# Roth Collection of Natural Products Data

Concise Descriptions
and Spectra

Edited by
Lutz Roth and Gabriele Rupp

VCH

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Distribution:
VCH, P.O. Box 101161, D-69451 Weinheim, Federal Republic of Germany
Switzerland: VCH, P.O. Box, CH-4020 Basel, Switzerland
United Kingdom and Ireland: VCH, 8 Wellington Court, Cambridge CB1 1HZ, United Kingdom
USA and Canada: VCH, 220 East 23rd Street, New York, NY 10010-4606, USA
Japan: VCH, Eikow Building, 10-9 Hongo 1-chome, Bunkyo-ku, Tokyo 113, Japan

ISBN 3-527-28180-0

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76185 Karlsruhe



Published jointly by
VCH Verlagsgesellschaft, Weinheim (Federal Republic of Germany)
VCH Publishers, New York, NY (USA)

Editorial Director: Dr. Hans-Joachim Kraus
Production Manager: Dipl.-Wirt.-Ing. (FH) Bernd Riedel

Titelbild: Chamomilla recutita (L.) Rauschert
von Kurt Kormann, Waldstr. 45, 75045 Walzbachtal

Library of Congress Card No. applied for

A catalogue record for this book is available from the British Library.

Die Deutsche Bibliothek – CIP-Einheitsaufnahme

**Roth collection of natural products data** : concise descriptions
and spectra / ed. by Lutz Roth and Gabriele Rupp. – Weinheim;
New York; Basel; Cambridge; Tokyo : VCH, 1995
ISBN 3-527-28180-0
NE: Roth, Lutz [Hrsg.]



Composition: Filmsatz Unger & Sommer GmbH, D-69469 Weinheim
Printing: Colordruck GmbH, D-69181 Leimen
Bookbinding: Wilh. Osswald + Co, D-67408 Neustadt

Printed in the Federal Republic of Germany

# Preface

More and more attention is being paid to natural products chemistry. The number of recently discovered and described natural products is growing exponentially, while increased analytical possibilities are contributing considerably to the information we already possess on natural products.

Scientific journals cannot, on the one hand, process the increasing amounts of information offered to them; on the other hand, they cannot sufficiently meet the growing need for information. The space they have for publications is inadequate, and the data on substances can only be printed in an extremely abbreviated form. In stark contrast to this lack of space is the constantly growing abundance of detailed information. Today databases are a significant aid in searching for special data. They are, however, not suitable for people wanting to acquire an overview of this gigantic field.

It is the aim of the Roth Collection of Natural Products Data to bridge this gap. It strikes a happy medium between a concise encyclopedic presentation and an introduction to the large field of primary literature by offering data on selected natural products in a clearly structured fashion. It proposes to introduce and inform, to stimulate and inspire.

This collection comprises short monographs on 75 natural products of plant origins, clearly presented and detailed. Each monograph contains information on physical and chemical properties, toxicology and risk potential, followed by spectroscopic and chromatographic data with clearly indicated measurement conditions and peaks. In addition, structural formulas and illustrations of NMR, mass and IR spectra are provided. Selected and annotated references on each natural product facilitate the search for further information.

In view of the abundance of natural products known to us, a collection such as this one must of course restrict itself to a few representatives from important classes of substances. At any rate, for a comprehensive collection of as much information as possible on as many natural products as possible, databases with their search possibilities are more suitable. However, one must bear in mind that data is often missing for products which were described some time ago; this occurs, for example, because only the more recent publications have been included in databases.

This collection includes data both on recently discovered and long known natural products. Although their values, properties etc can be found described in the literature, they are most widely scattered. Thus it is theoretically possible for a terpene expert to be uncertain as to where to find the NMR data for such a common substance as limonene. Consequently it appeared to be a good idea to compile the data on such common natural products as limonene, camphene or apigenine. This was a most work-intensive process, especially because many spectra had to be remeasured in order to achieve a certain standardization.

This work is the starting point for a reliable collection of natural products that is as comprehensive as possible and that includes particularly important and typical representatives of all classes of substances. A goal such as this one cannot be realized through one single book but rather through a dedicated continuation. Seen in those terms, this collection is a beginning waiting to be continued.

Karlsruhe, October 1994

L. Roth
G. Rupp

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*Patulin*
*Penicillic acid*
*Mycorrhizin A*
*Aurantioglíocladin*
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*Epicorazine A*
*Oosporein*
*Verrucarin A*
*Verrucarin B*
*Mellein*
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*Cardanol, Triene*
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*Deacetoxymatricarin*
*Honokiol*
*Magnolol*
*Peimine*
*Peiminine*
*Yomogin*

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*Apometzgerin 6,8-di-C-arabinoside*
*Bryoflavone*
*5',3'''-Dihydroxyrobustaflavone*
*2,3-Dihydro-5'-hydroxyamentoflavone*
*2,3-Dihydro-5',3'''-dihydroxyamentoflavone*
*Tricin-6,8-di-C-arabinoside*
*Heterobryoflavone*
*Isofurcatain-7-0-β-D-glucoside*
*Saponarin*
*Lucenin-2*
*Tricetin-6-C-arabinoside-8-C-glucoside*
*Tricetin 6,8-di-C-glucoside*

# List of Compounds

In the following collection substances can be found in alphabetical order according to the names written in boldface type.

| Synonyms and Systematic Names | Common Names |
|---|---|
| (-)-(3S,3aR,4S,9aS,9bS)-4-Acetoxy-2,3,3a,4,5,9,9a,9b-octanhydro-9-hydroxy-3,6,9-trimethyl-azuleno[4,5-b]-furan-2-one | **Matricine** |
| **Alantolactone** | |
| Anacardol | **Cardanol, saturated** |
| **Apigenin** | |
| **Apigenin-7-glucoside** | |
| Apigenin-7-β-D-glucoside | **Apigenin-7-glucoside** |
| Apigenol | **Apigenin** |
| Apigetrin | **Apigenin-7-glucoside** |
| Apometzgerin-6,8-di-C-α-arabinopyranoside | **Apometzgerin 6,8-di-C-arabinoside** |
| **Apometzgerin-6,8-di-C-arabinoside** | |
| 6-Arabinopyranosyl-8-β-D-glucopyranosyl-5,7-dihydroxy-2-(3,4,5-trihydroxyphenyl)-4*H*-1-benzopyran-4-one | **Tricetin 6-C-arabinoside-8-C-glucoside** |
| 6-C-α-L-Arabinopyranosyl-8-C-β-D-glucopyranosyl-tricetin | **Tricetin 6-C-arabinoside-8-C-glucoside** |
| **Artecanin** | |
| **Aurantiogliocladin** | |
| Austracamphene | **(+)-Camphene** |
| Axillin | **Deacetoxymatricarin** |
| 5′,6″-Biluteolin | **5′,3‴-Dihydroxyrobustaflavone** |
| [1,1′-Biphenyl]-2,2′-diol, 5,5′-di-2-propenyl- | **Magnolol** |
| [1,1′-Biphenyl]-2,4′-diol, 3′,5-di-2-propenyl- | **Honokiol** |
| **(-)-α-Bisabolol** | |
| **Bryoflavone** | |
| Cajeputene | **(±)-Limonene** |
| **Camphene** | |
| **Candelabrone** | |
| Cardanol 15:0 | **Cardanol, saturated** |
| Cardanol 15:1 (n-7) | **Cardanol, Monoene** |
| Cardanol 15:2 (n-4) | **Cardanol, Diene** |
| Cardanol 15:3 (n-1) | **Cardanol, Triene** |
| **Cardanol, Diene** | |
| **Cardanol, Monoene** | |
| **Cardanol, saturated** | |
| **Cardanol, Triene** | |
| Cardanol-diolefin | **Cardanol, Diene** |
| Cardanol-monoolefin | **Cardanol, Monoene** |
| Cardanol-triolefin | **Cardanol, Triene** |
| Cardol | **Cardol, saturated** |

| Synonyms and Systematic Names | Common Names |
|---|---|
| Cardol 15:0 | **Cardol, saturated** |
| Cardol 15:1 (n-7) | **Cardol, Monoene** |
| Cardol 15:2 (n-4) | **Cardol, Diene** |
| Cardol 15:3 (n-1) | **Cardol, Triene** |
| **Cardol, Diene** | |
| **Cardol, Monoene** | |
| **Cardol, Triene** | |
| Cardol-diolefin | **Cardol, Diene** |
| Cardol-monoolefin | **Cardol, Monoene** |
| **Cardol, saturated** | |
| Cardol-triolefin | **Cardol, Triene** |
| Carvene | **(+)-Limonene** |
| 5α,14α-Cevanine-3β,20β-dihydroxy-6-one | **Peiminine** |
| 5α, 14α-Cevanine-3β,6α,20β-triol | **Peimine** |
| Chaetomidin | **Oosporein** |
| **Chamazulene** | |
| **Chlorophyll a** | |
| **Chlorophyll b** | |
| Cinene | **(±)-Limonene** |
| Citrene | **(+)-Limonene** |
| Clavacin | **Patulin** |
| Clavatin | **Patulin** |
| Claviformin | **Patulin** |
| Coriandrol | **S-Linalool** |
| Cosmetin | **Apigenin-7-glucoside** |
| Cosmosiin | **Apigenin-7-glucoside** |
| Cynene | **(±)-Limonene** |
| Daturine | **L-Hyoscyamine** |
| **Deacetoxymatricarin** | |
| **Dehydrocorybulbine** | |
| 3-{[6-0-(6-Deoxy-α-L-mannopyranosyl)-≈β-D-glucopyranosyl]-oxy}-5,7-dihydroxy-2-≈(4-hydroxy-3-methoxyphenyl)-4*H*-1-benzopyran-4-one | **Narcissin** |
| 6-(6-Deoxy-α-L-mannopyranosyl)-7-(β-D-glucopyranosyloxy)-5-hydroxy-2-(4-hydroxyphenyl)4*H*-1-benzopyran-4-one | **Isofurcatain-7-O-β-D-glucoside** |
| 6,8-Di-C-α-L-arabinopyranosylapometzgerin | **Apometzgerin-6,8-di-C-arabinoside** |
| 6,8-Diarabinopyranosyl-5,7-dihydroxy-2-(3-hydroxy-4,5-dimethoxyphenyl)-4*H*-1-benzopyran-4-one | **Apometzgerin-6,8-di-C-arabinoside** |
| 6,8-Diarabinopyranosyl-5,7-dihydroxy-2-(4-hydroxy-3,5-dimethoxyphenyl)-4*H*-1-benzopyran-4-one | **Tricin 6,8-di-C-arabinoside** |
| 6,8-Di-C-α-L-arabinopyranosyl-3',4'-dimethoxytricetin | **Apometzgerin-6,8-di-C-arabinoside** |
| 6,8-Di-C-α-L-arabinopyranosyltricin | **Tricin 6,8-di-C-arabinoside** |
| 8-Dichloroacetyl-2,7-dimethyl-5-hydroxy-1,4-naphthoquinone | **Mollisin** |
| 6,8-Di-β-D-glucopyranosyl-5,7-dihydroxy-2-(3,4,5-trihydroxyphenyl)-4*H*-1-benzopyran-4-one | **Tricetin 6,8-di-C-glucoside** |

| Synonyms and Systematic Names | Common Names |
|---|---|
| 6,8-Di-C-β-D-glucopyranosyl-luteolin | **Lucenin-2** |
| 6,8-Di-C-β-D-glucopyranosyl-tricetin | **Tricetin-6,8-di-C-glucoside** |
| **II-2,3-Dihydroamentoflavone** | |
| **2,3-Dihydro-5′,3‴-dihydroxyamentoflavone** | |
| (-)-3,4-Dihydro-6,8-dihydroxy-3-methyl-1*H*-2-benzopyran-1-one | **(-)-3,4-Dihydro-6,8-dihydroxy-3-methylisocoumarin** |
| **(-)-3,4-Dihydro-6,8-dihydroxy-3-methylisocoumarin** | |
| 3,4-Dihydro-4,8-dihydroxy-1(2H)-naphthalenone | **Isosclerone** |
| 8-[5-(2,3-Dihydro-5,7-dihydroxy-4-oxo-4*H*-1-benzopyran-2-yl)-2,3-dihydroxy-phenyl]-5,7-dihydroxy-2-(3,4-dihydroxyphenyl)-4*H*-1-benzopyran-4-one | **2,3-Dihydro-5′,3‴-dihydroxyamentoflavone** |
| 8-[5-(2,3-Dihydro-5,7-dihydroxy-4-oxo-4H-1-benzopyran-2-yl)-2,3-dihydroxyphenyl]-5,7-dihydroxy-2-(4-hydroxyphenyl)-4*H*-1-benzopyran-4-one | **2,3-Dihydro-5'-hydroxyamentoflavone** |
| **2,3-Dihydro-5′-hydroxyamentoflavone** | |
| 3,4-Dihydro-8-hydroxy-3,5-dimethyl-1H-2-benzopyran-1-one | **Methylmellein** |
| (-)-2,5-Dihydro-5-hydroxy-4-[4′-hydroxyphenyl]-furan-2-one | **Hydroxybutenolide** |
| 3,4-Dihydro-8-hydroxy-3-methyl-[1H]-2-benzopyran-1-one | **Mellein** |
| 5,6-Dihydro-3-hydroxy-2,9,10-trimethoxy-13-methyl-dibenzo-[a,g]-quinolizinium | **Dehydrocorybulbine** |
| 1,3-Dihydroxy-2-hydroxymethyl-9,10-anthraquinone | **Lucidin** |
| 5,7-Dihydroxy-2(4-hydroxyphenyl)-4*H*-1-benzopyran-4-one | **Apigenin** |
| 8,12-Dihydroxy-4-methyl-11,16-dioxosenecionanium | **Senkirkine** |
| (-)-6,8-Dihydroxy-3-methylisochroman-1-one | **(-)-3,4-Dihydro-6,8-dihydroxy-3-methylisocoumarin** |
| 8-[5-(5,7-Dihydroxy-4-oxo-4*H*-1-benzopyran-2-yl)-2,3-dihydroxyphenyl]-5,7-dihydroxy-2-(4-hydroxyphenyl)4*H*-1-benzopyran-4-one | **5′-Hydroxyamentoflavone** |
| 6-[5-(5,7-Dihydroxy-4-oxo-4*H*-1-benzopyran-3-yl)-2,3-dihydroxyphenyl]-2-(3,4-dihydroxyphenyl)-5,7-dihydroxy-4*H*-1-benzopyran-4-one | **Bryoflavone** |
| 6-[5-(5,7-Dihydroxy-4-oxo-4*H*-1-benzopyran-2-yl)-2,3-dihydroxyphenyl]-2-(3,4-dihydroxyphenyl)-5,7-dihydroxy-4H-1-benzopyran-4-one | **5′,3‴-Dihydroxyrobustaflavone** |
| 8-[5-(5,7-Dihydroxy-4-oxo-4*H*-1-benzopyran-3-yl)-2,3-dihydroxyphenyl]-2-(3,4-dihydroxyphenyl)-5,7-dihydroxy-4*H*-1-benzopyran-4-one | **Heterobryoflavone** |
| 8-[5-(5,7-Dihydroxy-4-oxo-4*H*-1-benzopyran-2-yl)-2-hydroxyphenyl]-2,3-dihydro-5,7-dihydroxy-2-(4-hydroxyphenyl)-4*H*-1-benzopyran-4-one | **II-2,3-Dihydroamentoflavone** |
| 2-(3,4-Dihydroxyphenyl)-6,8-di-β-D-glucopyranosyl-5,7-dihydroxy-4*H*-1-benzopyran-4-one | **Lucenin-2** |
| **5′,3‴-Dihydroxyrobustaflavone** | |
| 4,8-Dihydroxy-1-tetralone | **Isosclerone** |
| Diisoprene | **(±)-Limonene** |
| 2,3-Dimethoxy-5,6-dimethyl-1,4-benzoquinone | **Aurantiogliocladin** |
| Dimethulene | **Chamazulene** |
| 2,2-Dimethyl-3-methylenebicyclo[1.2.2]heptane | **Camphene** |
| 2,2-Dimethyl-3-methylenenorbornane | **Camphene** |

