methyl-5-(3-methyl-2-butenyl)phenyl] ethanone, 984
[55483-00-6]	1-[3-(β-D-Glucopyranosyloxy)-4-hydroxyphenyl]ethanone, 987
[55736-04-4]	1-(2-Chloro-6-hydroxyphenyl)ethanone, 685
[55736-69-1]	1-(2-Bromo-6-hydroxyphenyl)ethanone, 679
[55736-71-5]	1-(3,4-Dichloro-2-hydroxyphenyl)ethanone, 671
[55736-72-6]	1-(3,6-Dichloro-2-hydroxyphenyl)ethanone, 672
[55742-65-9]	1-(2,5-Dihydroxy-3,4,6-trimethoxyphenyl)ethanone, 892
[56002-87-0]	1-[2-Hydroxy-3,5,6-trimethoxy-4-(1-methylethoxy)phenyl] ethanone, 995
[56146-52-2]	1-[2,4-Dihydroxy-5-(3-methylbutyl)phenyl]ethanone, 957
[56358-73-7]	1-(2,3-Dihydroxy-6-methoxyphenyl)ethanone, 784
[56358-74-8]	1-(3-Hydroxy-2,6-dimethoxyphenyl)ethanone, 839
[56394-40-2]	1-(4,5-Diethyl-2-hydroxyphenyl)ethanone, 911

[60640-95-1]	1-[2,4-Dihydroxy-5-(2-phenylethyl)phenyl]ethanone, 1017
[60840-18-8]	1-[2-Hydroxy-3,4,6-tris(phenylmethoxy)phenyl]ethanone, 1088
[60840-21-3]	1-[3,4,6-Trihydroxy-2-(1-methylethoxy)phenyl]ethanone, 892
[60965-25-5]	1-(5-Bromo-2,4-dihydroxyphenyl)ethanone, 683
[60990-39-8]	1-(3-Bromo-2,4-dihydroxyphenyl)ethanone, 682
[61124-56-9]	1-(4-Chloro-3-hydroxyphenyl)ethanone, 687
[61270-14-2]	1-[2-Hydroxy-6-(2-phenoxyethoxy)phenyl]ethanone, 1022
[61270-17-5]	1-[2-Hydroxy-5-(3-phenylpropyl)phenyl]ethanone, 1031
[61270-18-6]	1-[6-[(5-Bromopentyl)oxy]-2-hydroxy-3-(2-propenyl)phenyl] ethanone, 1024
[61270-23-3]	1-[4-[(5-Bromopentyl)oxy]-2-hydroxy-3-(2-propenyl)phenyl] ethanone, 1024
[61270-24-4]	1-[2-Hydroxy-4-(oxiranylmethoxy)phenyl]ethanone, 856
[61270-28-8]	1-(4-Hydroxy-3-propylphenyl)ethanone, 869
[61300-15-0]	1-[2-Hydroxy-5-(phenylmethyl)phenyl]ethanone, 1001
[61405-64-9]	1-(4-Hydroxy-2,3,5-trimethylphenyl)ethanone, 870
[61405-65-0]	1-[4-Hydroxy-3-methyl-2-(1-methylethyl)phenyl]ethanone, 918
[61775-18-6]	1-[4-(β-D-Glucopyranosyloxy)-2-hydroxy-6-methoxyphenyl] ethanone *(Annphenone)*, 1012
[61791-99-9]	1-(2-Bromo-4-hydroxyphenyl)ethanone, 679
[62069-33-4]	1-(2,4-Dihydroxy-5-iodophenyl)ethanone, 696
[62069-34-5]	1-(2,4-Dihydroxy-3,5-diodophenyl)ethanone, 676
[62615-24-1]	1-(4-Hydroxy-3-iodophenyl)ethanone, 695
[62615-25-2]	1-(5-Bromo-2,4-dihydroxy-3-methoxyphenyl)ethanone, 741
[62615-26-3]	1-(2,4-Dihydroxy-3-methoxyphenyl)ethanone, 784
[62615-64-9]	1-(6-Hydroxy-3,4-dimethoxy-2-methylphenyl)ethanone, 884
[63013-36-5]	1-[2-(Acetyloxy)-6-hydroxy-4-methoxyphenyl]ethanone, 857
[63359-84-2]	1-[2-Hydroxy-4-(2-phenylethoxy)phenyl]ethanone, 1019
[63359-85-3]	1-[2-Hydroxy-5-(2-phenylethoxy)phenyl]ethanone, 1019
[63359-86-4]	1-[2-Hydroxy-4-(3-phenylpropoxy)phenyl]ethanone, 1032
[63359-87-5]	1-[2-Hydroxy-5-(3-phenylpropoxy)phenyl]ethanone, 1033
[63359-88-6]	1-[2-Hydroxy-4-(4-phenylbutoxy)phenyl]ethanone, 1046
[63411-82-5]	1-(3-Butyl-2,6-dihydroxyphenyl)ethanone, 922
[63411-84-7]	1-(3,5-Dibromo-2,6-dihydroxyphenyl)ethanone, 667
[63411-85-8]	1-[2-[(3,4-Dichlorophenyl)methoxy]-6-hydroxyphenyl] ethanone, 997
[63411-86-9]	1-[2-[(2,4-Dichlorophenyl)methoxy]-6-hydroxyphenyl] ethanone, 996
[63411-87-0]	1-(2,4-Dihydroxy-5-propylphenyl)ethanone, 872
[63411-88-1]	1-(5-Hexyl-2,4-dihydroxyphenyl)ethanone, 992
[63437-82-1]	1-[3-[2-(Acetyloxy)ethoxy]-4-hydroxyphenyl]ethanone, 905
[63437-85-4]	1-[4-Hydroxy-3-(2-hydroxyethoxy)phenyl]ethanone, 842
[63437-86-5]	1-[3-Hydroxy-5-(2-hydroxyethoxy)phenyl]ethanone, 841
[63437-94-5]	1-[4-Hydroxy-3-(2-hydroxypropoxy)phenyl]ethanone, 885
[63438-68-6]	1-(2-Butoxy-6-hydroxyphenyl)ethanone, 921

[63542-37-0]	1-(2-Hydroxy-3,4-dimethoxy-6-methylphenyl)ethanone, 881
[63609-52-9]	1-[5-Hydroxy-2-(methylamino)phenyl]ethanone, 794
[63609-62-1]	1-[2-[(1,1-Dimethylethyl)amino]-5-hydroxyphenyl]ethanone, 937
[63635-39-2]	1-(2,3,4,6-Tetrahydroxyphenyl)ethanone, 724
[63635-41-6]	1-(2-Ethoxy-3,4,6-trihydroxyphenyl)ethanone, 846
[63854-17-1]	1-[2-Hydroxy-6-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl] ethanone, 947
[63990-67-0]	1-(3,5-Dibromo-2,4,6-trihydroxyphenyl)ethanone, 667
[64648-09-5]	1-[2-Hydroxy-6-methyl-3-phenyl-4-(phenylmethyl)phenyl] ethanone, 1067
[64794-45-2]	1-(2,5-Dihydroxy-3,4,6-trimethylphenyl)ethanone, 874
[65039-99-8]	1-[5-Hydroxy-2,4-dimethoxy-3-(phenylmethoxy)phenyl] ethanone, 1035
[65490-08-6]	1-[2-Hydroxy-4-(methoxymethoxy)phenyl]ethanone, 842
[65490-09-7]	1-[2-Hydroxy-4,6-bis(methoxymethoxy)phenyl]ethanone, 935
[65883-24-1]	1-(2,3-Dibromo-4,5,6-trihydroxyphenyl)ethanone, 667
[66003-50-7]	1-(6-Hydroxy-1,3-benzodioxol-5-yl)ethanone, 736
[66108-30-3]	1-(2-Hydroxy-5-methyl-3-nitrophenyl)ethanone, 752
[66264-55-9]	1-(4-Hydroxy-3-mercaptophenyl)ethanone, 713
[66264-56-0]	1-[4-Hydroxy-3-(methylthio)phenyl]ethanone, 767
[66296-84-2]	1-(3,4-Dihydroxy-2-methylphenyl)ethanone, 773
[66296-85-3]	1-(2,3,4,5-Tetrahydroxy-6-methylphenyl)ethanone, 791
[66625-03-4]	1-[2,4,6-Trihydroxy-3-[(trifluoromethyl)thio]phenyl]ethanone, 730
[66625-04-5]	1-[2,4,6-Trihydroxy-3,5-bis[(trifluoromethyl)thio]phenyl] ethanone, 796
[66842-24-8]	1-(2,6-Dihydroxy-3,4,5-trimethylphenyl)ethanone, 874
[66883-87-2]	1-[3-Chloro-5-(chloromethyl)-2-hydroxyphenyl]ethanone, 733
[66901-79-9]	1-[3-(Acetyloxy)-6-hydroxy-2,4,5-trimethylphenyl]ethanone, 944
[67088-16-8]	1-[2,4-Dihydroxy-3-(phenylmethyl)phenyl]ethanone, 1001
[67127-96-2]	1-[2-Hydroxy-3-(1-propenyl)phenyl]ethanone, 849
[67589-15-5]	1-[2-Hydroxy-5-(trifluoromethyl)phenyl]ethanone, 730
[67895-11-8]	1-(2,6-Dihydroxy-4-pentylphenyl)ethanone, 958
[68034-24-2]	1-[4-Hydroxy-3-(4-hydroxy-3-methyl-2-butenyl)phenyl] ethanone, 942
[68301-59-7]	1-(2-Chloro-4-hydroxyphenyl)ethanone, 684
[68531-86-2]	1-(3,6-Dihydroxy-4-methoxy-2-methylphenyl)ethanone, 832
[69027-37-8]	1-(2-Hydroxy-3,5-dinitrophenyl)ethanone, 677
[69051-59-8]	1-(3-Ethyl-2-hydroxy-4,6-dimethylphenyl)ethanone, 914
[69079-91-0]	1-[2-Hydroxy-6-(4-phenoxybutoxy)phenyl]ethanone, 1046
[69079-92-1]	1-[2-Hydroxy-3-(3-phenoxypropoxy)phenyl]ethanone, 1034
[69079-93-2]	1-[2-Hydroxy-6-(3-phenylpropoxy)phenyl]ethanone, 1033
[69082-35-5]	1-(2,4-Dihydroxy-3,6-dimethylphenyl)ethanone, 820
[69114-99-4]	1-[2,3-Dihydroxy-4-(phenylmethoxy)phenyl]ethanone, 1006
[69240-96-6]	1-(2-Chloro-3-hydroxyphenyl)ethanone, 684
[69240-97-7]	1-(2-Chloro-4,5-dihydroxyphenyl)ethanone, 689

[87165-49-9]	1-[3-(Chloromethyl)-2-hydroxyphenyl]ethanone, 743
[87165-50-2]	1-[2-Hydroxy-3-(methoxymethyl)phenyl]ethanone, 830
[87165-59-1]	1-[5-Chloro-2-hydroxy-3-(methoxymethyl)phenyl]ethanone, 807
[87165-62-6]	1-[3-(Chloromethyl)-2-hydroxy-5-methylphenyl]ethanone, 807
[87165-63-7]	1-[2-Hydroxy-3-(methoxymethyl)-5-methylphenyl]ethanone, 877
[87165-70-6]	1-[3-(Chloromethyl)-2-hydroxy-5-methoxyphenyl]ethanone, 808
[87165-71-7]	1-[2-Hydroxy-5-methoxy-3-(methoxymethyl)phenyl]ethanone, 885
[87239-37-0]	1-(4-Chloro-2-hydroxy-6-methylphenyl)ethanone, 742
[87472-78-4]	1-(2-Amino-4-hydroxy-3-propylphenyl)ethanone, 893
[87953-86-4]	1-(3-Chloro-6-hydroxy-2-methoxyphenyl)ethanone, 747
[87953-91-1]	1-(3-Chloro-2-hydroxy-6-methoxyphenyl)ethanone, 746
[87953-93-3]	1-(3-Chloro-2,6-dihydroxyphenyl)ethanone, 689
[87953-94-4]	1-(3,5-Dichloro-2-hydroxy-6-methoxyphenyl)ethanone, 735
[87953-95-5]	1-(3,5-Dichloro-2,6-dihydroxyphenyl)ethanone, 674
[88086-96-8]	1-[2-Hydroxy-3-[(2,3,4,6-tetra-O-acetyl-β-D-galactopyranosyl) oxy]-phenyl]ethanone, 1072
[88086-97-9]	1-[3-(β-D-Galactopyranosyloxy)-2-hydroxyphenyl]ethanone, 985
[88086-98-0]	1-[2-Hydroxy-5-[(2,3,4,6-tetra-O-acetyl-β-D-galactopyranosyl) oxy]-phenyl]ethanone, 1072
[88086-99-1]	1-[5-(β-D-Galactopyranosyloxy)-2-hydroxyphenyl]ethanone, 986
[88087-00-7]	1-[2-Hydroxy-4,6-bis[(2,3,4,6-tetra-O-acetyl-β-D-galactopyranosyl)oxy]-phenyl]ethanone, 1091
[88087-01-8]	1-[2,4-Bis-(β-D-galactopyranosyloxy)-6-hydroxyphenyl] ethanone, 1064
[88087-03-0]	1-[2-(Benzoyloxy)-5-hydroxyphenyl]ethanone, 997
[88087-04-1]	1-[2-(β-D-Galactopyranosyloxy)-6-hydroxyphenyl]ethanone, 985
[88661-97-6]	1-[3-(3,7-Dimethyl-2,6-octadienyl)-2,4-dihydroxyphenyl] ethanone, 1048
[88771-46-4]	1-(4-Chloro-2-hydroxy-3,6-dimethoxyphenyl)ethanone, 809
[88771-47-5]	1-(3,6-Diethoxy-2-hydroxyphenyl)ethanone, 928
[88771-57-7]	1-(4-Fluoro-2-hydroxy-3,6-dimethoxyphenyl)ethanone, 809
[88771-58-8]	1-(3-Chloro-6-hydroxy-2,5-dimethoxyphenyl)ethanone, 809
[88771-59-9]	1-(2-Hydroxy-3,6-dimethoxy-5-nitrophenyl)ethanone, 812
[88771-63-5]	1-(3,4-Dichloro-6-hydroxy-2,5-dimethoxyphenyl)ethanone, 799
[88772-48-9]	1-(4-Fluoro-2,5-dihydroxyphenyl)ethanone, 693
[89684-58-2]	1-(2,4-Dihydroxy-3-nitrophenyl)ethanone, 701
[89877-53-2]	1-(3-Hydroxy-2-methyl-4-nitrophenyl)ethanone, 752
[89880-47-7]	1-(3-Ethoxy-2-hydroxy-4,6-dimethoxyphenyl)ethanone, 932
[89942-63-2]	1-(3-Hydroxy-4-nitrophenyl)ethanone, 699
[90004-97-0]	1-(3-Bromo-2-hydroxy-5-nitrophenyl)ethanone, 663
[90004-98-1]	1-(3-Bromo-4-hydroxy-5-nitrophenyl)ethanone, 663
[90005-55-3]	1-(3-Amino-2-hydroxyphenyl)ethanone *(Hydrochloride)*, 727
[90033-64-0]	1-(2-Amino-4-hydroxyphenyl)ethanone, 725
[90110-31-9]	1-(4-Chloro-2,5-dihydroxyphenyl)ethanone, 690
[90110-32-0]	1-(5-Chloro-2,4-dihydroxyphenyl)ethanone, 690

[90377-24-5]	1-(3,6-Dihydroxy-2-methoxy-4-methylphenyl)ethanone, 832
[90564-25-3]	1-(2-Hydroxy-5-methoxy-3-nitrophenyl)ethanone, 755
[90743-02-5]	1-(2-Hydroxy-3,6-dimethylphenyl)ethanone, 815
[90743-03-6]	1-(3-Chloro-2-hydroxy-5,6-dimethylphenyl)ethanone, 806
[90971-91-8]	1-(4-Bromo-2-hydroxy-5-methoxyphenyl)ethanone, 740
[91060-92-3]	1-(4-Hydroxy-2,6-dimethylphenyl)ethanone, 818
[91061-75-5]	1-(5-Hydroxy-2,4-dimethoxyphenyl)ethanone, 841
[91124-33-3]	1-[3-(1,1-Dimethylethyl)-2,6-dihydroxyphenyl]ethanone, 923
[91497-16-4]	1-[2-Hydroxy-5-methoxy-4-(2-propenyloxy)phenyl]ethanone, 904
[91664-14-1]	1-[5-(2-Butenyl)-2,3,4-trihydroxyphenyl]ethanone, 902
[91664-16-3]	1-[3-(2-Butenyl)-2,4-dihydroxyphenyl]ethanone, 898
[91664-17-4]	1-[5-(2-Butenyl)-2,4-dihydroxyphenyl]ethanone, 898
[91664-19-6]	1-[5-(2-Butenyl)-2,4-dihydroxy-3-iodophenyl]ethanone, 896
[91664-20-9]	1-[2,4-Dihydroxy-6-methoxy-3-(1-methyl-2-propenyl)phenyl] ethanone, 945
[91664-21-0]	1-[4,6-Dihydroxy-2-methoxy-3-(1-methyl-2-propenyl)phenyl] ethanone, 945
[91664-22-1]	1-[3-(2-Butenyl)-2,4-dihydroxy-6-methoxyphenyl]ethanone, 945
[91664-23-2]	1-[3-(2-Butenyl)-4,6-dihydroxy-2-methoxyphenyl]ethanone, 945
[91664-24-3]	1-[3-(2-Butenyl)-2-hydroxy-4-methoxyphenyl]ethanone, 940
[91969-72-1]	1-[2-Hydroxy-4-(1-methylethyl)phenyl]ethanone, 867
[92119-05-6]	1-(2,4-Dichloro-3-hydroxyphenyl)ethanone, 670
[92518-06-4]	1-[4-(4-Bromobutoxy)-2-hydroxy-3-propylphenyl]ethanone, 1013
[92518-46-2]	1-[4-[(6-Bromohexyl)oxy]-2-hydroxy-3-propylphenyl] ethanone, 1040
[92831-82-8]	1-[2,5-Dihydroxy-4-(2-propenyloxy)phenyl]ethanone, 855
[93339-98-1]	1-(2-Fluoro-6-hydroxyphenyl)ethanone, 691
[93344-48-0]	1-[2-Hydroxy-4,6-bis(1-methylethoxy)phenyl]ethanone, 993
[93344-49-1]	1-[3,6-Dihydroxy-2,4-bis(1-methylethoxy)phenyl]ethanone, 994
[93344-50-4]	1-[6-Hydroxy-3-methoxy-2,4-bis(1-methylethoxy)phenyl] ethanone, 1015
[93344-52-6]	1-[2-Hydroxy-3,6-dimethoxy-4-(1-methylethoxy)phenyl] ethanone, 963
[93351-16-7]	1-(4-Ethyl-2-hydroxy-5-methylphenyl)ethanone, 864
[93434-27-6]	1-[2-Hydroxy-4-methoxy-5-(phenylmethyl)phenyl]ethanone, 1018
[93578-16-6]	1-(2,4-Dihydroxy-5-methylphenyl)ethanone, 769
[93898-99-8]	1-[2,4-Dihydroxy-5-(phenylmethyl)phenyl]ethanone, 1002
[93915-84-5]	1-(2-Hydroxy-3-propylphenyl)ethanone, 868
[94245-10-0]	1-[2-Hydroxy-3-methoxy-4-(1-methylpropoxy)phenyl] ethanone, 960
[94649-69-1]	1-(2,3,6-Trichloro-4-hydroxy-5-methoxyphenyl)ethanone, 730
[94649-70-4]	1-(2-Chloro-4-hydroxy-3,5-dimethoxyphenyl)ethanone, 808
[94649-71-5]	1-(2,6-Dichloro-4-hydroxy-3,5-dimethoxyphenyl)ethanone, 798
[94650-96-1]	1-(Trichloro-4-hydroxyphenyl)ethanone, 661
[95102-24-2]	1-[4-Hydroxy-3-(phenylmethyl)phenyl]ethanone, 1001

[95165-66-5]	1-[2-Hydroxy-3-iodo-4,6-bis(phenylmethoxy)phenyl]ethanone, 1067
[95604-05-0]	1-[4-(Acetyloxy)-2-hydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone, 1009
[95832-44-3]	1-[2,4-Dihydroxy-3,5-bis(phenylmethyl)phenyl]ethanone, 1067
[95832-45-4]	1-[2-Hydroxy-4-methoxy-3-(phenylmethyl)phenyl]ethanone, 1018
[95832-46-5]	1-[2-Hydroxy-4-methoxy-3,5-bis(phenylmethyl)phenyl]ethanone, 1076
[96501-84-7]	1-[6-Hydroxy-2,4-dimethoxy-3-(1-methylethoxy)phenyl]ethanone, 964
[96864-14-1]	1-[2-Heptyl-6-hydroxy-4-(phenylmethoxy)phenyl]ethanone, 1074
[97066-04-1]	1-[2-Hydroxy-4-methyl-6-(phenylamino)phenyl]ethanone, 1009
[97066-06-3]	1-[2-(Dimethylamino)-6-hydroxy-4-methylphenyl]ethanone, 894
[97066-07-4]	1-[2-(Diethylamino)-6-hydroxy-4-methylphenyl]ethanone, 966
[97066-15-4]	1-(2-Amino-6-hydroxy-4-methylphenyl)ethanone, 793
[97066-16-5]	1-[2-Hydroxy-4-methyl-6-[(phenylmethyl)amino]phenyl]ethanone, 1023
[97304-17-1]	1-(2,4-Dihydroxy-5-pentylphenyl)ethanone, 957
[97565-35-0]	1-(3-Hydroxy-2,4,5-trimethoxyphenyl)ethanone, 890
[97582-36-0]	1-[4-(Chloromethyl)-2-hydroxy-3-propylphenyl]ethanone, 908
[97582-37-1]	1-[4-(Chloromethyl)-2-hydroxy-3-methylphenyl]ethanone, 807
[97582-38-2]	1-[4-(Chloromethyl)-3-ethyl-2-hydroxyphenyl]ethanone, 859
[97582-41-7]	1-[4-(Chloromethyl)-2-hydroxy-3-(2-methylpropyl)phenyl]ethanone, 950
[97761-88-1]	1-(4-Hydroxy-2-methoxy-3,6-dimethylphenyl)ethanone, 877
[97871-70-0]	1-(4-Hydroxy-3-nitrosophenyl)ethanone, 697
[98619-07-9]	1-(2-Fluoro-4-hydroxyphenyl)ethanone, 691
[99217-72-8]	1-[2-Hydroxy-4-(methoxymethoxy)-5-(3-methyl-2-butenyl)phenyl]ethanone, 1012
[99283-88-2]	1-[2-Hydroxy-4-(octadecyloxy)phenyl]ethanone, 1086
[99370-47-5]	1-[2-Hydroxy-4-(2-propenyloxy)-3-propylphenyl]ethanone, 982
[99370-48-6]	1-[2,4-Dihydroxy-5-(2-propenyl)-3-propylphenyl]ethanone, 980
[99453-85-7]	1-[4-[(5-Bromopentyl)oxy]-2-hydroxy-3-propylphenyl]ethanone, 1025
[99892-62-3]	1-(3-Hydroxy-2,4,5-trimethylphenyl)ethanone, 870
[99892-63-4]	1-(3-Hydroxy-2,4-dimethylphenyl)ethanone, 817
[100245-06-5]	1-[5-(1,1-Dimethylethyl)-2-hydroxy-3-nitrophenyl]ethanone, 909
[100612-87-1]	1-[3-Hydroxy-4,6-dimethoxy-2-(2-propenyl)phenyl]ethanone, 946
[101002-29-3]	1-[2-Hydroxy-4-(pentyloxy)phenyl]ethanone, 959
[101002-31-7]	1-(4-Butoxy-5-hexyl-2-hydroxyphenyl)ethanone, 1056
[101140-07-2]	1-[3-(Benzoyloxy)-4-hydroxyphenyl]ethanone, 998
[101140-09-4]	1-[3-Hydroxy-4-[(2-methoxyethoxy)methoxy]phenyl]ethanone, 934
[101140-11-8]	1-[4-Hydroxy-3-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy]phenyl]ethanone (*Tetraacetylpungenin)*, 1074

