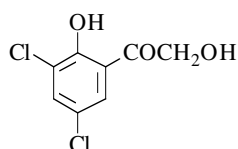


**Chapter 6. Compounds derived from hydroxyacetic acids****1-(3,5-Dichloro-2-hydroxyphenyl)-2-hydroxyethanone**

[58483-53-7]

C<sub>8</sub>H<sub>6</sub>Cl<sub>2</sub>O<sub>3</sub>

mol.wt. 221.04



Synthesis

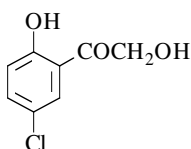
-Refer to: [268] (Romanian patent).

**1-(5-Chloro-2-hydroxyphenyl)-2-hydroxyethanone**

[52728-05-9]

C<sub>8</sub>H<sub>7</sub>ClO<sub>3</sub>

mol.wt. 186.59



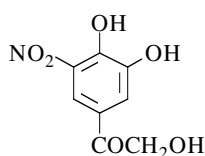
Synthesis

-Obtained by oxidative rearrangement of 5-chloro-2-hydroxy- $\alpha$ -bromoacetophenone in moist DMSO for 16 h at 20° (56%) [416].m.p. 98-99° [416]; <sup>1</sup>H NMR [416].**1-(3,4-Dihydroxy-5-nitrophenyl)-2-hydroxyethanone**

[134612-56-9]

C<sub>8</sub>H<sub>7</sub>NO<sub>6</sub>

mol.wt. 213.15



Synthesis

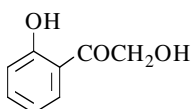
-Refer to: [163].

**2-Hydroxy-1-(2-hydroxyphenyl)ethanone**

[17375-96-1]

C<sub>8</sub>H<sub>8</sub>O<sub>3</sub>

mol.wt. 152.15



Syntheses

-Preparation by treatment of 2-hydroxy- $\alpha$ -bromoacetophenone with refluxing water for 16 h (88%) [145].  
 -Also obtained by hydrolysis of 2-hydroxy- $\alpha$ -(benzoyloxy)acetophenone with 50% aqueous potassium hydroxide in

refluxing ethanol for 34 h (73%) [416].

-Also obtained by oxidative rearrangement of 2-hydroxy- $\alpha$ -bromoacetophenone in moist DMSO for 16 h at 20° (31%) [416].

-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 [658].

-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  $-40^{\circ}$  for 1 h, then the temperature was slowly (1 h) raised to r.t. and stirring was continued for 30 min (25%) [1033].
- 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%) [271].
  - Also obtained from  $\alpha$ -chloro-o-hydroxyacetophenone by hydrolysis with boiling water for 15-20 h (20%) [1524].
  - Also refer to: [23] [24] [341] [390] [524] [823] [847].

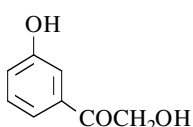
m.p.  $66-67^{\circ}$  [658],  $65^{\circ}$  [271] [1524],  $64-65^{\circ}$  [145] [416] [1033];  
 $^1\text{H}$  NMR [271] [416] [658], IR [271], UV [271].

### 2-Hydroxy-1-(3-hydroxyphenyl)ethanone

[131341-58-7]

 $\text{C}_8\text{H}_8\text{O}_3$ 

mol.wt. 152.15



Synthesis

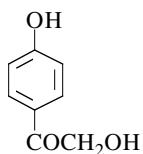
-Refer to: [607].