| Synonyms and Systematic Names | Common Names |
|---|---|
| 3,7-Dimethyl-1,6-octadien-3-ol | **Linalool** |
| Dipentene | **(±)-Limonene** |
| Duboisine | **L-Hyoscyamine** |
| **Epicorazine A** | |
| (8S,9R,9aS,10aS)-9-Ethenyl-8-(β-D-glucopyranosyloxy)-2,3,9,9a,10,10a-hexahydro-5H,8H-pyrano[4,3-d]thiazolo-[3,2-a]pyridine-5-one | **Xylostosidine** |
| (1S,8S,9R,9aS,10aS)-9-Ethenyl-8-(β-D-glucopyranosyloxy)-2,3,9,9a,10,10a-hexahydro-5H,8H-pyrano[4,3-d]thiazolo-[3,2-a]pyridine-5-one-1-oxide | **Loxylostosidine A** |
| 7-Ethyl-1,4-dimethylazulene | **Chamazulene** |
| Eudesma-4(14),11(13)-dien-12-oic acid,8β-hydroxy-, γ-lactone | **Isoalantolactone** |
| 4αH-Eudesma-5,11(13)-dien-12-oic acid,8β-hydroxy-, γ-lactone | **Alantolactone** |
| Expansin | **Patulin** |
| **Fabiatrin** | |
| Fritillarine | **Peiminine** |
| 6-C-β-D-Glucopyranosyl-apigenin-7-O-β-glucopyranoside | **Saponarin** |
| 6-β-D-Glucopyranosyl-7-(β-D-glucopyranosyloxy)-5-hydroxy-2-(4-hydroxyphenyl)-4*H*-1-benzopyran-4-one | **Saponarin** |
| 3-β-D-Glucopyranosyloxy-2-methoxy-6-hydroxy-benzoic acid-2′-β-D-glucopyranosyl-oxybenzylester | **Leiocarposide** |
| Helenin | **Alantolactone** |
| Hesperidene | **(+)-Limonene** |
| **Heterobryoflavone** | |
| 3,3a,4,5,9a,9b-Hexahydro-3,6,9-trimethyl-azuleno-[4,5-b]-furan-2,7-dione | **Deacetoxymatricarin** |
| 1a,2,12,12a,15a,18-Hexahydro-1a,12a,17-trimethyl-spiro[12,14-methano-3H,19H,20H-oxireno[8,9][1,6,12]trioxacyclooctadecino-[3,4-d]-[1]benzopyran-13(14H),2′-oxirane]-5,10,22,(22aH)-trione | **Verrucarin B** |
| **Honokiol** | |
| 1-Hydroxy-6-acetoxyguaj-2,4-(10)-dien-8,12-olide | **Matricine** |
| **5′-Hydroxyamentoflavone** | |
| **Hydroxybutenolide** | |
| p-Hydroxy-β-[carboxymethyl]cinnamic acid | **Sphagnum acid** |
| 1(S),6(S),9(R)-6-Hydroxy-8,8-dimethyl-4-(1-chloro-propenyl)-tricyclo-[4.4.0.0$^{1,9}$]-7-oxa-dec-3-ene-2,5-dione | **Mycorrhizin A** |
| 4-Hydroxy-4H-furo-[3,2-c]pyran-2[6H]-one | **Patulin** |
| 2-(4-Hydroxyphenyl)-5-hydroxy-7-β-D-glucosyl-4*H*-1-benzopyran-4-one | **Apigenin-7-glucoside** |
| 3-[4-Hydroxy-phenyl]pentenedioic acid | **Sphagnum acid** |
| **L-Hyoscyamine** | |
| **Isoalantolactone** | |
| **Isofurcatain-7-O-β-D-glucoside** | |
| Isohelenin | **Isoalantolactone** |

| Synonyms and Systematic Names | Common Names |
|---|---|
| Iso-oosporein | **Oosporein** |
| 4-Isopropenyl-1-methylcyclohexene | **Limonene** |
| **Isosclerone** | |
| Isoterebenthene | **(±)-Limonene** |
| Isovitexin-7-O-glucoside | **Saponarin** |
| Kautschine | **(±)-Limonene** |
| **Kingiside** | |
| **Leiocarposide** | |
| (+)-Leukodin | **Deacetoxymatricarin** |
| Levomenol | **(-)-α-Bisabolol** |
| l-Licareol | **R-Linalool** |
| **Limonene** | |
| **Linalool** | |
| **Loganin** | |
| Loganoside | **Loganin** |
| Loniceroside | **Secologanin** |
| **Loxylostosidine A** | |
| **Lucenin-2** | |
| **Lucidin** | |
| Luteolin-6,8-di-C-β-D-glucopyranoside | **Lucenin-2** |
| Luteolin-6,8-di-C-glucoside | **Lucenin-2** |
| Magnesium, 3-[(17S)-8-ethyl-13$^2$t-methoxycarbonyl-2,7,12,18t-tetramethyl-13$^1$-oxo-2-vinyl-13,13$^2$,17,18-tetrahydro-cyclopenta[at]-porphyrin-17r-yl]-propionato-[(7R,11R)-trans-phytylester] | **Chlorophyll a** |
| Magnesium, 3-[(17S)-7-formyl-8-ethyl-13$^2$-methoxycarbonyl-2,12,18t-trimethyl-13$^1$-oxo-3-vinyl-13$^1$,13$^2$,17,18-tetrahydro-cyclopenta[at]-porphyrin-17r-yl]-propionato-[(7R,11R)-trans-phytylester] | **Chlorophyll b** |
| **Magnolol** | |
| **Matricine** | |
| Melaleucol | **trans-Nerolidol** |
| **Mellein** | |
| *p*-Mentha-1,8-diene | **Limonene** |
| (13$^2$R)-13$^2$-Methoxycarbonyl-3$^1$,3$^2$-didehydro-phytochlorin-[7R,11R)-trans-phytylester] | **Chlorophyll a** |
| 3-Methoxy-5-methyl-4-oxo-2,5-hexadiene | **Penicillic acid** |
| 6-Methoxy-7-[(6-0-β-D-xylopyranosyl-β-D-glucopyranosyl)oxy]-2*H*-1-benzopyran-2-one | **Fabiatrin** |
| Methyl-(1S,5S,9R)-5-(formylmethyl)-1-(β-D-glucopyranosyloxy)-9-vinyl-5,9-dihydro-1H-pyran-4-carboxylate | **Secologanin** |
| Methyl-(1S,5S,8S,9S)-1-(β-D-glucopyranosyloxy)-1,5,6,7,8,9-hexahydro-8-methyl-7-one-4-pyrano[3,4-c]-pyran-4-carboxylate | **Kingiside** |
| Methyl-(1S,5S,7S,8R,9R)-1-(β-D-glucopyranosyloxy)-1,5,6,7,8,9-hexahydro-7-hydroxy-8-methylcylopenta(c)pyran-4-carboxylate | **Loganin** |
| **Methylmellein** | |

| Synonyms and Systematic Names | Common Names |
|---|---|
| (-)-(1'S,2S)-6-Methyl-2-(4'-methyl-3'-cyclohexen-1'-yl)-5-hepten-2-ol | **(-)-α-Bisabolol** |
| 1-Methyl-4-(1-methylethenyl)-cyclohexene | **Limonene** |
| 5-Methylochracin | **Methylmellein** |
| **Mollisin** | |
| Muconomycin A | **Verrucarin A** |
| Muconomycin B | **Verrucarin B** |
| Mycoin $C_3$ | **Patulin** |
| **Mycorrhizin A** | |
| 1,4-Naphthoquinone, 8-(dichloroacetyl)-5-hydroxy-2,7-dimethyl | **Mollisin** |
| Narcisoside | **Narcissin** |
| **Narcissin** | |
| **trans-Nerolidol** | |
| Ochracin | **Mellein** |
| **9,12-Octadecadien-6-ynoic acid** | |
| **9,12,15-Octadecatrien-6-ynoic acid** | |
| **9-Octadecen-6-ynoic acid** | |
| 4,4a,7,7a,11,11a,14,14a-Octahydro-4,11-dihydroxy-[4S-(4α,4aα,6aβ,7aβ,11α,11a α,13aβ,14aβ)]-8H,13H-6a,13a-epidithio-1H,6H-pyrazino[1,2-a:4,5-a']-diindole-1,6,8,13-tetrone | **Epicorazine A** |
| Octahydro-6-hydroxy-6,8a-dimethyl-3-methylene-4H-bis-oxireno[1,8a:2,3]-azuleno[4,5-b]furan-2(3H)-one | **Artecanin** |
| 4,5,6,7,16,16a,19a,22-Octahydro-4-hydroxy-5,16a,21-trimethyl-spiro[16,18-methano-1H,3H,23H-[1,6,12]trioxacyclooctadecino-[3,4-d]-[1]benzopyran-17(18H),2'-oxirane]-3,9,14-trione | **Verrucarin A** |
| **Oosporein** | |
| **Patulin** | |
| **Peimine** | |
| **Peiminine** | |
| Pelargidenon | **Apigenin** |
| Penicidin | **Patulin** |
| **Penicillic acid** | |
| (Z,Z)-5-n-(8,11-Pentadecadienyl)-1,3-benzenediol | **Cardol, Diene** |
| (Z,Z)-3-n-(8,11-Pentadecadienyl)-phenol | **Cardanol, Diene** |
| Pentadecadienylresorcinol | **Cardol, Diene** |
| (Z,Z)-5-n-(8,11,14-Pentadecatrienyl)-1,3-benzenediol | **Cardol, Triene** |
| (Z,Z)-3-n-(8,11,14-Pentadecatrienyl)-phenol | **Cardanol, Triene** |
| Pentadecatrienylresorcinol | **Cardol, Triene** |
| (Z)-5-n-(8-Pentadecenyl)-1,3-benzenediol | **Cardol, Monoene** |
| (Z)-3-n-(8-Pentadecenyl)-phenol | **Cardanol, Monoene** |
| Pentadecenylresorcinol | **Cardol, Monoene** |
| 5-n-Pentadecyl-1-3-benzenediol | **Cardol, saturated** |
| 3-n-Pentadecylphenol | **Cardanol, saturated** |
| Pentadecylresorcinol | **Cardol, saturated** |

| Synonyms and Systematic Names | Common Names |
|---|---|
| Renardine | **Senkirkine** |
| 6-C-α-L-Rhamnopyranosyl-apigenin-7-O-β-gluco-pyranoside | **Isofurcatain-7-O-β-D-glucoside** |
| **Saponarin** | |
| **Secologanin** | |
| **Senkirkine** | |
| **Sphagnum acid** | |
| **Squarrogenin 1** | |
| **Squarrogenin 2** | |
| Terecamphene | **(-)-Camphene** |
| 3a,8a,9,9a-Tetrahydro-5,8a-dimethyl-3-methylene-naphtho-[2,3-b]-furan-2,6(3H,4H)-dione | **Yomogin** |
| 3,3′,6,6′-Tetrahydroxy-5,5′-dimethyl-2,2′-bi-p-benzoquinone | **Oosporein** |
| Tricetin-6-C-α-L-arabinopyranoside-8-C-β-D-gluco-pyranoside | **Tricetin 6-C-arabinoside-8-C-glucoside** |
| **Tricetin 6-C-arabinoside-8-C-glucoside** | |
| Tricetin 6,8-di-C-β-D-glucopyranoside | **Tricetin 6,8-di-C-glucoside** |
| **Tricetin 6,8-di-C-glucoside** | |
| Tricin 6,8-di-C-α-L-arabinopyranoside | **Tricin 6,8-di-C-arabinoside** |
| **Tricin 6,8-di-C-arabinoside** | |
| (+)-(5*R*,10*S*)-11,12,14-Trihydroxy-8,11,13-abietatriene-3,7-dione | **Candelabrone** |
| 4′,5,7-Trihydroxyflavone | **Apigenin** |
| 21(R),22(S),23(R),3β,22β,30-Trihydroxy-21-methoxy-21,23-epoxycycloart-24-ene | **Squarrogenin 1** |
| 21(S),22(S),23(R),3β,22β,30-Trihydroxy-21-methoxy-21,23-epoxycycloart-24-ene | **Squarrogenin 2** |
| 3,7,11-Trimethyl-1,6,10-dodecatrien-3-ol | **trans-Nerolidol** |
| 1αH,5αH-Tropan-3α-ol(-)-tropate | **L-Hyoscyamine** |
| 3α-Tropanyl-S-(-)-tropate | **L-Hyoscyamine** |
| **Verrucarin A** | |
| **Verrucarin B** | |
| Versulin | **Apigenin** |
| Verticine | **Peimine** |
| **Xylostosidine** | |
| **Yomogin** | |

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# General Abbreviations

| | |
|---|---|
| *[α]* | specific optical rotation: $[\alpha]_D^{24}$ at 24 °C for D (sodium) line<br>Example: $[\alpha]_D^{20}$ + 220° (c = 1, MeOH), meaning 220 g of the substance dissolved in 100 ml methanol; when no solvent is given, the solvent is water. |
| AcOH | acetic acid |
| ADR | International regulations governing the transport of dangerous goods |
| alc | alcohol, ethanol |
| Ar | aryl |
| bp | boiling point (the pressure, if different from one atm, is indicated in brackets) |
| BRN | Beilstein registry number |
| c | concentration by volume (after optical rotations only) |
| CA | Chemical Abstracts |
| CAS | Chemical Abstracts Service |
| conc. | concentration |
| cor, corr. | corrected |
| *d* | density; specific gravity ($d_4^{20}$ specific gravity at 20° referred to water at 4°) |
| dec, decomp. | decomposition |
| DMSO | dimethyl sulfoxide |
| e.g. | *exempli gratia*, example given, for example |
| EtOH | ethanol |
| GGVE/GGVS | Gefahrgutverordnung Eisenbahn/Straße (german regulations governing the transport of dangerous goods) |
| GLC | gas-liquid chromatography |
| HPLC | high performance (pressure, power) liquid chromatography |
| I | intensity |
| i.v. | intravenous |
| ID | Inhibition dose |
| INN | International Nonproprietary Name |
| IR | Infrared spectrometry |
| IUPAC | International Union of Pure and Applied Chemistry |
| J | coupling constant |
| k′ | retention time |
| kg | kilogram |
| LD | Lethal Dose; $LD_{50}$, a dose which is lethal to 50% of the animals tested |
| $M^+$ | molecular ion |
| m/e | mass to charge ratio |
| max | maximum |
| MeOH | methanol |
| min | minute(s) |
| mp | melting point |
| MS | Mass spectrometry |
| m/z | mass to charge ratio |
| *n* | index of refraction ($n_D^{20}$ for 20° and sodium light) |
| NMR | nuclear magnetic resonance |
| no. | number |
| ORD | optical rotatory dispersion |
| PDM | Perdeuteromethylether |
| PM | Permethylether |
| ppm | parts per million |
| R | Nature of special risks attributed to dangerous substances |
| ref. | refer, reference |
| Rf | in paper chromatography ratio of movement of the band to the front of the solvent |
| RID | international regulations governing the transport of dangerous goods |
| RTECS | Registry of Toxic Effects of Chemical Substances |
| S | Safety advice concerning dangerous substances |
| s, sec | seconds |

| | |
|---|---|
| spp. | species (plural) |
| temp. | temperature |
| TLC | thin-layer chromatography |
| TMS | tetramethylsilane |
| UN | United Nations number, numbers assigned to many substances by a United Nations committee used for transport purposes |
| uncorr. | uncorrected |
| UV | ultraviolet spectrometry |
| v/v | % "volume in volume" expresses the number of milliliters of an active constituent in 100 milliliters of solution |
| ε | molar extinction coefficient (conc. in g-moles/l) |
| ≈ | approximately |
| * | There is no illustration for this spectrum |

# Hazard Labeling

| | |
|---|---|
| C | corrosive |
| E | explosive |
| F | highly flammable |
| F+ | extremely flammable |
| N | Dangerous for the environment |
| O | oxidizing |
| T | toxic |
| T+ | very toxic |
| Xi | irritant |
| Xn | harmful |

# Abbreviations in IR and NMR Spectroscopy

| | |
|---|---|
| vs | very strong |
| w | weak |
| m | medium |
| s | strong |
| vw | very weak |
| sh | shoulder |
| br | broad |
| s | singlet |
| d | doublet |
| dd | doublet of doublets |
| t | triplet |
| q, qu | quartet |
| quint | quintet |
| sext | sextet |
| sept | septet |
| m, mult | multiplet |

Superscripts in the Data Form indicate the References at the end of each form.

# CAS-Registry Numbers

| | |
|---|---|
| 78-70-6 | **Linalool (racemic)** |
| 79-92-5 | **Camphene** |
| 90-65-3 | **Penicillic acid** |
| 101-31-5 | **L-Hyoscyamine** |
| 126-90-9 | **S-(+)-Linalool** |
| 126-91-0 | **R-(-)-Linalool** |
| 149-29-1 | **Patulin** |
| 470-17-7 | **Isoalantolactone** |
| 475-54-7 | **Oosporein** |
| 478-08-0 | **Lucidin** |
| 479-61-8 | **Chlorophyll a** |
| 480-33-1 | **R-Mellein** |
| 483-54-5 | **Aurantiogliocladin** |
| 501-26-8 | **Cardanol, Monoene** |
| 501-24-6 | **Cardanol, saturated** |
| 519-62-0 | **Chlorophyll b** |
| 520-36-5 | **Apigenin** |
| 528-43-8 | **Magnolol** |
| 529-05-5 | **Chamazulene** |
| 546-43-0 | **Alantolactone** |
| 578-74-5 | **Apigenin-7-glucoside** |
| 604-80-8 | **Narcissin** |
| 667-92-5 | **Mollisin** |
| 1119-38-6 | **S-(+)-trans-Nerolidol** |
| 1200-93-7 | **(±)-Mellein** |
| 2290-11-1 | **Verrucarin B** |
| 2318-18-5 | **Senkirkine** |
| 3148-09-2 | **Verrucarin A** |
| 3158-56-3 | **Cardol, saturated** |
| 5794-03-6 | **(+)-Camphene** |
| 5794-04-7 | **(-)-Camphene** |
| 5989-27-5 | **(+)-Limonene** |
| 5989-54-8 | **(-)-Limonene** |
| 7705-14-8 | **(±)-Limonene** |
| 7734-92-1 | **R-Methylmellein** |
| 10067-18-2 | **Yomogin** |
| 17397-85-2 | **Mellein** |
| 17946-87-1 | **Deacetoxymatricarin** |
| 18059-10-4 | **Peiminine** |
| 18309-73-4 | **Fabiatrin** |
| 18524-94-2 | **Loganin** |
| 19314-92-2 | **(-)-3,4-Dihydro-6,8-dihydroxy-3-methylisocoumarin** |

19351-63-4 **Secologanin**
20310-89-8 **Saponarin**
22910-86-7 **Cardol, Monoene**

23089-26-1 **(-)-α-Bisabolol**
23496-41-5 **Peimine**
25406-67-1 **Kingiside**

29041-35-8 **Matricine**
29428-58-8 **Lucenin-2**
35354-74-6 **Honokiol**

40716-66-3 **trans-Nerolidol (racemic)**
51546-63-5 **Cardanol, Diene**
53766-42-0 **9,12,15-Octadecatrien-6-ynoic acid** (Z,Z,Z-isomer)