[101161-93-7]	1-[3,5-Bis(diphenylmethyl)-2,4-dihydroxy-6-methoxyphenyl] ethanone, 1090
[101161-94-8]	1-[3-(Diphenylmethyl)-2,4-dihydroxy-6-methoxyphenyl] ethanone, 1069
[101161-95-9]	1-[3-(Diphenylmethyl)-4,6-dihydroxy-2-methoxyphenyl] ethanone, 1069
[101161-96-0]	1-[3-(Diphenylmethyl)-2-hydroxy-4,6-dimethoxyphenyl] ethanone, 1076
[101161-97-1]	1-[3-(Diphenylmethyl)-6-hydroxy-2,4-dimethoxyphenyl] ethanone, 1077
[102056-82-6]	1-[2,4-Dihydroxy-3-[(2-hydroxyphenyl)methyl]-6-methoxyphenyl]ethanone, 1022
[102056-83-7]	1-[2-Hydroxy-3-[(2-hydroxyphenyl)methyl]-6-methoxy-4-(phenylmethoxy)phenyl]ethanone, 1078
[102104-05-2]	1-[2-Hydroxy-6-methoxy-4-(phenylmethoxy)-3-[[2-(phenylmethoxy)phenyl]methyl]phenyl]ethanone, 1088
[102297-89-2]	1-(5-Chloro-2,4-dihydroxy-3-iodophenyl)ethanone, 669
[102624-59-9]	1-(3-Chloro-2,6-dihydroxy-5-propylphenyl)ethanone, 859
[102624-71-5]	1-(3-Fluoro-2,6-dihydroxy-5-propylphenyl)ethanone, 861
[102877-53-2]	1-(2-Hydroxy-4-methoxy-5-nitrophenyl)ethanone, 755
[103323-22-4]	1-(3-Ethyl-2-hydroxyphenyl)ethanone, 813
[103440-57-9]	1-(2-Hydroxy-3-iodo-6-methoxyphenyl)ethanone, 749
[103440-59-1]	1-(4-Hydroxy-3-iodo-5-methoxyphenyl)ethanone, 750
[103633-31-4]	1-[2,4-Dihydroxy-6-methoxy-3-[[2-(phenylmethoxy)phenyl] methyl]phenyl]ethanone, 1077
[103633-32-5]	1-[4,6-Dihydroxy-2-methoxy-3-[[2-(phenylmethoxy)phenyl] methyl]phenyl]ethanone, 1078
[103633-36-9]	1-[2,4-Dihydroxy-3-[[2-(phenylmethoxy)phenyl]methyl]phenyl] ethanone, 1069
[103633-37-0]	1-[2,4-Dihydroxy-5-[[2-(phenylmethoxy)phenyl]methyl]phenyl] ethanone, 1069
[103633-38-1]	1-[2,4-Dihydroxy-3-[(2-hydroxyphenyl)methyl]phenyl] ethanone, 1006
[103633-39-2]	1-[2-Hydroxy-4-methoxy-3-[(2-methoxyphenyl)methyl]phenyl] ethanone, 1033
[103633-40-5]	1-[2-Hydroxy-4-(phenylmethoxy)-3-[[2-(phenylmethoxy)phenyl] methyl]phenyl]ethanone, 1087
[103633-43-8]	1-[2-Hydroxy-4-(phenylmethoxy)-5-[[2-(phenylmethoxy)phenyl] methyl]phenyl]ethanone, 1087
[103633-46-1]	1-[2-Hydroxy-4-[[2-(phenylmethoxy)phenyl]methoxy]-3-[[2-(phenyl-methoxy)phenyl]methyl]phenyl]ethanone, 1090
[103653-14-1]	1-(3-Bromo-4-hydroxy-5-methoxyphenyl)ethanone, 739
[103777-42-0]	1-(4,6-Dihydroxy-2,3-dimethoxyphenyl)ethanone, 846
[103777-43-1]	1-[4-(Dodecyloxy)-6-hydroxy-2,3-dimethoxyphenyl]ethanone, 1075
[103777-44-2]	1-[4-(Dodecyloxy)-2-hydroxy-3,6-dimethoxyphenyl]ethanone, 1075

[103777-45-3]	1-(3-Hydroxy-2,4,6-trimethoxyphenyl)ethanone, 890
[103777-47-5]	1-[3-(Dodecyloxy)-2-hydroxy-4,6-dimethoxyphenyl]ethanone, 1075
[103867-84-1]	1-[2-Hydroxy-3-(2-methyl-1,3-dioxolan-2-yl)phenyl]ethanone, 905
[103867-85-2]	1-[2-Hydroxy-5-methoxy-3-(2-methyl-1,3-dioxolan-2-yl)phenyl]ethanone, 948
[103867-86-3]	1-[2-Hydroxy-6-methoxy-3-(2-methyl-1,3-dioxolan-2-yl)phenyl]ethanone, 948
[103867-87-4]	1-[2-Hydroxy-5-(2-methyl-1,3-dioxolan-2-yl)phenyl]ethanone, 905
[103867-88-5]	1-[4-Hydroxy-3-(2-methyl-1,3-dioxolan-2-yl)phenyl]ethanone, 905
[104074-07-9]	1-[4-(2-Chloroethoxy)-2-hydroxy-3-propylphenyl]ethanone, 950
[104174-27-8]	1-(4-Hydroxy-2-propylphenyl)ethanone, 868
[104175-18-0]	1-[2-Hydroxy-3-(1-methylethyl)phenyl]ethanone, 866
[104481-00-7]	1-[6-Hydroxy-2,4-dimethoxy-3-(methoxymethoxy)phenyl] ethanone, 934
[104654-33-3]	1-[3,6-Bis(acetyloxy)-2-hydroxyphenyl]ethanone, 895
[104676-26-8]	1-[2-Hydroxy-5-[1-(4-hydroxyphenyl)-1-methylethyl]phenyl] ethanone, 1032
[105277-74-5]	1-[2-(3-Chloropropoxy)-6-hydroxyphenyl]ethanone, 860
[105337-34-6]	1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]ethanone, 916
[105337-35-7]	1-[6-Hydroxy-2-methyl-3-(1-methylethyl)phenyl]ethanone, 919
[105340-27-0]	1-[3-Bromo-5-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone, 906
[105342-70-9]	1-(3-Ethoxy-2,6-dihydroxy-4-methoxyphenyl)ethanone, 887
[105342-72-1]	1-(3,6-Diethoxy-2-hydroxy-4-methoxyphenyl)ethanone, 962
[105485-44-7]	1-[2,3,4-Trihydroxy-5-(phenylmethyl)phenyl]ethanone, 1007
[105485-45-8]	1-[2-Hydroxy-4-(phenylmethoxy)-3-(phenylmethyl)phenyl] ethanone, 1068
[105485-46-9]	1-[2-Hydroxy-3,4-bis(phenylmethoxy)-5-(phenylmethyl)phenyl]ethanone, 1086
[105485-47-0]	1-[2-Hydroxy-4-(phenylmethoxy)-3,5-bis(phenylmethyl)phenyl]ethanone, 1086
[105485-48-1]	1-[2-Hydroxy-4-(phenylmethoxy)-5-(phenylmethyl)phenyl] ethanone, 1068
[105485-57-2]	1-[2-Hydroxy-3,4-dimethoxy-5-(phenylmethyl)phenyl] ethanone, 1033
[105533-69-5]	1-(4-Chloro-5-fluoro-2-hydroxyphenyl)ethanone, 668
[106627-20-7]	1-[2-Hydroxy-4-[(6-hydroxyhexyl)oxy]-3-propylphenyl] ethanone, 1042
[106627-33-2]	1-[4-[(7-Bromoheptyl)oxy]-2-hydroxy-3-propylphenyl] ethanone, 1055
[106627-34-3]	1-[4-[(8-Bromooctyl)oxy]-2-hydroxy-3-propylphenyl] ethanone, 1061
[106627-35-4]	1-[4-[(10-Bromodecyl)oxy]-2-hydroxy-3-propylphenyl] ethanone, 1066
[106627-36-5]	1-[4-[(12-Bromododecyl)oxy]-2-hydroxy-3-propylphenyl] ethanone, 1084

[127371-46-4]	1-[5-(1,1-Dimethylethyl)-2-hydroxy-4-methoxyphenyl]ethanone, 958
[127371-47-5]	1-[3-Bromo-5-(1,1-dimethylethyl)-2-hydroxy-4-methoxyphenyl]ethanone, 949
[127701-70-6]	1-(5-Hydroxy-2,3-dimethylphenyl)ethanone, 819
[127870-07-9]	1-[3,6-Dihydroxy-2-(2-methyl-2-propenyl)phenyl]ethanone, 899
[127923-55-1]	1-(3-Bromo-4-hydroxy-5-methylphenyl)ethanone, 738
[127940-12-9]	1-(2,3-Dihydroxy-4-methoxy-6-methylphenyl)ethanone, 831
[128546-82-7]	1-(4-Ethyl-2-hydroxy-6-methoxyphenyl)ethanone, 876
[129375-13-9]	1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methyl-5-(1-methylethyl)phenyl]ethanone, 1027
[129399-54-8]	1-(2,6-Dihydroxy-4-methoxy-3,5-dimethylphenyl)ethanone *(Mallophenone)*, 879
[130435-29-9]	1-(3-Chloro-2,4,6-trihydroxyphenyl)ethanone, 690
[130471-75-9]	1-[4-(Benzoyloxy)-2,6-dihydroxyphenyl]ethanone, 999
[130600-90-7]	1-[2-Hydroxy-4,6-bis[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone, 1053
[131303-37-2]	1-[2-Hydroxy-4,6-bis(methoxymethoxy)-3,5-bis(3-methyl-2-butenyl)phenyl]ethanone, 1074
[131359-44-9]	1-(5-Bromo-4-ethoxy-2-hydroxyphenyl)ethanone, 803
[131845-25-5]	1-(5′-Ethyl-4-hydroxy-2′-methoxy-[1,1′-biphenyl]-3-yl)ethanone, 1032
[133186-55-7]	1-(3,5-Difluoro-4-hydroxyphenyl)ethanone, 675
[133393-99-4]	1-[4-Hydroxy-3-methoxy-5-(1-methylethyl)phenyl]ethanone, 925
[134255-78-0]	1-(2-Hydroxy-3,5-dimethoxy-4-methylphenyl)ethanone, 882
[134255-79-1]	1-(2-Hydroxy-4,5-dimethoxy-3-methylphenyl)ethanone, 882
[134700-74-6]	1-(3-Bromo-2-hydroxy-4-methoxy-5-nitrophenyl)ethanone, 731
[134716-11-3]	1-(3-Bromo-4-ethoxy-2-hydroxy-5-nitrophenyl)ethanone, 798
[135936-88-8]	1-[2-Hydroxy-5-(methylthio)phenyl]ethanone, 767
[136257-82-4]	1-[2,4,6-Trihydroxy-3,5-bis(tetrahydro-2*H*-pyran-2-yl)phenyl]ethanone, 1053
[136257-83-5]	1-[2,4-Dihydroxy-3-(tetrahydro-2*H*-pyran-2-yl)-6-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone, 1053
[136257-85-7]	1-[2,6-Dihydroxy-4-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone, 948
[136257-86-8]	1-[2,4-Dihydroxy-6-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone, 948
[136258-09-8]	1-[2-Hydroxy-4-methoxy-6-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]ethanone, 985
[136258-10-1]	1-[2,6-Dihydroxy-4-methoxy-3-(tetrahydro-2*H*-pyran-2-yl)phenyl]ethanone, 984
[136450-03-8]	1-(3-Amino-2-hydroxyphenyl)ethanone *(Hydrobromide)*, 726
[136608-20-3]	1-[2-Hydroxy-5-(2-phenylethyl)phenyl]ethanone, 1017
[136819-93-7]	1-(3-Hydroxy[1,1′-biphenyl]-2-yl)ethanone, 970

[137170-49-1]	1-[2-Hydroxy-4-(phenylmethoxy)-3-(2-propenyl)phenyl] ethanone, 1044
[138151-67-4]	1-(5-Ethoxy-3-hydroxy-2-methyl [1,1′-biphenyl]-4-yl) ethanone, 1032
[139140-13-9]	1-[4-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-2,6-dihydroxyphenyl]ethanone, 995
[139545-92-9]	1-[2-Hydroxy-6-methoxy-4-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy]phenyl]ethanone, 1080
[140155-06-2]	1-(4-Chloro-2-hydroxy-6-methoxyphenyl)ethanone, 747
[140660-31-7]	1-[5-(1,1-Dimethylethyl)-2,4-dihydroxyphenyl]ethanone, 924
[140660-34-0]	1-(5-Ethyl-2,4-dihydroxy-3-methylphenyl)ethanone, 875
[140660-35-1]	1-[4-[(5-Bromopentyl)oxy]-5-ethyl-2-hydroxy-3-methylphenyl] ethanone, 1025
[140660-37-3]	1-[4-(4-Azidobutoxy)-2-hydroxy-5-(2-propenyl)phenyl] ethanone, 1010
[140675-42-9]	1-(3,5-Difluoro-2-hydroxyphenyl)ethanone, 674
[141215-43-2]	1-[2-Hydroxy-6-methoxy-3-(3-methyl-1,3-butadienyl)phenyl] ethanone *(Z)*, 976
[142045-74-7]	1-[2,3,4-Trihydroxy-5-[(4-hydroxy-3,5-dimethylphenyl)methyl] phenyl]ethanone, 1037
[142608-87-5]	1-[2-Hydroxy-4-methoxy-5-[(3-methyl-2-butenyl)oxy]phenyl] ethanone, 984
[142905-38-2]	1-[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2,6-dihydroxy-3-(3-methyl-2-butenyl)phenyl]ethanone *(E)*, 1082
[142905-39-3]	1-[2-[(3,7-Dimethyl-2,6-octadienyl)oxy]-4,6-dihydroxyphenyl] ethanone (*E*), 1051
[142905-40-6]	1-[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2,6-dihydroxyphenyl] ethanone *(E)*, 1051
[143286-85-5]	1-[4-(Hexyloxy)-2-hydroxyphenyl]ethanone, 993
[143286-86-6]	1-[4-(Decyloxy)-2-hydroxyphenyl]ethanone, 1056
[143286-87-7]	1-[4-(Hexadecyloxy)-2-hydroxyphenyl]ethanone, 1084
[144152-29-4]	1-[2-(Acetyloxy)-5-hydroxyphenyl]ethanone, 800
[144152-30-7]	1-[2-(Acetyloxy)-5-hydroxy-4-methoxyphenyl]ethanone, 857
[144152-31-8]	1-[2,3-Bis(acetyloxy)-4-hydroxyphenyl]ethanone, 894
[144224-86-2]	1-[4-(Acetyloxy)-2-hydroxy-3-methylphenyl]ethanone, 854
[144224-87-3]	1-[3-(Acetyloxy)-2-hydroxyphenyl]ethanone, 800
[144406-93-9]	1-[2-(Benzoyloxy)-4-hydroxyphenyl]ethanone, 997
[144691-35-0]	1-(2-Hydroxy-3-iodo-4-phenoxyphenyl)ethanone, 969
[144691-36-1]	1-[2-Hydroxy-5-iodo-4-phenoxy-3-(2-propenyl)phenyl] ethanone, 1030
[145194-40-7]	1-[4-(β-D-Glucopyranosyloxy)-2-hydroxy-6-methoxy-3-methylphenyl]ethanone, 1025
[145300-04-5]	1-(2-Fluoro-5-hydroxyphenyl)ethanone, 691
[145489-48-1]	1-(5-Bromo-2-hydroxy-3-iodo-4-phenoxyphenyl)ethanone, 966
[145489-93-6]	1-(2-Hydroxy-3-iodo-5-nitro-4-phenoxyphenyl)ethanone, 967

[145666-17-7]	1-(3,5-Dibromo-2-hydroxy-4-methylphenyl)ethanone, 732
[145666-18-8]	1-(3,4-Dibromo-2-hydroxyphenyl)ethanone, 665
[145666-19-9]	1-(3,4,5-Tribromo-2-hydroxyphenyl)ethanone, 660
[145723-28-0]	1-[2-(Acetyloxy)-3-hydroxyphenyl]ethanone, 799
[145746-54-9]	1-[3,6-Dihydroxy-2-(phenylsulfonyl)phenyl]ethanone, 974
[145747-37-1]	1-[2-(Acetyloxy)-3-(diphenylmethyl)-4-hydroxyphenyl] ethanone, 1076
[145747-38-2]	1-[2-(Acetyloxy)-5-(diphenylmethyl)-4-hydroxyphenyl] ethanone, 1076
[145747-39-3]	1-[2-(Acetyloxy)-3,5-bis(diphenylmethyl)-4-hydroxyphenyl] ethanone, 1090
[145747-40-6]	1-[2,6-Bis(acetyloxy)-4-hydroxy-3-[(4-methoxyphenyl)methyl] phenyl]ethanone, 1061
[145797-51-9]	1-(2,3,5,6-Tetrafluoro-4-hydroxyphenyl)ethanone, 659
[146575-61-3]	1-[2-Hydroxy-5-(phenylsulfonyl)phenyl]ethanone, 973
[146575-64-6]	1-[2-Hydroxy-5-(trifluoromethoxy)phenyl]ethanone, 730
[146954-92-9]	1-[5-(3,7-Dimethyl-2,6-octadienyl)-2,4-dihydroxyphenyl] ethanone, 1049
[147816-49-7]	1-[5-[[4-(Acetyloxy)phenyl]sulfonyl]-2-hydroxyphenyl] ethanone, 1016
[147816-50-0]	1-[2-Hydroxy-5-[(4-hydroxyphenyl)sulfonyl]phenyl]ethanone, 974
[147816-51-1]	1-[2-Hydroxy-5-[(4-methylphenyl)sulfonyl]phenyl]ethanone, 1008
[147904-71-0]	1-[4-Hydroxy-2-[(4-hydroxy-3-methoxyphenyl)methyl]-3,5-dimethoxyphenyl]ethanone, 1046
[147904-74-3]	1-[4-Hydroxy-2-[(4-hydroxy-3,5-dimethoxyphenyl)methyl]-3,5-dimethoxyphenyl]ethanone, 1058
[148204-58-4]	1-(3,5-Dihydroxy-4-methoxyphenyl)ethanone, 789
[148254-30-2]	1-(4-Chloro-2-fluoro-5-hydroxyphenyl)ethanone, 668
[149105-11-3]	1-[4-Hydroxy-3-(trifluoromethyl)phenyl]ethanone, 730
[149454-53-5]	1-[2-Hydroxy-4-(2-hydroxybutoxy)phenyl]ethanone, 930
[149454-57-9]	1-[2-Hydroxy-4-(2-hydroxypropoxy)phenyl]ethanone, 884
[149475-52-5]	1-[2-(β-D-Glucopyranosyloxy)-5-hydroxyphenyl]ethanone *(Bungeiside A)*, 987
[149475-54-7]	1-[2-Hydroxy-4-[(6-O-β-D-xylopyranosyl-β-D-glucopyranosyl) oxy]phenyl]ethanone (*Bungeiside D)*, 1060
[149561-88-6]	1-[2-(β-D-Glucopyranosyloxy)-4-hydroxyphenyl]ethanone *(Cynanoneside B; Bungeiside B)*, 986
[149810-09-3]	1-[4-(Acetyloxy)-2-hydroxy-3-iodophenyl]ethanone, 797
[149810-10-6]	1-[4-(Acetyloxy)-2-hydroxy-3-(2-propenyl)phenyl]ethanone, 938
[149876-26-6]	1-[2,3,4-Trihydroxy-6-(3-methyl-2-butenyl)phenyl]ethanone, 947
[150313-75-0]	1-[4-Hydroxy-3-(1-methylpropyl)-5-nitrophenyl]ethanone, 910
[151027-43-9]	1-(4-Hydroxy-2-methoxy-3-propylphenyl)ethanone, 926
[151148-87-9]	1-[2,5-Dihydroxy-3,4-bis(phenylmethoxy)phenyl]ethanone, 1071
[151340-06-6]	1-(2-Chloro-4-hydroxy-3-methoxyphenyl)ethanone, 745
[151719-65-2]	1-[4-(3-Chloropropoxy)-3-hydroxyphenyl]ethanone, 860

[152810-05-4]	1-[2-Hydroxy-5-(1-methylethoxy)phenyl]ethanone, 877
[152810-06-5]	1-(5-Butoxy-2-hydroxyphenyl)ethanone, 921
[153356-01-5]	1-[3-Chloro-5-(1,1-dimethylethyl)-4-hydroxyphenyl]ethanone, 907
[153356-02-6]	1-[3-(1,1-Dimethylethyl)-4-hydroxy-5-iodophenyl]ethanone, 908
[153356-03-7]	1-[3-(1,1-Dimethylethyl)-5-fluoro-4-hydroxyphenyl]ethanone, 908
[153356-04-8]	1-[3-(1,1-Dimethylethyl)-4-hydroxy-5-nitrophenyl]ethanone, 909
[153356-09-3]	1-[3-(1,1-Dimethylethyl)-4-hydroxy-5-methoxyphenyl] ethanone, 958
[153356-10-6]	1-[3-Bromo-5-(1,1-dimethylethyl)-4-hydroxyphenyl]ethanone, 907
[153399-38-3]	1-[4,6-Dihydroxy-3-(3-methyl-2-butenyl)-2-[(3-methyl-2-butenyl) oxy]-phenyl]ethanone, 1050
[153399-41-8]	1-[4,6-Dihydroxy-3-(3-hydroxy-3-methylbutyl)-2-methoxyphenyl] ethanone, 994
[153404-65-0]	1-(3-Amino-5-chloro-2-hydroxyphenyl)ethanone *(Hydrochloride)*, 704
[154389-63-6]	1-[2-Hydroxy-4,6-dimethoxy-3-(methylthio)phenyl]ethanone, 885
[154520-54-4]	1-[3-Hydroxy-2-(3-methyl-2-butenyl)phenyl]ethanone, 939
[154603-69-7]	1-[3-(Azidomethyl)-4-hydroxyphenyl]ethanone, 757
[154638-85-4]	1-(2,3-Dichloro-4-hydroxy-5-methoxyphenyl)ethanone, 735
[154638-86-5]	1-(3-Chloro-4,5-dihydroxyphenyl)ethanone, 690
[154638-87-6]	1-(2,3,6-Trichloro-4,5-dihydroxyphenyl)ethanone, 661
[155818-27-2]	1-(3-Bromo-2,4,6-trihydroxyphenyl)ethanone, 683
[155982-91-5]	1-[2-(1,1-Dimethylethyl)-4-hydroxyphenyl]ethanone, 911
[156483-08-8]	1-(4-Hydroxy-2,3,6-trimethylphenyl)ethanone, 871
[156499-51-3]	1-[2,4,6-Trihydroxy-3-(3,7,11-trimethyl-2,6,10-dodecatrienyl) phenyl]ethanone, 1083
[156499-52-4]	1-[2,6-Dihydroxy-4-[(3,7,11-trimethyl-2,6,10-dodecatrienyl)oxy] phenyl]ethanone1081
[158013-69-5]	1-[6-[(1,1-Dimethylethyl)amino]-3-hydroxy-2-methylphenyl] ethanone, 966
[158148-88-0]	1-[2-Hydroxy-5-methoxy-3,4-bis(phenylmethoxy)phenyl] ethanone, 1079
[158499-95-7]	1-[4,6-Dihydroxy-3-(3-methyl-2-butenyl)-2-[[(4-methylphenyl) sulfonyl]-oxy]phenyl]ethanone, 1061
[158499-96-8]	1-[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-6-hydroxy-3-(3-methyl-2-butenyl)- 2-[[(4-methylphenyl)sulfonyl]oxy]phenyl] ethanone (*E)*, 1088
[158499-97-9]	1-[6-Hydroxy-3-(3-methyl-2-butenyl)-4-[(3-methyl-2-butenyl) oxy]-2-[[(4-methylphenyl)-sulfonyl]oxy]phenyl]ethanone, 1085
[158499-98-0]	1-[2,6-Dihydroxy-3-(3-methyl-2-butenyl)-4-[(3-methyl-2-butenyl) oxy]-phenyl]ethanone, 1050
[159848-01-8]	1-[3-Hydroxy-4,6-dimethoxy-2-methyl-5-(1-methylethyl)phenyl]-ethanone, 993
[159977-36-3]	1-(5-Cyclohexyl-2,4-dihydroxyphenyl)ethanone, 979
[160246-79-7]	1-(2,6-Dihydroxy-5-nitro[1,1′-biphenyl]-3-yl)ethanone, 970

[161358-64-1]	1-[2-Hydroxy-6-(methoxymethyl)phenyl]ethanone, 830
[162853-19-2]	1-[2-Hydroxy-3-methyl-6-(1-methylethyl)phenyl]ethanone, 915
[162853-20-5]	1-(4-Hydroxy-5-methoxy-2-methylphenyl)ethanone, 829
[163429-79-6]	1-(2-Hydroxy-3,4,6-trimethylphenyl)ethanone, 869
[165186-29-8]	1-[2,4-Dihydroxy-6-(2-hydroxyethyl)-3-methoxyphenyl] ethanone, 886
[167211-56-5]	1-[2,4-Dihydroxy-3-(2-hydroxypropyl)phenyl]ethanone, 879
[167211-59-8]	1-[2-Hydroxy-3-(2-hydroxypropyl)-4-mercaptophenyl] ethanone, 878
[167211-71-4]	1-[2-Hydroxy-3-(3-hydroxypropyl)-4-mercaptophenyl] ethanone, 879
[169130-25-0]	1-[2-Hydroxy-4,5-dimethoxy-3,6-bis(1-methylethoxy)phenyl] ethanone, 1029
[169130-27-2]	1-[2-Hydroxy-4,5-dimethoxy-6-(1-methylethoxy)-3-(phenylmethoxy)phenyl]ethanone, 1062
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[705963-54-8]	1-[5-Chloro-2,4-bis(phenylmethoxy)phenyl]ethanone, 1108
[741264-99-3]	1-(3-Hydroxy-4-methoxy-5-methylphenyl)ethanone, 1157
[747413-68-9]	1-[5-Bromo-2,4-bis(phenylmethoxy)phenyl]ethanone, 1103
[747414-06-8]	1-[5-(1,1-Dimethylethyl)-2,4-bis(phenylmethoxy)phenyl] ethanone, 1173
[747414-17-1]	1-[2,4-Dihydroxy-5-(1-methylethyl)phenyl]ethanone, 1165
[747414-18-2]	1-[5-(1-Methylethyl)-2,4-bis(phenylmethoxy)phenyl] ethanone, 1165
[784177-14-6]	1-(5-Butyl-2-methoxyphenyl)ethanone, 1172
[841298-81-5]	1-(4′-Chloro-4-methoxy[1,1′-biphenyl]-2-yl)ethanone, 1188
[865451-01-0]	1-(5-Chloro-4-fluoro-2-hydroxyphenyl)ethanone, 1097
[868702-20-9]	1-(4-Hydroxy-2-methoxy-5-methylphenyl)ethanone, 1157
[870480-17-4]	8-Acetyl-3-iodo-5,6,7-trimethoxy-4*H*-1-benzopyran-4-one, 1168
[870480-50-5]	1-(3-Acetyl-2-hydroxy-4,5,6-trimethoxyphenyl)-3-(dimethylamino)(2*E*)-2-propen-1-one, 1193
[870480-53-8]	8-Acetyl-5,7-dihydroxy-3-iodo-6-methoxy-4*H*-1-benzopyran-4-one, 1168
[870652-37-2]	1-(5-Bromo-2,3,4-trihydroxyphenyl)ethanone, 1103
[870652-73-6]	1-[2-(2-Chloroethoxy)-6-hydroxyphenyl]ethanone, 1150
[872057-13-1]	1-[2-Hydroxy-4,6-dimethoxy-3-(1-methyl-4-piperidinyl)phenyl] ethanone, 1194
[872415-44-6]	1-(2-Fluoro-4-methoxy-3-methylphenyl)ethanone, 1133
[872415-45-7]	1-(2-Fluoro-4-hydroxy-3-methylphenyl)ethanone, 1133
[873211-41-7]	1-[2-Hydroxy-5-(3-methyl-2-butenyl)phenyl]ethanone, 1178
[873211-43-9]	1-[3-(1,1-Dimethyl-2-propenyl)-2-hydroxyphenyl]ethanone, 1178
[873222-85-6]	1-(2,5-Dihydroxy-3,4-dipropylphenyl)ethanone, 1187
[873222-91-4]	1-(3,4-Diethyl-2,5-dihydroxyphenyl)ethanone, 1172
[873222-92-5]	1-(2,5-Dihydroxy-4-methyl-3-propylphenyl)ethanone, 1172
[873222-93-6]	1-(2,5-Dihydroxy-3-methyl-4-propylphenyl)ethanone, 1172
[873222-94-7]	1-[3-[2-(Acetyloxy)ethyl]-4-ethyl-2,5-dihydroxyphenyl] ethanone, 1186
[873222-95-8]	1-[4-[2-(Acetyloxy)ethyl]-3-ethyl-2,5-dihydroxyphenyl] ethanone, 1187
[880479-07-2]	1-(6-Hydroxy-4-methoxy-7-propoxy-5-benzofuranyl) ethanone, 1184
[880479-08-3]	1-[6-Hydroxy-4-methoxy-7-(pentyloxy)-5-benzofuranyl] ethanone, 1192
[880479-09-4]	1-[6-Hydroxy-4-methoxy-7-(phenylmethoxy)-5-benzofuranyl] ethanone, 1194
[880479-11-8]	1-[6-Hydroxy-7-methoxy-4-(pentyloxy)-5-benzofuranyl] ethanone, 1193