### 2-Hydroxy-1-(4-hydroxyphenyl)ethanone

[5706-85-4]

 $\text{C}_8\text{H}_8\text{O}_3$ 

mol.wt. 152.15



Syntheses

- Preparation by reaction of p-hydroxy- $\alpha$ -bromoacetophenone with formic acid in the presence of DBU, \*in benzene at  $0^{\circ}$ , followed by saponification of the intermediate formate ester with sodium hydroxide in methanol (99%) [1139];
- \*in methylene chloride, with the same treatment (49%) [560].
- Preparation by demethylation of  $\alpha$ -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  $\alpha$ -chloro-p-methoxyacetophenone with potassium acetate in ethanol [279].
- Preparation by action of boron trifluoride etherate with p-hydroxyphenyl diazomethyl ketone (SM1) in nitromethane under nitrogen at  $22^{\circ}$  for 15 min (81%). SM1, preparation given, melted at  $145-150^{\circ}$  (d) [143].
- Preparation from  $\alpha$ -acetoxy-4-hydroxyacetophenone (m.p.  $133^{\circ}$ ), \*by heating with 16% aqueous sodium hydroxide for 15 min on a steam bath (quantitative yield) [1238];
- \*in methanolic solution by treatment with 0.5 N aqueous sodium hydroxide at r.t. for 15 min (67%) [1577].
- Preparation by adding excess of concentrated hydrochloric acid to a warm concentrated aqueous solution of the potassium salt and cooling the solution [902].
- Preparation by treatment of the sodium salt with aqueous hydrochloric acid [1445].
- 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.  $109-110^{\circ}$ ) after treatment in dioxane with 2 N sulfuric acid at r.t. for 20 min, then at  $40^{\circ}$  until no more nitrogen evolved [931].
- Also obtained by condensation of glyoxal with phenol, \*in the presence of butylamine at  $33^{\circ}$  for 3 h (29%) [1039];
- \*in the presence of aqueous sodium hydroxide at  $33^{\circ}$  for 6 h (25%) [948];
- \*in the presence of hydrogen chloride at  $80^{\circ}$  for 4 h (< 5%) [947].

- 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° [504].
  - 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 [1387].
  - Also obtained by peroxidatic degradation of 7,4'-dihydroxyflavanone or 7,4'-dihydroxy-3'-methoxyflavanone [1159].
  - Also refer to: [823] [1038].
- N.B.:** Na salt [1238] [1445], K salt [902].

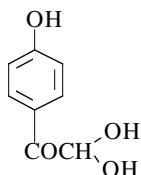
m.p. 177-178° [279] [902], 173-174° [931], 173° [1577], 170-177° [504],  
170-172° [1445], 170-171° [143], 165-167° [560] [1139];  
<sup>1</sup>H NMR [143] [1139], IR [143] [1139], MS [1139].

### 2,2-Dihydroxy-1-(4-hydroxyphenyl)ethanone

[197447-05-5]

C<sub>8</sub>H<sub>8</sub>O<sub>4</sub>

mol.wt. 168.15



#### Syntheses

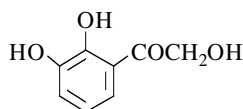
- Obtained by oxidation of p-hydroxyacetophenone with selenium oxide [1560].
- Also refer to: [296] (compound 1d) and [1521] [1522].

### 1-(2,3-Dihydroxyphenyl)-2-hydroxyethanone

[58483-49-1]

C<sub>8</sub>H<sub>8</sub>O<sub>4</sub>

mol.wt. 168.15



#### Synthesis

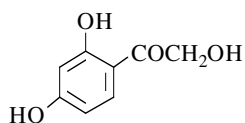
- Refer to: [851].

### 1-(2,4-Dihydroxyphenyl)-2-hydroxyethanone (*Fisetol*)

[487-47-8]

C<sub>8</sub>H<sub>8</sub>O<sub>4</sub>

mol.wt. 168.15



#### Syntheses

- Obtained by hydrolysis of α-acetoxyresacetophenone (m.p. 164°5) [287],  
\*with 5% aqueous sodium carbonate on a steam bath for 3 h (35%) [287];  
\*with 10% aqueous sodium hydroxide for 2 h at r.t. (38%) [287].
- Also obtained by hydrolysis of 2,4,α-triacetoxyacetophenone (m.p. 94°) [287],  
\*with 2 N sodium hydroxide [287];  
\*with 5 N methanolic ammonia for 8 days in the cold [1090].
- Also obtained by demethylation of α-methoxyresacetophenone with 40% hydrobromic acid for 3 h on a boiling water bath (16%) [592].
- Also obtained by treatment of α-[(methoxycarbonyl)oxy]resacetophenone (m.p. 157-158°) or