56795-52-9 **9,12-Octadecadien-6-ynoic acid** (Z,Z-isomer)
57100-28-4 **Sphagnum acid**
59870-72-3 **Dehydrocorybulbine**

59905-46-3 **Artecanin**
62256-05-7 **Epicorazine A**
62332-73-4 **Isosclerone**

62623-84-1 **S-Mellein**
64356-85-0 **Mycorrhizin A**
71953-77-0 **Leiocarposide**

74518-57-3 **Xylostosidine**
76491-13-9 **Apometzgerin 6,8-di-C-arabinoside**
76491-11-7 **Tricetin 6,8-di-C-glucoside**

77551-75-8 **R-(-)-trans-Nerolidol**
78119-20-7 **Loxylostosidine A**
79353-39-2 **Cardanol, Triene**

79473-25-9 **Cardol, Diene**
79473-24-8 **Cardol, Triene**
89886-37-3 **Apometzgerin 6,8-di-C-arabinoside**

86022-78-8 **Tricetin 6-C-arabinoside-8-C-glucoside**
89886-36-2 **Tricin 6,8-di-C-arabinoside**
83463-97-2 **Isofurcatain-7-O-β-D-glucoside**

104056-04-4 **5′,3‴-Dihydroxyrobustaflavone**
106577-42-8 **II-2,3-Dihydroamentoflavone**
111200-22-7 **Bryoflavone**

111200-23-8 **Heterobryoflavone**
113545-42-9 **Candelabrone**
114865-39-3 **5′-Hydroxyamentoflavone**

115374-25-9 **9-Octadecen-6-ynoic acid** (Z-isomer)
122475-58-5 **2,3-Dihydro-5′-hydroxyamentoflavone** (S-isomer)
122475-59-6 **2,3-Dihydro-5′,3‴-dihydroxyamentoflavone** (S-isomer)

123564-56-7 **Hydroxybutenolide**
125445-28-5 **Squarrogenin 1**
125445-29-6 **Squarrogenin 2**

# Part I
# Data Sheets

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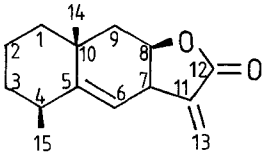
# Alantolactone

*H. Häberlein*

## 1. Name of Compound

| | | |
|---|---|---|
| Common name<br>**Alantolactone** | | |
| Synonyms<br>Helenin [1] | | |
| Systematic name<br>4αH-Eudesma-5,11(13)-dien-12-oic acid,8β-hydroxy-,γ-lactone | | |
| Substance<br>Terpene | Subgroup<br>Sesquiterpene lactone | |
| CAS registry number and other numbers<br>[546-43-0] | Merck Index *11*, 198 | BRN 13423 |

## 2. Formulas and Molecular Weight

| | |
|---|---|
| Molecular formular<br>$C_{15}H_{20}O_2$<br><br>Molecular weight<br>232.33 | Structural formula<br>  |

## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline solid | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>79 °C [2] | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>275 °C [2] | $[\alpha]_D^{20}$:<br>220.0° (c = 1,MeOH) [3] | Saponification value<br>not applicable |
| Flash point<br>not applicable | Soluble in<br>chloroform, dichloromethane, methanol, diethyl ether, acetone | |
| Color<br>white | Odor<br>indifferent | |

## 4. Occurrence

| |
|---|
| Main constituent from Inula species [4] |

## 5. Health Hazard Data

| | |
|---|---|
| Toxicology<br>Irritating substance<br>In vitro cytotoxicity<br>on human lung<br>Carcinoma Cell Line | Hazard labeling Xn<br>Harmful by inhalation, in contact with skin and if swallowed.<br>May cause sensitization. |
| | $ID_{50}$: 4.6 µg/ml [5] (ID = inhibition dose) |
| Waste disposal procedures<br>Dissolve or mix the material with a combustible solvent and burn in a chemical incinerator equipped with an afterburner and scrubber. | |

## 6. Transportation and Storage Instructions

| |
|---|
| Storage temperature: 0–5 °C |

## 7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | m/e 217 | Molecular ion | m/e 232.32545 |
| Ionization energy | 70 eV | Ion source temp. | 120 °C |
| Acceleration voltage | 4000 V | Emission current | 0.2 mA |
| Resolution | 1000 | Scan rate | 2.6 s/decade |
| Spectrometer type and manufacturer | | Vacuum Generators Micromass 7070 H | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>2960, 2940, 2900, 2830, 1725, 1640, 1450, 1380, 1250, 1240,<br>1155, 1120, 1050, 1030, 975, 920, 855, 850, 810 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | IR Spectrometer 398, Perkin Elmer | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts | | | |
| 6.18 (d, 1H, *H*C-13);<br>5.60 (d, 1H, *H*C-13);<br>5.13 (d, 1H, *H*C-6);<br>4.80 (ddd, 1H, $H_\alpha$C-8); | 3.56 (m, 1H, $H_\alpha$C-7);<br>2.43 (m, 1H, $H_\alpha$C-4);<br>2.09 (dd, 1H, $H_\alpha$C-9);<br>1.53 (dd, 1H, $H_\beta$C-9 | 1.17 (s, 3H, $H_{3\beta}$C-14)<br>1.07 (d, 3H, $H_{3\beta}$C-15) ppm | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 23 °C |
| Spectrometer type and manufacturer | | Bruker WH 400 | |

| NMR * | $^1$H-decoupled | Nucleus | $^{13}$C |
|---|---|---|---|
| Chemical Shifts | | | |
| 170.2 (s, C-12); | 149.0 (s, C-5); | 139.9 (s, C-11); | 121.3 (t, C-13); |
| 119.8 (d, C-6); | 76.3 (d, C-8); | 42.7 (t, C-9); | 41.7 (t, C-3); |
| 39.5 (d, C-7); | 37.5 (d, C-4); | 32.7 (t, C-1); | 32.7 (s, C-10); |
| 28.5 (qu, C-14); | 22.5 (qu, C-15); | 16.7 (t, C-2) ppm | |
| Frequency | 100 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 23 °C |
| Spectrometer type and manufacturer | | Bruker WH 400 | |

| UV | not applicable |
|---|---|

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.30 | | |
| Solvent | acetone | Solvent system | toluene/ethyl acetate (97:3) |
| Saturated atmosphere | yes | Detection/Color | 5% solution of $AlCl_3$ in ethanol, 10 min at 120 °C, brown under UV (366 nm) |
| Plate manufacturer | Riedel de Haën | Plate type/Product no. | DC-Mikrokarten Si $F_{254nm}$ Art. 37341 |

| GLC | For application of GLC for the separation of sesquiterpene lactones see ref. [6] and [7]. |
|---|---|

| HPLC | see ref. [3] | | |
|---|---|---|---|
| Retention time | k' = 6.33 | | |
| Column | 4.0 mm × 250 mm | Stationary phase | silica gel (5 m) |
| Mobile phase | n-pentane/diethyl ether (90:10) | Flow rate | 1 ml/min |
| Column temp. | room temperature | Pressure | 150 bar |
| Detector | UV 210 nm | Sample solvent | n-pentane/diethyl ether (90:10) |
| Sample size | 10 µl | Sample conc. | $8.62 \times 10^{-4}$ mol/l |
| Chromatograph type and manufacturer | Pump L-6200, UV-Detector 655A-23 Chromato-Integrator D-2500, Merck-Hitachi | | |

9. Remarks

| Used in the chemotaxonomy of higher plants and in several methods of chromatography as reference substance. |
|---|

10. References


[1] Karrer, W., *Konstitution und Vorkommen der organischen Pflanzenstoffe*, Birkhäuser Verlag, Basel 1958, No. 1900

[2] Glasby, J.S., *Encyclopaedia of the Terpenoids*, John Whiley & Sons (1982) pp. 124

[3] Häberlein, H., unpublished data

[4] Bohlmann, F., Mahanta, P.K., Jakupovic, J., Rastogi, R.C., Natu, A., *Phytochemistry 17* (1978) 1165-1172

[5] Woerdenbag, H.J., Meijer, C., Mulder, N.H., de Vries, E.G.E., Hendriks, H., Malingre, Th.M., *Planta Med. 2* (1986) 112-114

[6] Zinchenko, V.V., Khvorost, P.P., Bakai, S.I., Biryuk, V.A., Tarusin, A.D., Kravchina, T.S., *Rastit Resur. 19(4)*, (1983) 544-548

[7] Rosik, G.G., Zinchenko, V.V., Reznichenko, A.A., Koralev, I.P., *Khim.-Farm Zh. 21(5)*, (1987) 632-634

Additional analytical data are found in:

[8] Schmalle, H.W., *Acta Crystallogr., Sect. C: Cryst. Struct. Commun.* C42(6), (1986) 705-708 *(x-ray crystallography)*

# Apigenin

*R. Carle*

## 1. Name of Compound

Common name

**Apigenin** [1]

Synonyms

Pelargidenon, Apigenol, 4′,5,7-Trihydroxyflavon, Versulin

Systematic name

5,7-Dihydroxy-2(4-hydroxyphenyl)-4*H*-1-benzopyran-4-one

Substance

Flavonoid

CAS registry number and other numbers

[520-36-5] BRN 262620

## 2. Formulas and Molecular Weight

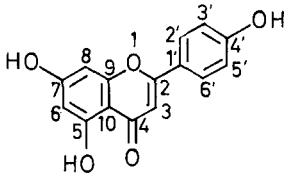
Molecular formular

$C_{15}H_{10}O_5$

Molecular weight

278.23 [1]

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline, needles [1] | $d_4^{20}$<br>not applicable | Iodine value<br>not determined |
| Melting Point mp<br>344-347 °C [2] | $n_D^{20}$<br>not applicable | Acid value<br>not determined |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20}$<br>not applicable | Saponification value<br>not applicable |
| Flash point<br>not applicable | Soluble in<br>hot alcohol, diluted KOH [1] | |
| Color<br>yellow [1] | Odor<br>odorless | |

4. Occurrence

| |
|---|
| E.g. in chamomile, yarrow [2, 3, 4] |

5. Health Hazard Data

| Toxicology | Hazard labeling | not necessary |
|---|---|---|
| not known [5] | $LD_{50}$ | 100 mg/kg (mouse i.v.)<br>1000 mg/kg (mouse, oral) |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage in closed containers |

7. Spectroscopic Data

| | |
|---|---|
| MS | see ref. [6] |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3320, 2920, 1610, 1500, 1360, 1240 $cm^{-1}$ | | | |
| Sample preparation | KBr-tablet | Resolution | – |
| Spectrometer type and manufacturer | | Perkin Elmer 1420 | |

| NMR * | see ref. [7] | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>6.20 (C-6); 6.48 (C-8); 6.77 (C-3); 6.93 (C-3′/C-5′); 7.92 (C-2′/C-6′) ppm | | | |
| Frequency | 500 MHz | Solvent | DMSO-$d_6$ |
| Standard | TMS | Sample temp. | 30 °C |
| Spectrometer type and manufacturer | | NMR Bruker AMX 500 | |

| NMR * | see ref. [8] | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts | | | |
| 95.57 (C-6); | 100.27 (C-8); | 104.33 (C-3); | 105.06 (C-10); |
| 117.32 (C-3′ and C-5′); | 122.72 (C-1′); | 129.79 (C-2′ and C-6′); | 158.67 (C-5); |
| 161.79 (C-4′); | 162.03 (C-9); | 164.93 (C-7); | 165.49 (C-2) ppm |
| Frequency | 25.15 MHz | Solvent | $CHCl_3$ |
| Standard | TMS | Sample temp. | 95 °C |
| Spectrometer type and manufacturer | | JEOL P-100 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 330, 267, 211 nm | $\varepsilon_{max}$ | 18.862 (330 nm) |
| Solvent | Methanol | | |
| Sample conc. | $3.7 \times 10^{-5}$ mole/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Lambda 5 UV/VIS, Perkin Elmer | |

8. Chromatographic Data

| TLC | see ref. [10] | | |
|---|---|---|---|
| Rf Value | 0.92–0.94 | | |
| Solvent | methanol | Solvent system | Ethylacetate/Formic Acid/Water (100+10+15) |
| Saturated atmosphere | yes | Detection/Color | 1% Diphenylboric-acid-β-aminoethylester in methanol: UV 365: green |
| Plate manufacturer | Merck | Plate type/Product no. | Silicagel |

| GLC | underivatized not possible |
|---|---|

| HPLC | | | |
|---|---|---|---|
| Retention time | 10.6 min | | |
| Column | 125 mm × 4 mm | Stationary phase | RP-18, particle size 5 µm |
| Mobile phase | A: 2000 ml $KH_2PO_4$ (0.005 mol/l), 14 ml dil. $H_3PO_4$ (pH appr. 2.6)<br>B: 1200 ml acetonitrile + 600 ml methanol | Flow rate | 1.0 ml/min |
| Column temp. | 37 °C | Pressure | 72 bar |
| Detector | Variable wavelength detector HP 1084 B/79875 A, 335 nm | Sample solvent | ethanol 96% (v/v) |
| Sample size | 15 µl | Sample conc. | 1.2 mg/100 ml |
| Chromatograph type and manufacturer | | Hewlett Packard HP 1084 B | |

9. Remarks

10. References


[1] Windholz, M. (Ed.); *The Merck Index*, 9th Ed., Merck & Co., Rahway 1976 *(Name, occurrence)*

[2] Power, F., Browning, H. jun., *Chem. Soc. Ldn. 105* (1914) 2280–2291 *(Occurrence)*

[3] Sorm, P., Cekan, Z., Herout, V., Raskova, H., *Chem. Listy 46* (1952), 308–309 *(Occurrence, chemical properties)*

[4] Karrer, W. in: *Konstitution und Vorkommen der organischen Pflanzenstoffe (exklusive Alkaloide)* Erg.-Bd. 2, Birkhäuser Verlag, Basel, Boston, Stuttgart 1981 pp. 313 *(Occurrence)*

[5] Janku, I., *C.* R. *Seances Soc. Biol. 151* (1957) 241 *(Toxicology)*

[6] Audier, H., *Bull. Soc. Chim. France* (1966) 2892–2899 *(Mass Spectrometry)*

[7] Batterham, T.J., Highet, R.J., Aust., *J. Chem. 17* (1964) 428–439 *(Proton NMR)*

[8] Ternai, B., Markham, K.R., *Tetrahedron 32* (1976) 565–569 *(Carbon-13 NMR)*

[9] Mabry, T.J., Markham, K.R., Thomas, M.B.: The *Systematic Identification of Flavonoids*, Springer Verlag Berlin 1970 *(UV-Spectroscopy)*

[10] Kunde, R., Isaac, 0., *Planta Med. 37* (1979) 124–130 *(TLC)*

[11] Dölle, B., Carle, R., Müller, W., *Dtsch. Apoth. Ztg. 43 Supp.I* (1985) 14–19 *(HPLC)*

# Apigenin-7-glucoside

*R. Carle*

## 1. Name of Compound

Common name

**Apigenin-7-glucoside**

Synonyms

Apigenin-7-β-D-glucoside, Apigetrin, Cosmosiin, Cosmetin [1]

Systematic name

2-(4-Hydroxyphenyl)-5-hydroxy-7-β-D-glucosyl-4*H*-1-benzopyran-4-one

Substance

Flavonoid

CAS registry number and other numbers

[578-74-5] BRN 65669

## 2. Formulas and Molecular Weight

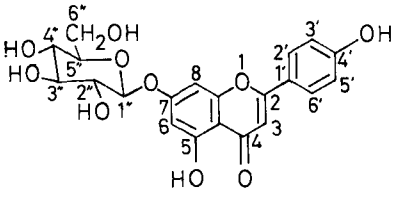
Molecular formular

$C_{21}H_{20}O_{10}$ [1]

Molecular weight

432.39 [1]

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline powder [1] | $d_4^{20}$<br>not applicable | Iodine value<br>not determined |
| Melting Point mp<br>240-242 °C [2] | $n_D^{20}$<br>not applicable | Acid value<br>not determined |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20}$<br>not applicable | Saponification value<br>not applicable |
| Flash point<br>not applicable | Soluble in<br>water, diluted alcohol [1] | |
| Color<br>light yellow | Odor<br>odorless | |

## 4. Occurrence

| |
|---|
| E.g. in chamomile and yarrow [2, 3] |

## 5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| | $LD_{50}$ | – |
| Waste disposal procedures<br>Combustion | | |