[2161-85-5]	1,1′-(4,6-Dihydroxy-1,3-phenylene)bis-ethanone, 1566
[2161-86-6]	1,1′-(2,4,6-Trihydroxy-1,3-phenylene)bis-ethanone, 1568
[2161-87-7]	1,1′,1″-(2,4,6-Trihydroxy-1,3,5-benzenetriyl)tris-ethanone, 1578
[2163-12-4]	1,1′-(2,4-Dihydroxy-1,3-phenylene)bis-ethanone, 1564
[2491-31-8]	1-(2-Hydroxyphenyl)-2-phenylethanone, 1404
[2491-32-9]	1-(4-Hydroxyphenyl)-2-phenylethanone, 1406
[2491-34-1]	1-(2-Hydroxy-4-methylphenyl)-2-phenylethanone, 1415
[2491-36-3]	2-Bromo-1-(2-hydroxyphenyl)ethanone, 1206
[2491-37-4]	2-Bromo-1-(3-hydroxyphenyl)ethanone, 1207
[2491-38-5]	2-Bromo-1-(4-hydroxyphenyl)ethanone, 1207
[2491-39-6]	2-Bromo-1-(2,4-dihydroxyphenyl)ethanone, 1208
[2491-40-9]	2-Bromo-1-(2,6-dihydroxyphenyl)ethanone, 1209
[2495-77-4]	1-(2,4-Diethoxy-6-hydroxyphenyl)-2-methoxyethanone, 1338
[2551-38-4]	1,1′-(4,4′-Dihydroxy-2,2′-dimethoxy[1,1′-biphenyl]-3,3′-diyl) bis-ethanone, 1593
[2551-44-2]	1,1′-(2,2′,4,4′-Tetrahydroxy[1,1′-biphenyl]-3,3′-diyl) bis-ethanone, 1591
[2589-80-2]	1-(5-Benzoyl-2-hydroxyphenyl)ethanone, 1628
[2631-85-8]	2-(3,4-Dimethoxyphenyl)-1-(6-hydroxy-4-methoxy-1,3-benzodioxol-5-yl)ethanone, 1510
[2652-17-7]	1-(6-Hydroxy-4-methoxy-1,3-benzodioxol-5-yl)-2-phenylethanone, 1424
[2746-88-5]	2-(1,3-Benzodioxol-5-yl)-1-(2-hydroxy-4,5-dimethoxyphenyl) ethanone, 1492
[2746-89-6]	2-(1,3-Benzodioxol-5-yl)-1-(2,5-dihydroxy-4-methoxyphenyl) ethanone, 1476
[2746-90-9]	2-(1,3-Benzodioxol-5-yl)-1-(6-hydroxy-1,3-benzodioxol-5-yl) ethanone, 1474
[2828-14-0]	2-(1,3-Benzodioxol-5-yl)-1-(2,4,5-trihydroxyphenyl)ethanone, 1462
[2970-79-8]	2-(Dimethylamino)-1-(4-hydroxyphenyl)ethanone *(Hydrochloride)*, 1302
[2999-24-8]	1,1′-(2,4,5-Trihydroxy-1,3-phenylene)bis-ethanone, 1568
[2999-42-0]	1,1′-(2,4,6-Trihydroxy-5-methyl-1,3-phenylene)bis-ethanone, 1576
[3098-38-2]	1,1′-(2,4-Dihydroxy-6-methoxy-1,3-phenylene)bis-ethanone, 1575
[3136-47-8]	2-Phenyl-1-(2,4,6-trihydroxy-3-methylphenyl)ethanone, 1423
[3207-38-3]	1-(6-Hydroxy-4-methoxy-1,3-benzodioxol-5-yl)-2-(4-hydroxyphenyl)ethanone, 1476
[3207-42-9]	1-(6-Hydroxy-4-methoxy-1,3-benzodioxol-5-yl)-2-(4-methoxyphenyl)ethanone, 1494
[3511-69-1]	1,1′-[(1-Methylethylidene)bis(6-hydroxy-3,1-phenylene)]bis-ethanone, 1611
[3606-32-4]	1-(2-Hydroxy-3,4-dimethoxyphenyl)-2-(4-methoxyphenyl) ethanone, 1502
[3669-41-8]	1-(2,4-Dihydroxyphenyl)-2-phenylethanone, 1407
[3669-46-3]	1-(4-Hydroxyphenyl)-2-(4-methoxyphenyl)ethanone, 1466

[3669-47-4]	1,2-Bis(4-hydroxyphenyl)ethanone, 1456
[3669-50-9]	1-(4-Hydroxy-2-methylphenyl)-2-phenylethanone, 1417
[4108-04-7]	1-(3-Bromo-5-fluoro-2-hydroxyphenyl)-2-phenylethanone, 1399
[4108-05-8]	1-(3-Chloro-5-fluoro-2-hydroxyphenyl)-2-phenylethanone, 1400
[4324-56-5]	2-Ethoxy-1-(2-ethoxy-6-hydroxy-3,4-dimethoxyphenyl)ethanone, 1349
[4324-58-7]	1-(2,4-Diethoxy-3,6-dihydroxyphenyl)-2-methoxyethanone, 1339
[4324-59-8]	1-(2,4-Diethoxy-6-hydroxy-3-methoxyphenyl)-2-methoxyethanone, 1341
[4873-38-5]	1-(4-Hydroxyphenyl)-2,2-diphenylethanone, 1539
[4940-44-7]	1-(2-Hydroxy-4-methoxyphenyl)-2-methoxyethanone, 1324
[4970-24-5]	1-(2-Hydroxyphenyl)-2,2-diphenylethanone, 1539
[5029-61-8]	2-Bromo-1-(4-hydroxy-3-nitrophenyl)ethanone, 1204
[5037-70-7]	2-Bromo-1-(2-hydroxy-5-nitrophenyl)ethanone, 1204
[5071-47-6]	1-(2-Hydroxy-3,4,5,6-tetramethoxyphenyl)-2-methoxyethanone, 1340
[5090-29-9]	2-Amino-1-(3,4-dihydroxyphenyl)ethanone, 1296
[5128-46-1]	1-(4,6-Diethoxy-2-hydroxy-3-methoxyphenyl)-2-methoxyethanone, 1342
[5128-49-4]	1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-(4-methoxyphenyl)ethanone, 1502
[5128-54-1]	1-(2,4-Dihydroxy-5-methoxyphenyl)-2-(4-methoxyphenyl)ethanone, 1485
[5128-56-3]	2-(1,3-Benzodioxol-5-yl)-1-(2-hydroxy-4-methoxyphenyl)ethanone, 1475
[5653-25-8]	2-(1,3-Benzodioxol-5-yl)-1-(2,4-dihydroxyphenyl)ethanone, 1461
[5706-85-4]	2-Hydroxy-1-(4-hydroxyphenyl)ethanone, 1370
[6305-04-0]	2-Chloro-1-(4-hydroxyphenyl)ethanone, 1233
[6344-28-1]	2-Chloro-1-(4-hydroxy-3-methoxyphenyl)ethanone, 1244
[6502-87-0]	2-(2,4-Dimethoxyphenyl)-1-(2-hydroxy-3,4-dimethoxyphenyl)ethanone, 1516
[7294-92-0]	1-(2-Hydroxy-3-methylphenyl)-2-phenylethanone, 1414
[7354-81-6]	1-(4-Hydroxy-3-methylphenyl)-2-phenylethanone, 1417
[7507-92-8]	2-Chloro-1-(2-hydroxy-3,4-dimethoxyphenyl)ethanone, 1248
[7622-42-6]	1,2-Bis(2-hydroxyphenyl)ethanone, 1455
[7741-43-7]	1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-2-methoxyethanone, 1335
[7741-48-2]	2-(Benzoyloxy)-1-(2,4-dihydroxy-3,6-dimethoxyphenyl)ethanone, 1391
[7741-49-3]	2-(Benzoyloxy)-1-(2-hydroxy-3,4,6-trimethoxyphenyl)ethanone, 1392
[10048-37-0]	2-(Benzoyloxy)-1-[2-hydroxy-3,6-dimethoxy-4-(phenylmethoxy)phenyl]ethanone, 1393
[10508-84-6]	1,1′-[Methylenebis(2,4-dihydroxy-3,1-phenylene)]bis-ethanone, 1600
[13043-37-3]	1-(3-Benzoyl-4-hydroxyphenyl)ethanone, 1627

[23937-90-8]	1,1′-[1,5-Pentanediylbis[oxy(6-hydroxy-4-methoxy-2,1-phenylene)]]bis-ethanone, 1619
[24126-91-8]	1-(2,4-Dihydroxy-5-methoxyphenyl)-2-(3,4-dimethoxyphenyl) ethanone, 1505
[24126-94-1]	1-(2,5-Dihydroxy-4-methoxyphenyl)-2-(3,4-dimethoxyphenyl) ethanone, 1506
[24126-98-5]	1-(2,4-Dihydroxyphenyl)-2-(3,4-dimethoxyphenyl)ethanone, 1487
[24195-21-9]	1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-(2,4,5-trimethoxyphenyl)-ethanone, 1524
[24195-22-0]	2-(3,4-Dimethoxyphenyl)-1-(2-hydroxy-4,5-dimethoxyphenyl) ethanone, 1518
[24195-23-1]	1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-(7-methoxy-1,3-benzodioxol-5-yl)ethanone, 1511
[24195-24-2]	1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-(6-methoxy-1,3-benzodioxol-5-yl)ethanone, 1510
[24195-30-0]	1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-(2-methoxyphenyl) ethanone, 1502
[24195-31-1]	1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-phenylethanone, 1429
[24258-63-7]	1-(2-Hydroxy-5-methylphenyl)-2-phenylethanone, 1416
[24483-75-8]	2-Chloro-1-(5-chloro-2-hydroxyphenyl)ethanone, 1232
[24852-33-3]	1-(2-Hydroxy-3,4-dimethoxyphenyl)-2-phenylethanone, 1429
[24852-34-4]	2-(4-Chlorophenyl)-1-(2-hydroxy-3,4-dimethoxyphenyl) ethanone, 1478
[24852-43-5]	1-(5-Bromo-2-hydroxy-3,4-dimethoxyphenyl)-2-(4-methoxyphenyl)ethanone, 1495
[24863-50-1]	2-(3-Chlorophenyl)-1-(2-hydroxy-3,4-dimethoxyphenyl) ethanone, 1478
[25015-91-2]	2-Bromo-1-(2,5-dihydroxyphenyl)ethanone, 1208
[25015-92-3]	2-Chloro-1-(2,4-dihydroxyphenyl)ethanone, 1234
[25666-51-7]	2,2,2-Trifluoro-1-(2-hydroxyphenyl)ethanone, 1268
[27171-77-3]	1-[5-[2-(3-Acetyl-4-hydroxyphenyl)ethyl]-2-methoxyphenyl] ethanone, 1610
[27171-78-4]	1-[5-[1-(3-Acetyl-4-hydroxyphenyl)-1-methylethyl]-2-methoxyphenyl]ethanone, 1612
[27171-79-5]	1-[5-[2-(3-Acetyl-4-hydroxyphenyl)ethyl]-2-ethoxyphenyl] ethanone, 1612
[27171-80-8]	1-[5-[2-(3-Acetyl-4-hydroxyphenyl)ethyl]-2-propoxyphenyl] ethanone, 1613
[27693-62-5]	1-(3,4-Dihydroxyphenyl)-2-[(1-methylethyl)amino]ethanone *(Sulfate)*, 1308
[28441-16-9]	2-(Acetyloxy)-1-[3,5-bis(1,1-dimethylethyl)-4-hydroxyphenyl] ethanone, 1386
[28466-42-4]	1,1′-[Methylenebis(2-hydroxy-4-methoxy-3,1-phenylene)]bis-ethanone, 1602
[28467-08-5]	1,1′-[Oxybis(6-hydroxy-3,1-phenylene)]bis-ethanone, 1614
[28467-22-3]	1,1′-[Methylenebis(6-hydroxy-3,1-phenylene)]bis-ethanone, 1600

[35287-64-0]	1,1′-(2,4′-Dihydroxy-2′,4-dimethoxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone, 1597
[35292-40-1]	1,1′-(2,2′-Diethoxy-4,4′-dihydroxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone, 1595
[35486-77-2]	1-[2-Hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-2-phenylethanone, 1437
[35817-38-0]	1-[2-Hydroxy-6-methoxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-2-(4-methoxyphenyl)ethanone, 1528
[35817-95-9]	1-[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-(4-methoxyphenyl)ethanone, 1527
[35817-96-0]	1-[4,6-Dihydroxy-2-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-(4-methoxyphenyl)ethanone, 1528
[35928-53-1]	2,2-Dibromo-1-(4-hydroxy-3-nitrophenyl)ethanone, 1225
[35928-54-2]	2,2-Dibromo-1-(3-bromo-4-hydroxy-5-nitrophenyl)ethanone, 1224
[35930-51-9]	1-(2,5-Dihydroxy-4-methoxyphenyl)-2-methoxyethanone, 1327
[36039-26-6]	1-(5-Acetyl-2-hydroxyphenyl)-1-propanone, 1631
[36414-93-4]	1-[2-(2,4-Dihydroxybenzoyl)phenyl]ethanone, 1639
[36754-72-0]	1-(2,4-Dihydroxyphenyl)-2-(3-hydroxy-4-methoxyphenyl) ethanone, 1470
[37086-37-6]	1,1′-[1,5-Pentanediylbis[oxy(2-hydroxy-4,1-phenylene)]]bis-ethanone, 1617
[37126-05-9]	1,1′-[2-Hydroxy-4-(phenylmethoxy)-6-(2-propenyloxy)-1,3-phenylene]bis-ethanone, 1589
[37126-08-2]	1,1′-(4,6-Dihydroxy-2-methoxy-5-propyl-1,3-phenylene)bis-ethanone, 1587
[37126-09-3]	1,1′-[2,4-Dihydroxy-6-methoxy-5-(2-propenyl)-1,3-phenylene] bis-ethanone, 1585
[37126-10-6]	1,1′-(2,4-Dihydroxy-6-methoxy-5-propyl-1,3-phenylene)bis-ethanone, 1586
[37879-22-4]	1,1′-(2,4′-Dihydroxy-2′,4,6,6′-tetramethoxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone, 1597
[37879-23-5]	1,1′-(2-Hydroxy-2′,4,4′,6,6′-pentamethoxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone, 1598
[37904-71-5]	2-Chloro-1-(4-hydroxy-2-methylphenyl)ethanone, 1240
[38319-83-4]	1-(2-Hydroxy-4,6-dimethylphenyl)-2-phenylethanone, 1426
[38412-59-8]	2-(4-Methoxyphenyl)-1-(2,3,4-trihydroxyphenyl)ethanone, 1473
[38412-65-6]	1-(2,3-Dihydroxy-4-methoxyphenyl)-2-(4-methoxyphenyl) ethanone, 1484
[38782-67-1]	1,1′-[Methylenebis(4-hydroxy-5-methyl-3,1-phenylene)]bis-ethanone, 1601
[38782-68-2]	1,1′-[Methylenebis(4-hydroxy-3,1-phenylene)]bis-ethanone, 1599
[38782-69-3]	1-[3,5-Bis[(5-acetyl-2-hydroxy-3-methylphenyl)methyl]-4-hydroxyphenyl]ethanone, 1606
[38987-02-9]	1-[2,4-Dihydroxy-3-(2-propenyl)phenyl]-2-phenylethanone, 1431
[38987-03-0]	1-[2,4-Dihydroxy-5-(2-propenyl)phenyl]-2-phenylethanone, 1431

[39022-25-8]	1-[4-Hydroxy-2-methoxy-5-(2-propenyl)phenyl]-2-phenylethanone, 1435
[39066-18-7]	2-Chloro-1-(3-chloro-4-hydroxyphenyl)ethanone, 1231
[39068-36-5]	1-(2-Hydroxyphenyl)-2-(methylsulfonyl)ethanone, 1544
[39125-99-0]	1,1′-(3,6-Dihydroxy-1,2-phenylene)bis-ethanone, 1565
[39126-03-9]	1,1′-(2,3-Dihydroxy-1,4-phenylene)bis-ethanone, 1563
[39548-95-3]	1-[2-Hydroxy-4,6-bis(phenylmethoxy)-3-(phenylmethyl)phenyl]-2-phenylethanone, 1448
[39548-96-4]	1-[2-Hydroxy-4,6-bis(phenylmethoxy)phenyl]-2-phenylethanone, 1448
[39548-97-5]	1-[2,4-Dihydroxy-6-(phenylmethoxy)-3-(phenylmethyl)phenyl]-2-phenylethanone, 1447
[39548-98-6]	2-(1,3-Benzodioxol-5-yl)-1-(2,4,6-trihydroxyphenyl)ethanone, 1462
[39548-99-7]	2-(1,3-Benzodioxol-5-yl)-1-[2-hydroxy-4,6-bis(phenylmethoxy)-3-(phenylmethyl)phenyl]ethanone, 1538
[39549-00-3]	2-(1,3-Benzodioxol-5-yl)-1-[2-hydroxy-4,6-bis(phenylmethoxy) phenyl]ethanone, 1536
[39549-01-4]	2-(1,3-Benzodioxol-5-yl)-1-[2,4-dihydroxy-6-(phenylmethoxy)-3-(phenyl-methyl)phenyl]ethanone, 1536
[39581-98-1]	1-(2,4-Dihydroxy-3-methylphenyl)-2-phenylethanone, 1418
[39604-64-3]	1-(2-Hydroxy-4-methoxyphenyl)-2-(4-methoxyphenyl) ethanone, 1483
[39604-65-4]	1-(2-Hydroxy-4-methoxy-3-methylphenyl)-2-(4-methoxyphenyl) ethanone, 1498
[39604-66-5]	1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-phenylethanone, 1430
[39604-67-6]	1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-2-phenylethanone, 1433
[39604-68-7]	1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-(4-methoxyphenyl) ethanone, 1504
[39604-69-8]	2-(2,4-Dimethoxyphenyl)-1-(2-hydroxy-4,6-dimethoxyphenyl) ethanone, 1517
[39604-80-3]	1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-2-phenylethanone, 1441
[39604-84-7]	2-(2,4-Dimethoxyphenyl)-1-[2-hydroxy-4,6-bis(phenylmethoxy) phenyl]ethanone, 1538
[39878-43-8]	2-Chloro-1-(3,5-dihydroxyphenyl)ethanone, 1235
[39954-75-1]	1-(4-Benzoyl-3-hydroxyphenyl)ethanone, 1627
[39954-81-9]	1-(4-Benzoyl-5-hydroxy-2-methylphenyl)ethanone, 1628
[40131-99-5]	2-Bromo-1-(3,4-dihydroxyphenyl)ethanone, 1209
[40231-09-2]	2-(Acetyloxy)-1-(2-hydroxyphenyl)ethanone, 1383
[40449-66-9]	1,1′-(5-Butyl-4,6-dihydroxy-1,3-phenylene)bis-ethanone, 1586
[40456-49-3]	1-(2,4-Dihydroxyphenyl)-2-(4-hydroxy-3-methoxyphenyl) ethanone, 1471
[40584-06-3]	1-(2-Hydroxy-6-methoxyphenyl)-2-phenylethanone, 1421
[40943-24-6]	2-Chloro-1-(4-hydroxy-3-methylphenyl)ethanone, 1240
[40943-25-7]	2-Chloro-1-(4-hydroxy-3,5-dimethylphenyl)ethanone, 1247

[52945-23-0]	1-(4-Hydroxy-3,5-dimethoxyphenyl)-2-(methylsulfonyl) ethanone, 1547
[52977-39-6]	1-[2-[(3-Acetyl-4-hydroxyphenyl)methyl]-5-hydroxyphenyl] ethanone, 1599
[53074-73-0]	2-Chloro-1-(2-hydroxyphenyl)ethanone, 1232
[53084-05-2]	2-(3,4-Dimethoxyphenyl)-1-(2-hydroxy-4-methoxyphenyl) ethanone, 1501
[53084-06-3]	2-(3,4-Dimethoxyphenyl)-1-(2,4,6-trihydroxyphenyl) ethanone, 1490
[54735-43-2]	2,2-Dibromo-1-(2-hydroxyphenyl)ethanone, 1226
[54794-31-9]	1-(3-Hydroxyphenyl)-2-methoxyethanone, 1322
[54903-53-6]	1-(4-Amino-3-hydroxyphenyl)-2-phenylethanone, 1411
[54917-81-6]	1-(3-Benzoyl-2,4-dihydroxy-5-nitrophenyl)ethanone, 1627
[54917-83-8]	1-[3-Benzoyl-4-(β-D-galactopyranosyloxy)-2-hydroxyphenyl] ethanone, 1630
[54918-25-1]	1-[3-Benzoyl-4-(β-D-glucopyranosyloxy)-2-hydroxyphenyl] ethanone, 1630
[54921-24-3]	1-(3-Bromo-2-hydroxy-4,5-dimethoxyphenyl)-2-phenylethanone, 1425
[54943-18-9]	1-[3-Hydroxy-4-(methylamino)phenyl]-2-phenylethanone, 1423
[54963-60-9]	1-[5-Acetyl-2-hydroxy-3-(3-hydroxy-3-methyl-1-butenyl)phenyl]-3-methyl-1-butanone *(E)*, 1634
[54981-34-9]	1-(5-Bromo-2-hydroxyphenyl)-2-phenylethanone, 1400
[54981-35-0]	1-(4-Bromo-2-hydroxyphenyl)-2-phenylethanone, 1400
[55108-28-6]	1,1′-(2-Hydroxy-5-methyl-1,3-phenylene)bis-ethanone, 1570
[55168-30-4]	1,1′-[5-(Acetyloxy)-2,4-dihydroxy-1,3-phenylene] bis-ethanone, 1578
[55313-03-6]	2-Hydroxy-1-(2,4,6-trihydroxyphenyl)ethanone, 1374
[55317-02-7]	2-Methoxy-1-(2,4,6-trihydroxyphenyl)ethanone, 1323
[55338-29-9]	1-(2,4-Dihydroxy-6-methylphenyl)-2-phenylethanone, 1419
[55607-18-6]	1-(2,4-Dihydroxy-6-methoxyphenyl)-2-phenylethanone, 1422
[55607-20-0]	1-[2,4-Dihydroxy-6-methoxy-3,5-bis(3-methyl-2-butenyl)phenyl]-2-phenylethanone, 1444
[55607-21-1]	1-[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-phenylethanone, 1438
[55607-22-2]	1-[4,6-Dihydroxy-2-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-phenylethanone, 1439
[55607-23-3]	1-[2-Hydroxy-4,6-dimethoxy-3-(3-methyl-2-butenyl)phenyl]-2-phenylethanone, 1442
[55607-25-5]	1-[6-Hydroxy-2,4-dimethoxy-3-(3-methyl-2-butenyl)phenyl]-2-phenylethanone, 1442
[55607-36-8]	2-(1,3-Benzodioxol-5-yl)-1-(2,4-dihydroxy-6-methoxyphenyl) ethanone, 1476
[55607-37-9]	2-(1,3-Benzodioxol-5-yl)-1-[2,4-dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]ethanone, 1527