- $\alpha$ -[(ethoxycarbonyl)oxy]resacetophenone (m.p. 107°) with 2 N sodium hydroxide for 2 h at r.t. [1380].
- Also obtained by reaction of hydroxyacetonitrile with resorcinol [803], (41%) (Hoesch reaction) [768].
  - 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%) [271].
  - Also obtained by treatment of *Fisetin* (3,7,3',4'-tetrahydroxyflavone) — m.p. 330° (d) — with boiling ethanolic potassium hydroxide [637].
  - Also refer to: [303] [593] [691] [1028] [1371].

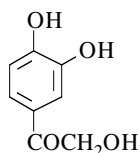
m.p. 191° [803], 189° [287] [768] [1090] [1380], 187-188° [592], 185-186° [271];  
<sup>1</sup>H NMR [271], IR [271], UV [271] [592] [1574].

### 1-(3,4-Dihydroxyphenyl)-2-hydroxyethanone (*DOPKET*)

[29477-54-1]

C<sub>8</sub>H<sub>8</sub>O<sub>4</sub>

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- $\alpha$ -chloroacetophenone with potassium acetate in refluxing acetic anhydride for 15 min (quantitative yield) [1510].
- Also obtained by heating N-acetyldopamine with 1 N hydrochloric acid [1031].
- 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 [48].
- 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* [1290].

#### Isolation from natural sources

- From sclerotization of the adult cuticle (*Leucophaea maderae*) [354].
- Also obtained by mild acid hydrolysis of sclerotized cuticles from locusts (*Schistocerca gregaria*) and beetles (*Pachynoda sinuata*) [45].
- From acid hydrolysates of insect hard cuticle [44].
- 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*) [46].
- By acid hydrolysis from exuviae of last instar larvae of the cicada *Tibicen pruinosus* [140].
- From the seed coat tamarind (*Tamarindus indica* L.) [1472].
- From the skins of tamarind seeds [1548].
- From mild acid hydrolysates of tanning pharate pupae cuticle from *Manduca sexta* [1120].
- 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* [1486].
- 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*) [91].
- 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*) [91];
  - \***Hemiptera** (*Nezara viridula*) [91];
  - \***Lepidoptera** (*Papilio aegeus* and *Antheraea helena*) [91];
  - \***Coleoptera** (*Anthrenus australis*) [91].
- in acidic extracts of insect cuticles (preparia) in refluxing 1 N hydrochloric acid for 1.5 h, i. e.:

\***Diptera** (*Lucilia cuprina*) [91].

-in acid extracts of insect cuticles (prepal cuticles):

\***Lepidoptera** (*Papilio aegaeus*) [91].

-Also refer to: [326] [645] [890] [1411] [1470] [1471].

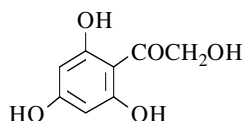
m.p. 195° [1510]; UV [44] [45], MS [44] [45] [1242];  
electrophoresis [354]; column chromatography [45];  
TLC [44] [45] [354]; LCEC chromatography [354]; GC [91]; HPLC [1120] [1290];  
HPLC-MS [1031].

## 2-Hydroxy-1-(2,4,6-trihydroxyphenyl)ethanone

[55313-03-6]

C<sub>8</sub>H<sub>8</sub>O<sub>5</sub>

mol.wt. 184.15



### Syntheses

-Preparation according to Hoesch reaction from  
phloroglucinol,  
\*with acetoxyacetonitrile (81%) [291];  
\*with hydroxyacetonitrile (63%) [1574].

-Also obtained from dihydrokaempferol (3,5,7,4'-tetrahydroxyflavanone) (*Aromadendrin*) by basic hydrolysis and subsequent oxidation [1091].