## 6. Transportation and Storage Instructions

| |
|---|
| Storage in closed containers, protected from humidity |

## 7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS * | see ref. [4] | | |
| Base peak | 270 | Molecular ion | 432 |
| Ionization energy | 70 eV | Ion source temp. | 250 °C |
| Acceleration voltage | – | Emission current | – |
| Resolution | 20000 | Scan rate | – |
| Spectrometer type and manufacturer | | MS 9 | |

| | | | |
|---|---|---|---|
| IR * | see ref. [5] | | |
| Characteristic peaks<br>3420s, 3100w, 2930w, 2905w, 1710m, 1620s, 1595m, 1500m, 1320m, 1185m, 1095m, 845m $cm^{-1}$ | | | |
| Sample preparation | KBr-tablet | Resolution | – $cm^{-1}$ |
| Spectrometer type and manufacturer | | Perkin Elmer 1420 | |

| | | | |
|---|---|---|---|
| NMR * | see ref. [5] | Nucleus | $^1H$ |
| Chemical Shifts<br>3.1–3.8 (m, 6H, H-2″ to H-6″); 5.7 (d, 1H, H-1″); 6.46 (d, 1H, H-6);<br>6.82 (d, 1H, H-8); 6.83 (s, 1H, H-3); 6.93 (d, 2H, H-3′ and H-5′);<br>7.94 (d, 2H, H-2′ and H-6′); 12.95 (br s, 1H, OH-5) ppm | | | |
| Frequency | | Solvent | – |
| Standard | | Sample temp. | |
| Spectrometer type and manufacturer | | – | |

| NMR * | see ref. [8] | Nucleus | $^{13}C$ |
|---|---|---|---|

Chemical Shifts

| | | | | |
|---|---|---|---|---|
| 182.3 (C-4); | 164.6 (C-7); | 163.3 (C-2); | 161.9 (C-9); | 161.3 (C-4'); |
| 157.2 (C-5); | 128.8 (C-2'/C-6'); | 121.1 (C-1'); | 116.3 (C-3'/C-5'); | 105.6 (C-10); |
| 103.3 (C-3); | 100.2 (C-1''); | 99.7 (C-6); | 95.1 (C-8); | 77.4 (C-3''); |
| 76.6 (C-5''); | 73.3 (C-2''); | 69.8 (C-4''); | 60.9 (C-6'') ppm | |

| Frequency | 15.08 MHz | Solvent | DMSO-$d_6$; MeOH-$d_4$ |
|---|---|---|---|
| Standard | TMS | Sample temp. | – |
| Spectrometer type and manufacturer | | BRUKER WP 60 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 267 and 337 nm | $\varepsilon_{max}$ | 1.11 (335 nm) [6] |
| Solvent | $KH_2PO_4$ solution 0.005 mol/l acetonitrile + methanol (100+80+40) | | |
| Sample conc. | 2 mg/100 ml | Cell thickness | – |
| Spectrometer type and manufacturer | | Hewlett Packard HP 1084 B | |

8. Chromatographic Data

| TLC | see ref. [7] | | |
|---|---|---|---|
| Rf Value | 0.42–0.51 | | |
| Solvent | methanol | Solvent system | Acetic acid ethylester/Formic acid/Water(100 : 10 : 15) |
| Saturated atmosphere | yes | Detection/Color | 1% Diphenylboric-acid-β-aminoethylester in methanol: UV 366 nm: green |
| Plate manufacturer | Merck | Plate type/Product no. | Silicagel |

| GLC | underivatized not possible |
|---|---|

| HPLC | | | |
|---|---|---|---|
| Retention time | 3.2 min | | |
| Column | 125 mm × 4 mm | Stationary phase | RP-18, particle size 5 µm |
| Mobile phase | 2000 ml $KH_2PO_4$ (0.005 mol/l) + Acetonitrile/Methanol (100+80+40) | Flow rate | 1.0 ml/min |
| Column temp. | 37 °C | Pressure | – |
| Detector | Variable wavelength detector HP 1084 B/79875 A, 335 nm | Sample solvent | Ethanol 96% (v/v) |
| Sample size | 15 µl | Sample conc. | 5 mg/100 ml |
| Chromatograph type and manufacturer | | Hewlett Packard HP 1084 B | |

9. Remarks

Post harvest enzymatic degradation into apigenin.

10. References


[1] Windholz, M. (Ed.); *The Merck Index*, 9th Ed., Merck & Co., Rahway 1976 *(Name, occurrence, physical properties)*

[2] Lang, W., Schwandt, K., *Dtsch. Apoth. Ztg. 97* (1957) 149–151 *(Occurrence)*

[3] Karrer, W. in: *Konstitution und Vorkommen der organischen Pflanzenstoffe (exclusive Alkaloide)*, Erg.-Bd. 2, Birkhäuser Verlag, Basel, Boston, Stuttgart 1981, pp. 314 *(Occurrence)*

[4] Prox, A., *Tetrahedron 24* (1968) 3697–3715 *(Mass Spectrometry)*

[5] Carle, R. (unpublished) *(UV-,IR-Spectrometry, Proton-NMR)*

[6] Poethke, W., Bulin, P., *Pharm. Zentralhalle 108* (1969) 733–747 *(UV-Spectrometry)*

[7] Dölle, B., Carle, R., Müller, W., *Dtsch. Apoth. Ztg. 43 Suppl.I* (1985) 14–19 *(HPLC)*

[8] Schreiber, A., Carle, R., Reinhard, E., *Planta Med. 56* (1990) 179–181 *(Enzymatic degradation)*

[9] Chari, V.M., Jordan, M., Wagner, H., Thies, P.W., *Phytochemistry 16* (1977) 1110–1112 *($^{13}$C-NMR)*

# Apometzgerin-6,8-di-C-arabinoside

*S. Anhut, H. D. Zinsmeister, R. Mues*

## 1. Name of Compound

Common name
**Apometzgerin-6,8-di-C-arabinoside** [1, 2]

Synonyms
Apometzgerin-6,8-di-C-α-L-arabinopyranoside
6,8-Di-C-α-L-arabinopyranosylapometzgerin
6,8-Di-C-α-L-arabinopyranosyl-3′,4′-dimethoxytricetin

Systematic name
6,8-Diarabinopyranosyl-5,7-dihydroxy-2-(3-hydroxy-4,5-dimethoxyphenyl)-4*H*-1-benzopyran-4-one

Substance
Flavonoid

Subgroup
Flavone Glycoside
Flavone-C-glycoside

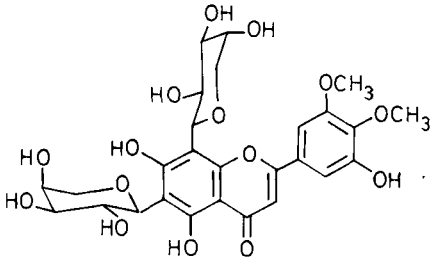
CAS registry number and other numbers
[76491-13-9] in *94*:84438a
[89886-37-3] in *95*:187589a

## 2. Formulas and Molecular Weight

Molecular formular
$C_{27}H_{30}O_{15}$

Molecular weight
594.53

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline | $d_4^{20}$<br>not applicable | Iodine value<br>not applicable |
| Melting Point mp<br>198-200 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20}$<br>not available | Saponification value<br>not applicable |
| Flash point<br>not applicable | Soluble in<br>methanol | |
| Color<br>pale yellow | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Apometzgeria pubescens (Hepaticae; Bryophytes) [1, 2, 3]<br>(only known from this species!) [5]. |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| not known | $LD_{50}$ | not known |
| Waste disposal procedures | – | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder; preferably in the dark. |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS | as Perdeuteromethylether (PDM)<br>as Permethylether (PM) [1] | | |
| Base peak | PM derivative: 689<br>PDM: 713 | Molecular ion | PM: 720;<br>PDM: 747 |
| Ionization energy | 90 eV | Ion source temp. | 150 °C |
| Acceleration voltage | 1.8 kV | Emission current | 2 mA |
| Resolution | 2000 | Scan rate | – |
| Spectrometer type and manufacturer | | Varian MAT 311A – 100 MS | |

| |
|---|
| IR for structure elucidation of Flavone-C-glycosides not practicable |

| | | | |
|---|---|---|---|
| NMR | see ref. [5] | Nucleus | $^1H$ |
| Chemical Shifts<br>Aglycone: 7.22 (s, H-2′, H-6′); 6.88 (s, H-3); 13.25 (s, 5-OH);<br>Sugars: 4.76 (d, J=9.5 Hz, H-1″); 4.58 (d, J=9.5 Hz, H-1‴)<br>3.88 (s, Methoxyl); 3.73 (s, Methoxyl) ppm | | | |
| Frequency | 400 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | ambient temp., ≈25 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | see ref. [4], [5] | Nucleus | $^{13}C$ **) see remarks |
|---|---|---|---|

Chemical Shifts

183.6 (C-4); 164.3 (C-2); 163.1 (C-7); 160.3 (C-5); 155.0 (C-9);
154.5 (C-3′); 151.8 (C-5′); 140.6 (C-4′); 126.8 (C-1′); 109.8 (C-6)**);
105.0 (C-10 and C-6′); 104.0 (C-8); 103.2 (C-3 and C-2′); 60.5 (4′-$OCH_3$);
6-C-Arabinose: 75.6 (C-3″); 74.5 (C-1″); 71.1 (C-5″); 69.3 (C-2″); 69.3 (C-4″);
8-C-Arabinose: 75.0 (C-3‴); 74.5 (C-1‴); 70.8 (C-5‴); 69.3 (C-2‴); 69.3 (C-4‴) ppm

| Frequency | 100 MHz | Solvent | DMSO-$d_6$ |
|---|---|---|---|
| Standard | DMSO-$d_6$ | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV | see ref. [1] | | |
|---|---|---|---|
| $I_{max}$ | 276, 331 nm | $\varepsilon_{max}$ | – |
| Solvent | Methanol (Uvasol)<br>(additional UV spectra with shift reagents were reported in [1]) | | |
| Sample conc. | about $2 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Super Scan 3 (Varian); UV-VIS-spectrophotometer, double beam | | |

## 8. Chromatographic Data

| TLC | I–III on Cellulose (Avicel); IV on Polyamide-6 [1] | | |
|---|---|---|---|
| Rf Value | System I: 0.52 System III: 0.37<br>System II: 0.81 System IV: 0.57 | | |
| Solvent system | I: Acetic Acid : Water (15 : 85); II: Acetic Acid : Water (40 : 60)<br>III: Butanol-(1) : Acetic Acid : Water (4 : 1 : 5) upper layer<br>IV: Water : Butanone(2): MeOH : Pentanedione (65 : 15 : 15 : 5) | | |
| Saturated atmosphere | – | Detection/Color | UV dark; UV, sprayed with Naturstoffreagenz A: green |
| Plate manufacturer | Schleicher & Schüll<br>Macherey & Nagel | Plate type/Product no. | Cellulose, Avicel F 1440<br>Polygram; 0.1 mm; Polyamide-TLC 6 |

| GLC | underivatized not possible |
|---|---|

| HPLC | | | |
|---|---|---|---|
| Retention time | 9.9 min | | |
| Column | Macherey & Nagel ET 250/8/4 Nucleosil 5C18 | Stationary phase | Nucleosil RP-18, 5 µm |
| Mobile phase | A: MeOH/B = $H_2O$ : AcOH (95 : 5), isocratic 40A – 60B | Flow rate | 1.0 ml/min |
| Column temp. | ambient temp., ≈25 °C | Pressure | – |
| Detector | UV Detector Waters 450 UV at 254 nm | Sample solvent | MeOH |
| Sample size | 10 µl | Sample conc. | – |
| Chromatograph type and manufacturer | Waters M 45 | | |

9. Remarks

Assignment for C-6 changed due to recent results of $^{13}$C-NMR/DEPT experiments.

10. References


[1] Theodor, R., Zinsmeister, H. D., Mues, R. and Markham, K. R.: Flavone C-glycosides of Apometzgeria pubescens. *Phytochemistry 19*, 1980, pp. 1695-1700

[2] Theodor, R., Markham, K. R., Mues, R. and Zinsmeister, H. D.: The structures of new Flavone-di-C-glycosides from Apometzgeria pubescens. *Phytochemistry 20/6*, 1981, pp. 1457-8

[3] Chopin, J. and Dellamonica, G.: C-Glycosylflavonoids. In: ‚The Flavonoids', Harborne, J.B. (Ed.), Chapman and Hall London, 1988

[4] Agrawal, P.K. and Bansal, M.C.: Flavonoid Glycosides.In ‚Carbon-13 NMR of Flavonoids', Agrawal, P.K. (Ed.), *Studies in Organic Chemistry 39*, pp. 333-4, Elsevier Science Pub., 1989

[5] Markham, K.R., Mues, R., Stoll, M. and Zinsmeister, H.D.: NMR Spectra of Flavone Di-C-glycosides from Apometzgeria pubescens and the Detection of Rotational Isomerism in 8-C-Hexosylflavones. *Z. Naturforsch. 42c*, 1987, pp 1039-42

# Artecanin

*Wu Chongming*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Artecanin** | |
| Synonyms | |
| Systematic name<br>Octahydro-6-hydroxy-6,8a-dimethyl-3-methylene-4H-bisoxireno[1,8a:2,3]-azuleno[4,5-b]furan-2(3H)-one | |
| Substance<br>Sesquiterpene lactone | Subgroup<br>Guaianolides lactone |
| CAS registry number and other numbers<br>[59905-46-3] | BRN 1432844 |

## 2. Formulas and Molecular Weight

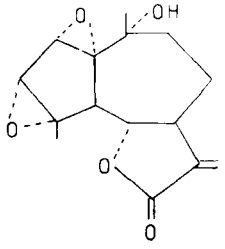
Molecular formular

$C_{15}H_{18}O_5$

Molecular weight

278.30

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>241-243 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{20} = +29.6°$ (25 °C)<br>(c = 0.42, EtOH) | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>ethyl acetate, methanol | |
| Color<br>colorless | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Several species of the genus Artemisia (e.g. A. gmelinii Web. ex Stechm) (Compositae) |

5. Health Hazard Data

| Toxicology | Hazard labeling |
|---|---|
| not available | $LD_{50}$ not available |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 111 | Molecular ion | 296 ($M+NH_4^+$, 100) CI/$NH_3$ |
| Ionization energy | 70 eV | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | MM 7070H | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3420, 2925, 1734, 1162, 1128, 1016, 997 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | PE-599B | |

| NMR | | Nucleus | $^1H$-$^1H$-correlation |
|---|---|---|---|
| Chemical Shifts<br>0.84 (s, 3H, $C_{10}$-$CH_3$); 1.32 (s, 3H, $C_4$-$CH_3$);<br>1.57 and 1.93 (m, 3H, $C_8$-$H_2$ and m, 1H, $C_9$-$H_2$);<br>2.68 (d, J=10 Hz, 1H, $C_5$-H); 3.23 (d, J=1 Hz, 1H, $C_2$-H);<br>3.46 (d, J=1 Hz, 1H, $C_3$-H); 3.77 (d, J=10 Hz, 1H, $C_6$-H);<br>4.95 (br, 1H, $C_{10}$-OH; disappear after $D_2O$);<br>5.42 (d, J=3 Hz, 1H, $C_{13}$-$H_a$); 5.88 (d, J=3 Hz, 1H, $C_{13}$-$H_b$) ppm | | | |
| Frequency | 90 MHz | Solvent | DMSO-$d_6$ |
| Standard | TMS | Sample temp. | – |
| Spectrometer type and manufacturer | | EM-390 | |

| | |
|---|---|
| UV | not available |

8. Chromatographic Data

| | |
|---|---|
| TLC | not available |

| | |
|---|---|
| GLC | not available |

| | |
|---|---|
| HPLC | not available |

9. Remarks

10. References

[1] Nageshvar, R.B. et al.: Phytochem. *14*(12), 1975, 2651

[2] Wu, Chongming, Tu, Youyou: Chinese Bulletin of Botany *3*(3), 1985, 34

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# Aurantiogliocladin

*C. Franke, K. Krohn*

## 1. Name of Compound

Common name
**Aurantiogliocladin**

Synonyms

Systematic name
2,3-Dimethoxy-5,6-dimethyl-1,4-benzoquinone

Substance
Benzoquinone

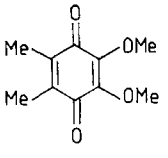
CAS registry number and other numbers
[483-54-5] BRN 1961187

## 2. Formulas and Molecular Weight

Molecular formular
$C_{10}H_{12}O_4$

Molecular weight
196.202

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>62.5 °C | $n_D^{20}$<br>not applicable | Acid value<br>not available |
| Boiling point bp<br>not available | $[\alpha]_D^{20}$<br>not applicable | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>petroleum ether | |
| Color<br>orange | Odor<br>like benzoquinone but milder | |

4. Occurrence

| |
|---|
| Isolated from the fungi Gliocladium sp. [1], Paecilomyces |

5. Health Hazard Data

| Toxicology | Hazard labeling |
|---|---|
| antibiotic activity | $LD_{50}$ |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 151 | Molecular ion | 196 |
| Ionization energy | 70 eV | Ion source temp. | room temperature |
| Acceleration voltage | 3 kV | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | 3 s/decade |
| Spectrometer type and manufacturer | | Finnigan MAT 8430 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>2950, 1670, 1645, 1610, 1455, 1385, 1330, 1270, 1210, 1160, 940, 925, 865, 735 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | 2.5 $cm^{-1}$ |
| Spectrometer type and manufacturer | | Perkin Elmer 1420 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>2.01 (s, 6H, $2CH_3$); 3.99 (s, 6H, 2 O-$CH_3$) ppm | | | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | $^{1}H$ decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>12.2 (q; $CH_3$); 61.2 (q; O-$CH_3$); 138.9 (s, C-5, C-6); 144.4 (s, C-2, C-3); 184.3 (s, C-1, C-6) ppm | | | |
| Frequency | 100 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 209, 276, 401 nm | $\varepsilon_{max}$ | 8575, 16069, 525 |
| Solvent | Methanol | | |
| Sample conc. | $5.912 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Beckmann | UV 5230 |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 2.30 | | |
| Solvent | – | Solvent system | 1% MeOH/99% $CH_2Cl_2$ |
| Saturated atmosphere | – | Detection/Color | 254 nm/violet |
| Plate manufacturer | Merck, Darmstadt | Plate type/Product no. | Kieselgel 60 $F_{254}$ no. 5562 |

| GLC | not available |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

10. References


[1] Brian, P.W. et al., *Experentia 7*, 1951, 266 *(isolation)*

[2] Savoie, J.Y., *Can. J. Chem.*, 1971, 3515 *(synthesis, IR, UV, NMR)*

[3] Koshi, K., *Chem. Pharm. Bull. 16*, 1968, 2343 *(synthesis, IR, UV, NMR)*

[4] Visher, E.B., *J. Chem. Soc.*, 1953, 815 *(structure)*

[5] Pettersson, G., *Acta Chem. Scand. 19*, 1965, 1827 *(biosynthesis)*

# (-)-α-Bisabolol

*R. Carle*

## 1. Name of Compound

Common name
**(-)-α-Bisabolol** [1]