[56923-35-4]	1,1′-[Sulfonylbis[4-(benzoyloxy)-6-hydroxy-3,1-phenylene]]bis-ethanone, 1625
[56923-41-2]	1,1′-[Thiobis(4,6-dihydroxy-3,1-phenylene)]bis-ethanone, 1622
[56923-42-3]	1,1′-[Thiobis(6-hydroxy-4-methoxy-3,1-phenylene)] bis-ethanone, 1622
[56923-49-0]	1,1′-[Thiobis[6-hydroxy-4-(phenylmethoxy)-3,1-phenylene]] bis-ethanone, 1623
[56923-50-3]	1,1′-[Thiobis[4-(benzoyloxy)-6-hydroxy-3,1-phenylene]]bis-ethanone, 1622
[56982-36-6]	1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-(4-nitrophenyl) ethanone, 1479
[56986-82-4]	1-(2-Hydroxyphenyl)-2-(methylthio)ethanone, 1543
[57097-17-3]	1-[2-Hydroxy-3-methyl-4-(2-propenyloxy)phenyl]-2-phenylethanone, 1436
[57165-58-9]	2-(3,4-Dihydroxyphenyl)-1-(2,3,4-trihydroxyphenyl)ethanone, 1460
[57272-98-7]	1-(2-Hydroxy-4-methoxyphenyl)-2-(4-nitrophenyl)ethanone, 1464
[57280-73-6]	1-[2-Hydroxy-4-(2-propenyloxy)phenyl]-2-methoxyethanone, 1333
[57280-75-8]	1-(2,4-Dihydroxyphenyl)-2-methoxyethanone, 1322
[58316-48-6]	1,1′,1″,1‴-[Methylenebis(2,4,6-trihydroxy-5,1,3-benzenetriyl)] tetrakis-ethanone, 1604
[58451-99-3]	1-(3-Hydroxy-4-methoxyphenyl)-2-phenylethanone, 1421
[58483-49-1]	1-(2,3-Dihydroxyphenyl)-2-hydroxyethanone, 1372
[58483-53-7]	1-(3,5-Dichloro-2-hydroxyphenyl)-2-hydroxyethanone, 1369
[58805-51-9]	1,1′-[5-(Ethoxymethyl)-4,6-dihydroxy-1,3-phenylene]bis-ethanone, 1585
[58805-52-0]	1,1′-(4,6-Dihydroxy-5-propyl-1,3-phenylene)bis-ethanone, 1584
[58805-53-1]	1,1′-[2-Hydroxy-4-methoxy-6-(2-propenyloxy)-1,3-phenylene] bis-ethanone, 1586
[58805-54-2]	1,1′-[4,6-Dihydroxy-5-(hydroxymethyl)-1,3-phenylene]bis-ethanone, 1574
[59108-68-8]	2-(4-Methylphenyl)-1-(2,4,6-trihydroxyphenyl)ethanone, 1469
[59108-69-9]	2-Cyclopentyl-1-(2,4-dihydroxyphenyl)ethanone, 1541
[59208-55-8]	1-(2,4-Dihydroxyphenyl)-2-(4-methylphenyl)ethanone, 1465
[59507-91-4]	1-(2-Hydroxy-5-nitrophenyl)-2-nitroethanone, 1396
[59719-58-3]	2-Chloro-1-(4-hydroxy-3,5-dimethoxyphenyl)ethanone, 1249
[60011-06-5]	1,1′-[Methylenebis(5-chloro-2-hydroxy-3,1-phenylene)] bis-ethanone, 1598
[60278-33-3]	1-(2-Hydroxy-4-methoxyphenyl)-2-(4-hydroxyphenyl) ethanone, 1469
[60312-44-9]	1,1′-(2,2′-Dihydroxy[1,1′-biphenyl]-3,3′-diyl)bis-ethanone, 1590
[60312-53-0]	1,1′-[Methylenebis(2-hydroxy-3,1-phenylene)]bis-ethanone, 1599
[60795-08-6]	1-(2-Hydroxy-3-methylphenyl)-2-nitroethanone, 1397
[60795-09-7]	1-(3-Chloro-2-hydroxyphenyl)-2-nitroethanone, 1395
[60795-10-0]	1-(2-Hydroxy-4-methylphenyl)-2-nitroethanone, 1397
[60795-11-1]	1-(4-Chloro-2-hydroxyphenyl)-2-nitroethanone, 1395

[63124-29-8]	2-[2,4-Dihydroxy-3-(3-methyl-2-butenyl)phenyl]-2-oxoethyl 2-methylpropanoate, 1388
[63186-92-5]	2-(4-Bromophenyl)-1-(4-hydroxyphenyl)ethanone, 1450
[63192-59-6]	1,2-Bis(3-hydroxyphenyl)ethanone, 1455
[63220-58-6]	1-[2-Hydroxy-5-(1-methylethyl)phenyl]-2-(methylsulfinyl) ethanone, 1548
[63411-83-6]	1,1′-(5-Ethyl-2,4-dihydroxy-1,3-phenylene)bis-ethanone, 1580
[63704-55-2]	2-Chloro-1-(2,3-dihydroxyphenyl)ethanone, 1234
[64184-96-9]	1-(2,4-Diethoxy-6-hydroxyphenyl)-2-ethoxyethanone, 1349
[64225-20-3]	2-(4-Aminophenyl)-1-(2,4,6-trihydroxyphenyl)ethanone, 1460
[64349-38-8]	1-(4-Hydroxy-3-methoxyphenyl)-2-methoxyethanone, 1325
[64349-40-2]	1-(3,4-Dihydroxyphenyl)-2-methoxyethanone, 1323
[64554-42-3]	1-(6-hydroxy-2,3,4-trimethoxyphenyl)-2-(3,4,5-trimethoxyphenyl)ethanone, 1526
[64640-60-4]	1-(2,4-Dihydroxy-3,6-dimethoxyphenyl)-2-(3-hydroxy-4-methoxyphenyl)ethanone, 1507
[64857-81-4]	1,1′-(2-Hydroxy-4-methoxy-1,3-phenylene)bis-ethanone, 1573
[64857-82-5]	1,1′,1″-(2,4-Dihydroxy-1,3,5-benzenetriyl)tris-ethanone, 1577
[65039-95-4]	2-Methoxy-1-(6-methoxy-2,4,5-trihydroxyphenyl)ethanone, 1328
[65134-36-3]	1-[2,4-Dihydroxy-3-(1-methylnonyl)phenyl]-2,2,2-trifluoroethanone, 1286
[65134-37-4]	1-[2,4-Dihydroxy-3-(1-methylundecyl)phenyl]-2,2,2-trifluoroethanone, 1287
[65220-47-5]	1-(2-Hydroxy-3-methoxyphenyl)-2-(methylsulfinyl)ethanone, 1545
[65233-60-5]	1-(2,4-Dihydroxy-3-methylphenyl)-2,2,2-trifluoroethanone, 1270
[65233-62-7]	1-[2-(Acetyloxy)-4-hydroxy-3-methylphenyl]-2,2,2-trifluoroethanone, 1275
[65233-62-7]	1-[4-(Acetyloxy)-2-hydroxy-3-methylphenyl]-2,2,2-trifluoroethanone, 1275
[65233-63-8]	1-(5-Chloro-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone, 1267
[65233-64-9]	1-[2-(Acetyloxy)-5-chloro-4-hydroxyphenyl]-2,2,2-trifluoroethanone, 1272
[65233-64-9]	1-[4-(Acetyloxy)-5-chloro-2-hydroxyphenyl]-2,2,2-trifluoroethanone, 1272
[65233-65-0]	1-[5-Chloro-2-hydroxy-4-(octadecanoyloxy)phenyl]-2,2,2-trifluoroethanone, 1288
[65233-65-0]	1-[5-Chloro-4-hydroxy-2-(octadecanoyloxy)phenyl]-2,2,2-trifluoroethanone, 1288
[65233-66-1]	1-[2-Hydroxy-3-methyl-4-(10-undecenoyloxy)phenyl]-2,2,2-trifluoroethanone, 1287
[65233-67-2]	1-[5-Chloro-2-hydroxy-4-(10-undecenoyloxy)phenyl]-2,2,2-trifluoroethanone, 1286
[65233-68-3]	2,2,2-Trifluoro-1-(5-hexyl-2,4-dihydroxyphenyl)ethanone, 1282
[65233-69-4]	1-[2-(Acetyloxy)-5-hexyl-4-hydroxyphenyl]-2,2,2-trifluoroethanone, 1285

[65240-08-6]	1-(5-Chloro-2,4-dihydroxy-3-methylphenyl)-2,2,2-trifluoroethanone, 1269
[65240-09-7]	1-(3,5-Dichloro-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone, 1266
[65240-10-0]	1-(3-Chloro-5-hexyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone, 1281
[65240-11-1]	1-(3,5-Dibromo-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone, 1266
[65240-12-2]	1-(3-Bromo-5-hexyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone, 1280
[65240-13-3]	1-[2,4-Dihydroxy-5-[(4-methylphenyl)sulfonyl]phenyl]-2,2,2-trifluoroethanone, 1284
[65240-14-4]	1-[2,4-Dihydroxy-3-[(4-methylphenyl)sulfonyl]phenyl]-2,2,2-trifluoroethanone, 1283
[65240-15-5]	1-(2,4-Dihydroxy-3-methyl-5-nitrophenyl)-2,2,2-trifluoroethanone, 1270
[65240-16-6]	1-(2,4-Dihydroxy-5-nitrophenyl)-2,2,2-trifluoroethanone, 1268
[65240-17-7]	1-(2,4-Dihydroxy-3,5-dinitrophenyl)-2,2,2-trifluoroethanone, 1266
[65240-18-8]	1-[2,4-Dihydroxy-3-(1-methylpentyl)phenyl]-2,2,2-trifluoroethanone, 1281
[65240-19-9]	1-(3-Cyclododecyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone, 1287
[65240-20-2]	1-(3-Cyclopentyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone, 1278
[65240-21-3]	1-(3-Cycloheptyl-2,4-dihydroxyphenyl)-2,2,2-trifluoroethanone, 1284
[65240-22-4]	1-[2,4-Dihydroxy-3-(1-methylheptyl)phenyl]-2,2,2-trifluoroethanone, 1285
[65240-25-7]	1-[4-(Decyloxy)-2-hydroxyphenyl]-2,2,2-trifluoroethanone, 1286
[65240-27-9]	1-(4-Butoxy-2-hydroxyphenyl)-2,2,2-trifluoroethanone, 1277
[65240-29-1]	1,1′-[Methylenebis(5-ethyl-2,4-dihydroxy-3,1-phenylene)]bis[2,2,2-trifluoroethanone, 1607
[65240-30-4]	1,1′-[Methylenebis(2,4-dihydroxy-5-methyl-3,1-phenylene)]bis[2,2,2-trifluoroethanone, 1607
[65240-31-5]	1,1′-[Methylenebis(2,4-dihydroxy-5-propyl-3,1-phenylene)]bis[2,2,2-trifluoroethanone, 1608
[65240-32-6]	1,1′-[Methylenebis(2,4-dihydroxy-5-pentyl-3,1-phenylene)]bis[2,2,2-trifluoroethanone, 1608
[65240-33-7]	1,1′-[Methylenebis(5-hexyl-2,4-dihydroxy-3,1-phenylene)]bis[2,2,2-trifluoroethanone, 1609
[65240-34-8]	1,1′-[Methylenebis(5-dodecyl-2,4-dihydroxy-3,1-phenylene)]bis[2,2,2-trifluoroethanone, 1609
[65240-35-9]	1,1′-[Methylenebis[2,4-dihydroxy-5-(1-methylethyl)-3,1-phenylene]]bis-[2,2,2-trifluoroethanone, 1607
[65240-36-0]	1,1′-[Methylenebis[2,4-dihydroxy-5-(phenylmethyl)-3,1-phenylene]]bis-[2,2,2-trifluoroethanone, 1609
[65240-37-1]	1,1′-[Methylenebis(5-cyclohexyl-2,4-dihydroxy-3,1-phenylene)]bis[2,2,2-trifluoroethanone, 1609

[73331-41-6]	2-Chloro-1-(2-hydroxy-6-methylphenyl)ethanone, 1240
[73898-24-5]	2-Bromo-1-(3-ethyl-4-hydroxyphenyl)ethanone, 1217
[73898-25-6]	2-Bromo-1-[4-hydroxy-3-(1-methylethyl)phenyl]ethanone, 1221
[73898-26-7]	2-Bromo-1-(3-cyclohexyl-4-hydroxyphenyl)ethanone, 1223
[73898-29-0]	2-Bromo-1-(4-hydroxy-3-iodophenyl)ethanone, 1204
[73898-30-3]	2-Bromo-1-(3-hydroxy-4-methylphenyl)ethanone, 1213
[73898-31-4]	2-Bromo-1-(4-ethyl-3-hydroxyphenyl)ethanone, 1218
[73898-32-5]	2-Bromo-1-[3-hydroxy-4-(1-methylethyl)phenyl]ethanone, 1221
[73898-33-6]	2-Bromo-1-(4-cyclohexyl-3-hydroxyphenyl)ethanone, 1223
[73898-34-7]	2-Bromo-1-(4-chloro-3-hydroxyphenyl)ethanone, 1203
[73898-35-8]	2-Bromo-1-(4-bromo-3-hydroxyphenyl)ethanone, 1205
[73898-36-9]	2-Bromo-1-(3-hydroxy-4-iodophenyl)ethanone, 1204
[73937-48-1]	1-[2-Hydroxy-4-(2-propenyloxy)phenyl]-2-(4-methoxyphenyl)-ethanone, 1510
[74047-41-9]	1-[2-Hydroxy-3-iodo-6-methoxy-4-(2-propenyloxy)phenyl]-2-methoxyethanone, 1338
[74047-42-0]	1-(2,4-Dihydroxy-3-iodo-6-methoxyphenyl)-2-methoxyethanone, 1323
[74384-31-9]	1-(4-Hydroxy-3-methylphenyl)-2-(4-methoxyphenyl) ethanone, 1480
[74384-32-0]	1-(4-Hydroxy-3,5-dimethylphenyl)-2-(4-methoxyphenyl) ethanone, 1496
[74384-33-1]	1-(2-Hydroxy-3-methylphenyl)-2-(4-methoxyphenyl) ethanone, 1480
[74384-34-2]	1-(2-Hydroxy-5-methylphenyl)-2-(4-methoxyphenyl) ethanone, 1480
[74384-35-3]	1-(2-Hydroxy-3,5-dimethylphenyl)-2-(4-methoxyphenyl) ethanone, 1496
[74384-36-4]	1-(2-Hydroxy-3-methylphenyl)-2-(2-methoxyphenyl) ethanone, 1479
[74384-37-5]	1-(4-Hydroxy-3,5-dimethylphenyl)-2-(2-methoxyphenyl) ethanone, 1496
[74384-38-6]	1-(2-Hydroxy-5-methylphenyl)-2-(2-methoxyphenyl) ethanone, 1480
[74384-39-7]	1-(2-Hydroxy-3,5-dimethylphenyl)-2-(2-methoxyphenyl) ethanone, 1496
[74730-79-3]	2-(Ethylamino)-1-(4-hydroxyphenyl)ethanone *(Hydrochloride)*, 1303
[74815-26-2]	2-Bromo-1-(3,5-dibromo-2,6-dihydroxyphenyl)ethanone, 1202
[74815-30-8]	2-Bromo-1-(5-cyclohexyl-2-hydroxyphenyl)ethanone, 1223
[75060-43-4]	2-Chloro-1-[5-(1,1-dimethylethyl)-2-hydroxyphenyl]ethanone, 1252
[75060-51-4]	1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-2-phenylethanone, 1436
[75060-56-9]	1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-2,2,2-trifluoroethanone, 1277

[85288-47-7]	1-(4-Hydroxy-2-methoxyphenyl)-2-phenylethanone, 1421
[85288-48-8]	1-(2-Hydroxy-4-methoxyphenyl)-2-(2,4,5-trimethoxyphenyl) ethanone, 1519
[85299-04-3]	2,2-Dichloro-1-(3-hydroxyphenyl)ethanone, 1256
[85450-67-5]	1,1′-(2-Hydroxy-4,6-dimethyl-5-nitro-1,3-phenylene) bis-ethanone, 1579
[85450-70-0]	1-(4-Benzoyl-3-hydroxy-5-methyl-6-nitro[1,1′-biphenyl]-2-yl) ethanone, 1630
[85450-76-6]	1,1′-(5-Amino-2-hydroxy-4,6-dimethyl-1,3-phenylene)bis-ethanone, 1582
[85450-81-3]	1-(6-Amino-4-benzoyl-3-hydroxy-5-methyl[1,1′-biphenyl]-2-yl)-ethanone, 1630
[85465-61-8]	2-Fluoro-1-(3-hydroxy-4-methoxyphenyl)ethanone, 1265
[85602-17-1]	2-Phenyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl] ethanone, 1437
[85602-22-8]	2-(4-Chlorophenyl)-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl) phenyl]ethanone, 1521
[85950-49-8]	2-Methoxy-1-(2,4,6-trihydroxy-3,5-dimethoxyphenyl) ethanone, 1332
[86828-07-1]	1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl) methyl]-2,4,6-trihydroxy-5-(3-methyl-2-butenyl)phenyl] ethanone, 1605
[86828-08-2]	1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl) methyl]-2,4,6-trihydroxy-5-(2-hydroxy-3-methyl-3-butenyl) phenyl]ethanone, 1606
[87538-40-7]	1-(2-Hydroxy-4-methoxy-3-methylphenyl)-2-phenylethanone, 1427
[87538-41-8]	1-(2-Hydroxy-4-methoxy-5-methylphenyl)-2-phenylethanone, 1427
[87669-75-8]	2-(3,5-Dichloro-2-hydroxyphenyl)-2-oxoethyl dimethylcarbamodithioate, 1547
[88092-53-9]	2-Chloro-1-(2-hydroxy-4,6-dimethoxyphenyl)-2-methoxyethanone, 1329
[88503-19-9]	2,2-Dibromo-1-(3-bromo-2-hydroxy-4,6-dimethoxyphenyl) ethanone, 1227
[88693-95-2]	2-[Bis(phenylmethyl)amino]-1-(4-hydroxyphenyl)ethanone, 1318
[89019-83-0]	1-(2,4-Dihydroxyphenyl)-2-(3-methoxyphenyl)ethanone, 1467
[89019-84-1]	1-(2,4-Dihydroxyphenyl)-2-(3-hydroxyphenyl)ethanone, 1457
[89019-87-4]	2-(4-Ethoxyphenyl)-1-(2-hydroxy-4-methoxyphenyl) ethanone, 1497
[89019-88-5]	1-(4-Hydroxy-2-methoxyphenyl)-2-(4-hydroxyphenyl) ethanone, 1469
[90005-54-2]	2-Amino-1-(3-hydroxyphenyl)ethanone, 1293
[90426-22-5]	2-Hydroxy-1-(4-hydroxy-3,5-dimethoxyphenyl)ethanone, 1377
[90464-79-2]	1,1′-(4-Hydroxy-1,2-phenylene)bis-ethanone, 1562
[90536-46-2]	2-Hydroxy-1-(3-hydroxy-4-methoxyphenyl)ethanone, 1376
[90971-90-7]	2-Bromo-1-(3-hydroxy-4-methoxyphenyl)ethanone, 1215
[91061-33-5]	2-Ethoxy-1-(4-hydroxyphenyl)ethanone, 1346

[107410-01-5]	1-(2,3-Dihydroxyphenyl)-2-phenylethanone, 1407
[107410-02-6]	1-(3,4-Dihydroxyphenyl)-2-phenylethanone, 1409
[107410-55-9]	1-(4-Chloro-2-hydroxyphenyl)-2-phenylethanone, 1402
[107584-64-5]	1-(4-Hydroxyphenyl)-2-(3-methylphenoxy)ethanone, 1359
[107584-67-8]	1-(4-Hydroxy-3-methoxyphenyl)-2-(3-methylphenoxy) ethanone, 1363
[107584-68-9]	1-(4-Hydroxy-3-methoxyphenyl)-2-(3-methoxyphenoxy) ethanone, 1364
[107584-69-0]	1-(4-Hydroxy-3-methoxyphenyl)-2-[3-(trifuoromethyl)phenoxy] ethanone, 1362
[107584-70-3]	1-(4-Hydroxy-3-methoxyphenyl)-2-(3-nitrophenoxy) ethanone, 1359
[107584-71-4]	1-(4-Hydroxy-3-methoxyphenyl)-2-(3-hydroxyphenoxy) ethanone, 1362
[107584-78-1]	2-Bromo-1-(4-hydroxy-2,5-dimethylphenyl) ethanone, 1218
[107584-79-2]	1-(4-Hydroxy-2,5-dimethylphenyl)-2-(3-methylphenoxy) ethanone, 1365
[107584-80-5]	1-(4-Hydroxy-2,5-dimethylphenyl)-2-[3-(trifluoromethyl) phenoxy]ethanone, 1365
[107602-85-7]	2-(3-Chlorophenoxy)-1-(4-hydroxy-3-methoxyphenyl) ethanone, 1358
[107700-04-9]	2-Bromo-1-[3-bromo-5-(chloromethyl)-4-hydroxyphenyl] ethanone, 1211
[107700-05-0]	2,2-Dibromo-1-[3-bromo-5-(chloromethyl)-4-hydroxyphenyl] ethanone, 1227
[108378-94-5]	1-(2-Hydroxyphenyl)-2-[(4-methylphenyl)thio]ethanone, 1553
[108378-95-6]	1-(2-Hydroxyphenyl)-2-[(4-methylphenyl)sulfinyl] ethanone (±), 1553
[108434-12-4]	2-([1,1′-Biphenyl]-2-yloxy)-1-(4-hydroxy-3-methoxyphenyl) ethanone, 1367
[108448-95-9]	1-(2-Hydroxyphenyl)-2-[(4-methylphenyl)sulfinyl]ethanone (R), 1553
[108708-11-8]	1-(5-Amino-2-hydroxyphenyl)-2-chloroethanone, 1237
[108708-12-9]	1-(3-Amino-4-hydroxyphenyl)-2-chloroethanone, 1237
[108708-13-0]	1-(4-Amino-2-hydroxyphenyl)-2-chloroethanone, 1237
[108909-49-5]	1,1′-(3-Hydroxy-4′,5-dimethyl[1,1′-biphenyl]-2,6-diyl) bis-ethanone, 1596
[108909-50-8]	1,1′-[4′-(Dimethylamino)-3-hydroxy-5-methyl[1,1′-biphenyl]-2,6-diyl]bis-ethanone, 1589
[109089-92-1]	1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-(3-methoxyphenyl) ethanone, 1503
[109089-93-2]	1-(4-Hydroxy-2,6-dimethoxyphenyl)-2-(4-methoxyphenyl) ethanone, 1504
[109091-12-5]	1-(2,4-Dihydroxy-6-methoxyphenyl)-2-(2,4-dimethoxyphenyl) ethanone, 1505

[122918-54-1]	1-(4-Hydroxy-3-methylphenyl)-2,2-diphenylethanone, 1540
[123716-19-8]	2,2,2-Trifluoro-1-(2-hydroxy-4-methoxyphenyl)ethanone, 1271
[124208-68-0]	1-[3-(3-Acetyl-4-hydroxybenzoyl)-4-hydroxyphenyl] ethanone, 1640
[124208-69-1]	1-[2-Hydroxy-5-(2-hydroxybenzoyl)phenyl]ethanone, 1639
[125617-37-0]	1-[4-(3-Bromopropoxy)-2-hydroxyphenyl]-2,2,2-trifluoroethanone, 1275
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[18430-72-3]	1-(3-Bromo-4-hydroxyphenyl)-1-propanone, 1747
[18430-73-4]	1-(2-Bromo-4-hydroxyphenyl)-1-propanone, 1746
[18430-74-5]	1-(3,5-Dichloro-2-hydroxyphenyl)-1-propanone, 1744
[18592-97-7]	1,1′-(6,6′-Dihydroxy-5,5′-dimethoxy[1,1′-biphenyl]-3,3′-diyl)bis-1-propanone, 2121
[18592-99-9]	1-(4′-Hydroxy-3′,5-dimethoxy-6-propionyloxy[1,1′-biphenyl]-3-yl)-1-propanone, 1952
[18593-00-5]	1-(4′,6-Dihydroxy-3′,5-dimethoxy[1,1′-biphenyl]-3-yl)-1-propanone, 1942
[18593-02-7]	1-[2′-Hydroxy-6-(2-hydroxy-3-methoxy-5-propionylphenoxy)-5,3′-dimethoxy-5′-propionyl[1,1′-biphenyl]-3-yl]-1-propanone, 2128
[18607-90-4]	1-(6-Hydroxy-1,3-benzodioxol-5-yl)-1-propanone, 1988
[18892-91-6]	1-[4,6-Dihydroxy-5-(3-methylbutyl)-2-(1-methylethyl)-7-benzofuranyl]-2-methyl-1-propanone, 2074
[18906-66-6]	1-(2-Hydroxyphenyl)-1-propanone copper complex, 1762
[18944-22-4]	1-[2,3-Dihydro-4,6-dihydroxy-2-(1-hydroxy-1-methylethyl)-5-(3-methylbutyl)-7-benzofuranyl]-2-methyl-1-propanone, 2079
[19647-29-1]	2-[[3-Bromo-5,7-dihydroxy-2,2-dimethyl-8-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-6-yl]methyl]-3,5-dihydroxy-4,4-dimethyl-6-(2-methyl-1-oxopropyl)-2,5-cyclohexadien-1-one, 2152
[19809-78-0]	3,5-Dihydroxy-4,4-dimethyl-2-(2-methyl-1-oxopropyl)-6-[[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)-5-(2-methyl-1-oxopropyl) phenyl]methyl]-2,5-cyclohexadien-1-one, 2154
[19809-79-1]	2-[[5,7-Dihydroxy-2,2-dimethyl-8-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-6-yl]methyl]-3,5-dihydroxy-4,4-dimethyl-6-(2-methyl-1-oxopropyl)-2,5-cyclohexadien-1-one, 2152
[19809-80-4]	2-[[3,4-Dihydro-5,7-Dihydroxy-2,2-dimethyl-8-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-6-yl]methyl]-3,5-dihydroxy-4,4-dimethyl-6-(2-methyl-1-oxopropyl)-2,5-cyclohexadien-1-one, 2153
[19809-81-5]	2-[[3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-6-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-8-yl]methyl]-3,5-dihydroxy-4,4-dimethyl-6-(2-methyl-1-oxopropyl)-2,5-cyclohexadien-1-one, 2153
[19937-86-1]	1-(7-Methoxy-1,3-benzodioxol-5-yl)-1-propanone, 1992
[20683-32-3]	1-[3-(1,1-Dimethylethyl)-4-hydroxyphenyl]-1-propanone, 1891
[20683-42-5]	1-[3-Bromo-5-(1,1-dimethylethyl)-4-hydroxyphenyl]-1-propanone, 1888
[20800-11-7]	1-[2-Hydroxy-4-(2-morpholinoethoxy)phenyl]-1-propanone 1(Hydrochloride), 1926
[20800-22-0]	1-[2-Hydroxy-4-(2-piperidinoethoxy)phenyl]-1-propanone (Hydrochloride), 1939
[20935-63-1]	1-(2,4-Dimethoxy-3,5-dimethylphenyl)-1-propanone, 1899
[21009-91-6]	1-(2-Hydroxy-4,6-dimethylphenyl)-2-methyl-1-propanone, 2032