-Dihydrokaempferol yields kaempferol (3,5,7,4'-tetrahydroxyflavone) with peroxides and alkaline conditions; subsequent thermolysis produces the titled ketone [1091].

-Quercetin (3,5,7,3',4'-pentahydroxyflavone) yields the same product under alkaline thermolysis (80°) [1091].

-Also refer to: [800].

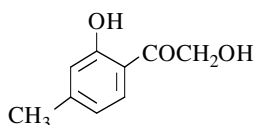
m.p. 226° [1574], 224° [291]; UV [1574]; GC-MS [1091].

## 2-Hydroxy-1-(2-hydroxy-4-methylphenyl)ethanone

[55960-03-7]

C<sub>9</sub>H<sub>10</sub>O<sub>3</sub>

mol.wt. 166.18



### Syntheses

-Obtained by a selective one-step synthesis from 3-methyl-phenoxy magnesium bromide (1 mol) and anhydrous monomeric glyoxal (1 mol) in boiling benzene for 20 h (48%) [271].

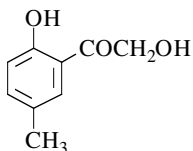
-Also refer to: [851].

m.p. 68-69° [271]; <sup>1</sup>H NMR [271], IR [271], UV [271].

## 2-Hydroxy-1-(2-hydroxy-5-methylphenyl)ethanone

C<sub>9</sub>H<sub>10</sub>O<sub>3</sub>

mol.wt. 166.18



### Synthesis

-Obtained by hydrolysis of 2-hydroxy-5-methyl-α-chloro-acetophenone with boiling water for 15-20 h (40%) [1524].

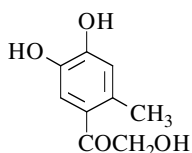
m.p. 76-77° [1524].

**1-(4,5-Dihydroxy-2-methylphenyl)-2-hydroxyethanone**

[61407-16-7]

C<sub>9</sub>H<sub>10</sub>O<sub>4</sub>

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 [1190].

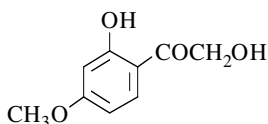
amorphous [1190]; <sup>1</sup>H NMR [1190], MS [1190].

**2-Hydroxy-1-(2-hydroxy-4-methoxyphenyl)ethanone**

[55960-07-1]

C<sub>9</sub>H<sub>10</sub>O<sub>4</sub>

mol.wt. 182.18

**Syntheses**

-Preparation from 2-hydroxy-4-methoxy- $\alpha$ -bromoacetophenone with refluxing water for 20 h (76%) [24].  
-Also obtained from fisetol 4-monomethyl ether diacetate (m.p. 86°) by heating with ethanolic potassium hydroxide [1438].

-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%) [271].  
-Also obtained by action of 40% aqueous hydrobromic acid with 2-hydroxy-4, $\alpha$ -dimethoxyacetophenone in acetic acid on a boiling water bath for 3 h (22%) [592].  
-Also refer to: [23] [303] [561] [593] [1371] [1382].

m.p. 128° [1438], 127° [592], 126-128° [24], 126-127° [271];

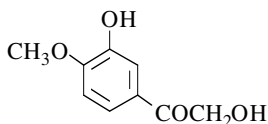
<sup>1</sup>H NMR [24] [271], IR [24] [271], UV [271] [592], MS [24].

**2-Hydroxy-1-(3-hydroxy-4-methoxyphenyl)ethanone**

[90536-46-2]

C<sub>9</sub>H<sub>10</sub>O<sub>4</sub>

mol.wt. 182.18

**Syntheses**

-Preparation by total hydrolysis of 4-methoxy-3, $\alpha$ -diacetoxyacetophenone (m.p. 82-83°) [478], (m.p. 81-82°) [454] in methanol with concentrated hydrochloric acid,  
\*for 4-5 h at r.t. (75%) [478];  
\*for 30 min at reflux (56%) [454].