Synonyms
Levomenol (INN)

Systematic name
(-)-(1′S,2S)-6-Methyl-2-(4′-methyl-3′-cyclohexen-1′-yl)-5-hepten-2-ol (IUPAC)

Substance
Sesquiterpene alcohol

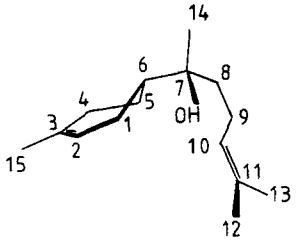
CAS registry number and other numbers
[23089-26-1] BRN 4292517

## 2. Formulas and Molecular Weight

Molecular formular
$C_{15}H_{26}O$

Molecular weight
222.36

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>viscous liquid | $d_4^{20}$<br>0.9211 g/cm$^3$ [1] | Iodine value<br>not determined |
| Melting Point mp<br>not determined | $n_D^{20}$<br>1.4965 [2] | Acid value<br>not applicable |
| Boiling point bp<br>153 °C (12 torr) | $[\alpha]_D^{20}$<br>−55.4 [2] | Saponification value<br>not applicable |
| Flash point<br>not determined | Soluble in<br>methanol, ethanol, isopropanol | |
| Color<br>light yellowish | Odor<br>sweetish | |

## 4. Occurrence

| |
|---|
| Vanillosmopsis erythropappa SCHULTZ-BIP. [3]<br>Chamomilla recutita (L.) RAUSCHERT [1] |

## 5. Health Hazard Data

| Toxicology | Hazard labeling not necessary |
|---|---|
| oral toxicity test | $LD_{50}$ 15 ml/kg (rat and mouse) [4] |
| Waste disposal procedures<br>Combustion | |

## 6. Transportation and Storage Instructions

| |
|---|
| Storage in closed containers, preferably in the dark |

## 7. Spectroscopic Data

| MS * | see ref. [5] | | |
|---|---|---|---|
| Base peak | 69 m/e | Molecular ion | 222 m/e |
| Ionization energy | 70 eV | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | AEI MS902 | |

| IR * | see ref. [6] | | |
|---|---|---|---|
| Characteristic peaks<br>3420, 3050, 3010, 2970, 2930, 1450, 1435, 1375, 1120, 915 $cm^{-1}$ | | | |
| Sample preparation | capillary film | Resolution | – |
| Spectrometer type and manufacturer | | Perkin Elmer 337 | |

| NMR * | see ref. [5] | Nucleus | $^{1}H$ |
|---|---|---|---|
| Chemical Shifts<br>1.10 (C-7, $CH_3$); 1.62 (C-11, $CH_3$); 1.64 (C-3, $CH_3$);<br>1.68 (H-12); 5.12 (t, J=7 Hz, H-10); 5.37 (br s, H-2) ppm | | | |
| Frequency | 270 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | – |
| Spectrometer type and manufacturer | | Bruker HFX-270 | |

| NMR * | | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>17.65 (C-13); 22.23 (C-9)[a]; 23.33 (C-14 and C-15); 23.44 (C-5)[a];<br>25.68 (C-12); 27.04 (C-1); 31.19 (C-4); 40.32 (C-8); 43.21 (C-6);<br>74.15 (C-7); 120.78 (C-2); 124.91 (C-10); 131.32 (C-11); 133.91 (C-3) ppm<br>([a] interchangeable) | | | |
| Frequency | 20 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | – |
| Spectrometer type and manufacturer | | CFT-20, Varian | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 203.5 nm | $\varepsilon_{max}$ | 6016.2 |
| Solvent | Methanol | | |
| Sample conc. | 0.0001 mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Lambda 5 UV/VIS, Perkin Elmer | |

## 8. Chromatographic Data

| TLC | see ref. [8] | | |
|---|---|---|---|
| Rf Value | 0.3 | | |
| Solvent | $CH_2Cl_2$ | Solvent system | $CH_2Cl_2$ (100%) |
| Saturated atmosphere | yes | Detection/Color | anisaldehyd/sulfuric-acid: violet-red |
| Plate manufacturer | Merck | Plate type/Product no. | silica gel |

| GLC | see ref. [9] | | |
|---|---|---|---|
| Retention time | 11.7 min | | |
| Column type | Chromosorb | Column length | 2 m |
| Column packing | Carbowax 20 M | Column temp. | 90–220 °C, 8 °C/min |
| Injector port temp. | 250 °C | Carrier gas | helium |
| Detector temp. | 300 °C | Sample solvent | cyclohexane |
| Sample size | 0.1 µl | Sample conc. | 100% |
| Chromatograph type and manufacturer | | HP 5890A, Hewlett Packard | |

| HPLC | see ref. [9] | | |
|---|---|---|---|
| Retention time | 11.7 min | | |
| Column | 125 mm × 4 mm | Stationary phase | Lichrospher RP 18 |
| Mobile phase | acetonitrile $KH_2PO_4$ 0.005 mol/l (80:59 v:v) | Flow rate | 1.20 ml/min |
| Column temp. | 40 °C | Pressure | 85 bar |
| Detector | HP 1084B/79875A | Sample solvent | methanol |
| Sample size | 20 µl | Sample conc. | 150 mg/100 ml |
| Chromatograph type and manufacturer | | Hewlett Packard HP 1084 B | |

9. Remarks

Four stereoisomers of α-bisabolol are possible and also occur in different plants [7, 10].
Synthetic α-bisabolol is not optically active [11].
The origin of (-)-α-bisabolol can be identified by means of IRMS [12].

10. References


[1] Sorm, F., Zaoral, M., Herout, *V., Collection Czechoslov. Chem. Commun. 16* (1951) 626–638 *(Name, Occurrence, Physical properties)*

[2] Isaac, 0., Schneider, H., Eggenschwiller, H., *Dtsch. Apoth. Ztg. 108* (1968) 293–298 *(Physical properties, GLC)*

[3] Gottlieb, 0.R., Magalhaes, M.T., *Perfum. Ess. Oil Record 49* (1958) 711–714 *(Occurrence, Characterization)*

[4] Habersang, S., Leuschner, F., Isaac, 0., Thiemer, K., *Planta Med. 37,* (1979) 115–123 *(Toxicology)*

[5] Schwartz, M.A., Swanson, G.C., *J. Org. Chem. 44* (1979) 953–958 *(Mass Spectrometry, Proton-NMR)*

[6] Kergomard, A., Veschambre, H., *Tetrahedron 33* (1977) 2215–2224 *(IR-Spectrometry)*

[7] Flaskamp, E., Nonnenmacher, G., Isaac, 0., *Z. Naturforsch. 36b* (1981) 114–118 and *36b* (1981) 536 *(Carbon-13 NMR, Stereoisomers)*

[8] Stahl, E., Schild, W., *Pharmazeutische Biologie 4, Drogenanalyse II: Inhaltsstoffe und Isolierungen,* Gustav Fischer Verlag Stuttgart, New York 1981 *(TLC, Isolation)*

[9] Carle, R., Fleischhauer, I., Fehr, D., *Dtsch. Apoth. Ztg. 127* (1987) 2451–2457 *(GLC)*

[10] Brunke, E.-J., Hammerschmidt, F.J., *Dragoco-Report 2* (1984) 37–43 *(Stereoisomers)*

[11] Kunde, R., Isaac, 0., *Planta Med. 35* (1979) 71–75 *(Adulteration)*

[12] Carle, R., Fleischhauer, I., Beyer, J., Reinhard, E., *Planta Med. 56* (1990) 456–460 *(Adulteration, Isotope Ratio Mass Spectrometry)*

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# Bryoflavone

*H. D. Zinsmeister, S. Anhut, R. Mues*

## 1. Name of Compound

Common name
**Bryoflavone** [1]

Synonyms

Systematic name
6-[5-(5,7-Dihydroxy-4-oxo-4*H*-1-benzopyran-3-yl)-2,3-dihydroxyphenyl]-2-(3,4-dihydroxyphenyl)-5,7-dihydroxy-4*H*-1-benzopyran-4-one

| Substance | Subgroup |
|---|---|
| Flavonoid | Biflavonoid |

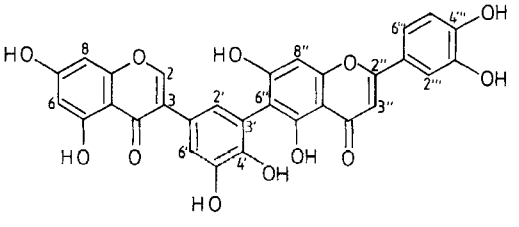
CAS registry number and other numbers
[111200-22-7]

## 2. Formulas and Molecular Weight

Molecular formular
$C_{30}H_{18}O_{12}$

Molecular weight
570.462

Structural formula



## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| crystalline | not applicable | not applicable |
| Melting Point mp | $n_D^{20}$ | Acid value |
| 250–252 °C (decomp.) | not applicable | not applicable |
| Boiling point bp | $[\alpha]_D^{20}$ | Saponification value |
| not applicable | not measurable | not applicable |
| Flash point | Soluble in | |
| not applicable | acetone, methanol | |
| Color | Odor | |
| yellow | odorless | |

4. Occurrence

| |
|---|
| Bryum capillare (Musci) [1, 2] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| not known | $LD_{50}$ | not known |
| Waste disposal procedures | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder; preferably in the dark. |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS | FD – Mass spectrum | | |
| Base peak | – | Molecular ion | 570 mu |
| Ionization energy | – | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | Varian MAT 311 with FD source | |

| | |
|---|---|
| IR | not available |

| | | | |
|---|---|---|---|
| NMR | | Nucleus | $^1H$ |
| Chemical Shifts<br>13.21 and 13.02 (s, OH-5″ and OH-5);<br>7.43 (dd, J=8, 2 Hz, H-6‴);<br>7.04 (d, J=2 Hz, H-6′);<br>6.76 (d, J=2 Hz, H-2′);<br>6.37 (d, J=2 Hz, H-8); | | 8.28 (s, Isoflav., H-2);<br>7.41 (d, J=2 Hz, H-2‴);<br>6.90 (d, J=8 Hz, H-5‴);<br>6.67 (s, Flavone, H-3″); 6.54 (H-8″, s);<br>6.21 (d, J=2 Hz, H-6) ppm | |
| Frequency | 400 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | see ref. [1, 3, 4] | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts | | | |
| 181.6 (C-4″); | 180.3 (C-4); | 164.1 (C-7); | 163.4 (C-2″); |
| 162.0 (C-7″, C-5); | 159.0 (C-5″); | 157.2 (C-9); | 156.1 (C-9″); |
| 153.8 (C-2); | 149.5 (C-4‴); | 145.7 (C-3‴); | 144.9/144.3 (C-4′, C-5′); |
| 122.8 (C-2′); | 122.4 (C-3); | 121.6 (C-1′); | 120.4/120.3 (C-1‴, C-3′); |
| 118.9 (C-6‴); | 116.0 (C-6′); | 114.9 (C-5‴); | 113.2 (C-2‴); |
| 109.9 (C-6″); | 104.5 (C-10); | 103.2 (C-10″); | 102.7 (C-3″); |
| 98.9 (C-6); | 93.6/93.4 (C-8, C-8″) ppm | | |
| Frequency | 100 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 262, 288sh, 344 nm | $\varepsilon_{max}$ | – |
| Solvent | Methanol (Uvasol)<br>additional UV-spectra with shift reagents according to [5] | | |
| Sample conc. | about $2 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Super Scan 3 (Varian); UV-VIS-spectrophotometer,double beam | | |

8. Chromatographic Data

| TLC | I-II on Cellulose (Avicel); III on Polyamide-6 | | |
|---|---|---|---|
| Rf Value | System I: 0.91 | II: 0.39 | III: 0.37 |
| Solvent system | I: BuOH(1) – Acetic Acid – Water (4 : 1 : 5) upper layer<br>II: Acetic Acid – Water (4 : 6)<br>III: Ethylacetate – Butanone(2) – Formic Acid – Water (5 : 3 : 1 : 1) | | |
| Saturated atmosphere | yes | Detection/Color | UV: deep purple;<br>UV, sprayed with Diphenylboric acid - β-aminomethylester (NA): yellow |
| Plate manufacturer | Schleicher & Schüll<br>Macherey & Nagel | Plate type/Product no. | Cellulose, Avicel F 1440<br>Polygram; 0.1 mm; Polyamide-TLC 6 |

| GLC | underivatized not possible |
|---|---|

| HPLC | | | |
|---|---|---|---|
| Retention time | 34.8 min | | |
| Column | Knauer, Berlin<br>250 × 4 mm | Stationary phase | Spherisorb ODS II, 5 µm<br>(Octadecylsilan II) |
| Mobile phase | A: MeOH – Acetic Acid (95 : 5)<br>B: Water – Acetic Acid (95 : 5) | Flow rate | 1.0 ml/min<br>Gradient; linear<br>0–5 min 30A – 70B<br>5–35 min 70A – 30B<br>35–40 min 70A – 30B |
| Column temp. | ambient temperature, ≈ 25 °C | Pressure | – |
| Detector | Waters 990<br>Photodioden-Array-Detector | Sample solvent | MeOH |
| Sample size | 10 µl | Sample conc. | – |
| Chromatograph type and manufacturer | | Waters Multisolvent Delivery System | |

9. Remarks

The substance could be isolated just in small amounts. For structure elucidation only NMR spectroscopic techniques were used, since they allow a subsequent recovery.

10. References


[1] Geiger, H., Stein, W., Mues, R. and Zinsmeister, H. D.: Bryoflavone and Heterobryoflavone, Two New Isoflavone- Flavone Dimers from Bryum capillare. *Z. Naturforsch. 42c*, 1987, pp 863–7 *(name, $^{13}$C-NMR)*

[2] Geiger, H. and Quinn, C.: Biflavonoids. In ‚The Flavonoids', Harborne, J.B. (Ed.), Chapman and Hall, London, 1988 *(name, occurrence)*

[3] Chari, V. M., Ilyas M., Wagner, H., Neszmélyi, A., Chen, F.-C., Chen, L.-K., Lin, Y.-C. and Lin, Y.-M.: $^{13}$C-NMR Spectroscopy of Biflavonoids. *Phytochemistry 16*, 1977, pp. 1273–8 *($^{13}$C-NMR)*

[4] Markham, K.R. and Chari, V.M.: Carbon-13 NMR Spectroscopy of Flavonoids. In 'The Flavonoids', Harborne, J. B. and Mabry, T. J. (Eds.), Chapman and Hall London, 1982 *($^{13}$C-NMR)*

[5] Mabry, T. J., Markham, K. R. and Thomas, M. B.: The Systematic Identification of Flavonoids. Springer Verlag, 1970 *(UV spectroscopy)*

# Camphene

*O. Vostrowsky*

## 1. Name of Compound

Common name
**Camphene** (+)-, (−)- and (±)-camphene

Synonyms
(+): Austracamphene (old) [1]
(−): Terecamphene (old) [1]

Systematic name
2,2-Dimethyl-3-methylenenorbornane
2,2-Dimethyl-3-methylenebicyclo[1.2.2]heptane

Substance
Terpene

Subgroup
Monoterpene hydrocarbon

CAS registry number and other numbers
[79-92-5]; Merck Index 10, 1708; RTECS EX1055000 BRN 1903765
(+): [5794-03-6]; Merck Index 11, 1736 (+): BRN 2323386
(−): [5794-04-7] (−): BRN 2204256

## 2. Formulas and Molecular Weight

Molecular formular
$C_{10}H_{16}$

Molecular weight
136.24

Structural formula


## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{55}$ 0.8412<br>(+): $d_4^{50}$ 0.8450<br>(±): $d_4^{50}$ 0.8410 g/cm$^3$ [1] | Iodine value<br>not available |
| Melting Point mp<br>(+): 52.5, 45-46<br>(−): 49.2-49.6<br>(±): 47.1 °C [1] | (−): $n_D^{54}$ 1.4564 [1]<br>(−): $n_D^{55}$ 1.4562 [1]<br>(±): $n_D^{55}$ 1.4562 [1] | Acid value<br>not applicable |
| Boiling point bp<br>(+): 158/760, 52/17<br>(−): 91.5/100, 157.8/743<br>79-80/58 °C/Torr [1] | $[\alpha]_D^{20}$: +112 (2,benzene) [1]<br>+106.8 (44,$CHCl_3$) [1]<br>−106.2 (40,benzene) [1]<br>$[\alpha]_D^{19}$: −104.7 (7,EtOH) [1] | Saponification value<br>not applicable |
| Flash point<br>(+): 36 °C<br>(−): 34 °C | Soluble in<br>ether, hydrocarbons, chloroform, benzene, ethanol etc. | |
| Color<br>colorless | Odor | |