[25069-71-0]	1-(2-Chloro-4,5-dihydroxyphenyl)-1-propanone, 1752
[25441-52-5]	1-[3-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-propanone, 1891
[25677-09-2]	2-Methyl-1-(2,4,6-trimethoxy-3,5-dimethylphenyl)-1-propanone, 2049
[25801-58-5]	1-(2-Methoxy-1-naphthalenyl)-1-propanone, 1972
[25944-41-6]	7-Hydroxy-8-methyl-6-(1-oxopropyl)-4-phenyl-2*H*-1-benzopyran-2-one, 2008
[25944-43-8]	7-Hydroxy-4,8-dimethyl-6-(1-oxopropyl)-2*H*-1-benzopyran-2-one, 2001
[27178-00-3]	1-[4-(Acetyloxy)-2,6-dihydroxyphenyl]-1-propanone, 1826
[28885-62-3]	1-[3,6-Dihydroxy-2-(2-propenyl)phenyl]-1-propanone, 1860
[28885-63-4]	1-(3,6-Dihydroxy-2-propylphenyl)-1-propanone, 1873
[29026-02-6]	1-[2-Hydroxy-5-(2-propenyloxy)phenyl]-1-propanone, 1861
[29048-54-2]	1-(2,4-Dihydroxyphenyl)-2-methyl-1-propanone, 2017
[29048-55-3]	1-(2-Hydroxy-4-methoxyphenyl)-2-methyl-1-propanone, 2027
[29373-18-0]	1-[3,4-Dihydro-5,7-dihydroxy-8-(3-methylbutyl)-2,2-dimethyl-2*H*-1-benzopyran-6-yl]-2-methyl-1-propanone, 2078
[29525-18-6]	1-[3,4-Dihydro-5,7-dihydroxy-6-(3-methylbutyl)-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone, 2078
[29525-19-7]	7-Hydroxy-6-isobutyryl-2,2-dimethyl-8,8-bis(3-methyl-2-butenyl)-5-(8*H*)-chromanone, 2078
[29525-23-3]	3,3a-Dihydro-4-hydroxy-2-(1-hydroxy-1-methylethyl)-5-isobutyryl-3a,7-bis-(3-methyl-2-butenyl)-6-(2*H*)-benzofuranone, 2077
[29525-24-4]	1-[2,3-Dihydro-4,6-dihydroxy-2-(1-hydroxy-1-methylethyl)-7-(3-methylbutyl)-5-benzofuranyl]-2-methyl-1-propanone, 2079
[29525-25-5]	1-[2,3,8,9-Tetrahydro-4-hydroxy-2-(1-hydroxy-1-methylethyl)-7,7-dimethyl-7*H*-furo[2,3-*f*]-1-benzopyran-5-yl]-2-methyl-1-propanone, 2077
[29578-81-2]	1-(5-Methoxy-2-methylphenyl)-1-propanone, 1841
[29578-84-5]	1-(3-Methoxy-5-methylphenyl)-1-propanone, 1840
[29725-94-8]	1-(5-Chloro-2-hydroxyphenyl)-1-propanone (Oxime), 1752
[30314-46-6]	1-(3,4-Dimethoxyphenyl)-2,2-dimethyl-1-propanone, 2093
[30574-34-6]	1-(2-Methoxy-5-methylphenyl)-2-methyl-1-propanone, 2033
[31826-78-5]	1-(2-Hydroxyphenyl)-1-propanone (Tetra-O-acetyl-β-D-glucopyranoside), 1763
[31826-79-6]	1-(2-Hydroxyphenyl)-1-propanone (β-D-Glucopyranoside), 1762
[31826-80-9]	1-(4-Hydroxyphenyl)-1-propanone (β-D-Glucopyranoside), 1766
[31827-85-7]	1-(3,5-Diiodo-4-methoxyphenyl)-1-propanone, 1783
[31867-16-0]	1-(4-Hydroxyphenyl)-1-propanone (Tetra-O-acetyl-β-D-glucopyranoside), 1766
[31918-60-2]	1-[5,7-Dihydroxy-2,2-dimethyl-6-(3-methyl-2-butenyl)-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone, 2074
[32154-22-4]	1-[5-Bromo-3-(1,1-dimethylethyl)-2-methoxyphenyl]-1-propanone (2,4-Dinitrophenylhydrazone), 1909

[35888-90-5]	2-Hydroxy-3-(1-oxopropyl)benzaldehyde, 2131
[35888-91-6]	1-[2-Hydroxy-3-(2-propenyl)phenyl]-1-propanone, 1859
[35888-92-7]	2-Hydroxy-3-(1-oxopropyl)benzoic acid, 1784
[35888-93-8]	2-Hydroxy-3-(1-oxopropyl)benzoic acid ethyl ester, 1862
[35932-36-6]	2-Methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-propanone, 2046
[35932-37-7]	1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl)-2-methyl-1-propanone, 2069
[35932-38-8]	1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-2-methyl-1-propanone, 2068
[36039-26-6]	1-(5-Acetyl-2-hydroxyphenyl)-1-propanone, 2132
[36198-81-9]	1-(4-Methoxy-1-naphthalenyl)-2-methyl-1-propanone, 2064
[36198-82-0]	1-(4-Methoxy-1-naphthalenyl)-2,2-dimethyl-1-propanone, 2101
[36677-69-7]	1,1′-(4,4′-Dihydroxy[1,1′-biphenyl]-3,3′-diyl)bis-1-propanone, 2117
[36871-54-2]	1-(2-Methoxy-4-methylphenyl)-1-propanone, 1839
[36871-55-3]	1-(4-Chloro-2-methoxyphenyl)-1-propanone, 1791
[36871-56-4]	1-(4-Methoxy-2,5-dimethylphenyl)-1-propanone, 1871
[36871-58-6]	1-(2-Methoxy-4,5-dimethylphenyl)-1-propanone, 1870
[36871-61-1]	1-(3-Bromo-6-methoxy-2,4-dimethylphenyl)-1-propanone, 1864
[37456-35-2]	1-[2-Hydroxy-4-(3-methylbutoxy)phenyl]-1-propanone, 1914
[37769-66-7]	1-(5-sec-Dodecyl-2-hydroxyphenyl)-1-propanone (Oxime), 1956
[37847-36-2]	1-[4-Hydroxy-2-methyl-5-(1-methylethyl) phenyl]-1-propanone, 1896
[37848-03-6]	1-(2-Hydroxyphenyl)-1-propanone zinc complex, 1762
[37848-04-7]	1-(2-Hydroxyphenyl)-1-propanone cadmium complex, 1762
[37848-05-8]	1-(2-Hydroxy-4-methylphenyl)-1-propanone zinc complex, 1802
[37848-06-9]	1-(2-Hydroxy-4-methylphenyl)-1-propanone cadmium complex, 1802
[37888-90-7]	1-(4-Methoxy-2-nitrophenyl)-1-propanone, 1797
[37951-49-8]	1-(3-Methoxyphenyl)-1-propanone, 1807
[38319-72-1]	1-(2-Methoxy-4,6-dimethylphenyl)-2-methyl-1-propanone, 2040
[38463-02-4]	1-(2-Ethyl-6-methoxyphenyl)-1-propanone, 1867
[38775-76-7]	2,6-dimethoxy-4-(1-hydroxyneopentyl)phenol, 2094
[39026-68-1]	1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)-1-propanone, 1851
[39027-10-6]	1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)-1-propanone (Glycoside), 1851
[39036-20-9]	5′-[3,4-Dihydro-5-methoxy-3,6-dimethyl-4-oxo-7-[(2,3,4-tri-*O*-methyl-β-D-glucopyranosyl)oxy]-2*H*-1-benzopyran-2-yl]-2′,4-dimethoxy-, intramol. 3,6‴-ester [1,1′-biphenyl]-3-carboxylic acid, 1851
[39084-13-4]	3-[5-[3,4-Dihydro-5-methoxy-3,6-dimethyl-4-oxo-7-[(2,3,4-tri-*O*-methyl-β-D-glucopyranosyl)oxy]-2*H*-1-benzopyran-2-yl]-2-methoxyphenyl]-2-methoxy-1,6′-lactone benzoic acid, 1851

[49582-15-2]	1-[3,5-Bis[[2,4-dihydroxy-6-methoxy-5-methyl-3-(1-oxopropyl) phenyl]-methyl]-2,4,6-trihydroxyphenyl]-1-butanone, 2145
[49710-99-8]	1-(2-Hydroxy-5-methoxyphenyl)-1-propanone, 1812
[49812-94-4]	1-(4,7-Dihydroxy-2,3-dimethyl-6-benzofuranyl)-1-propanone, 2000
[50342-15-9]	1-(2-Hydroxy-4,5-dimethylphenyl)-2-methyl-1-propanone, 2031
[50444-94-5]	1,1′-[Sulfinylbis(6-chloro-4-hydroxy-3,1-phenylene)]bis-1-propanone, 2117
[50816-73-4]	1-(3-Hydroxy-2,4,5,6-tetramethoxyphenyl)-1-propanone, 1904
[50874-47-0]	1-[2,4,6-Trihydroxy-3,5-bis(3-methyl-2-butenyl)phenyl]-1-propanone, 1951
[50916-44-4]	1-(4-Hydroxy-3-nitrophenyl)-1-propanone, 1758
[51233-75-1]	1-(2-Hydroxy-3,6-dimethylphenyl)-1-propanone, 1835
[51233-85-3]	1-[5-(1,1-Dimethylethyl)-2-hydroxy-3-methylphenyl]-1-propanone, 1911
[51233-86-4]	1-[3-Chloro-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-propanone, 1889
[51335-40-1]	1-(3,5-Dichloro-4-hydroxyphenyl)-1-propanone, 1744
[51379-76-1]	1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-propanone, 1881
[51451-24-2]	1-[5-(1,1-Dimethylethyl)-4-hydroxy-2-methylphenyl]-1-propanone, 1912
[51451-25-3]	1-[3-(1,1-Dimethylethyl)-6-hydroxy-2-methylphenyl]-1-propanone, 1911
[51451-26-4]	1-(2-Hydroxy-6-methylphenyl)-1-propanone, 1804
[52069-29-1]	1-[3-(1,1-Dimethylethyl)-4-hydroxy-5-methylphenyl]-1-propanone, 1911
[52099-20-4]	1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-propanone, 1881
[52376-21-3]	1-(4-Bromo-2,5-dihydroxyphenyl)-1-propanone, 1749
[52536-84-2]	1-(4-Hydroxy-3,5-dimethoxyphenyl)-1-propanone (Acetate), 1854
[52597-50-9]	1,1′-[4,6-Dihydroxy-5-(1-oxopropoxy)-1,3-phenylene]bis-1-propanone, 2114
[52749-67-4]	1-(7-Bromo-3,4-dihydroxy-2-naphthalenyl)-1-propanone, 1963
[52856-18-5]	1-(2,6-Dimethoxyphenyl)-2-methyl-1-propanone, 2035
[53107-35-0]	1-(2,3-Dichloro-4-methoxyphenyl)-2-methyl-1-propanone, 2021
[53347-07-2]	1-(5-Chloro-3-ethyl-2-hydroxyphenyl)-1-propanone, 1828
[53773-76-5]	1-(4-Methoxy-2-methylphenyl)-1-propanone, 1841
[54204-29-4]	1-(2-Hydroxyphenyl)-1-propanone uranium complex, 1762
[54204-30-7]	1-(2-Hydroxyphenyl)-1-propanone uranium complex, 1762
[54331-13-4]	1-(2-Hydroxyphenyl)-1-propanone uranium complex, 1762
[54362-59-3]	1,1′-[5-(1,1′-Dimethylethyl)-4-hydroxy-1,3-phenylene]bis-1-propanone, 2116
[54437-05-7]	1-[2-Hydroxy-5-(phenylmethoxy)phenyl]-1-propanone, 1936
[54437-06-8]	1-[2-Hydroxy-5-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]-1-propanone, 1907
[54556-10-4]	1-(2,4,6-Trihydroxy-3,5-dipropylphenyl)-1-propanone, 1930

[60884-04-0]	1-(2-Hydroxy-6-methoxyphenyl)-2,2-dimethyl-1-propanone, 2089
[60884-07-3]	1-(2,4-Dihydroxyphenyl)-2,2-dimethyl-1-propanone, 2085
[60884-08-4]	1-(2,4-Dimethoxyphenyl)-2,2-dimethyl-1-propanone, 2092
[60884-09-5]	1-(2,6-Dihydroxy-4-methylphenyl)-2,2-dimethyl-1-propanone, 2089
[61948-26-3]	1-(2-Hydroxy-3,4-dimethoxyphenyl)-1-propanone, 1851
[61983-10-6]	1-(3,4-Dihydroxy-2-naphthalenyl)-1-propanone, 1969
[61983-12-8]	1-(3,4-Dihydroxy-2-naphthalenyl)-2-methyl-1-propanone, 2064
[61983-21-9]	3,4-Dihydroxy-2-(1-oxopropyl)-1-naphthalenecarbonitrile, 1971
[61983-31-1]	1-(7-Bromo-3,4-dihydroxy-2-naphthalenyl)-2-methyl-1-propanone, 2062
[61983-39-9]	1-(3,4-Dihydroxy-7-methyl-2-naphthalenyl)-1-propanone, 1975
[61983-40-2]	1-(3,4-Dihydroxy-7-methyl-2-naphthalenyl)-2-methyl-1-propanone, 2065
[62545-34-0]	1-[2-(Acetyloxy)-4,6-dihydroxyphenyl]-2-methyl-1-propanone, 2030
[62545-45-3]	1-[4-(Acetyloxy)-2,6-dihydroxyphenyl]-2-methyl-1-propanone, 2030
[63213-30-9]	1-(2,4,5-Trihydroxyphenyl)-1-propanone (Triethyl ether), 1774
[63360-15-6]	1-[4-(3-Chloro-2-hydroxypropoxy)-2-hydroxy-3-(2-propenyl) phenyl]-1-propanone, 1923
[63360-36-1]	7-[2-Hydroxy-3-[3-hydroxy-4-(1-oxopropyl)-2-(2-propenyl) phenoxy]-propoxy]-4-(phenylmethoxy)-2*H*-1-benzopyran-2-one, 1961
[63386-37-8]	1-(3,4-Dimethoxyphenyl)-1-propanone-*3,3,3*-d_3, 1822
[63411-89-2]	1-(2,4-Dihydroxy-5-nitrophenyl)-1-propanone, 1759
[63411-90-5]	1-(4-Ethoxy-2-hydroxyphenyl)-1-propanone, 1845
[63411-91-6]	1-[2-Hydroxy-4-(pentyloxy)phenyl]-1-propanone, 1915
[63411-92-7]	1-[2-Hydroxy-4-(octyloxy)phenyl]-1-propanone, 1946
[63411-93-8]	1-[4-(2-Ethoxyethoxy)-2-hydroxyphenyl]-1-propanone, 1902
[63411-94-9]	1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1-propanone, 1936
[63411-95-0]	1-[4-[(3,4-Dichlorophenyl)methoxy]-2-hydroxyphenyl]-1-propanone, 1931
[63411-96-1]	1-[4-[(2,4-Dichlorophenyl)methoxy]-2-hydroxyphenyl]-1-propanone, 1931
[63437-97-8]	1-[4-Hydroxy-3-[2-(1-oxopropoxy)ethoxy]phenyl]-1-propanone, 1908
[63876-46-0]	1-(2,4-Dihydroxy-3-methylphenyl)-1-propanone, 1809
[63876-51-7]	3,4-Dichloro-7-hydroxy-6-(1-oxopropyl)-2*H*-1-benzopyran-2-one, 1993
[63876-52-8]	3,4-Dichloro-7-hydroxy-8-methyl-6-(1-oxopropyl)-2*H*-1-benzopyran-2-one, 1996
[63909-10-4]	1-(5-Ethyl-2-hydroxyphenyl)-1-propanone, 1834
[64030-63-3]	1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1-propanone, 1848
[64207-03-0]	1-(2-Hydroxy-5-methylphenyl)-2-methyl-1-propanone, 2024

[64603-55-0]	1-(3,5-Dibromo-2,4-dihydroxyphenyl)-1-propanone, 1742
[65208-26-6]	1-(4-Hexadecyl-2,5-dihydroxyphenyl)-1-propanone, 1960
[65282-38-4]	1-[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]-1-propanone radical ion (1^-), 1944
[65282-39-5]	1-[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]-1-propanone-*2,2,3,3,3-*d_5 radical ion (1^-), 1943
[65412-09-1]	1-[5-[(4-Ethylphenyl)azo]-2,4-dihydroxyphenyl]-1-propanone, 1940
[65412-10-4]	1-[2,4-Dihydroxy-5-[(2-methoxyphenyl)azo]phenyl]-1-propanone, 1933
[65412-16-0]	1-[2,4-Dihydroxy-5-[(4-nitrophenyl)azo]phenyl]-1-propanone, 1919
[65412-17-1]	1-[5-(2-Benzothiazolylazo)-2,4-dihydroxyphenyl]-1-propanone, 1931
[65412-18-2]	1-[2,4-Dihydroxy-5-[(6-nitro-2-benzothiazolyl)azo]phenyl]-1-propanone, 1931
[65561-66-2]	1-[2,4-Dihydroxy-5-[[4-(phenylazo)phenyl]azo]phenyl]-1-propanone, 1953
[65561-67-3]	1-[2,4-Dihydroxy-5-[[2-methyl-4-[(2-methylphenyl)azo]phenyl]azo]phenyl]-1-propanone, 1957
[65908-29-4]	1-[5-Hydroxy-2-phenyl-6-(phenylamino)-4-benzoxazolyl]-2,2-dimethyl-1-propanone, 2103
[66021-78-1]	1-(3,5-Dichloro-2-hydroxy-4,6-dimethoxyphenyl)-1-propanone, 1824
[66021-80-5]	1-(3,5-Dichloro-2-hydroxy-4-methoxyphenyl)-1-propanone, 1783
[66021-81-6]	1-(3,5-Dichloro-2,4-dihydroxyphenyl)-1-propanone, 1745
[66047-39-0]	1,1′-[Methylenebis[oxy(2-hydroxy-4,1-phenylene)]]bis-1-propanone, 2119
[66047-41-4]	1,1′-[1,2-Ethanediylbis[oxy(2-hydroxy-4,1-phenylene)]]bis-1-propanone, 2122
[66047-42-5]	1,1′-[1,3-Propanediylbis[oxy(2-hydroxy-4,1-phenylene)]]bis-1-propanone, 2123
[66047-44-7]	1,1′-[Methylenebis[oxy(2-hydroxy-4,1-phenylene)]]bis-1-propanone, (Dioxime), 2119
[66047-46-9]	1,1′-[1,2-Ethanediylbis[oxy(2-hydroxy-4,1-phenylene)]]bis-1-propanone, (Dioxime), 2122
[66047-47-0]	1,1′-[1,3-Propanediylbis[oxy(2-hydroxy-4,1-phenylene)]]bis-1-propanone, (Dioxime), 2123
[66265-14-3]	1-[4-Hydroxy-3-(methylthio)phenyl]-1-propanone, 1808
[66611-86-7]	3-Hydroxy-4-(1-oxopropyl)acetanilide, 1831
[66655-97-8]	1-[2,6-Dihydroxy-4-methoxy-3-methyl-5-[[2,4,6-trihydroxy-3-methyl-5-(1-oxopropyl)phenyl]methyl]phenyl]-1-butanone, 2144
[66711-55-5]	1-(3-Hexyl-2,4,6-trihydroxyphenyl)-1-propanone, 1930
[66711-57-7]	1-(3-Butyl-2,4,6-trihydroxyphenyl)-2-methyl-1-propanone, 2045
[66711-60-2]	1-(2,4,6-Trihydroxy-3-pentylphenyl)-1-propanone, 1916

[67127-81-5]	5-Ethyl-2-hydroxy-3-(1-oxopropyl)benzoic acid, 1862
[67166-42-1]	1-(X-Chloro-4-hydroxy-3-methoxyphenyl)-1-propanone, 1792
[67460-92-8]	1-(6-Methoxy-2-naphthalenyl)-2,2-dimethyl-1-propanone, 2102
[67474-13-9]	1-[4-Hydroxy-3-(phenylsulfonyl)phenyl]-1-propanone, 1922
[67474-14-0]	1-[4-Hydroxy-3-[(4-methylphenyl)sulfonyl]phenyl]-1-propanone, 1937
[67474-15-1]	1-[3-[(4-Chlorophenyl)sulfonyl]-4-hydroxyphenyl]-1-propanone, 1918
[67474-16-2]	1-[3-[(4-Bromophenyl)sulfonyl]-4-hydroxyphenyl]-1-propanone, 1918
[67474-17-3]	1-[4-Hydroxy-3-[(4-iodophenyl)sulfonyl]phenyl]-1-propanone, 1918
[67474-18-4]	1-[4-Hydroxy-3-[(4-methoxyphenyl)sulfonyl]phenyl]-1-propanone, 1938
[67752-18-5]	7-Hydroxy-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one, 1993
[67771-05-5]	Methyl 2,6-dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl)-benzoate, 2044
[68047-77-8]	1-(1-Hydroxy-5-methoxy-2-naphthalenyl)-1-propanone, 1975
[68079-14-1]	1-(2,4-Dihydroxyphenyl)-2-methyl-1-propanone (Uranium complex), 2018
[68223-29-0]	1,1′,1″,1‴-[Methylenebis(2,4,6-trihydroxy-5,1,3-benzenetriyl)] tetrakis-1-propanone, 2127
[68223-31-4]	1,1′,1″,1‴-[Methylenebis(2,4,6-trihydroxy-5,1,3-benzenetriyl)] tetrakis-[2-methyl-1-propanone, 2154
[68223-39-2]	1-[2,4,6-Trihydroxy-3-methyl-5-[[2,4,6-trihydroxy-3,5-bis (1-oxopropyl)phenyl]methyl]-phenyl]-1-butanone, 2144
[68223-40-5]	1-[2,4,6-Trihydroxy-3-methyl-5-[[2,4,6-trihydroxy-3,5-bis(2-methyl-1-oxopropyl)phenyl]methyl]phenyl]-1-butanone, 2164
[68223-53-0]	1-[2,4,6-Trihydroxy-3-(2-methyl-1-oxopropyl)-5-[[2,4,6-trihydroxy-3-methyl- 5-(1-oxobutyl)phenyl]methyl]phenyl]-1-butanone, 2164
[68241-22-5]	1-(6-Hydroxy-3-methyl-1,2-benzisoxazol-7-yl)-1-propanone, 1990
[68241-23-6]	1-(3-Ethyl-6-hydroxy-1,2-benzisoxazol-7-yl)-1-propanone, 1995
[68241-40-7]	1-(6-Hydroxy-3-methyl-1,2-benzisoxazol-7-yl)-1-propanone (Oxime), 1991
[68241-42-9]	1-(3-Ethyl-6-hydroxy-1,2-benzisoxazol-7-yl)-1-propanone (Oxime), 1995
[68597-43-3]	1-(3-Chloro-2-methoxyphenyl)-1-propanone, 1790
[68597-44-4]	1-(5-Chloro-2-methoxyphenyl)-1-propanone, 1791
[68597-45-5]	1-(2-Methoxy-3-methylphenyl)-1-propanone, 1839
[69076-17-1]	4-Hydroxy-7-[2-hydroxy-3-[3-hydroxy-4-(1-oxopropyl)-2-propylphenoxy]-propoxy]-3-nitro-2*H*-1-benzopyran-2-one, 1958
[69076-30-8]	1-[4-(3-Chloro-2-hydroxypropoxy)-2-hydroxy-3-propylphenyl]-1-propanone, 1926