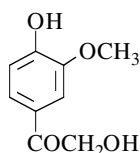
m.p. 177-178° [454], 176-177° [478]; IR [454], UV [454].

**2-Hydroxy-1-(4-hydroxy-3-methoxyphenyl)ethanone**

[18256-48-9]

C<sub>9</sub>H<sub>10</sub>O<sub>4</sub>

mol.wt. 182.18

**Syntheses**

-Preparation by total hydrolysis of 3-methoxy-4, $\alpha$ -diacetoxyacetophenone (m.p. 77-78°) [478], (m.p. 75-76°) [454] in methanol with concentrated hydrochloric acid,  
\*for 4-5 h at r.t. (78%) [478], (25%) [1124];  
\*during 14 h at 20°, then for 30 min at reflux (67%) [454].

- 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 [324].
- Also obtained by photorelease of  $\gamma$ -aminobutyric acid from 2-(4-hydroxy-3-methoxyphenyl)-2-oxoethyl  $\gamma$ -aminobutyrate [284043-11-4] with either 300 or 350 nm lamps in water or in deuterium oxide [324].
- N.B.:** Details of the synthesis and  $^1\text{H}$  NMR,  $^{13}\text{C}$  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 [324].
- Also obtained by treatment of 2-(acetoxy)-1-(4-hydroxy-3-methoxyphenyl)ethanone,
  - \*with boiling aqueous barium carbonate for 2 h [1198];
  - \*with 16% aqueous sodium hydroxide on the steam bath. The obtained sodium salt was treated with 2 N acetic acid (62%) [909].
- N.B.:** Na salt sesquihydrate (70%) [909].
- Also refer to: [1101].

## Isolation from natural sources

- From cell cultures of *Solanum khasianum* (Solanaceae) [1043] [1044].
- N.B.:** Microsomal preparations from heterotropic cell cultures of *Solanum khasianum* catalyse the hydroxylation of the  $\alpha$ -methyl group of acetovanillone. The reaction requires both oxygen and NADPH [1043].
- From the Namibian shrub *Salsola tuberculiformis* [1415].
- From the suprarenal capsules [454].
- Also refer to: [1386].

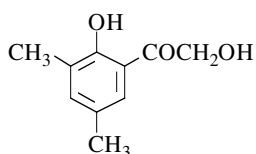
m.p. 160-161° [454] [1124], 159-160° [478], 158-160° (anhydrous) [909];  
 $^1\text{H}$  NMR [1044] [1124],  $^{13}\text{C}$  NMR [1044], IR [454] [1124],  
 UV [454] [1044], MS [1044] [1124] [1386];  
 fluorescence spectroscopy [938]; HPLC [1044]; GC/MS [1044] [1386].

**2-Hydroxy-1-(2-hydroxy-3,5-dimethylphenyl)ethanone**

[55960-05-9]

 $\text{C}_{10}\text{H}_{12}\text{O}_3$ 

mol.wt. 180.20



## Synthesis

-Obtained by a selective one-step synthesis for 2,4-dimethylphenoxymagnesium bromide (1 mol) and anhydrous monomeric glyoxal (1 mol) in boiling benzene for 20 h (35%) [271].

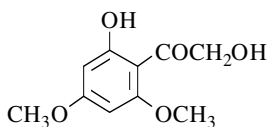
m.p. 100-101° [271];  
 $^1\text{H}$  NMR [271], IR [271], UV [271].

**2-Hydroxy-1-(2-hydroxy-4,6-dimethoxyphenyl)ethanone**

[83768-75-6]

 $\text{C}_{10}\text{H}_{12}\text{O}_5$ 

mol.wt. 212.20



## Syntheses

-Preparation by hydrolysis of 2-(2-hydroxy-4,6-dimethoxyphenyl)-2-oxoethyl benzoate in pyridine with aqueous sodium hydroxide under nitrogen atmosphere at r.t. for 1 h (80%) [1085].