4. Occurrence

| |
|---|
| Found in many essential oils; both optically active forms occur in plants |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology<br>RTECS EX 1055000<br>Schweizer Giftklasse 3 | Hazard labeling | Xn; R: 10-20/21 S: 16-22-24/25<br>Flammable. Harmful by inhalation and in contact with skin |
| | $LD_{50}$ | not available |
| Waste disposal procedures<br>by combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| GGVE/GGVS: 3/31c; IMDG: 3.3/III UN 2319; PAX: 309;<br>RID/ADR: 3/31c; IATA/ICAO: 3 UN 2319; CAO: 310 |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | m/z 93 | Molecular ion | m/z 136 (136.12520) |
| Ionization energy | 70 eV | Ion source temp. | 120 °C |
| Acceleration voltage | 4000 V | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | – |
| Spectrometer type and manufacturer | | Varian MAT 311A | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3074, 2970, 2886, 1660, 1464, 1379, 1366, 1157, 1110, 882 $cm^{-1}$ | | | |
| Sample preparation | GC-FTIR gas phase | Resolution | 2 $cm^{-1}$ |
| Spectrometer type and manufacturer | | Bruker IFS 48 | |

| NMR | assigned by 2D spectrum | Nucleus | $^{1}H$ |
|---|---|---|---|

Chemical Shifts

1.02 (s, 3H, $H_3C$-9), 1.05 (s, 3H, $H_3C$-10),
1.20 (d, 1H, *H*C-7 anti), 1.20–1.30 (m, 1H, *H*C-6 endo),
1.35–1.45 (m, 1H, *H*C-5 endo), 1.60–1.70 (m, 1H, *H*C-6 exo),
1.65–1.70 (m, 1H, *H*C-5 exo), 1.65–1.75 (m, 1H, *H*C-7 syn),
1.89 (mc, 1H, *H*C-4), 2.66 (mc, 1H, *H*C-1),
4.47 (s, 1H, *H*C=-8 syn), 4.72 (s, 1H, *H*C=-8 anti) ppm

| Frequency | 400 MHz | Solvent | $CDCl_3$ |
|---|---|---|---|
| Standard | TMS | Sample temp. | 20 °C |
| Spectrometer type and manufacturer | | JEOL JNM-GX 400 | |

| NMR | | Nucleus | $^{13}C$ |
|---|---|---|---|

Chemical Shifts

| | | | |
|---|---|---|---|
| 23.8 (t, C-5); | 25.8 (qu, C-9); | 28.9 (t, C-6); | 29.4 (qu, C-10); |
| 37.4 (t, C-7); | 41.8 (s, C-3); | 46.9 (d, C-1); | 48.1 (d, C-4); |
| 99.0 (t, C-8); | 166.3 (s, C-2) ppm | | |

| Frequency | 100 MHz | Solvent | $CDCl_3$ |
|---|---|---|---|
| Standard | TMS | Sample temp. | 20 °C |
| Spectrometer type and manufacturer | | JEOL JNM GX400 | |

| UV | not applicable |
|---|---|

## 8. Chromatographic Data

| | |
|---|---|
| TLC | not available |

| GLC | | | |
|---|---|---|---|
| Retention time | Kovats RI | a) $RI_{SE54}$ 948 | b) $RI_{CW20M}$ 1078 |
| Column type | fused silica capillary | Column length | a) 25 m b) 25 m |
| Column packing | coating a) SE54 b) CW20M | Column temp. | 4 min 60; 60–220 °C; 3 °/min |
| Injector port temp. | 220 °C | Carrier gas | 1 ml/min (22 cm/sec) $N_2$ |
| Detector temp. | 240 °C | Sample solvent | n-$C_6H_{14}$ |
| Sample size | 1 µl/Split | Sample conc. | 5 % |
| Chromatograph type and manufacturer | | HP 5890A FID | |

| HPLC | see ref. [4] |
|---|---|

## 9. Remarks

Commercially camphene is obtained by acid-catalyzed rearrangement of pinene, recognized many years prior to the isolation from natural sources.

## 10. References


[1] *Beilsteins Handbuch der Organischen Chemie* (Beilstein Institut), Vol V, 4th Ed., H pp. 156–162 (1922), EI pp. 82–85 (1930), EII pp. 105–108 (1943), EIII pp. 380–387 (1963), EIV pp. 461–464 (1976)

[2] Karrer, W., *Konstitution und Vorkommen der organischen Pflanzenstoffe*, Birkhäuser Verlag, Basel 1958, ff, No.

[3] Versatile chiral synthon:
a) Buchbauer, G. et al., *Monatsh. Chem. 115* (1984) 509
b) Bestmann, H.J., Röder, Th., *Angew. Chem. 95* (1983) 812

Additional analytical data and safety instructions are given in:

[4] Kubeczka, K.-H., in: Schreier, P. (Ed.), *Flavour '81*, Walter de Gruyter & Co, Berlin-New York 1981

[5] FT-IR: The Aldrich Library of FT-IR Spectra, 1st Ed., Vol. 3, 66B

[6] NMR: a) The Aldrich Library of NMR Spectra, 2nd Ed., Vol 1, 48C
b) Bremser, W., Ernst, L., Franke, B. (Eds.), *Carbon-13 NMR Spectral Data*, Verlag Chemie, Weinheim, New York 1978

[7] The Sigma-Aldrich Library of Chemical Safety Data, 2nd Ed., 261C, D

# Candelabrone

*S. Cañigueral*

## 1. Name of Compound

Common name
**Candelabrone**

Synonyms

Systematic name
(+)-(5*R*,10*S*)-11,12,14-Trihydroxy-8,11,13-abietatriene-3,7-dione

Substance
Diterpene

Subgroup
Abietane diterpene

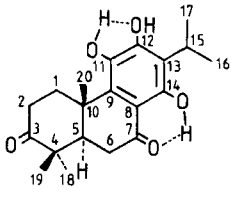
CAS registry number and other numbers
[113545-42-9]

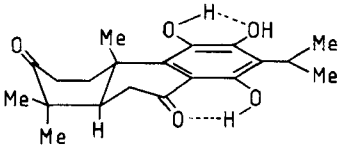
## 2. Formulas and Molecular Weight

Molecular formular
$C_{20}H_{26}O_5$

Molecular weight
346.1773

Structural formula





The proposed conformation

## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>224–226 °C<br>uncorr. (MeOH) | $n_D^{20}$<br>not applicable | Acid value<br>not available |
| Boiling point bp<br>not available | $[\alpha]_D^{30}$<br>+160 ° | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>chloroform, methanol | |
| Color<br>yellow | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Salvia candelabrum Boiss. (Lamiaceae) Leaves |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| not available | $LD_{50}$ | not available |
| Waste disposal procedures<br>not usual | | |

6. Transportation and Storage Instructions

| |
|---|
| Store as solid. Unstable in solution. |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | [M$^+$] 346 | Molecular ion | [M$^+$] 346 |
| Ionization energy | 70 eV | Ion source temp. | 200 °C |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | $2.4 \times 10^{-3}$ s/decade |
| Spectrometer type and manufacturer | | Hewlett Packard 5985B | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3600–3000 (OH's), 1690 (C=O at C-3), 1600 (chelated C=O at C-7) cm$^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | Perkin-Elmer | |

| NMR | | Nucleus | $^1$H |
|---|---|---|---|
| Chemical Shifts<br>1.13 (s, H-18 and H-19); 1.35 (d, J=7 Hz, H-16 and H-17);<br>1.40 (s, H-20); 1.9–2.4 (m, H-1α);<br>2.5–2.9 (m, H-2α, H-2β, H-5, H-6α and H-6β);<br>3–3.5 (m, H-1β and H-15); 5.05 (s, $C_{12}$-O*H*);<br>5.95 (s, $C_{11}$-O*H*); 13.40 (s, $C_{14}$-O*H*) ppm | | | |
| Frequency | 80 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 27 °C |
| Spectrometer type and manufacturer | | Bruker WP80SY | |

| NMR | | Nucleus | $^{1}H$ |
|---|---|---|---|
| Chemical Shifts<br>1.150 (s, $J_{15,16} = J_{15,17} = 7.1$ Hz,H-18,H-19);<br>1.430 (br s, $J_{1\alpha,20} < 0.1$ Hz, H-20);<br>2.421 (m, $J_{1\beta,2\beta} = 5.3$, H-5);<br>2.598 (m, $J_{1\alpha,2\beta} = 8.6$, H-2α);<br>2.780 (dd, $J_{5,6\alpha} = 2.9$ Hz, H-6β);<br>3.450 (sept, $J_{5,6\beta} = 14.6$ Hz, H-15) ppm | | 1.290 (d, $J_{6\alpha,6\beta} = -16.6$ Hz,H-16,H-17);<br>2.012 (dt, $J_{1\alpha,1\beta} = -13.9$ Hz, H-1α);<br>2.452 (m, $J_{2\alpha,2\beta} = -14.2$ Hz, H-6α);<br>2.637 (m, $J_{1\beta,2\alpha} = 7.0$ Hz, H-2β);<br>3.398 (m, $J_{1\alpha,2\alpha} = 8.3$, H-1β); | |
| Frequency | 400 MHz | Solvent | $CD_3OD$ |
| Standard | TMS | Sample temp. | 27 °C |
| Spectrometer type and manufacturer | | Bruker AM400 | |

| NMR | | | Nucleus | $^{13}C$ |
|---|---|---|---|---|
| Chemical Shifts | | | | |
| 18.93 (q, C-20); | 21.01 (q, C-16); | 21.03 (q, C-17); | | 21.77 (q, C-19); |
| 26.28 (d, C-15); | 27.79 (q, C-18); | 35.94 (t, C-2); | | 37.23 (t, C-6); |
| 37.79 (t, C-1); | 40.67 (s, C-10); | 48.61 (s, C-4); | | 50.90 (d, C-5); |
| 109.69 (s, C-8); | 120.94 (s, C-13); | 136.66 (s, C-11); | | 139.14 (s, C-9); |
| 156.80 (s, C-14); | 160.96 (s, C-12); | 205.17 (s, C-7); | | 219.46 (s, C-3) ppm |
| Frequency | 100 MHz | | Solvent | $CD_3OD$ |
| Standard | TMS | | Sample temp. | 27 °C |
| Spectrometer type and manufacturer | | | Bruker AM400 | |

| UV * | | | |
|---|---|---|---|
| $I_{max}$ | 289 and 352nm | $\varepsilon_{max}$ | 3.79 und 3.6 |
| Solvent | Methanol | | |
| Sample conc. | – | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Perkin Elmer 550 S | |

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.31 (1) and 0.31 (2) | | |
| Solvent | MeOH | Solvent system | 1) Toluene(40):MeOH(3):AcOH(2)<br>2) Hexane(40):*$CHCl_3$(40):MeOH(3)<br>* Stabilized with Ethanol |
| Saturated atmosphere | 20–25 °C | Detection/Color | UV 366 nm yellow |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel $60F_{254}$; Merck 5554 |

| GLC | not available |
|---|---|

| HPLC | | | |
|---|---|---|---|
| Retention time | 12.71 min | | |
| Column | 125 × 4.6 mm | Stationary phase | Sphersorb ODS-2 (C-18), 5 μm |
| Mobile phase | A = ACN B = $H_2O$(19):AcOH(1)<br>t=0 min 25% A, t=1 min rate=2,5% A/min | Flow rate | 1.8 ml/min |
| Column temp. | 35 °C | Pressure | – |
| Detector | Perkin Elmer LC-85B<br>UV at 254 nm | Sample solvent | MeOH |
| Sample size | – | Sample conc. | – |
| Chromatograph type and manufacturer | | Perkin Elmer Series 2 | |

## 9. Remarks

ORD (MeOH, sample conc. 0.0436 g/100 ml, 30 °C):

| | | | | |
|---|---|---|---|---|
| [α]: | +160° | +984° | 0° | −6979° |
| λ (nm): | 589 | 390 | 377 | 325 |

## 10. References


[1] Cañigueral, S., Iglesias, J., Sánchez-Ferrando, F., Virgili, A., *Phytochemistry*, 1988. *27*, 221 *(Isolation, Structure determination, Spectroscopy)*

[2] Cañigueral, S., *Contribución al estudio de los polifenoles de especies del género Salvia L.*. Barcelona: Universidad de Barcelona, 1986; Dissertation *(Isolation, Structure, TLC)*

[3] Manso, D. *Contribución al estudio fitoquimico de Salvia candelabrum Boiss.*. Tesina de Licenciatura, Barcelona: Universidad de Barcelona, 1986 *(HPLC)*

# Cardanol, Diene

*G. Skopp*

## 1. Name of Compound

Common name

**Cardanol, Diene**

Synonyms

Cardanol-diolefin, Cardanol 15 : 2 (n-4)

Systematic name

(Z,Z)-3-n-(8,11-Pentadecadienyl)-phenol

| Substance | Subgroup |
|---|---|
| Phenol | Long chain phenol |

CAS registry number and other numbers

[51546-63-5] BRN 2655043

## 2. Formulas and Molecular Weight

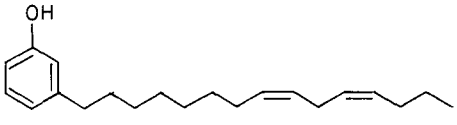
Molecular formular

$C_{31}H_{32}O$

Molecular weight

300.276

Structural formula




## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| liquid | not available | not available |
| **Melting Point mp** | $n_D^{20}$ | **Acid value** |
| not applicable | not available | not applicable |
| **Boiling point bp** | $[\alpha]_D^{20}$ | **Saponification value** |
| not available | not applicable | not applicable |
| **Flash point** | **Soluble in** | |
| not available | acetone, ether | |
| **Color** | **Odor** | |
| colorless | like light petroleum | |

## 4. Occurrence

| |
|---|
| Anacardium occidentale L. (Anacardiaceae) [2]<br>Schinus terebinthifolius Raddi (Anacardiaceae) [1] |

## 5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | irritant, sensitizing [3, 4] |
| – | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

## 6. Transportation and Storage Instructions

| |
|---|
| Storage in light resistant containers under nitrogen. |

## 7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 108 mu | Molecular ion | 300 mu |
| Ionization energy | 100 eV | Ion source temp. | 100 °C |
| Acceleration voltage | 3 kV | Emission current | about 1 mA |
| Resolution | 1000 | Scan rate | – s/decade |
| Spectrometer type and manufacturer | | Varian MAT 311 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3340 (OH), 3005 (C=C), 2930 and 2860 (H-C), 1460 ($CH_2$), 780 and 695 (m-subst. arom. ring) $cm^{-1}$ | | | |
| Sample preparation | film | Resolution | – |
| Spectrometer type and manufacturer | | Perkin-Elmer spectrometer 521 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>7.03 (dd, J=0.5, 7.5 Hz, 5-H);<br>6.46 (dt, J=0.5, 1.5, 2.4 Hz, 2-H);<br>3.90 (s, br., O*H*);<br>2.87 (t, 10'-H);<br>2.05 (td, 7', 13'-H);<br>1.34 (sext., 14'-H);<br>0.88 (t, 15'-H) ppm | | 6.70 (dtd, J=0.9, 1.5, 7.5 Hz, 4-H);<br>6.40 (ddd, J=0.9, 2.4, 8.1 Hz, 6-H);<br>5.41-5.52 (m, 8',9',11',12'-H);<br>2.46 (t, J=7.7 Hz, 1'-H);<br>1.50-1.55 (m, J=7.7 Hz, 2'-H);<br>1.18-1.28 (s, br., 3',4',5',6'-H); | |
| Frequency | 500 MHz | Solvent | $C_6D_6$ |
| Standard | TMS | Sample temp. | 21 °C |
| Spectrometer type and manufacturer | | Bruker AM 500 | |

| UV | not available |
|---|---|

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | a) 0.65, b) 0.65 | | |
| Solvent | – | Solvent system | a) light petroleum (40-60 °C) : ether 7 : 3,<br>b) acetonitrile : water 9 : 1 |
| Saturated atmosphere | yes | Detection/Color | UV, fast blue salt B 0.5%: yellow |
| Plate manufacturer | Merck | Plate type/Product no. | a) Silica gel 60 $F_{254}$,<br>b) HPTLC RP-18 $F_{254}$ |

| GLC | not available |
|---|---|

| HPLC | see ref. [5] |
|---|---|

9. Remarks

The substance could be isolated just in small amounts. For structure elucidation NMR spectroscopy techniques were preferred.