[71539-65-6]	1-[4-[[(2*E*)-3,7-Dimethyl-2,6-octadienyl]oxy]-2,6-dihydroxyphenyl]-2-methyl-1-propanone, 2056
[71539-69-0]	1-(3,4-Dihydro-5-hydroxy-7-methoxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-2-methyl-1-propanone, 2071
[72008-03-8]	1-[3-[(2*E*)-3,7-Dimethyl-2,6-octadienyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-propanone *(E)*, 2055
[72008-05-0]	1-[3-[(2*E*)-3,7-Dimethyl-2,6-octadienyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-propanone *(Z)*, 2055
[72008-14-1]	1-[3,4-Dihydro-5,7-dihydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone, 2073
[72008-16-3]	1-[3,4-Dihydro-5,7-dihydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2*H*-1-benzopyran-6-yl]-2-methyl-1-propanone, 2073
[72017-59-5]	1-(3,4-Dihydroxyphenyl)-2,2-dimethyl-1-propanone, 2086
[72051-68-4]	1-(2-Hydroxyphenyl)-1-propanone-*2,2,3,3,3-*d_5 ion (1^-), radical ion (1^-), 1737
[72051-72-0]	1-[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]-1-propanone radical ion (1^-), 1944
[72051-73-1]	1-[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]-1-propanone-*2,2,3,3,3-*d_5 ion (1^-), radical ion (1^-), 1943
[72051-76-4]	1-(2-Hydroxy-4-methylphenyl)-1-propanone radical ion (1^-), 1802
[72051-77-5]	1-(2-Hydroxy-4-methylphenyl)-1-propanone-*2,2,3,3,3-*d_5 ion (1^-), radical ion (1^-), 1777
[72051-80-0]	1-(4-Ethyl-2-hydroxyphenyl)-1-propanone ion (1^-), radical ion (1^-), 1833
[72051-81-1]	1-(4-Ethyl-2-hydroxyphenyl)-1-propanone-*2,2,3,3,3-*d_5 ion (1^-), 79 radical ion (1^-), 1821
[72051-84-4]	1-[2-Hydroxy-4-(1-methylethyl)phenyl]-1-propanone ion (1^-), radical ion (1^-), 1868
[72051-85-5]	1-[2-Hydroxy-4-(1-methylethyl)phenyl]-1-propanone-*2,2,3,3,3-*d_5 ion (1^-), radical ion (1^-), 1858
[72051-88-8]	1-[4-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-propanone ion (1^-), radical ion (1^-), 1891
[72051-89-9]	1-[4-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-propanone-*2,2,3,3,3-*d_5 ion (1^-), radical ion (1^-), 1882
[72337-80-5]	1-(6,7-Dimethoxy-2-naphthalenyl)-1-propanone, 1978
[72569-10-9]	1-(4-Hydroxyphenyl)-2,2-dimethyl-1-propanone, 2084
[72569-11-0]	1-(4-Hydroxy-3-methylphenyl)-2,2-dimethyl-1-propanone, 2087
[72569-12-1]	1-(4-Hydroxy-3,5-dimethylphenyl)-2,2-dimethyl-1-propanone, 2090
[72569-13-2]	1-(2-Hydroxy-5-methylphenyl)-2,2-dimethyl-1-propanone, 2087
[72569-14-3]	1-(2-Hydroxy-3,4-dimethylphenyl)-2,2-dimethyl-1-propanone, 2089
[72569-15-4]	1-(2-Hydroxy-3,5-dimethylphenyl)-2,2-dimethyl-1-propanone, 2090
[72569-16-5]	1-(2-Hydroxy-4,5-dimethylphenyl)-2,2-dimethyl-1-propanone, 2090
[72934-91-9]	2-[[5,7-Dihydroxy-2,2-dimethyl-8-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-6-yl]methyl]-3,5-dihydroxy-4,4-dimethyl-6-(3-methyl-1-oxobutyl)-2,5-cyclohexadien-1-one, 2165

[76288-10-3]	1,1′-[Methylenebis(1-hydroxy-4,2-naphthalenediyl)]bis-1-propanone, 2128
[76805-57-7]	1-(4-Methoxy-3-methylphenyl)-1-propanone, 1841
[77526-99-9]	2-Hydroxy-5-(1-oxopropyl)benzoic acid methyl ester, 1825
[77697-21-3]	1-(2-Hydroxy-4-propoxyphenyl)-1-propanone (Oxime), 1876
[77820-39-4]	3-[[3-(3,7-Dimethyl-2,6-octadienyl)-2,4,6-trihydroxy-5-(2-methyl-1-oxopropyl)-phenyl]methyl]-6-ethyl-4-hydroxy-5-methyl-2*H*-pyran- 2-one *(E)*, 2061
[77942-13-3]	1-(2,4-Dimethoxy-3-methylphenyl)-1-propanone, 1873
[77942-24-6]	1-(2,4-Dimethoxy-5-methylphenyl)-1-propanone, 1874
[78094-43-6]	1-(2-Hydroxy-3-methoxyphenyl)-1-propanone, 1811
[78094-44-7]	2-(2-Ethyl-1,3-dioxolan-2-yl)-6-methoxy-2,5-cyclohexadiene-1,4-dione, 2007
[78094-46-9]	5-Hydroxy-6-methoxy-2-methyl-4-(1-oxopropyl)-3-benzofurancarboxylic acid ethyl ester, 2007
[78377-68-1]	3,4-Dihydro-6,8-dihydroxy-7-methyl-1(2*H*)-naphthalenone, 2143
[78377-69-2]	3,4-Dihydro-6,8-dihydroxy-7-methyl-5-(1-oxopropyl)-1(2*H*)-naphthalenone, 2143
[78377-71-6]	6,8-Dimethoxy-7-methyl-5-(1-oxopropyl)-1,2-naphthalenedione, 1979
[78377-72-7]	1-(5-Hydroxy-2,4-dimethoxy-3-methyl-6-nitroso-1-naphthalenyl)-1-propanone, 1979
[78377-74-9]	N-[1,6-Dihydroxy-8-methoxy-7-methyl-5-(1-oxopropyl)-2-naphthalenyl]acetamide, 1982
[78411-76-4]	3-[[2,6-Dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl) phenyl]-methyl]-2,4,6-trihydroxy-5-(3-methyl-1-oxobutyl) benzaldehyde, 2161
[78417-99-9]	2-Hydroxy-5-(1-oxopropyl)benzoic acid, 1784
[79010-36-9]	1-(3-Acetyl-4-hydroxyphenyl)-1-propanone, 2132
[79387-88-5]	1-(1-Hydroxy-2-naphthalenyl)-2-methyl-1-propanone, 2062
[79558-49-9]	1-(2,4-Dihydroxy-3-propylphenyl)-1-propanone, 1872
[79744-62-0]	1-(2,4,5-Trihydroxyphenyl)-1-propanone, 1773
[79744-64-2]	1-(2,4-Dihydroxy-5-methoxyphenyl)-1-propanone, 1818
[79925-34-1]	1-(2-Hydroxy-4-nitrophenyl)-1-propanone, 1757
[79925-35-2]	1-(5-Amino-2-hydroxyphenyl)-1-propanone, 1777
[80427-25-4]	1-[2-Methoxy-5-(1-oxopropoxy)phenyl]-1-propanone, 1887
[80427-31-2]	1-(5-Hydroxy-2-methoxyphenyl)-1-propanone, 1818
[81336-14-3]	1-(4,6-Dimethoxy-1-naphthalenyl)-1-propanone, 1978
[81336-21-2]	1-(6-Methoxy-1-naphthalenyl)-1-propanone, 1974
[81336-23-4]	1-(4-Hydroxy-6-methoxy-1-naphthalenyl)-1-propanone, 1976
[81389-57-3]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-2-methyl-1-propanone (O-methyloxime), 2054
[81389-67-5]	1-[3-(1,1-Dimethylethyl)-4,5-dihydroxyphenyl]-2,2-dimethyl-1-propanone, 2096
[81421-70-7]	1-(2-Hydroxy-3,4-dimethoxy-6-methylphenyl)-1-propanone, 1877

[81421-71-8]	1-(2,3,4-Trimethoxy-6-methylphenyl)-1-propanone, 1903
[81484-62-0]	1-(5-Bromo-4-methoxy-2-nitrosophenyl)-1-propanone, 1780
[82053-90-5]	1-(2-Methoxy-3-methylphenyl)-2-methyl-1-propanone, 2032
[82350-84-3]	1-(4-Hydroxy-3-nitrophenyl)-2-methyl-1-propanone, 2015
[82490-57-1]	1-(6-Hydroxy-2,3,4-trimethylphenyl)-2-methyl-1-propanone, 2039
[82620-73-3]	1-(2-Methoxy-5-methylphenyl)-1-propanone, 1839
[82623-49-2]	1-(2-Methoxy-5-methylphenyl)-1-propanone (2,4-Dinitrophenylhydrazone), 1840
[82667-80-9]	1-(5-Chloro-2,4-dihydroxyphenyl)-1-propanone (Oxime), 1753
[82846-20-6]	1-(4-Fluoro-3-methoxyphenyl)-1-propanone, 1793
[83016-68-6]	1-(7-Hydroxy-1,3-benzodioxol-5-yl)-1-propanone, 1989
[83294-23-9]	1-(4-Amino-2-hydroxyphenyl)-1-propanone, 1776
[83569-68-0]	1-[4-Hydroxy-3-methoxy-5-(1-methylethyl)phenyl]-1-propanone, 1900
[83569-69-1]	1-[3,4-Dimethoxy-5-(1-methylethyl)phenyl]-1-propanone, 1913
[83674-89-9]	1-[4-Methoxy-3-(phenylmethoxy)phenyl]-1-propanone, 1942
[85052-32-0]	1-[5-(1,1-Dimethylethyl)-2-hydroxy-3-nitrophenyl]-1-propanone, 1890
[85131-64-2]	1-(5-Chloro-2,4-dihydroxyphenyl)-1-propanone, 1753
[85602-23-9]	1-[2,4,6-Trihydroxy-3-(2-propenyl)phenyl]-1-propanone, 1863
[86690-30-4]	1-[2-(1,1-Dimethylethyl)-6-hydroxyphenyl]-1-propanone, 1890
[86774-65-4]	1-(2,4-Dimethoxyphenyl)-2-methyl-1-propanone, 2034
[87061-00-5]	1-[2-Hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-1-propanone, 1907
[87108-26-7]	1-[2-[(3,7-Dimethyl-2,6-octadienyl)oxy]-4-hydroxyphenyl]-1-propanone *(E)*, 1951
[87544-99-8]	1-[2-Hydroxy-5-(1-oxopropyl)phenyl]-3-phenyl-2-propen-1-one, 2139
[87545-01-5]	1-[2-Hydroxy-5-(1-oxopropyl)phenyl]-3-(4-methoxyphenyl)-2-propen-1-one, 2140
[88466-30-2]	2-Hydroxy-3-(1-oxopropyl)benzoic acid methyl ester, 1825
[88521-75-9]	1-(2-Hydroxy-5-nitrophenyl)-2-methyl-1-propanone, 2015
[88771-65-7]	1-(4-Chloro-2-hydroxy-3,6-dimethoxyphenyl)-2-methyl-1-propanone, 2030
[88772-49-0]	1-(4-Chloro-2,5-dihydroxyphenyl)-2-methyl-1-propanone, 2014
[88792-61-4]	1-(1-Hydroxy-4,8-dimethoxy-2-naphthalenyl)-1-propanone, 1978
[89106-18-3]	1-(4-Chloro-3-methoxyphenyl)-1-propanone, 1791
[89106-39-8]	1-(3-Chloro-5-methoxyphenyl)-1-propanone, 1790
[89106-46-7]	1-(2-Fluoro-5-methoxyphenyl)-1-propanone, 1792
[89556-60-5]	1-(2,5-Dimethoxyphenyl)-2-methyl-1-propanone, 2035
[89880-48-8]	1-(2,3,4,6-Tetramethoxyphenyl)-1-propanone, 1903
[89880-49-9]	1-(3-Ethoxy-2-hydroxy-4,6-dimethoxyphenyl)-1-propanone, 1903
[90363-43-2]	1-(1,4-Dihydro-6,8-dimethoxy-7-methyl-1,4-epoxynaphthalene-5-yl)-1-propanone, 1980
[90363-44-3]	1-(8-Hydroxy-2,4-dimethoxy-3-methyl-1-naphthalenyl)-1-propanone, 1980

[90363-46-5]	3,5-Dimethoxy-4-methyl-2*H*-naphtho[1,8-*bc*]furan-2-one, 1980
[90363-47-6]	2-Bromo-5,7-dimethoxy-6-methyl-8-(1-oxopropyl)-1,4-naphthalenedione, 2143
[90363-48-7]	5,7-Dimethoxy-6-methyl-8-(1-oxopropyl)-1,4-naphthalenedione, 2143
[90363-49-8]	1-(7-Bromo-2,4,5,8-tetramethoxy-3-methyl-1-naphthalenyl)-1-propanone, 1983
[90363-50-1]	1-(2,4,5,8-Tetramethoxy-3-methyl-1-naphthalenyl)-1-propanone, 1984
[90363-51-2]	1-(7-Bromo-2,4,5,8-tetramethoxy-3-methyl-6-nitronaphthalenyl)-1-propanone, 1983
[90363-52-3]	1-(2,4,5,8-Tetramethoxy-3-methyl-6-nitro-1-naphthalenyl)-1-propanone, 1983
[90363-53-4]	1-(6-Amino-2,4,5,8-tetramethoxy-3-methyl-1-naphthalenyl)-1-propanone, 1984
[90363-54-5]	N-[1,4,6,8-Tetramethoxy-7-methyl-5-(1-oxopropyl)-2-naphthalenyl]acetamide, 1984
[90536-26-8]	1-(2,3-Dihydroxyphenyl)-1-propanone, 1767
[90537-41-0]	1-(5-Chloro-2-hydroxy-3-nitrophenyl)-1-propanone, 1743
[90725-67-0]	1-(5-Bromo-2-hydroxy-3-nitrophenyl)-1-propanone, 1741
[90743-04-7]	1-(5-Chloro-2-hydroxyphenyl)-2-methyl-1-propanone, 2013
[90852-26-9]	1-(4-Methoxy-2,3-dimethylphenyl)-1-propanone, 1871
[90922-89-7]	1-(2-Hydroxy-3-methyl-5-nitrophenyl)-1-propanone, 1795
[90922-90-0]	1-(4-Hydroxy-2-methyl-5-nitrophenyl)-1-propanone, 1796
[91061-55-1]	1-(2,3-Dihydroxy-5-methylphenyl)-1-propanone, 1808
[91134-62-2]	1-(4,5-Dimethoxy-2-nitrophenyl)-1-propanone, 1832
[91143-39-4]	4-Methoxy-3-(1-oxopropyl)benzoic acid, 1826
[91143-73-6]	1-[4-(Acetyloxy)-2-hydroxyphenyl]-1-propanone, 1824
[91211-02-8]	1-(2-Hydroxy-3,5-dinitrophenyl)-1-propanone, 1746
[91307-45-8]	1-(5-Hydroxy-1-naphthalenyl)-1-propanone, 1967
[91496-09-2]	1-[4-Hydroxy-3-(2-propenyl)phenyl]-1-propanone, 1859
[91496-99-0]	1-(3-Acetyl-2-hydroxy-5-methylphenyl)-1-propanone, 2134
[91497-18-6]	4-Methoxy-3-(1-oxopropyl)benzoic acid methyl ester, 1863
[91497-44-8]	1,1′-(2,5-Dihydroxy-1,4-phenylene)bis-1-propanone, 2109
[91555-68-9]	1-(2,4-Dihydroxy-6-methoxy-3-methylphenyl)-2-methyl-1-propanone, 2036
[91569-25-4]	8-Hydroxy-5-(1-oxopropyl)quinoline, 1994
[91641-62-2]	4-Hydroxy-3-(1-oxopropyl)propionanilide, 1865
[91889-34-8]	1-[4-(Hydroxy-*d*)phenyl]-1-propanone-*2,2*-d_2, 1739
[91889-35-9]	1-(4-Methoxyphenyl)-1-propanone-*2,2*-d_2, 1807
[91902-70-4]	1-(3-Hydroxy-2-naphthalenyl)-1-propanone, 1966
[91970-96-6]	1-(2-Hydroxy-3-methoxy-5-methylphenyl)-1-propanone, 1847
[91970-97-7]	1-(4-Hydroxy-3-methoxy-5-methylphenyl)-1-propanone, 1849
[91970-98-8]	1-(5-Hydroxy-4-methoxy-2-methylphenyl)-1-propanone, 1850
[91991-98-9]	1-(2-Hydroxy-3-nitrophenyl)-1-propanone, 1756
[92156-94-0]	1-(2-Ethyl-4,5-dimethoxyphenyl)-1-propanone, 1899

[92189-66-7]	1-(5-Bromo-6-methoxy-2-naphthalenyl)-1-propanone, 1971
[92301-90-1]	2-Methyl-1-(2,4,6-trimethoxy-3-methylphenyl)-1-propanone, 2046
[92554-09-1]	1-(4-Hydroxy-5-nitro[1,1′-biphenyl]-3-yl)-1-propanone, 1919
[92729-83-4]	1-(2-Hydroxy-3,5-dipropylphenyl)-1-propanone, 1928
[92920-81-5]	1-(1-Hydroxy-5-methoxy-2-naphthalenyl)-2-methyl-1-propanone, 2066
[92920-82-6]	1-(1-Hydroxy-5-methoxy-2-naphthalenyl)-2,2-dimethyl-1-propanone, 2103
[94190-87-1]	1,1′-(2-Hydroxy-4,6-dimethoxy-1,3-phenylene)bis-1-propanone, 2114
[94190-88-2]	1-[2-Hydroxy-4,6-dimethoxy-3-(1-oxopropoxy)phenyl]-1-propanone, 1908
[94190-89-3]	1-(2,3-Dihydroxy-4,6-dimethoxyphenyl)-1-propanone, 1854
[95102-26-4]	1-[5-(1,1-Dimethylethyl)-2,4-dihydroxyphenyl]-1-propanone, 1899
[95102-28-6]	1-[5-(1,1-Dimethylpropyl)-2,4-dihydroxyphenyl]-1-propanone, 1914
[95102-29-7]	1-(6-Hydroxy[1,1′-biphenyl]-3-yl)-1-propanone, 1920
[95125-20-5]	1,1′-[Thiobis(6-hydroxy-3,1-phenylene)]bis-1-propanone, 2118
[95185-71-0]	1-(3-Cyclohexyl-4-hydroxyphenyl)-1-propanone, 1923
[95455-11-1]	1-(2-Hydroxy-1-naphthalenyl)-2-methyl-1-propanone, 2063
[95699-99-3]	1,1′-[Sulfonylbis(6-hydroxy-3,1-phenylene)bis]-1-propanone, 2118
[95818-32-9]	1-(2,4-Dihydroxy-6-pentadecylphenyl)-1-propanone, 1959
[96552-58-8]	1-[2,3-Dihydro-4,6-dihydroxy-2-(1-methylethenyl)-5-benzofuranyl]-2-methyl-1-propanone (R), 2068
[96573-29-4]	2,4,6-Trihydroxy-3-(1-oxopropyl)benzaldehyde, 2131
[96573-37-4]	2,4,6-Trihydroxy-3-methyl-5-(1-oxopropyl)benzaldehyde, 2133
[96573-38-5]	3-Ethyl-2,4,6-trihydroxy-5-(1-oxopropyl)benzaldehyde, 2134
[96573-39-6]	2,4,6-Trihydroxy-3-(1-oxopropyl)-5-propylbenzaldehyde, 2135
[96573-40-9]	1-[2,4,6-Trihydroxy-3-(1-oxopropyl)phenyl]-1-butanone, 2135
[96573-42-1]	2,4,6-Trihydroxy-5-(1-oxopropyl)-1,3-benzenedicarboxaldehyde, 2132
[96573-44-3]	2,4,6-Trihydroxy-3-(1-oxopropyl)benzaldehyde (1-oxime), 1798
[96573-45-4]	2-[[2,4,6-Trihydroxy-3-(1-oxopropyl)phenyl]methylene] hydrazinecarboxamide, 1833
[96756-26-2]	2-Methyl-1-(2,4,6-trihydroxy-3-pentylphenyl)-1-propanone, 2049
[96756-27-3]	1-[2,4,6-Trihydroxy-3-(3-methylbutyl)phenyl]-1-propanone, 1916
[96853-74-6]	1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl) methyl]-2,4,6-trihydroxy-5-(3-methyl-2-butenyl)phenyl]-2-methyl-1-propanone, 2162
[97139-82-7]	1-(2-Hydroxyphenyl)-1-propanone (Acetate), 1763
[97304-06-8]	1-[2,4-Dihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-propanone, 1906
[97304-11-5]	1-[2,4-Dihydroxy-5-(3-methyl-2-butenyl)phenyl]-1-propanone, 1906

[99814-61-6]	1,1′-[(6-Methylheptylidene)bis(3,4-dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-6,8-diyl)]bis[2-methyl-1-propanone, 2157
[99842-69-0]	4-Hydroxy-3-(1-oxopropyl)phenylacetonitrile, 1823
[99842-70-3]	2-Methoxy-3-(1-oxopropyl)benzonitrile, 1823
[99853-35-7]	5-Bromo-2,4-dihydroxy-3-(1-oxopropyl)benzoic acid methyl ester, 1822
[99854-30-5]	5-Chloro-2,4-dihydroxy-3-(1-oxopropyl)benzoic acid methyl ester, 1822
[99855-34-2]	4-Hydroxy-3-(1-oxopropyl)acetanilide, 1831
[99860-74-9]	1-[3,5-Bis(chloromethyl)-2-hydroxyphenyl]-1-propanone, 1824
[99964-98-4]	1-(2,4-Dihydroxy-3,5-dimethoxyphenyl)-1-propanone, 1855
[99965-00-1]	1-[2-Hydroxy-3,5-dimethoxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-1-propanone, 1938
[100059-02-7]	1-(2,3-Dihydroxyphenyl)-2,2-dimethyl-1-propanone, 2085
[100117-91-7]	4-Hydroxy-3-(1-oxopropyl)benzoic acid ethyl ester, 1862
[100126-81-6]	1-[5-(Chloromethyl)-2-methoxyphenyl]-1-propanone, 1830
[100246-22-8]	1-(3-Bromo-2,4-dihydroxy-5-nitrophenyl)-1-propanone, 1741
[100257-32-7]	1-[5-(Ethoxymethyl)-2-hydroxyphenyl]-1-propanone, 1875
[100393-42-8]	8-Hydroxy-2-methyl-5-(1-oxopropyl)quinoline, 2000
[100523-86-2]	1-(3,5-Dihydroxyphenyl)-1-propanone, 1772
[100612-28-0]	1-(5,6,7,8-Tetrahydro-1-hydroxy-2-naphthalenyl)-1-propanone, 1970
[100884-41-1]	3-[2,4-Dimethoxy-3-(1-oxopropyl)phenyl]-2-propenoic acid, 1905
[100886-06-4]	1-(6-Hydroxy-2-naphthalenyl)-2-methyl-1-propanone, 2063
[100886-53-1]	7-Hydroxy-2,3-dimethyl-8-(1-oxopropyl)-4*H*-1-benzopyran-4-one, 2142
[100953-75-1]	5-Bromo-6-hydroxy-3-methyl-7-(1-oxopropyl)benzofuran, 1993
[100972-94-9]	1-(2-Acetyl-3,5,6-trimethoxyphenyl)-1-propanone, 2135
[100976-03-2]	1-(1-Hydroxy-2-naphthalenyl)-2,2-dimethyl-1-propanone, 2100
[101002-22-6]	1-[4-(Cyclohexyloxy)-2-hydroxyphenyl]-1-propanone (Oxime), 1924
[101002-26-0]	1-(4-Dodecyl-2-hydroxyphenyl)-1-propanone (Oxime), 1955
[101002-35-1]	1-[4-(Cyclohexyloxy)-2-hydroxyphenyl]-1-propanone, 1924
[101002-16-8]	1-[4-(Decyloxy)-2-hydroxyphenyl]-1-propanone (Oxime), 1952
[101002-30-6]	1-[4-(Decyloxy)-2-hydroxyphenyl]-1-propanone, 1951
[101012-66-2]	5-Bromo-2,4-dihydroxy-3-(1-oxopropyl)benzoic acid, 1778
[101103-44-0]	1-(2,3-Dihydroxyphenyl)-2,2-dimethyl-1-propanone (Diacetate), 2085
[101386-02-1]	1-(2,6-Dihydroxyphenyl)-2-methyl-1-propanone, 2018
[101597-03-9]	1-(5-Benzoyl-2-hydroxy-3-methylphenyl)-1-propanone, 2137
[101724-90-7]	8-Hydroxy-5-(2-methyl-1-oxopropyl)quinoline, 2067
[102020-39-3]	1-(5-Decyl-2-hydroxyphenyl)-1-propanone, 1951
[102092-19-3]	1-(2,4-Dihydroxy-6-methoxyphenyl)-2-methyl-1-propanone, 2027
[102168-44-5]	1-[4-Hydroxy-2-methyl-5-(1-methylethyl)-3-(phenylmethyl)phenyl]-1-propanone, 1953