- Also obtained by degradation of 2-[2-(2-hydroxy-4,6-dimethoxyphenyl)-2-oxoethoxy]-2-methylpropionic acid in refluxing mixture of concentrated hydrochloric acid/methanol (1 vol/5 vol) for 1 h (42%) [61].
- Also obtained by action of 40% hydrobromic acid with 2-hydroxy- $\alpha$ ,4,6-trimethoxyacetophenone in acetic acid by heating on a boiling water bath for 3 h (32%) [592].
- Also refer to: [593].

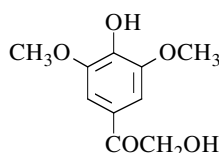
m.p. 140-142° [61], 139-140° [592], 131-132° [1085]; TLC [1085];  
<sup>1</sup>H NMR [61] [1085], IR [1085], UV [592], MS [1085].

## 2-Hydroxy-1-(4-hydroxy-3,5-dimethoxyphenyl)ethanone (*Danielone*)

[90426-22-5]

C<sub>10</sub>H<sub>12</sub>O<sub>5</sub>

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%) [935].
  - Obtained by hydrolysis of its diacetate (SM) with 5% hydrochloric acid in 70% dilute ethanol at 80° for 1.5 h [1378]. SM was prepared according to [757].
  - 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 [324].
  - Also obtained by photorelease of  $\gamma$ -aminobutyric acid from 2-(4-hydroxy-3,5-dimethoxyphenyl)-2-oxoethyl  $\gamma$ -aminobutyrate [284043-12-5] with either 300 or 350 nm lamps, in water or in deuterium oxide [324].
- N.B.:** Details of the synthesis and <sup>1</sup>H NMR, <sup>13</sup>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 [324].
- Also obtained from 1,2-bis(4-hydroxy-3,5-dimethoxyphenyl)-propane-1,3-diol, a  $\beta$ -1-lignin substructure model compound, by degradation with laccase of *Coriolus versicolor* (Fr.) Quel. [775].
  - Also refer to: [1116] [1379] [1464].

### Isolation from natural sources

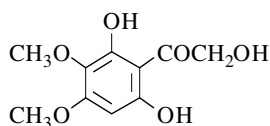
- From *Nicotiana tabacum* [1378] [1396] and *Atropa belladonna* root cultures [1378].
- From cell. suspension cultures of *Hyoscyamus albus* [1004].
- From *Carica papaya* fruit slices (Caricaceae) [440].
- Isolated as virulence gene inducing compounds of *Agrobacterium* from the hairy root cultures of belladonna [1377].
- Also refer to: [1386].

m.p. 145° [440], 109-110° [1378]. One of the reported melting points is obviously wrong.  
 TLC [440]; GC/MS [1386] [1396];  
<sup>1</sup>H NMR [440] [1378], <sup>13</sup>C NMR [440] [1378], IR [440] [935],  
 UV [440] [1378] [1396], MS [440] [935] [1378] [1386] [1396].



**1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-2-hydroxyethanone** (*Methyldegeranylmelicopol*)C<sub>10</sub>H<sub>12</sub>O<sub>6</sub>

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%) [1237]. SM was isolated from the leaves of *Melicope broadbentiana* F. M. Bail (Rutaceae)

[128] [1237]. **N.B.:** In the paper [1237], the formulas (VII) as well as (XI) representing the titled compound were erroneous [128].

m.p. 181-183° [1237];

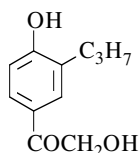
<sup>1</sup>H NMR [1237], IR [1237], UV [1237].

**2-Hydroxy-1-(4-hydroxy-3-propylphenyl)ethanone**

[178978-33-1]

C<sub>11</sub>H<sub>14</sub>O<sub>3</sub>

mol.wt. 194.23



## Synthesis

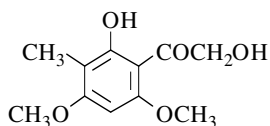
-Refer to: [616].