10. References


[1] Skopp, G., Über phenolische Inhaltsstoffe aus Schinus terebinthifolius Raddi – n-Alkylphenole und Biflavonoide – Diss., 1986 *(name, MS, IR, $^1$H-NMR)*

[2] Symes, W.F., Dawson, C.R., *J. Am. Chem. Soc. 75*, 1953, pp. 4952-7 *(occurrence)*

[3] Lewin, L., Gifte und Vergiftungen, Haug Verlag, Ulm, 1962 *(inflammatory properties)*

[4] Morton, F.J., *Econ. Bot. 32(4)*, 1978, pp. 353-9 *(impact on environment)*

[5] Tyman J.H.P., Tychopoulos, V., Chan, P., *J. Chrom. 303*, 1984, pp. 137-50 *(high performance liquid chromatography)*

# Cardanol, Monoene

*G. Skopp*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Cardanol, Monoene** | |
| Synonyms<br>Cardanol-monoolefin, Cardanol 15:1 (n-7) | |
| Systematic name<br>(Z)-3-n-(8-Pentadecenyl)-phenol | |
| Substance<br>Phenol | Subgroup<br>Long chain phenol |
| CAS registry number and other numbers<br>[501-26-8] | BRN 1977267 |

## 2. Formulas and Molecular Weight

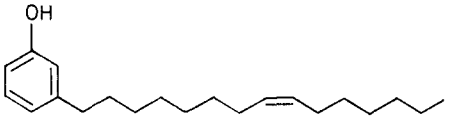
Molecular formular

$C_{21}H_{34}O$

Molecular weight

302.292

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>liquid | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>not applicable | $n_D^{20}$<br>not available | Acid value<br>not applicable |
| Boiling point bp<br>237-242 °C | $[\alpha]_D^{20}$<br>not applicable | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>acetone, ether | |
| Color<br>colorless | Odor<br>like light petroleum | |

4. Occurrence

| |
|---|
| Rhus typhina L. (Anacardiaceae) [2], Schinus terebinthifolius Raddi (Anacardiaceae) [5], Semecarpus vitiensis Engl. (Anacardiaceae) [3], Knema elegans Warb. (Myristicaceae) [4] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | irritant, sensitizing [5] |
| – | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage in light resistant containers under nitrogen. |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 108 mu | Molecular ion | 302 mu |
| Ionization energy | 100 eV | Ion source temp. | 75 °C |
| Acceleration voltage | 3 kV | Emission current | about 1 mA |
| Resolution | 1000 | Scan rate | – |
| Spectrometer type and manufacturer | | Varian MAT 311 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3330 (OH), 3005 (C=C), 2930 and 2860 (H-C), 1460 ($CH_2$), 780 and 695 (m-subst. arom. ring) $cm^{-1}$ | | | |
| Sample preparation | film | Resolution | – |
| Spectrometer type and manufacturer | | Perkin-Elmer spectrometer 521 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>7.03 (dd, J=0.5, 7.5 Hz, 5-H);<br>6.46 (dt, J=0.5, 1.5, 2.4 Hz, 2-H);<br>3.90 (s, br., O*H*);<br>2.45 (t, J=7.0 Hz, 1'-H);<br>1.50-1.56 (m, J=7.0 Hz, 2'-H);<br>0.89 (t, 15'-H) ppm | | 6.70 (dtd, J=0.9, 1.5, 7.5 Hz, 4-H);<br>6.40 (ddd, J=0.9, 2.4, 8.1 Hz, 6-H);<br>5.49 (m, J=-1.4, 6.8, 10.6 Hz, 8',9'-H);<br>2.08 (m, J=−1.4, −0.2, 6.8 Hz, 7', 10'-H);<br>1.23-1.40 (s, br, 3',4',5',6',11',12',13',14'-H); | |
| Frequency | 500 MHz | Solvent | $C_6D_6$ |
| Standard | TMS | Sample temp. | 21 °C |
| Spectrometer type and manufacturer | | Bruker AM 500 | |

| UV | see ref. [3] |
|---|---|

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | a) 0.65, b) 0.48 | | |
| Solvent | — | Solvent system | a) light petroleum (40-60 °C) : ether 7 : 3,<br>b) acetonitrile : water 9 : 1 |
| Saturated atmosphere | yes | Detection/Color | UV, fast blue salt B 0.5 %<br>yellow |
| Plate manufacturer | Merck | Plate type/Product no. | a) Silica gel 60 $F_{254}$,<br>b) HPTLC RP-18 $F_{254}$ |

| GLC | not available |
|---|---|

| HPLC | see ref. [6] |
|---|---|

## 9. Remarks

10. References


[1] Skopp, G., Über phenolische Inhaltsstoffe aus Schinus terebinthifolius Raddi – n-Alkylphenole und Biflavonoide – Diss., 1986 *(name, MS, IR, $^1$H-NMR)*

[2] Bestmann, H.-J., Classen B., Kobold, U., Vostrowsky, O., Klingauf, F., *Phytochemistry 27/1*, 1988, 85-90 *(occurrence)*

[3] Pramono, S., Gleye, J., Moulis, C., Debray, M., Stanislas, E., *Plantes medicinales et phytotherapie 19*, 1985, pp. 159-62 *(occurrence, UV, $^1$H-NMR)*

[4] Spencer, G.F., Tjarks, L.W., Kleiman, R., *J. Nat. Products 43*, 1980, pp. 724-30 *(occurrence)*

[5] Stahl, E., Keller, K., Blinn, C., *Planta medica 48*, 1983, pp. 5-9 *(occurrence, TLC, irritating effect)*

[6] Tyman J.H.P., Tychopoulos, V., Chan, P., *J. Chrom. 303*, 1984, pp. 137-50 *(high performance liquid chromatography)*

# Cardanol, saturated

*G. Skopp*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Cardanol, saturated** | |
| Synonyms<br>Anacardol, Cardanol 15 : 0 | |
| Systematic name<br>3-n-Pentadecylphenol | |
| Substance<br>Phenol | Subgroup<br>Long chain phenol |
| CAS registry number and other numbers<br>[501-24-6] | BRN 2053142 |

## 2. Formulas and Molecular Weight

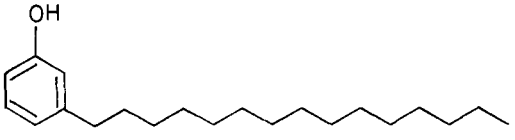
Molecular formular

$C_{21}H_{36}O$

Molecular weight

304.308

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline | $d_4^{20}$<br>not applicable | Iodine value<br>not applilable |
| Melting Point mp<br>50–53 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>190–195 °C (1 mm Hg) | $[\alpha]_D^{20}$<br>not applicable | Saponification value<br>not applicable |
| Flash point<br>>110 °C | Soluble in<br>acetone, ether | |
| Color<br>white | Odor<br>like light petroleum | |

## 4. Occurrence

| |
|---|
| Schinus terebinthifolius Raddi (Anacardiaceae) [1]<br>Anacardium occidentale L. (Anacardiaceae) [1], Schistochila appendiculata [3] |

## 5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | irritant, sensitizing [4, 5, 6] |
| – | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

## 6. Transportation and Storage Instructions

| |
|---|
| Storage in tight, light resistant containers. |

## 7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 108 mu | Molecular ion | 304 mu |
| Ionization energy | 100 eV | Ion source temp. | 105 °C |
| Acceleration voltage | 3 kV | Emission current | about 1 mA |
| Resolution | 1000 | Scan rate | – |
| Spectrometer type and manufacturer | | Varian MAT 311 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3340 (OH), 2920 and 2860 (H-C), 1465 ($CH_2$), 1380 ($CH_3$), 785 and 695 (m-substitution) $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | Perkin-Elmer spectrometer | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>7.14 (dd, J=8, 1 Hz, 5-H); 6.77 (m, 4-H); 6.60–6.70 (m, 6-H);<br>6.67 (d, J=1 Hz, 2-H); 4.70 (s, br., *OH*); 2.55 (t, 1′-H);<br>1.56–1.61 (m, 2′-H); 1.21–1.35 (s, br., 3′-14′-H);<br>0.88 (t, distorted, 15′-H) ppm | | | |
| Frequency | 250 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 21 °C |
| Spectrometer type and manufacturer | | Bruker WM 250 | |

| UV | see ref. [7] |
|---|---|

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | a) 0.65, b) 0.42 | | |
| Solvent | – | Solvent system | a) light petroleum (40–60 °C) : ether 7 : 3,<br>b) acetonitrile : water 9 : 1 |
| Saturated atmosphere | yes | Detection/Color | UV, fast blue salt B 0.5%: yellow |
| Plate manufacturer | Merck | Plate type/Product no. | a) Silica gel 60 $F_{254}$,<br>b) HPTLC RP-18 $F_{254}$ |

| GLC | not available |
|---|---|

| HPLC | see ref. [8] |
|---|---|

## 9. Remarks

10. References


[1] Skopp, G., Opferkuch, H.-D., Schwenker, G., *Z. Naturforsch. 42c*, 1987, 7–16 *(name, $^1$H-NMR)*

[2] Symes, W.F., Dawson, C.R., *J. Am. Chem. Soc. 75*, 1953, pp. 4952–7 *(occurrence, chromatographic separation)*

[3] Asakawa, Y., Masuya, T., Tori, M., Campbell, E.O., *Phytochemistry 26(3)*, 1987, pp. 735–8 *(occurrence)*

[4] Lewin, L., Gifte und Vergiftungen, Haug Verlag, Ulm, 1962 *(inflammatory properties)*

[5] Morton, F.J., *Econ. Bot. 32(4)*, 1978, pp. 353–9 *(impact on environment)*

[6] Roth, L., Daunderer, M., Kormann, K., Giftpflanzen, Pflanzengifte, Ecomed Verlag, München, 1984 *(irritation symptom)*

[7] Tyman, J.H.P., *J. Chrom. 166*, 1978, pp. 159–172 *(TLC, densitometry, UV-spectrophotometry)*

[8] Tyman, J.H.P., Tychopoulos, V., Chan, P., *J. Chrom. 303*, 1984, pp. 137–50 *(high performance liquid chromatography)*

# Cardanol, Triene

*G. Skopp*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Cardanol, Triene** | |
| Synonyms<br>Cardanol-triolefin, Cardanol 15 : 3 (n-1) | |
| Systematic name<br>(Z,Z)-3-n-(8,11,14-Pentadecatrienyl)-phenol | |
| Substance<br>Phenol | Subgroup<br>Long chain phenol |
| CAS registry number and other numbers<br>[79353-39-2] | BRN 3143188 |

## 2. Formulas and Molecular Weight

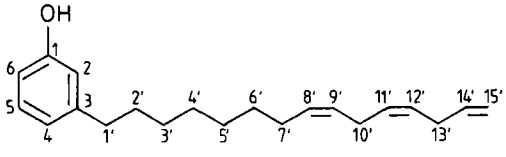
Molecular formular

$C_{21}H_{30}O$

Molecular weight

398.260

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>liquid | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>not applicable | $n_D^{20}$<br>not available | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{20}$<br>not applicable | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>acetone, ether | |
| Color<br>colorless | Odor<br>like light petroleum | |

4. Occurrence

| |
|---|
| Anacardium occidentale L. (Anacardiaceae) [2]<br>Schinus terebinthifolius Raddi (Anacardiaceae) [1] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | irritant, sensitizing [3] |
| $ID_{50}$ = 32 µg/ear | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage in light resistant containers under nitrogen. |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 107 mu | Molecular ion | 298 mu |
| Ionization energy | 100 eV | Ion source temp. | 80 °C |
| Acceleration voltage | 3 kV | Emission current | about 1 mA |
| Resolution | 1000 | Scan rate | – s/decade |
| Spectrometer type and manufacturer | | Varian MAT 311 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3300 (OH), 3080 ($CH_2$=), 3010 (RCH=), 2930 and 2860 (H-C), 1460 ($CH_2$), 780 and 695 (m-subst. arom. ring) $cm^{-1}$ | | | |
| Sample preparation | film | Resolution | – |
| Spectrometer type and manufacturer | | Perkin-Elmer spectrometer 521 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>7.03 (dd, J=0.5, 7.5 Hz, 5-H);<br>6.46 (dt, J=0.5, 1.5, 2.4 Hz, 2-H);<br>3.90 (s, br., O*H*);<br>5.41-5.52 (m, 8',9',11',12'-H);<br>4.98 (tdd, J=1.5, 1.8, 10.1 Hz, 15'-H);<br>2.76 (t, 13'-H);<br>2.04 (td, 7'-H);<br>1.16-1.33 (s, br., 3', 4', 5', 6'-H) ppm | | 6.70 (dtd, J=0.9, 1.5, 7.5 Hz, 4-H);<br>6.40 (ddd, J=0.9, 2.4, 8.1 Hz, 6-H);<br>5.77 (tdd, J=6.1, 10.1, 17.1 Hz, 14'-H);<br>5.07 (tdd, J=1.5, 1.8, 17.1 Hz, 15'-H);<br>2.81 (t, 10'-H);<br>2.45 (t, J=7.7 Hz, 1'-H);<br>1.50-1.55 (m, 2'-H); | |
| Frequency | 500 MHz | Solvent | $C_6D_6$ |
| Standard | TMS | Sample temp. | 21 °C |
| Spectrometer type and manufacturer | | Bruker AM 500 | |

| UV | not available |
|---|---|

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | a) 0.65, b) 0.71 | | |
| Solvent | – | Solvent system | a) light petroleum (40-60 °C : ether 7 : 3,<br>b) acetonitrile : water 9 : 1 |
| Saturated atmosphere | yes | Detection/Color | UV, fast blue salt B 0.5%/ yellow |
| Plate manufacturer | Merck | Plate type/Product no. | a) Silica gel 60 $F_{254}$,<br>b) HPTLC RP-18 $F_{254}$ |

| GLC | not available |
|---|---|

| HPLC | see ref. [4] |
|---|---|

## 9. Remarks

10. References


[1] Skopp, G., Über phenolische Inhaltsstoffe aus Schinus terebinthifolius Raddi – n-Alkylphenole und Biflavonoide – Diss., 1986 *(name, occurrence)*

[2] Symes, W.F., Dawson, C.R., *J. Am. Chem. Soc. 75*, 1953, pp. 4952–7 *(occurrence)*

[3] Evans, F.J., Schmidt, R.J., *Planta medica 38*, 1980, 289–316 *(contact dermatitis)*

[4] Tyman J.H.P., Tychopoulos, V., Chan, P., *J. Chrom. 303*, 1984, pp. 137–50 *(high performance liquid chromatography)*

# Cardol, Diene

*G. Skopp*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Cardol, Diene** | |
| Synonyms<br>Cardol-diolefin, Cardol 15 : 2 (n-4), Pentadecadienylresorcinol | |
| Systematic name<br>(Z,Z)-5-n-(8,11-Pentadecadienyl)-1,3-benzenediol | |
| Substance<br>Phenol | Subgroup<br>Long chain phenol |
| CAS registry number and other numbers<br>[79473-25-9] | BRN 5062771 |

## 2. Formulas and Molecular Weight

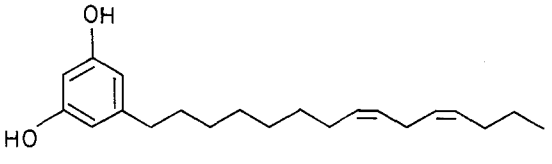
Molecular formular

$C_{21}H_{32}O_2$

Molecular weight

316.275

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>liquid | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>not applicable | $n_D^{20}$<br>not available | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{20}$<br>not applicable | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>acetone, ether | |
| Color<br>colorless | Odor<br>like petroleum ether, repulsive | |

4. Occurrence

| |
|---|
| Anacardium occidentale L. (Anacardiaceae) [1]<br>Schinus terebinthifolius Raddi (Anacardiaceae) [2] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | irritant, sensitizing [7] |
| – | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage in light resistant containers under nitrogen |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 124 mu | Molecular ion | 316 mu |
| Ionization energy | 100 eV | Ion source temp. | 90 °C |
| Acceleration voltage | 3 kV | Emission current | about 1 mA |
| Resolution | 1000 | Scan rate | – s/decade |
| Spectrometer type and manufacturer | | Varian MAT 311 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3330 (OH), 2930 and 2860 (H-C), 1470 ($CH_2$), 840 and 695 (1,3,5-substitution) $cm^{-1}$ | | | |
| Sample preparation | film | Resolution | – |
| Spectrometer type and manufacturer | | Perkin-Elmer spectrometer 521 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>6.25 (d, J=2.5 Hz, 4,6-H);<br>4.67 (s, br., O*H*);<br>2.76 (td, 10′-H);<br>2.05 (7′,13′-H);<br>1.25–1.45 (3′,4′,5′,6′,14′-H); | | 6.17 (t, J=2.5 Hz, 2-H);<br>5.25–5.40 (m, 8′,9′,11′,12′-H);<br>2.49 (t, J=7.8 Hz, 1′-H);<br>1.50–1.65 (m, 2′-H);<br>0.92 (t, J=7.0 Hz, 15′-H) ppm | |
| Frequency | 250 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 21 °C |
| Spectrometer type and manufacturer | | Bruker WM 250 | |

| UV | not available |
|---|---|

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | a) 0.18, | b) 0.53 | |
| Solvent | – | Solvent system | a) light petroleum (40–60 °C: ether 7 : 3 (v/v), b) aceto-nitrile : water 8 : 2 (v/v) |
| Saturated atmosphere | yes | Detection/Color | UV, fast blue salt B 0.5%/red |
| Plate manufacturer | Merck | Plate type/Product no. | a) Silica gel 60 $F_{254}$,<br>b) HPTLC RP-18 $F_{254}$ |

| GLC | see ref. [9] |
|---|---|

| HPLC | see ref. [10] |
|---|---|

9. Remarks

| |
|---|

10. References


[1] Symes, W.F., Dawson, C.R., *Nature 171*, 1953, pp. 841-2 *(occurrence)*

[2] Skopp, G., Über phenolische Inhaltsstoffe aus Schinus terebinthifolius Raddi – n-Alkylphenole und Biflavonoide – Diss., 1986 *(name, occurrence)*

[3] List, P.H., Hörhammer, L., Hagers Handbuch der pharmazeutischen Praxis 4, Springer Verlag, Heidelberg, 1973 *(occurrence)*

[4] Cojocaru, M., Droby, S., Glotter, E., Goldmann, A., Gottlieb, H.E., Jacoby, B., Prusky, D., *Phytochemistry 25(5)*, 1986, pp. 1093-5 *(occurrence)*

[5] Barrero, A.F., Sanchez, J.F., Corrales, F., Rodrigues, I., *Phytochemistry 28(1)*, 1989, pp. 161-4 *(occurrence)*

[6] Morton, F.J., *Econ. Bot. 32(4)*, 1978, pp. 353-9 *(impact on environment)*

[7] Evans, F.J., Schmidt, R.J., *Planta medica 38*, 1980, 289-316 *(contact dermatitis)*

[8] Tyman, J.H.P., *J. Chrom. 166*, 1978, pp. 159-72 *(UV spectrophotometry)*

[9] Tyman, J.H.P., *J. Chrom. 138*, 1977, pp. 97-110 *(gas-liquid chromatography)*

[10] Tyman J.H.P., Tychopoulos, V., Chan, P., *J. Chrom. 303*, 1984, pp. 137-50 *(high performance liquid chromatography)*