[103784-19-6]	3-[[5,7-Dihydroxy-2,2-dimethyl-6-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-8-yl]methyl]-6-ethyl-4-hydroxy-5-methyl-2*H*-pyran-2-one, 2082
[103784-23-2]	2-Methyl-1-[5,7,8-trihydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2*H*-1-benzopyran-6-yl]-1-propanone, 2073
[103858-21-5]	1,1′-[Thiobis(4-hydroxy-5-methyl-3,1-phenylene)]bis-1-propanone, 2121
[103863-52-1]	1,1′-[Thiobis(2-hydroxy-5-methyl-3,1-phenylene)]bis-1-propanone, 2120
[104008-43-7]	1-(3,5-Diethyl-4-methoxyphenyl)-1-propanone, 1910
[104008-46-0]	1-(3,5-Diethyl-4-hydroxyphenyl)-1-propanone, 1890
[104095-32-1]	5-Chloro-6-hydroxy-3-methyl-7-(1-oxopropyl)benzofuran, 1994
[104129-04-6]	1-[3-(Aminomethyl)-5-bromo-4-hydroxyphenyl]-1-propanone, 1798
[104129-05-7]	1-[3-(Aminomethyl)-5-bromo-4-hydroxyphenyl]-1-propanone (Oxime), 1798
[104129-07-9]	1-[3-(Aminomethyl)-4-hydroxy-5-iodophenyl]-1-propanone (Oxime), 1799
[104129-08-0]	1-[3-(Aminomethyl)-5-chloro-4-hydroxyphenyl]-1-propanone (Oxime), 1799
[104129-14-8]	1-[3-(Aminomethyl)-5-bromo-4-hydroxyphenyl]-2-methyl-1-propanone (Oxime), 2023
[104129-15-9]	1-[3-(Aminomethyl)-5-bromo-4-hydroxyphenyl]-2-methyl-1-propanone, 2023
[104216-16-2]	1-(4-Hydroxy-5-methoxy-2-methylphenyl)-1-propanone, 1849
[104216-40-2]	1-(3-Methoxy-2-methylphenyl)-1-propanone, 1840
[104557-28-0]	1-(4-Hydroxy-3-nonylphenyl)-1-propanone, 1949
[105041-56-3]	1-(3-Chloro-6-hydroxy-2,4-dimethylphenyl)-1-propanone, 1829
[105290-18-4]	1,1′-(2-Hydroxy-5-methyl-1,3-phenylene)bis-1-propanone, 2111
[105329-87-1]	1-(2,3-Dimethoxyphenyl)-2-methyl-1-propanone, 2034
[105339-17-1]	5-Ethyl-2,4-dihydroxy-3-(1-oxopropyl)benzoic acid, 1864
[105630-20-4]	1-[3,5-Bis(diphenylmethyl)-2,4,6-trihydroxyphenyl]-1-propanone, 1962
[105630-21-5]	1-[3,5-Bis(diphenylmethyl)-2,4,6-trimethoxyphenyl]-1-propanone, 1962
[105630-22-6]	1-[3-(Diphenylmethyl)-2,4,6-trihydroxyphenyl]-1-propanone, 1956
[105630-23-7]	1-[3-(Diphenylmethyl)-2,4,6-trimethoxyphenyl]-1-propanone, 1960
[105838-23-1]	2-Ethyl-3-methoxy-5-(1-oxopropyl)benzonitrile, 1884
[105905-84-8]	1,1′-(4,6-Dihydroxy-5-nitro-1,3-phenylene)bis-1-propanone, 2107
[105910-12-1]	1,1′-(2,4-Dihydroxy-5-nitro-1,3-phenylene)bis-1-propanone, 2107
[106141-17-7]	1-(2-Hydroxy-3,5-dimethylphenyl)-2-methyl-1-propanone, 2031
[106214-15-7]	5-Ethyl-2,4-dihydroxy-3-(1-oxopropyl)benzoic acid methyl ester, 1887

[109218-76-0]	5-Chloro-6-hydroxy-3-methyl-7-(1-oxopropyl)benzofuran-2-carboxylic acid, 1998
[109314-49-0]	1-[3-(Aminomethyl)-4-hydroxyphenyl]-1-propanone (Hydrochloride), 1821
[109314-56-9]	1-[3-(Aminomethyl)-4-hydroxy-5-iodophenyl]-1-propanone, 1799
[109314-57-0]	1-[3-(Aminomethyl)-5-chloro-4-hydroxyphenyl]-1-propanone, 1799
[109402-62-2]	6-Ethyl-7-hydroxy-4-methyl-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one, 2005
[109402-64-4]	5-Ethyl-6-hydroxy-3-methyl-7-(1-oxopropyl)-2-benzofurancarboxylic acid, 2005
[109469-57-0]	1-(4-Hydroxy-5-methoxy-2-methylphenyl)-1-propanone (Benzoate), 1850
[110030-74-5]	6-Chloro-7-hydroxy-4-methyl-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one, 1998
[110054-61-0]	3-Chloro-7-hydroxy-4-methyl-8-(1-oxopropyl)-2*H*-1-benzopyran-2-one, 1998
[111039-01-1]	1-(3-Ethyl-4-methoxyphenyl)-2-methyl-1-propanone, 2039
[111122-82-8]	2,2-Dimethyl-1-(2,4,6-trimethoxyphenyl)-1-propanone, 2094
[111122-92-0]	2-(1,1-Dimethylethyl)-2-(2,4,6-trimethoxyphenyl)-1,3-benzoxathiole, 2094
[111983-96-1]	1-(5,7-Dihydroxy-2,2,6-trimethyl-2*H*-1-benzopyran-8-yl)-2-methyl-1-propanone, 2070
[111983-98-3]	1,1′-(1,7a,13a,13b-Tetrahydro-5,8,10-trihydroxy-2,2,6,9,13,13-hexamethyl-2*H*,-13*H*-bis[1]benzopyrano[5,4-*bc*:3′,4′-*e*]pyran-4,11-diyl)bis[2-methyl-1-propanone (7aα, 13aα, 13bα), 2155
[112450-17-6]	5-Hydroxy-4-(1-oxopropyl)-1,3-benzoxathiol-2-one, 1988
[112450-18-7]	5-Hydroxy-4-(2-methyl-1-oxopropyl)-1,3-benzoxathiol-2-one, 2067
[112450-27-8]	1-(2,5-Dihydroxyphenyl)-2-methyl-1-propanone, 2018
[112613-99-7]	2-Methyl-1-[1,7a,13a,13b-tetrahydro-5,8,10-trihydroxy-2,2,6,9,13,13-hexamethyl-4-(2-methyl-1-oxopropyl)-2*H*,13*H*-bis-1-benzopyrano[5,4-*bc*:3′,4′-*e*]pyran-11-yl]-1-butanone, 2165
[112614-00-3]	2-Methyl-1-[1,7a,13a,13b-tetrahydro-5,8,10-trihydroxy-2,2,6,9,13,13-hexa-methyl-11-(2-methyl-1-oxopropyl)-2*H*,13*H*-bis-1-benzopyrano[5,4-*bc*:3′,4′-*e*]- pyran-4-yl]-1-butanone, 2166
[112709-78-1]	1,1′-(1,7a,13a,13b-Tetrahydro-5,8,10-trihydroxy-2,2,6,9,13,13-hexamethyl-1-2*H*, 13*H*-bis-1-benzopyrano[5,4-*bc*:3′,4′-*e*]pyran-4,11-diyl)bis[2-methylpropanone, 2155
[113730-35-1]	1-(2-Amino-3,4-dichloro-5-methoxyphenyl)-1-propanone, 1792
[113730-37-3]	1-(3,4-Dichloro-5-methoxyphenyl)-1-propanone, 1782
[113730-39-5]	1-(3,4-Dichloro-5-hydroxyphenyl)-1-propanone, 1743
[113730-43-1]	1-[4,5-Dichloro-3-hydroxy-2-(2-propenyl)phenyl]-1-propanone, 1858
[114113-06-3]	1-(5-Ethyl-2,4-dihydroxyphenyl)-1-propanone (Oxime), 1847
[114477-31-5]	1-[3-[(Hexyloxy)methyl]-4-hydroxyphenyl]-1-propanone, 1940

[114477-32-6]	1-[4-Hydroxy-3-(propoxymethyl)phenyl]-1-propanone, 1901
[115048-28-7]	4-Hydroxy-3-(1-oxopropyl)phenylacetic acid, 1825
[116074-75-0]	1-[2,3-Dihydro-5-hydroxy-3,3-dimethyl-2-(4-morpholinyl)-4-benzofuranyl]-1-propanone, 2007
[116235-78-0]	1-(4-Hydroxy-3-methoxyphenyl)-1-propanone ion (1^-), 1816
[116557-45-0]	1-(2-Hydroxy-4-methylphenyl)-2-methyl-1-propanone, 2024
[116867-95-9]	1-(3-Ethyl-2,6-dihydroxyphenyl)-1-propanone, 1847
[116964-16-0]	1-[6-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl) methyl]-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone, 2162
[117844-76-5]	1-(3,4-Dihydro-5-hydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-1-propanone, 2003
[117844-77-6]	1-(3,4-Dihydro-7-hydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-1-propanone, 2003
[117844-79-8]	1-(2,4-Dihydroxy-3-iodophenyl)-1-propanone, 1756
[117844-80-1]	1-(3,4-Dihydro-7-hydroxy-8-iodo-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-1-propanone, 2003
[117844-90-3]	1-(3,4-Dihydro-7-hydroxy-2,2,8-trimethyl-2*H*-1-benzopyran-6-yl)-1-propanone, 2006
[117952-43-9]	1-(2,4-Dihydroxy-5-methylphenyl)-1-propanone, 1809
[117970-66-8]	1-(2-Hydroxy-3,4,5,6-tetramethoxyphenyl)-1-propanone, 1904
[118585-45-8]	1-(5-Hydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-1-propanone, 2002
[118609-36-2]	1-[4-[(1,1-Dimethyl-2-propynyl)oxy]-2-hydroxyphenyl]-1-propanone, 1904
[118683-25-3]	4-[[3-Hydroxy-4-(1-oxopropyl)-2-propylphenoxy]methyl]-3-methoxybenzoic acid, 1954
[119232-80-3]	1-(2,5-Dihydroxy-3,4,6-trimethoxyphenyl)-1-propanone, 1881
[119257-50-0]	1-(3-Fluoro-2,6-dimethoxyphenyl)-1-propanone, 1831
[119426-01-6]	1-(4-Hydroxy-7-methoxy-1,3-benzodioxol-5-yl)-1-propanone, 1993
[119691-92-8]	1-(2,4,6-Trihydroxy-3-nitrophenyl)-1-propanone, 1760
[119691-98-4]	1-(2,4,6-Trihydroxy-3-nitro-5-propylphenyl)-1-propanone, 1866
[119994-05-7]	1-(4-Fluoro-2-hydroxy-5-nitrophenyl)-2-methyl-1-propanone, 2012
[119994-06-8]	1-(4-Fluoro-2-hydroxy-5-nitrophenyl)-2-methyl-1-propanone (Oxime), 2012
[120072-80-2]	1-(2,4-Dihydroxy-3-propylphenyl)-2-methyl-1-propanone, 2040
[120350-19-8]	1-[4-(1,1-Dimethylethyl)-2,5-dimethoxyphenyl]-1-propanone, 1929
[120350-21-2]	1-[4-(1,1-Dimethylethyl)-2,5-dimethoxyphenyl]-2-methyl-1-propanone, 2052
[120716-99-6]	3-Butyl-2,4,6-trihydroxy-5-(1-oxopropyl)benzaldehyde, 2135
[121194-61-4]	1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-propanone, 1895
[121194-62-5]	1-[2-Hydroxy-4-methyl-5-(1-methylethyl)phenyl]-1-propanone, 1894

[154185-29-2]	1-[4-Hydroxy-3-(1-oxopropyl)phenyl]-3-phenyl-2-propen-1-one, 2139
[154185-30-5]	3-(4-Chlorophenyl)-1-[4-hydroxy-3-(1-oxopropyl)phenyl]-2-propen-1-one, 2138
[154185-31-6]	1-[4-Hydroxy-3-(1-oxopropyl)phenyl]-3-(4-methylphenyl)-2-propen-1-one, 2140
[154185-32-7]	1-[4-Hydroxy-3-(1-oxopropyl)phenyl]-3-(4-methoxyphenyl)-2-propen-1-one, 2140
[154185-33-8]	3-(3,4-Dimethoxyphenyl)-1-[4-hydroxy-3-(1-oxopropyl)phenyl]-2-propen-1-one, 2141
[154603-70-0]	1-[3-(Azidomethyl)-4-hydroxyphenyl]-1-propanone, 1798
[154783-68-3]	1-[3-[(Cyclopentyloxy)methyl]-4-hydroxyphenyl]-1-propanone, 1924
[154921-37-6]	1-[2-[(Difluoroboryl)oxy]-4-hydroxy-6-methylphenyl]-1-propanone, 1785
[155969-61-2]	1-(4-Hydroxy-3-methoxyphenyl)-1-propanone (β-D-Glucopyranoside), 1817
[157732-52-0]	1-(5-Bromo-2,4-dihydroxyphenyl)-1-propanone, 1749
[158153-03-8]	1-(5-Ethyl-2,4-dimethoxyphenyl)-1-propanone, 1900
[158153-04-9]	1-(5-Ethyl-2,4-dihydroxyphenyl)-1-propanone, 1847
[158897-28-0]	1-(2-Fluoro-6-methoxyphenyl)-2,2-dimethyl-1-propanone, 2086
[158897-29-1]	1-(2-Fluoro-6-hydroxyphenyl)-2,2-dimethyl-1-propanone, 2083
[159186-06-8]	1-(3-Ethoxy-4-hydroxyphenyl)-1-propanone, 1845
[159977-37-4]	1-(5-Cyclohexyl-2,4-dihydroxyphenyl)-1-propanone, 1924
[160308-44-1]	1-(4-Ethoxy-2-hydroxyphenyl)-1-propanone Polymer with 1,4-butanediol, 1846
[161140-14-3]	1-(4-Butoxy-2-hydroxyphenyl)-1-propanone (Oxime), 1898
[161319-29-5]	1-[4-Hydroxy-3-[(2,4,7-trinitro-9*H*-fluorene-9-ylidene)amino]phenyl]-2-methyl-1-propanone, 2059
[161429-78-3]	1-[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]-2,2-dimethyl-1-propanone (Oxime), 2099
[161450-86-8]	1-(4-Ethoxy-2-hydroxyphenyl)-1-propanone Polymer with 1,2-ethanediol, 1846
[161583-88-6]	1-[2,3-Bis(acetyloxy)-4-hydroxyphenyl]-1-propanone, 1883
[162052-63-3]	1-(2-Methoxy-6-methylphenyl)-2,2-dimethyl-1-propanone, 2091
[162052-64-4]	2,2-Dimethyl-1-(2,3,4-trimethoxy-6-methylphenyl)-1-propanone, 2096
[163706-93-2]	1-(4-Ethoxy-2-hydroxyphenyl)-1-propanone Polymer with 1,3-propanediol, 1846
[164072-22-4]	1-(2-Fluoro-3-hydroxyphenyl)-1-propanone, 1754
[166973-19-9]	1-(2-Amino-5-methoxyphenyl)-2-methyl-1-propanone, 2029
[171609-26-0]	1-[2-Hydroxy-6-(2-methyl-1,3-dioxolan-2-yl)phenyl]-2,2-dimethyl-1-propanone, 2094
[171609-27-1]	1-(2-Acetyl-6-hydroxyphenyl)-2,2-dimethyl-1-propanone, 2171
[171609-28-2]	1-(2-Acetyl-6-hydroxyphenyl)-2-methyl-1-propanone, 2159

[173867-31-7]	1-(2-Hydroxy-4,6-dimethoxy-3,5-dimethylphenyl)-2-methyl-1-propanone, 2045
[175226-44-5]	1-(3-Hydroxy-1-naphthalenyl)-1-propanone, 1966
[175226-45-6]	1-(6-Hydroxy-1-naphthalenyl)-1-propanone, 1967
[175226-46-7]	1-(7-Hydroxy-2-naphthalenyl)-1-propanone, 1968
[175226-47-8]	1-(7-Hydroxy-1-naphthalenyl)-1-propanone, 1968
[175413-69-1]	1-[2-[2,6-Dihydroxy-3-(3-methyl-2-butenyl)benzoyl]-3-hydroxy-5-methylphenyl]-2-methyl-1-propanone, 2160
[176642-56-1]	1-(2-Hydroxy-5-methoxyphenyl)-2-methyl-1-propanone, 2027
[176843-49-5]	1-[5-[4,6-Bis(2,4-dimethylphenyl)-1,3,5-triazin-2-yl]-2,4-dihydroxyphenyl]-1-propanone, 1961
[177028-17-0]	1-(1,4-Dimethoxy-2-naphthalenyl)-2,2-dimethyl-1-propanone, 2103
[177028-19-2]	1-(4-Methoxy-2-naphthalenyl)-2,2-dimethyl-1-propanone, 2102
[178374-78-2]	1-(3,5-Difluoro-4-hydroxyphenyl)-1-propanone, 1745
[178375-14-9]	1-[3-Hydroxy-4-(phenylmethoxy)phenyl]-1-propanone, 1937
[178678-85-8]	1-(7-Hydroxy-2,2-diphenyl-1,3-benzodioxol-5-yl)-1-propanone, 2009
[178693-81-7]	1-(4-Butoxy-2-hydroxyphenyl)-1-propanone, 1898
[179113-58-7]	1-(3-Fluoro-5-hydroxyphenyl)-1-propanone, 1754
[179930-43-9]	1-(6-Methoxy-2-naphthalenyl)-2-methyl-1-propanone, 2065
[181236-19-1]	1-[6-Hydroxy-3-[3-(3-methoxypropoxy)propyl]-2-naphthalenyl]-1-propanone, 1985
[181260-67-3]	1-(4-Methoxy-3-methylphenyl)-2,2-dimethyl-1-propanone, 2092
[182184-03-8]	1-(4-Butoxy-2-hydroxy-5-nitrophenyl)-1-propanone, 1890
[183280-17-3]	1-(5-Fluoro-2-hydroxyphenyl)-2-methyl-1-propanone, 2015
[184963-79-9]	1-(4-Hydroxy-3,5-dimethoxyphenyl)-1-propanone (Ethyl ether), 1854
[184963-80-2]	1-(4-Hydroxy-3,5-dimethoxyphenyl)-1-propanone (Isobutyl ether), 1854
[184963-81-3]	1-(4-Ethyl-3,5-dimethoxyphenyl)-1-propanone, 1900
[184963-86-8]	1-(4-Hydroxy-3,5-dimethoxyphenyl)-1-propanone (Propyl ether), 1854
[185207-91-4]	1-[6-Methoxy-2-(methoxymethoxy)-3,4-dimethylphenyl]-1-propanone, 1916
[185207-93-6]	1-(2-Hydroxy-6-methoxy-3,4-dimethylphenyl)-1-propanone, 1875
[185413-93-8]	1-(2-Hydroxy-3-methyl-1-naphthalenyl)-1-propanone, 1972
[185413-94-9]	1-[2-Hydroxy-6-methoxy-3-methyl-5-(phenylmethoxy)-1-naphthalenyl]-1-propanone, 1986
[185413-95-0]	1-[2-Hydroxy-3-methyl-6,8-bis(phenylmethoxy)-1-naphthalenyl]-1-propanone, 1987
[185413-96-1]	N-[6-Hydroxy-7-methyl-5-(1-oxopropyl)-4-(phenylmethoxy)-2-naphthalenyl]acetamide, 1986
[185437-45-0]	1-(8-Hydroxy-5-quinazolinyl)-1-propanone, 1990
[186962-22-1]	1-[5-(1,1-Dimethylethyl)-2-methoxyphenyl]-2,2-dimethyl-1-propanone, 2097

[186962-23-2]	1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-2,2-dimethyl-1-propanone, 2095
[186962-24-3]	1-[4-(1,1-Dimethylethyl)-2-methoxyphenyl]-2,2-dimethyl-1-propanone, 2096
[186962-25-4]	1-[4-(1,1-Dimethylethyl)-2-hydroxyphenyl]-2,2-dimethyl-1-propanone, 2095
[187276-37-5]	1-[2-Hydroxy-3-(1-propenyl)phenyl]-1-propanone *(E)*, 1859
[188435-69-0]	1-(2,5-Difluoro-4-hydroxyphenyl)-1-propanone, 1745
[188527-69-7]	1-[4-Methoxy-2-(2-pyridinyl)phenyl]-1-propanone, 2005
[188579-52-4]	1-[2-Methoxy-6-(8,11-pentadecadiynyl)phenyl]-1-propanone, 1960
[188984-62-5]	1-[4-Methoxy-3-[(4-nitrophenyl)methyl]phenyl]-1-propanone, 1940
[190777-99-2]	1-[2-Hydroxy-6-[(2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl)oxy]phenyl]-1-propanone, 1958
[193693-93-5]	1-(3-Hydroxy-5-nitrophenyl)-1-propanone (Dysprosium salt), 1758
[193693-96-8]	1-(3-Hydroxy-5-nitrophenyl)-1-propanone, 1758
[194608-83-8]	1-[4-(3-Bromopropoxy)-2-hydroxy-3-propyl]-1-propanone, 1925
[194791-95-2]	1-[4-[3-[[2-Chloro-4-(1*H*-tetrazol-5-ylmethyl)phenyl]thio]propoxy]-2-hydroxy-3-propylphenyl]-1-propanone, 1957
[194791-97-4]	1-[4-[3-[[2-Chloro-4-(1*H*-tetrazol-5-ylmethyl)phenyl]thio]propoxy]-2-hydroxy-3-propylphenyl]-1-propanone (Oxime), 1957
[194791-98-5]	1-[4-[4-[[2-Chloro-4-(1*H*-tetrazol-5-ylmethyl)phenyl]thio]butoxy]-2-hydroxy-3-propylphenyl]-1-propanone, 1959
[194791-99-6]	1-[4-[4-[[2-Chloro-4-(1*H*-tetrazol-5-ylmethyl)phenyl]thio]butoxy]-2-hydroxy-3-propylphenyl]-1-propanone (Oxime), 1959
[194792-34-2]	1-[3-(Cyclopropylmethyl)-2,4-dihydroxyphenyl]-1-propanone, 1885
[194792-37-5]	1-[2-Hydroxy-4-(4-hydroxy-1-butynyl)-3-propylphenyl]-1-propanone, 1938
[194792-38-6]	1-[2-Hydroxy-4-(4-hydroxybutyl)-3-propylphenyl]-1-propanone, 1940
[194792-39-7]	1-[4-(4-Bromobutyl)-2-hydroxy-3-propylphenyl]-1-propanone, 1939
[194792-40-0]	1-[4-(3-Bromopropoxy)-2-hydroxy-3-(2-propenyl)phenyl]-1-propanone, 1923
[194792-41-1]	1-(4-Hydroxy-3-propylphenyl)-1-propanone, 1869
[194793-05-0]	1-[4-(4-Bromobutoxy)-2-hydroxy-3-propylphenyl]-1-propanone, 1939
[194854-84-7]	1-[4-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]-2-hydroxy-3-propylphenyl]-1-propanone, 1949
[195729-19-2]	1-(1-Ethyl-6-hydroxy-2-naphthalenyl)-1-propanone, 1977
[195729-50-1]	1-(1-Ethyl-6-methoxy-2-naphthalenyl)-1-propanone, 1979
[195729-54-5]	1-(6-Methoxy-1-propyl-2-naphthalenyl)-1-propanone, 1982
[195729-60-3]	1-(6-Hydroxy-1-propyl-2-naphthalenyl)-1-propanone, 1979

[231957-52-1]	N-(5-Cyano-2-pyridinyl)-N′-[(1*R*,2*R*)-2-[6-fluoro-2-hydroxy-3-(1-oxopropyl)phenyl]cyclopropyl]urea, 1950
[231957-54-3]	N-(5-Cyano-2-pyridinyl)-N′-[(1*S*,2*S*)-2-[6-fluoro-2-hydroxy-3-(1-oxopropyl)phenyl]cyclopropyl]urea, 1950
[231958-06-8]	1-(4-Fluoro-2-methoxyphenyl)-1-propanone, 1793
[241131-36-2]	1-[4-(β-D-Glucopyranosyloxy)-2,6-dihydroxy-3,5-dimethylphenyl]-2-methyl-1-propanone, 2054
[245052-19-1]	2,4,6-Trihydroxy-5-(2-methyl-1-oxopropyl)-1,3-benzenedicarboxaldehyde, 2159
[245407-09-4]	1-(2-Hydroxy-3-iodo-4-phenoxyphenyl)-1-propanone, 1918
[245407-10-7]	1,1′-(4-Hydroxy-5-iodo-6-phenoxy-1,3-phenylene)bis-1-propanone, 2116
[245407-11-8]	1-(2-Hydroxy-3-iodo-5-nitro-4-phenoxyphenyl)-1-propanone, 1917
[245407-13-0]	1-(5-Bromo-2-hydroxy-3-iodo-4-phenoxyphenyl)-1-propanone, 1917
[246041-90-7]	1-(3-Methoxy-4-nitrophenyl)-1-propanone, 1797
[247230-90-6]	N-(5-Chloro-2-pyridinyl)-N′-[(1*R*,2*R*)-2-[6-fluoro-2-hydroxy-3-(1-oxopropyl)phenyl]cyclopropyl]urea, 1946
[247230-91-7]	N-(5-Chloro-2-pyridinyl)-N′-[(1*S*,2*S*)-2-[6-fluoro-2-hydroxy-3-(1-oxopropyl)phenyl]cyclopropyl]urea, 1947
[247230-94-0]	1-(4-Fluoro-2-hydroxyphenyl)-1-propanone, 1755
[251463-58-8]	1-[2-(Acetyloxy)-4-hydroxyphenyl]-2-methyl-1-propanone, 2029
[256335-72-5]	1-(3-Hydroxy-4-nitroso-2-naphthalenyl)-1-propanone, 1963
[261928-44-3]	1-[3-[4-(Acetyloxy)-3-methyl-2-butenyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-propanone, 2053
[263010-96-4]	1-(4-Fluoro-2,6-dimethoxyphenyl)-2-methyl-1-propanone, 2031
[263010-97-5]	1-[2,6-Dimethoxy-4-(4-morpholinyl)phenyl]-2-methyl-1-propanone, 2052
[266310-09-2]	1-(2-Hydroxy-3-methoxyphenyl)-2-methyl-1-propanone, 2026
[267001-65-0]	2,2-Dimethyl-1-(2,4,5-trihydroxy-3,6-dimethylphenyl)-1-propanone, 2093
[267001-71-8]	1-(2,4-Dihydroxy-3,5-dimethylphenyl)-2-methyl-1-propanone, 2034
[267001-74-1]	1-(2,4-Dihydroxy-3,5-dimethylphenyl)-2,2-dimethyl-1-propanone, 2092
[267008-04-8]	1-(2-Ethyl-4,5-dihydroxyphenyl)-1-propanone, 1846
[267410-40-2]	1-(3,4,5-Trihydroxyphenyl)-1-propanone, 1775
[268234-16-8]	1-[2-Methoxy-5-(methoxymethoxy)phenyl]-1-propanone, 1878
[270084-45-2]	1-[2-Hydroxy-4-[(tetrahydro-2*H*-pyran-2-yl)oxy]phenyl]-1-propanone, 1907
[275803-01-5]	1-(1,4,5,8-Tetramethoxy-2-naphthalenyl)-1-propanone, 1982
[276690-11-0]	1-[2-Hydroxy-4,6-dimethoxy-3-(methoxymethoxy)phenyl]-1-propanone, 1904
[288401-09-2]	1-(4,5-Dichloro-2-hydroxyphenyl)-1-propanone, 1744