**2-Hydroxy-1-(2-hydroxy-4,6-dimethoxy-3-methylphenyl)ethanone**

[184706-61-4]

C<sub>11</sub>H<sub>14</sub>O<sub>5</sub>

mol.wt. 226.23



## Isolation from natural sources

-From the stem wood of *Euphorbia quinquecostata* Volk. (Euphorbiaceae) [982].

m.p. 164-166° [982];

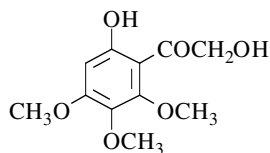
<sup>1</sup>H NMR [982], <sup>13</sup>C NMR [982], IR [982], UV [982], MS [982].

**2-Hydroxy-1-(6-hydroxy-2,3,4-trimethoxyphenyl)ethanone** (*Dimethyldegeranylmelicopol*)

[51117-08-9]

C<sub>11</sub>H<sub>14</sub>O<sub>6</sub>

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%) [128].

-Also obtained by hydrogenolysis of *dimethylmelicopol* (VIII) (SM) [1237]. SM was obtained by partial methylation of *methylmelicopol* (VII) [1237], itself isolated from the leaves of *Melicope broadbentiana* F. M. Bail. (Rutaceae) [128] [1237]. In the paper [1237], the formulas (VII) and (VIII), as well as (XIX) representing the titled compound were erroneous [128].

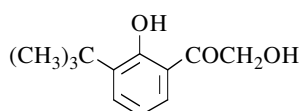
m.p. 87° [1237], 86-87° [128];  
<sup>1</sup>H NMR [1237], IR [1237], UV [1237].

**1-[3-(Dimethylethyl)-2-hydroxyphenyl]-2-hydroxyethanone**

[55960-04-8]

C<sub>12</sub>H<sub>16</sub>O<sub>3</sub>

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%) [271].

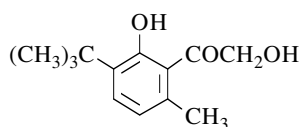
oil [271]; <sup>1</sup>H NMR [271], IR [271], UV [271].

**1-[3-(Dimethylethyl)-2-hydroxy-6-methylphenyl]-2-hydroxyethanone**

[55960-06-0]

C<sub>13</sub>H<sub>18</sub>O<sub>3</sub>

mol.wt. 222.28



Synthesis

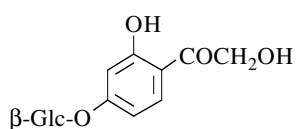
-Obtained by a selective one-step synthesis from 2-tert-butyl-5-methylphenoxymagnesium bromide (1 mol) and anhydrous monomeric glyoxal (1 mol) in boiling benzene for 20 h (25%) [271].

m.p. 76-77° [271]; <sup>1</sup>H NMR [271], IR [271], UV [271].

**1-[4-(β-D-Glucopyranosyloxy)-2-hydroxyphenyl]-2-hydroxyethanone**

C<sub>14</sub>H<sub>18</sub>O<sub>9</sub>

mol.wt. 330.29



Synthesis

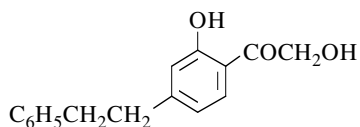
-Refer to: [1499].

**2-Hydroxy-1-[2-hydroxy-4-(2-phenylethyl)phenyl]ethanone**

[132197-47-8]

C<sub>16</sub>H<sub>16</sub>O<sub>3</sub>

mol.wt. 256.30



Synthesis

-Refer to: [523] (Japanese patent).

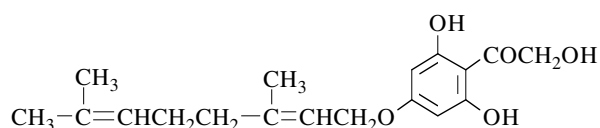
**1-[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2,6-dihydroxyphenyl]-2-hydroxyethanone**

[142905-41-7]

C<sub>18</sub>H<sub>24</sub>O<sub>5</sub>

mol.wt. 320.38

Isolation from natural source



-From the fruit of *Evodia Merrillii* Kanehira & Sasaki ex Kanehira (Rutaceae) [304].

m.p. 106-108° [304]; column chromatography [304];  
<sup>1</sup>H NMR [304], <sup>13</sup>C NMR [304], IR [304], UV [304], MS [304].

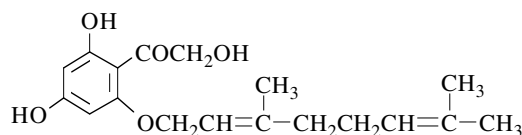
**1-[2-[(3,7-Dimethyl-2,6-octadienyl)oxy]-4,6-dihydroxyphenyl]-2-hydroxyethanone (E)**

[149492-42-2]

C<sub>18</sub>H<sub>24</sub>O<sub>5</sub>

mol.wt. 320.38

Isolation from natural source



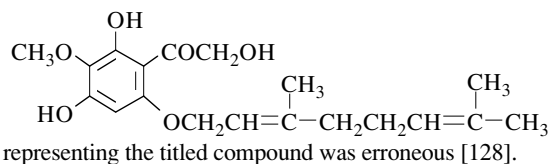
-From the fruits of *Evodia merrillii* Kanehira & Sasaki ex Kanehira (Rutaceae) [926].

m.p. 144°5-146° [926];

<sup>1</sup>H NMR [926], <sup>13</sup>C NMR [926], IR [926], UV [926], MS [926].
**1-[6-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2,4-dihydroxy-3-methoxyphenyl]-2-hydroxyethanone (*Melicopol*)**C<sub>19</sub>H<sub>26</sub>O<sub>6</sub>

mol.wt. 350.41

Isolation from natural source



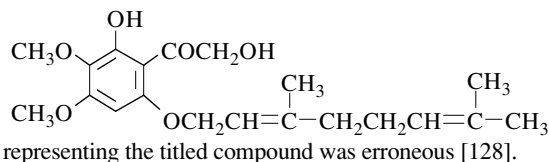
-From the leaves of *Melicope broadbentiana* F. M. Bail. (Rutaceae) [128] [1237]. **N.B.:** In the paper [1237], the formula (VI) representing the titled compound was erroneous [128].

m.p. 133-134° [1237];

<sup>1</sup>H NMR [1237], <sup>1</sup>H NMR NOE [128], IR [1237], UV [128] [1237].
**1-[3,4-Dimethoxy-6-[(3,7-dimethyl-2,6-octadienyl)oxy]-2-hydroxyphenyl]-2-hydroxyethanone (*Methylmelicopol*)**C<sub>20</sub>H<sub>28</sub>O<sub>6</sub>

mol.wt. 364.44

Isolation from natural source



-From the leaves of *Melicope broadbentiana* F. M. Bail. (Rutaceae) [128] [1237]. **N.B.:** In the paper [1237], the formula (VII) representing the titled compound was erroneous [128].

m.p. 103° [1237];

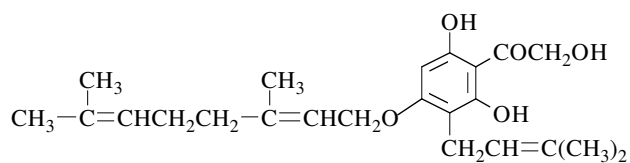
<sup>1</sup>H NMR [1237], <sup>1</sup>H NMR NOE [128], IR [1237], UV [128] [1237].

**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<sub>23</sub>H<sub>32</sub>O<sub>5</sub>

mol.wt. 388.26



Isolation from natural source

-From the fruits of *Evodia merrillii* Kanehira & Sasaki ex Kanehira (Rutaceae) [926].

m.p. 136-137° [926];

<sup>1</sup>H NMR [926], <sup>13</sup>C NMR [926], IR [926], UV [926], MS [926].