[404964-94-9]	1-(6,7-Dihydroxy-2-naphthalenyl)-2-methyl-1-propanone, 2064
[432547-82-5]	1-(4-Hydroxy-3-methoxyphenyl)-1-propanone (Tetra-O-acetyl-β-D-glucopyranoside), 1817
[438490-65-4]	1-[6-Hydroxy-3-(4-methoxyphenyl)-2-phenyl-5-benzofuranyl]-1-propanone, 2009
[438490-68-7]	1-[6-Hydroxy-3-(4-methylphenyl)-2-phenyl-5-benzofuranyl]-1-propanone, 2009
[449779-73-1]	1-(2,4-Dihydroxy-3,5-dipropylphenyl)-1-propanone, 1929
[449779-75-3]	1-(4-Hydroxy-3,5-dipropylphenyl)-1-propanone, 1928
[449779-85-5]	1-(2-Fluoro-4-hydroxy-3,5-dipropylphenyl)-1-propanone, 1926
[453518-19-9]	1-(5-Hydroxy-2-nitrophenyl)-1-propanone, 1759
[457628-03-4]	1-[2-Hydroxy-4-(2-propynyloxy)phenyl]-1-propanone, 1858
[457628-04-5]	1-(5-Hydroxy-2*H*-1-benzopyran-6-yl)-1-propanone, 1994
[459124-92-6]	1-[2-Hydroxy-5-(1-oxopropoxy)phenyl]-1-propanone, 1862
[473807-71-5]	1-(2,4-Dihydroxy-5-nitrophenyl)-1-propanone (Oxime), 1759
[475502-03-5]	2,2-Dimethyl-1-(2,2′,6,6′-tetramethoxy[1,1′-biphenyl]-3-yl)-1-propanone, 2099
[477904-75-9]	1-(1,4-Dimethoxy-2-naphthalenyl)-1-propanone, 1978
[479580-94-4]	1-(2-Hydroxy-4-phenoxyphenyl)-1-propanone, 1922
[488106-61-2]	1-(2-Chloro-4-methoxy-5-methylphenyl)-1-propanone, 1829
[501374-02-3]	1-(5-Methoxy[1,1′-biphenyl]-2-yl)-2,2-dimethyl-1-propanone, 2098
[501374-20-5]	1-(4′-Methoxy[1,1′-biphenyl]-2-yl)-2,2-dimethyl-1-propanone, 2098
[502924-41-6]	1-(5-Bromo-2-methoxyphenyl)-1-propanone, 1787
[502924-43-8]	1-(5-Benzoyl-2-methoxyphenyl)-1-propanone, 2138
[502924-47-2]	1-(2-Methoxy-5-phenoxyphenyl)-1-propanone, 1937
[502924-49-4]	1-(3-Hydroxy-5-methoxyphenyl)-1-propanone, 1814
[502924-51-8]	1-(3-Methoxy-5-phenoxyphenyl)-1-propanone, 1937
[507272-84-6]	1-(7-Methoxy-1-naphthalenyl)-1-propanone, 1975
[540495-19-0]	1-(5′-Hydroxy-2′-methyl-2-propyl[1,1′-biphenyl]-4-yl)-1-propanone, 1950
[540495-26-9]	1-(3-Ethyl-4-hydroxyphenyl)-1-propanone, 1833
[540495-32-7]	1-(2,2′-Diethyl-5′-hydroxy[1,1′-biphenyl]-4-yl)-1-propanone, 1950
[540495-37-2]	1-(2′-Ethyl-5′-hydroxy-2-propyl[1,1′-biphenyl]-4-yl)-1-propanone, 1952
[540495-43-0]	1-[2′-Ethyl-5′-hydroxy-2-(1-methylethyl)[1,1′-biphenyl]-4-yl]-1-propanone, 1952
[551002-14-3]	1-[5-Hydroxy-2-(4-hydroxyphenyl)-7-benzofuranyl]-1-propanone, 2007
[561046-07-9]	1-[4-Hydroxy-3-(3-methyl-2-butenyl)phenyl]-1-propanone, 1905
[568600-66-8]	1,1′-(4,4′-Dihydroxy[1,1′-biphenyl]-3,3′-diyl)bis-1-propanone polymer with 3-methoxy-1,2-benzenediamine, 2118
[574001-78-8]	1-(3-Hydroxy-2-naphthalenyl)-2,2-dimethyl-1-propanone, 2100

[574004-35-6]	1,1′-[(1*R*)-2,2′-Dihydroxy[1,1′-binaphthalene]-3,3′-diyl]bis[2,2-dimethyl-1-propanone, 2170
[612812-31-4]	1-(5-Chloro-2,4-dihydroxy-3-propylphenyl)-1-propanone, 1865
[658702-60-4]	1-[(2*R*,3*S*)-3,4-Dihydro-5,7-dihydroxy-2-methyl-3-(3-methyl-2-butenyl)-2-(4-methyl-3-pentenyl)-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone (+), 2082
[658702-62-6]	1-[3,4-Dihydro-5,7-dihydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2*H*-1-benzopyran-8-yl]-2-methyl-1-propanone (+), 2073
[682320-25-8]	1-(2-Methoxy-5-nitrophenyl)-1-propanone, 1796
[687184-53-7]	1-(4-Hydroxy-3-methyl-6-phenyl-2-benzofuranyl)-1-propanone, 2008
[687184-57-1]	1-(4-Hydroxy-3-methyl-6-phenyl-2-benzofuranyl)-2,2-dimethyl-1-propanone, 2103
[718613-15-1]	1-(2-Amino-5-hydroxyphenyl)-1-propanone-*3,3,3*-d_3, 1745
[722457-94-5]	2-Methyl-1-[2,4,6-trihydroxy-3-(3-hydroxy-3,7-dimethyl-6-octenyl)phenyl]-1-propanone (+), 2057
[722457-95-6]	1-[(4a*R*,9a*R*)-2,3,4,4a,9,9a-Hexahydro-6,8-dihydroxy-1,1,4a-trimethyl-1*H*-xanthen-7-yl]-2-methyl-1-propanone, 2075
[722457-96-7]	1-[3,4-Dihydro-5,7-dihydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2*H*-1-benzopyran-6-yl]-2-methyl-1-propanone (-), 2073
[761459-40-9]	1-(4-Hydroxy-2-methylphenyl)-2-methyl-1-propanone, 2025
[761459-41-0]	1-(4-Hydroxy-1-naphthalenyl)-2-methyl-1-propanone, 2063
[777067-72-8]	1-(3,4-Dichloro-2-hydroxyphenyl)-1-propanone, 1743
[777067-73-9]	1-(3′-Hydroxy[1,1′:2′,1″-terphenyl]-4′-yl)-1-propanone, 1953
[820990-87-2]	1-(2,4,6-Trihydroxyphenyl)-1-propanone (Compound with 4,4′-(1*E*)-1,2-ethenediylbis[pyridine]), 1775
[842121-75-9]	1-[2,3-Dihydro-6-hydroxy-2-(1-hydroxy-1-methylethyl)-7-benzofuranyl]-2-methyl-1-propanone, 2069
[847344-76-7]	1-(5-Ethyl-2-hydroxyphenyl)-2-methyl-1-propanone, 2031
[848734-12-3]	2-(4-Fluorophenyl)-5,7-dihydroxy-6-(1-oxopropyl)-4*H*-1-benzopyran-4-one, 2143
[851036-32-3]	1-[7-Methoxy-2,2-dimethyl-4-(1-methylethyl)-2*H*-1-benzopyran-6-yl]-1-propanone, 2008
[852290-70-1]	1-(4′-Methoxy[1,1′-biphenyl]-2-yl-*3,4,5,6*-d_4)-2,2-dimethyl-1-propanone, 2098
[852290-72-3]	1-(4′-Methoxy[1,1′-biphenyl]-2-yl-3-*d*)-2,2-dimethyl-1-propanone, 2098
[852612-18-1]	1-[2-Hydroxy-4-methoxy-6-[(1*E*)-2-[4-(methoxymethoxy)phenyl] ethenyl]-phenyl]-2-methyl-1-propanone, 2058
[853577-59-0]	1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-2-methyl-1-propanone, 2043
[859403-57-9]	1-(2,3-Dihydro-7-hydroxy-1,4-benzodioxin-6-yl)-1-propanone, 1991
[860152-45-0]	1-(3-Bromo-4,6-dihydroxy[1,1′-biphenyl]-2-yl)-1-propanone, 1917

[860152-81-1]	1-[4,6-Dihydroxy-2-(2-methoxyethyl)[1,1′-biphenyl]-3-yl]-2-methyl-1-propanone, 2055
[860152-82-5]	1-[4,6-Dihydroxy-2-(2-methoxyethyl)[1,1′-biphenyl]-3-yl]-1-propanone, 1947
[862666-40-8]	1-(2,3-Dihydroxyphenyl)-2-methyl-1-propanone, 2017
[864287-73-0]	1-(5′-Fluoro-2′-methoxy[1,1′-biphenyl]-4-yl)-1-propanone, 1933
[864866-61-5]	1-(2-Fluoro-4,6-dimethoxyphenyl)-1-propanone, 1831
[864866-62-6]	1-(2-Fluoro-4,6-dihydroxyphenyl)-1-propanone, 1755
[868266-10-8]	1-(4-Methoxy[1,1′-biphenyl]-3-yl)-2,2-dimethyl-1-propanone, 2098
[868266-11-9]	1-(4-Hydroxy[1,1′-biphenyl]-3-yl)-2,2-dimethyl-1-propanone, 2097
[868266-15-3]	1-(4-Methoxy[1,1′-biphenyl]-3-yl)-2-methyl-1-propanone, 2052
[868266-16-4]	1-(4-Hydroxy[1,1′-biphenyl]-3-yl)-2-methyl-1-propanone, 2050
[868606-10-4]	1-(5-Bromo-2-hydroxy-3-iodophenyl)-1-propanone, 1740
[868606-11-5]	1-(2-Hydroxy-3-iodo-5-methylphenyl)-1-propanone, 1794
[868634-82-6]	1-[4-(β-D-Glucopyranosyloxy)-2,6-dihydroxyphenyl]-2-methyl-1-propanone, 2051
[868731-80-0]	1-[2-(1,1-Dimethylethoxy)-6-hydroxyphenyl]-1-propanone, 1899
[868853-56-9]	1-[5-Hydroxy-2-(4-hydroxyphenyl)-7-benzoxazolyl]-1-propanone, 2006
[869562-75-4]	1-[2-Hydroxy-4-methoxy-5-(2-propenyl)phenyl]-1-propanone, 1885
[870456-80-7]	1-(4-Methoxyphenyl-2,6-d_2)-2-methyl-1-propanone, 2021
[870701-66-9]	1-(2,3,5-Trihydroxyphenyl)-1-propanone, 1773
[870701-67-0]	1-(2,3,6-Trihydroxyphenyl)-1-propanone, 1773
[872630-73-4]	1-[2-Methoxy-6-(2-pyridinyl)phenyl]-1-propanone, 2004
[872630-76-7]	1-[5-Methoxy-2-(2-pyridinyl)phenyl]-1-propanone, 2005
[873222-90-3]	1-(2,5-Dihydroxy-3,4-dipropylphenyl)-1-propanone, 1929
[874183-64-9]	1,1′-(2,2′-Dihydroxy[1,1′-binaphthalene]-6,6′-diyl)bis[2,2-dimethyl-1-propanone (racemic), 2170
[874187-24-3]	1,1′-(2,2′-Dihydroxy[1,1′-binaphthalene]-6,6′-diyl)bis[2,2-dimethyl-1-propanone (1*R*), 2170
[874187-25-4]	1,1′-(2,2′-Dihydroxy[1,1′-binaphthalene]-6,6′-diyl)bis[2,2-dimethyl-1-propanone (1*S*), 2170
[878555-18-1]	2,2-Dimethyl-1-(3′,4′,5′-trimethoxy[1,1′-biphenyl]-2-yl)-1-propanone, 2099
[879339-62-5]	1-(3-Fluoro-2-hydroxyphenyl)-2-methyl-1-propanone, 2014
[879339-65-8]	1-(3-Fluoro-4-hydroxyphenyl)-2-methyl-1-propanone, 2014
[879339-67-0]	1-(3-Fluoro-4-methoxyphenyl)-2-methyl-1-propanone, 2023
[879339-86-3]	1-(3-Fluoro-2-hydroxyphenyl)-1-propanone, 1754
[879339-88-5]	1-(3-Fluoro-2-methoxyphenyl)-1-propanone, 1793
[879420-45-8]	4-(1-Acetoxypropyl)-5,7-dihydroxy-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxopropyl)-2*H*-1-benzopyran-2-one (-), 2080
[880134-95-2]	1,1′-[Oxybis(4-hydroxy-3,1-phenylene)]bis-1-propanone, 2118

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Common Abbreviations

Volume 1

Common abbreviations used in the dictionary for organic chemistry

Å	Angström units
b.p.	Boiling point (for example, b.p.$_{0.1}$ 100° means boils at 100° if the pressure is 0.1 mm Hg)
(d)	Decomposition
20°	20 degrees Celsius
d	Density (for example, d^{20} specific gravity at 20°C referred to water at 4°C)
DDQ	2,3-Dichloro-5,6-dicyanobenzoquinone
EPR	Electron paramagnetic resonance
HMPT	Hexamethylphosphoric Triamide
^{13}C NMR	Nuclear magnetic resonance relative to carbon 13
(E)	Geometric stereodescriptor used for compounds having achiral elements resulting from double bonds where the groups of highest priority are on the opposite sides of the vertical reference plane
^{19}F NMR	Nuclear magnetic resonance relative to fluorine 19
GC	Gas chromatography
GC-MS	Gas chromatography–mass spectrometry
GLC	Gas–liquid chromatography
h	Hour
^{1}H NMR	Nuclear magnetic resonance relative to proton
HPLC	High pressure liquid chromatography
IR	Infrared (spectra)
iso-	Aliphatic hydrocarbon having two methyl groups on the terminal carbon atom of the chain (for example, isoamyl $(CH_3)_2CH\text{-}CH_2\text{-}CH_2\text{-})$
LDA	lithium diisopropylamide
m-	Meta-
M	Molar (concentration)
min	Minute

mol	Molecule
mol.wt.	Molecular weight
m.p.	Melting point
MS	Mass spectra
n-	Normal, as n-butyl
N	Normal (equivalents per liter, as applied to concentration)
NA	Not available
N.B.:	Nota bene
n_D^{20}	Index of refraction (n_D^{20} for 20°C and sodium light)
o-	Ortho-
p-	Para-
Pa	pascal
Pd/C	Palladium on charcoal
pK_a	Log of the reciprocal of the dissociation constant, 1/log K_a
Rh/C	Rhodium on charcoal
r.t.	Room temperature
Sadtler	Sadtler Research Laboratories, Philadelphia (USA)
SDS	Sodium dodecyl sulfate
sec-	Secondary
SM	Starting material
tert-	Tertiary-
TFAA	Trifluoroacetic anhydride
TFMS	Trifluoromethanesulfonic acid
TLC	Thin layer chromatography
UV	Ultraviolet (spectra)
Vol.	Volume
(Z)	Opposite of *(E)*

Volume 2

Common abbreviations used in the dictionary for organic chemistry

Å	Angström units
$(\alpha)_D^{20}$	Specific optical rotation at 20°C for D (sodium) line
b.p.	Boiling point (for example, b.p.$_{0.1}$ 100° means boils at 100° if the pressure is 0.1 mm Hg)
d	Density (for example, d_4^{20} specific gravity at 20°C referred to water at 4°C)
20°	20 degrees Celsius
DEAD	Diethyl azodicarboxylate
dl	Racemic
DME	1,2-Dimethoxyethane (glyme)
DMF	Dimethylformamide
DMSO	Dimethyl sulfoxide

EIMS	Electron impact mass spectra
GC	Gas chromatography
GLC	Gas liquid chromatography
HMPA	Hexamethylphosphoramide (hexamethylphosphoric triamide),
HMPT	Hexamethylphosphorous triamide
HPLC	High performance (pressure, power) liquid chromatography
^{13}C NMR	Nuclear magnetic resonance relative to carbon 13
(E)	Geometric stereodescriptor used for compounds having achiral elements resulting from double bonds where the groups of highest priority are on the opposite sides of the vertical reference plane
^{19}F NMR	Nuclear magnetic resonance relative to fluorine 19
h	Hour
HREIMS	High resolution electron impact mass spectra
HRMS	High resolution mass spectra
^{1}H NMR	Nuclear magnetic resonance relative to proton
IR	Infrared spectra
iso-	Aliphatic hydrocarbon having two methyl groups on the terminal carbon atom of the chain (for example, isoamyl $(CH_3)_2CH$-CH_2-CH_2-)
m-	Meta-
M	Molar (concentration)
min	Minute
mol	Molecule
mol. equiv.	Molecular equivalent
mol.wt.	Molecular weight
m.p.	Melting point
MS	Mass spectra
n-	Normal (as n-butyl)
N	Normal (equivalents per liter, as applied to concentration)
NA	Not available
N.B.:	Nota bene
NBS	N-Bromosuccinimide
n_D^{20} =	Index of refraction (n_D^{20} for 20°C and sodium light)
o-	Ortho-
p-	Para-
Pd/C	Palladium on charcoal
$PdCl_2/C$	Palladium chloride on charcoal
PdO/C	Palladium oxide on charcoal
pH	Log of reciprocal of hydrogen ion concentration
pK_a	Log of the reciprocal of the dissociation constant, 1/log K_a
Pt/C	Platinum on charcoal
r.t.	Room temperature
sec-	Secondary (as sec-butyl)
SM	Starting material
TBAI	Tetrabutylammonium iodide

tert-	Tertiary (as tert-butyl)
TFE	2,2,2-Trifluoroethanol
THF	Tetrahydrofuran
TLC	Thin layer chromatography
UV	Ultraviolet spectra
(Z)	Opposite of *(E)*

Volume 3

Å	Angström units
$(\alpha)_D^{20}$	Specific optical rotation at 20°C for D (sodium) line
ART	2-Amino-1-(3,4-dihydroxyphenyl)ethanone
b.p.	Boiling point (for example, b.p.$_{0.1}$ 100° means boils at 100° if the pressure is 0.1 mm Hg)
CAN	Ceric ammonium nitrate
m-CPBA	m-Chloroperoxybenzoic acid
20°	20 degrees Celsius
d	Density (for example, d^{20} specific gravity at 20°C referred to water at 4°C)
(d)	with decomposition
DEAD	Diethyl azodicarboxylate
dl	Racemic
DME	1,2-Dimethoxyethane (glyme)
DMF	Dimethylformamide
DMSO	Dimethyl sulfoxide
DMPU	1,3-Dimethyl-3,4,5,6-tetrahydro-2-*[1H]*-pyrimidinone
DOPKET	1-(3,4-Dihydroxyphenyl)-2-hydroxyethanone
equiv	Equivalent
GC	Gas chromatography
HMPA	Hexamethylphosphoramide (hexamethylphosphoric triamide)
HMPT	Hexamethylphosphorous triamide
HPLC	High performance (pressure, power) liquid chromatography
^{13}C NMR	Nuclear magnetic resonance relative to carbon 13
(E)	Geometric stereodescriptor used for compounds having achiral elements resulting from double bonds where the groups of highest priority are on the opposite sides of the vertical reference plane
^{19}F NMR	Nuclear magnetic resonance relative to fluorine 19
h	Hour
HR-MS	High resolution mass spectra
^{1}H NMR	Nuclear magnetic resonance relative to proton
IR	Infrared spectra
iso-	Aliphatic hydrocarbon having two methyl groups on the terminal carbon atom of the chain (for example, isoamyl $(CH_3)_2CH\text{-}CH_2\text{-}CH_2\text{-}$)

LCEC	Liquid chromatography with electrochemical detection
LDA	Lithium diisopropylamide
m-	Meta-
M	Molar (concentration)
min	Minute
mol	Molecule
mol.wt.	Molecular weight
MOM-	Methoxymethyl-
m.p.	Melting point
MS	Mass spectra
n-	Normal (as n-butyl)
N	Normal (equivalents per litre, as applied to concentration)
N.B.	Nota bene
NBS	N-Bromosuccinimide
n_D^{20}	Index of refraction (n_D^{20} for 20°C and sodium light)
N,N′-MBA	N,N′-Methylenebisacrylamide
NPFA	Nitropentafluoroacetone
o-	Ortho-
p-	Para-
Pd/C	Palladium on charcoal
$PdCl_2/C$	Palladium chloride on charcoal
PdO/C	Palladium oxide on charcoal
pH	Log of reciprocal of hydrogen ion concentration
pK_A	Log of the reciprocal of the dissociation constant, $1/\log K_A$
pK_B	Log of the reciprocal of the dissociation constant, $1/\log K_B$
Ψ	pseudo
psi	per square inch
Pt/C	Platinum on charcoal
r.t.	Room temperature
sec-	Secondary (as sec-butyl)
SM	Starting material
TBAI	Tetrabutylammonium iodide
TEAF	Triethylammonium formate
tert-	Tertiary (as tert-butyl)
TFA	Trifluoroacetic acid
TFE	2,2,2-Trifluoroethanol
THF	Tetrahydrofuran
TLC	Thin layer chromatography
TMS	Tetramethylsilane
UV	Ultraviolet spectra
w/w	per cent “weight in weight” expresses the number of grams of an active constituent in 100 grams of solution or mixture
(Z)	Opposite of *(E)*

Volume 4

Common abbreviations used in the dictionary for organic chemistry

$(\alpha)_D^{20}$	Specific optical rotation at 20°C for D (sodium) line
atm	atmosphere
b.p.	Boiling point (for example, b.p.$_{0.1}$ 100° means boils at 100° if the pressure is 0.1 mm Hg)
BF_3	Boron trifluoride
BF_3-OBu_2	Boron trifluoride-n-Butyl ether complex
BF_3-OAc	Boron trifluoride-acetic anhydride complex
CCL	Candida cylindracea lipase
^{13}C NMR	Nuclear magnetic resonance relative to carbon 13
d	Density (for example, d_4^{20} specific gravity at 20°C referred to water at 4°C)
(d)	with decomposition
20°	20 degrees Celsius
DDQ	2,3-Dichloro-5,6-dicyano-1,4-benzoquinone
DMA	Dimethylacetamide
DMF	Dimethylformamide
DMSO	Dimethyl sulfoxide
3D QSAR	Three-dimensional quantitative structure-activity relationship
(E)	Geometric stereodescriptor used for compounds having achiral elements resulting from double bonds where the groups of highest priority are on the opposite sides of the vertical reference plane
equiv	Equivalent
ESR	Electron spin resonance
EtOH	Ethyl alcohol
^{19}F NMR	Nuclear magnetic resonance relative to fluorine 19
GC	Gas chromatography
GLC	Gas liquid chromatography
h	Hour
HMG-coA	3-Hydroxy-3-methylglutaryl co-enzyme A reductase
^{1}H NMR	Nuclear magnetic resonance relative to proton
$HNTf_2$	Bis(trifluoromethanesulfonyl)amide
HPLC	High performance (pressure, power) liquid chromatography
HRMS	High resolution mass spectra
IR	Infrared spectra
iso-	Aliphatic hydrocarbon having two methyl groups on the terminal carbon atom of the chain (for example, isoamyl $(CH_3)_2CH$-CH_2-CH_2-)
LD_{50}	Median lethal dose, the quantity of a chemical that is estimated to be fatal to 50% of the organisms tested
LDA	Lithium diisopropylamide
m-	Meta-

μCi	Microcurie
min	Minute
mol	Molecule
mol.	equiv. Molecular equivalent
mol.wt.	Molecular weight
m.p.	Melting point
M	Molar(concentration)
MAO	Monoamine oxidase
MNDO	Modified Neglect of Diatomic Overlap
MS	Mass spectra
n-	Normal (as n-butyl)
N	Normal (equivalents per liter, as applied to concentration)
N.B.:	Nota bene
n_D^{20} =	Index of refraction (n_D^{20} for 20°C and sodium light)
nm	nanometre
NMDA	N-methyl-D-aspartic acid
o-	Ortho-
^{17}O	NMR Nuclear magnetic resonance relative to oxygen 17
p-	Para-
PCC	pyridinium chlorochromate
Pd/C	Palladium on charcoal
pH	Log of reciprocal of hydrogen ion concentration
PIDA	Phenyliodonium diacetate
pK_a	Log of the reciprocal of the dissociation constant, 1/log K_a
PPL	Porcine pancreas lipase
r.t.	Room temperature
sec-	Secondary (as sec-butyl)
SM	Starting material
tert-	Tertiary (as tert-butyl)
TfOH	Trifluoromethanesulfonic acid (Triflic acid)
THF	Tetrahydrofuran
TLC	Thin layer chromatography
UV	Ultraviolet spectra
VPS	Valerophenone synthase
w/w	per cent "weight in weight" expresses the number of grams of an active constituent in 100 grams of solution or mixture
(Z)	Opposite of *(E)*