# Cardol, Monoene

*G. Skopp*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Cardol, Monoene** | |
| Synonyms<br>Cardol-monoolefin, Cardol 15 : 1 (n-7), Pentadecenylresorcinol | |
| Systematic name<br>(Z)-5-n-(8-Pentadecenyl)-1,3-benzenediol | |
| Substance<br>Phenol | Subgroup<br>Long chain phenol |
| CAS registry number and other numbers<br>[22910-86-7] | BRN 1987389 |

## 2. Formulas and Molecular Weight

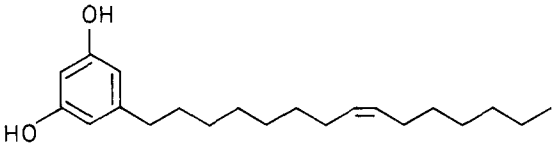
Molecular formular

$C_{21}H_{34}O_2$

Molecular weight

318.291

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>liquid | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>not applicable | $n_D^{20}$<br>not available | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{20}$<br>not applicable | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>acetone, ether | |
| Color<br>colorless | Odor<br>like petroleum ether, repulsive | |

4. Occurrence

| |
|---|
| Anacardium occidentale L. (Anacardiaceae) [1]; Schinus terebinthifolius Raddi (Anacardiaceae) [2]; Gingko biloba L. (gingkoaceae) [3]; Mangifera indica L. (Anacardiaceae) [4]; Ononis speciosa lag (Leguminosae) [5] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | irritant, sensitizing [7] |
| – | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage in light resistant containers under nitrogen. |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 124 mu | Molecular ion | 318 mu |
| Ionization energy | 100 eV | Ion source temp. | 115 °C |
| Acceleration voltage | 3 kV | Emission current | about 1 mA |
| Resolution | 1000 | Scan rate | – s/decade |
| Spectrometer type and manufacturer | | Varian MAT 311 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3340 (OH), 2930 and 2860 (H-C), 1470 ($CH_2$), 840 and 690 (1,3,5-substitution) $cm^{-1}$ | | | |
| Sample preparation | film | Resolution | – |
| Spectrometer type and manufacturer | | Perkin-Elmer spectrometer 521 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>6.25 (d, J=2.5 Hz, 4,6-H);<br>5.35 (8′,9′-H);<br>1.52–1.63 (2′-H);<br>0.88 (t, J=7.0 Hz, 15′-H) ppm | | 6.17 (t, J=2.5 Hz, 2-H);<br>2.49 (t, 1′-H, J=7.8 Hz);<br>1.24–1.57 (3′,4′,5′,6,′11′,12′,13′,14′-H); | 4.67 (s, br., O*H*);<br>2.02 (7′,10′-H); |
| Frequency | 250 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 21 °C |
| Spectrometer type and manufacturer | | Bruker WM 250 | |

| UV | not available |
|---|---|

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | a) 0.18, | b) 0.44 | |
| Solvent | – | Solvent system | a) light petroleum (40–60 °C: ether 7 : 3 (v/v), b) acetonitrile : water 8 : 2 (v/v) |
| Saturated atmosphere | yes | Detection/Color | UV, fast blue salt B 0.5%/red |
| Plate manufacturer | Merck | Plate type/Product no. | a) Silica gel 60 $F_{254}$,<br>b) HPTLC RP-18 $F_{254}$ |

| GLC | see ref. [9] |
|---|---|

| HPLC | see ref. [10] |
|---|---|

9. Remarks

| |
|---|

## 10. References


[1] Symes, W.F., Dawson, C.R., *Nature 171*, 1953, pp. 841-2 *(occurrence)*

[2] Skopp, G., Über phenolische Inhaltsstoffe aus Schinus terebinthifolius Raddi – n-Alkylphenole und Biflavonoide – Diss., 1986 *(name, occurrence)*

[3] List, P.H., Hörhammer, L., Hagers Handbuch der pharmazeutischen Praxis 4, Springer Verlag, Heidelberg, 1973 *(occurrence)*

[4] Cojocaru, M., Droby, S., Glotter, E., Goldmann, A., Gottlieb, H.E., Jacoby, B., Prusky, D., *Phytochemistry 25(5)*, 1986, pp. 1093-5 *(occurrence)*

[5] Barrero, A.F., Sanchez, J.F., Corrales, F., Rodrigues, I., *Phytochemistry 28(1)*, 1989, pp. 161-4 *(occurrence)*

[6] Morton, F.J., *Econ. Bot. 32(4)*, 1978, pp. 353-9 *(impact on environment)*

[7] Evans, F.J., Schmidt, R.J., *Planta medica 38*, 1980, 289-316 *(contact dermatitis)*

[8] Tyman, J.H.P., *J. Chrom. 166*, 1978, pp. 159-72 *(UV spectrophotometry)*

[9] Tyman, J.H.P., *J. Chrom. 138*, 1977, pp. 97-110 *(gas-liquid chromatography)*

[10] Tyman J.H.P., Tychopoulos, V., Chan, P., *J. Chrom. 303*, 1984, pp. 137-50 *(high performance liquid chromatography)*

# Cardol, saturated

*G. Skopp*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Cardol, saturated** | |
| Synonyms<br>Cardol, Cardol 15 : 0, Pentadecylresorcinol | |
| Systematic name<br>5-n-Pentadecyl-1,3-benzenediol | |
| Substance<br>Phenol | Subgroup<br>Long chain phenol |
| CAS registry number and other numbers<br>[3158-56-3] | BRN 1982742 |

## 2. Formulas and Molecular Weight

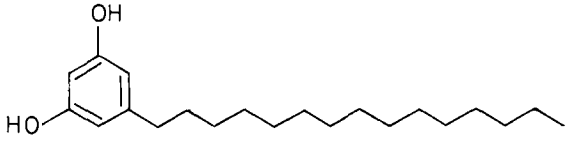
Molecular formular

$C_{21}H_{36}O_2$

Molecular weight

320.307

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>94-96 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20}$<br>not applicable | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>acetone, ether | |
| Color<br>white | Odor<br>like petroleum ether, repulsive | |

## 4. Occurrence

| |
|---|
| Anacardium occidentale L. (Anacardiaceae) [1]<br>Schinus terebinthifolius Raddi (Anacardiaceae) [2] |

## 5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | irritant, sensitizing [7] |
| – | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

## 6. Transportation and Storage Instructions

| |
|---|
| Storage in light resistant containers |

## 7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 124 mu | Molecular ion | 320 mu |
| Ionization energy | 100 eV | Ion source temp. | 95 °C |
| Acceleration voltage | 3 kV | Emission current | about 1 mA |
| Resolution | 1000 | Scan rate | – s/decade |
| Spectrometer type and manufacturer | | Varian MAT 311 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3330 (OH), 2920 and 2850 (H-C), 1470 ($CH_2$), 1380 ($CH_3$), 835 and 695 (1,3,5-substitution) $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | Perkin-Elmer spectrometer 521 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>6.24 (d, J=2.5 Hz, 4,6-H); 6.17 (t, J=2.5 Hz, 2-H); 4.67 (s, br, O*H*);<br>2.48 (t, 1''-H, J=7.8 Hz); 1.50–1.65 (m, 2'-H);<br>1.20–1.38 (s, br, 3',4',5',6,',7,',8',9',10',11',12',13',14'-H);<br>0.88 (t, J=7.0 Hz, 15'-H) ppm | | | |
| Frequency | 250 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 21 °C |
| Spectrometer type and manufacturer | | Bruker WM 250 | |

| UV | see ref. [8] |
|---|---|

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | a) 0.18, b) 0.36 | | |
| Solvent | – | Solvent system | a) light petroleum (40–60 °C : ether 7 : 3 (v/v)<br>b) acetonitrile : water 8 : 2 (v/v) |
| Saturated atmosphere | yes | Detection/Color | UV, fast blue salt B 0.5%/red |
| Plate manufacturer | Merck | Plate type/Product no. | a) Silica gel 60 $F_{254}$,<br>b) HPTLC RP-18 $F_{254}$ |

| GLC | see ref. [9] |
|---|---|

| HPLC | see ref. [10] |
|---|---|

## 9. Remarks

10. References


[1] Symes, W.F., Dawson, C.R., *Nature 171*, 1953, pp. 841-2 *(occurrence)*

[2] Skopp, G., Über phenolische Inhaltsstoffe aus Schinus terebinthifolius Raddi – n-Alkylphenole und Biflavonoide – Diss., 1986 *(name, occurrence)*

[3] List, P.H., Hörhammer, L., Hagers Handbuch der pharmazeutischen Praxis 4, Springer Verlag, Heidelberg, 1973 *(occurrence)*

[4] Cojocaru, M., Droby, S., Glotter, E., Goldmann, A., Gottlieb, H.E., Jacoby, B., Prusky, D., *Phytochemistry 25(5)*, 1986, pp. 1093-5 *(occurrence)*

[5] Barrero, A.F., Sanchez, J.F., Corrales, F., Rodrigues, I., *Phytochemistry 28(1)*, 1989, pp. 161-4 *(occurrence)*

[6] Morton, F.J., *Econ. Bot. 32(4)*, 1978, pp. 353-9 *(impact on environment)*

[7] Evans, F.J., Schmidt, R.J., *Planta medica 38*, 1980, 289-316 *(contact dermatitis)*

[8] Tyman, J.H.P., *J. Chrom. 166*, 1978, pp. 159-72 *(UV spectrophotometry)*

[9] Tyman, J.H.P., *J. Chrom. 138*, 1977, pp. 97-110 *(gas-liquid chromatography)*

[10] Tyman J.H.P., Tychopoulos, V., Chan, P., *J. Chrom. 303*, 1984, pp. 137-50 *(high performance liquid chromatography)*

# Cardol, Triene

*G. Skopp*

## 1. Name of Compound

Common name

**Cardol, Triene**

Synonyms

Cardol-triolefin, Cardol 15 : 3 (n-1), Pentadecatrienylresorcinol

Systematic name

(Z,Z)-5-n-(8,11,14-Pentadecatrienyl)-1,3-benzenediol

| Substance | Subgroup |
|---|---|
| Phenol | Long chain phenol |

| CAS registry number and other numbers | |
|---|---|
| [79473-24-8] | BRN 5066815 |

## 2. Formulas and Molecular Weight

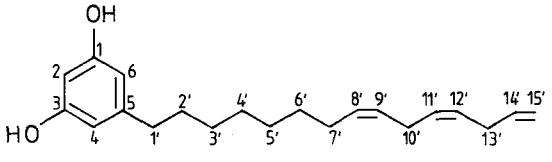
Molecular formular

$C_{21}H_{30}O_2$

Molecular weight

314.259

Structural formula




## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| liquid | not available | not available |
| **Melting Point mp** | $n_D^{20}$ | **Acid value** |
| not applicable | not available | not applicable |
| **Boiling point bp** | $[\alpha]_D^{20}$ | **Saponification value** |
| not available | not applicable | not applicable |
| **Flash point** | **Soluble in** | |
| not available | acetone, ether | |
| **Color** | **Odor** | |
| colorless | like petroleum ether, repulsive | |

## 4. Occurrence

| |
|---|
| Anacardium occidentale L. (Anacardiaceae) [1]<br>Schinus terebinthifolius Raddi (Anacardiaceae) [2, 3] |

## 5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | irritant, sensitizing [7] |
| – | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

## 6. Transportation and Storage Instructions

| |
|---|
| Storage in light resistant containers under nitrogen. |

## 7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 124 mu | Molecular ion | 314 mu |
| Ionization energy | 100 eV | Ion source temp. | 90 °C |
| Acceleration voltage | 3 kV | Emission current | about 1 mA |
| Resolution | 1000 | Scan rate | – s/decade |
| Spectrometer type and manufacturer | | Varian MAT 311 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3325 (OH), 3080 (=$CH_2$), 3010 (CHR), 2930 and 2860 (H-C), 1470 ($CH_2$),<br>840 and 695 (1,3,5-substitution) $cm^{-1}$ | | | |
| Sample preparation | film | Resolution | – |
| Spectrometer type and manufacturer | | Perkin-Elmer spectrometer 521 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>6.25 (d, J=2.5 Hz, 4,6-H);<br>5.83 (14′-H);<br>2.80 (10′,13′-H);<br>1.48–1.61 (2′-H); | | 6.17 (t, J=2.5 Hz, 2-H);<br>5.27–5.50 (8′,9′,11′,12′-H);<br>2.46 (t, J=7.8 Hz, 1′-H);<br>1.30–1.40 (3′,4′,5′,6′-H) ppm | 4.67 (s, br., O*H*);<br>4.95–5.10 (15′-H);<br>2.03 (td, 7′-H); |
| Frequency | 250 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 21 °C |
| Spectrometer type and manufacturer | | Bruker WM 250 | |

| UV | not available |
|---|---|

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | a) 0.18, | b) 0.60 | |
| Solvent | – | Solvent system | a) light petroleum (40–60 °C: ether 7 : 3 (v/v)<br>b) acetonitrile : water 8 : 92 |
| Saturated atmosphere | yes | Detection/Color | UV, fast blue salt B 0.5%/red |
| Plate manufacturer | Merck | Plate type/Product no. | a) Silica gel 60 $F_{254}$,<br>b) HPTLC RP-18 $F_{254}$ |

| GLC | see ref. [9] |
|---|---|

| HPLC | see ref. [10] |
|---|---|

9. Remarks

| |
|---|

## 10. References


[1] Symes, W.F., Dawson, C.R., *Nature 171*, 1953, pp. 841–2 *(occurrence)*

[2] Skopp, G., Über phenolische Inhaltsstoffe aus Schinus terebinthifolius Raddi – n-Alkylphenole und Biflavonoide – Diss., 1986 *(name, occurrence)*

[3] List, P.H., Hörhammer, L., Hagers Handbuch der pharmazeutischen Praxis 4, Springer Verlag, Heidelberg, 1973 *(occurrence)*

[4] Cojocaru, M., Droby, S., Glotter, E., Goldmann, A., Gottlieb, H.E., Jacoby, B., Prusky, D., *Phytochemistry 25(5)*, 1986, pp. 1093–5 *(occurrence)*

[5] Barrero, A.F., Sanchez, J.F., Corrales, F., Rodrigues, I., *Phytochemistry 28(1)*, 1989, pp. 161–4 *(occurrence)*

[6] Morton, F.J., *Econ. Bot. 32(4)*, 1978, pp. 353–9 *(impact on environment)*

[7] Evans, F.J., Schmidt, R.J., *Planta medica 38*, 1980, 289–316 *(contact dermatitis)*

[8] Tyman, J.H.P., *J. Chrom. 166*, 1978, pp. 159–72 *(UV spectrophotometry)*

[9] Tyman, J.H.P., *J. Chrom. 138*, 1977, pp. 97–110 *(gas-liquid chromatography)*

[10] Tyman J.H.P., Tychopoulos, V., Chan, P., *J. Chrom. 303*, 1984, pp. 137–50 *(high performance liquid chromatography)*

# Chamazulene

*R. Carle*

## 1. Name of Compound

Common name

**Chamazulene** [1]

Synonyms

Dimethulene

Systematic name

7-Ethyl-1,4-dimethylazulene

Substance

Azulene

CAS registry number and other numbers

[529-05-5] BRN 1306577

## 2. Formulas and Molecular Weight

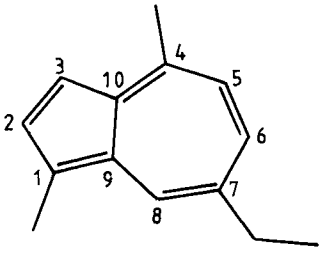
Molecular formular

$C_{14}H_{16}$

Molecular weight

184.27

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>oil [2] | $d_4^{20}$<br>0.9883 g/cm$^3$ | Iodine value<br>not determined |
| Melting Point mp<br>132 °C<br>(trinitrobenzenate) [3] | $n_D^{20}$<br>not determined | Acid value<br>not applicable |
| Boiling point bp<br>161 °C (12 torr) [2] | $[\alpha]_D^{20}$<br>±0 | Saponification value<br>not applicable |
| Flash point<br>not determined | Soluble in<br>methanol, ethanol, n-hexane | |
| Color<br>blue [2] | Odor<br>odorless | |

## 4. Occurrence

| |
|---|
| Chamomile, yarrow [1] |

## 5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not necessary |
| oral toxicity test | $LD_{50}$ | 3 g/kg (white mice) [2] |
| Waste disposal procedures<br>Combustion | | |

## 6. Transportation and Storage Instructions

| |
|---|
| Storage in closed containers, cool, protected from light. |

## 7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS * | see ref. [4] | | |
| Base peak | 169 m/e | Molecular ion | 184 m/e |
| Ionization energy | 70 eV | Ion source temp. | – °C |
| Acceleration voltage | 1.8 kV | Emission current | – mA |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | Finnigan 4023 | |

| | | | |
|---|---|---|---|
| IR | | | |
| Characteristic peaks | see ref. [4] | | |
| Sample preparation | film between CsI-plates | Resolution | – |
| Spectrometer type and manufacturer | | Perkin Elmer 325 | |

| | | | |
|---|---|---|---|
| NMR * | see ref. [4] | Nucleus | $^1H$ |
| Chemical Shifts<br>8.15 (d, 2Hz, H-8); 7.60 (d, 4.0 Hz, H-2); 7.33 (dd, 2.0/10.5 Hz, H-6);<br>7.23 (d, 4.0 Hz, H-3); 6.92 (d, 10.5 Hz, H-5); 2.80 (q, 7.5 Hz, C-7, $C_2H_5$);<br>2.78 (s, C-4, $CH_3$) ppm | | | |
| Frequency | 60 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | – |
| Spectrometer type and manufacturer | | Varian EM 360 A | |

| NMR * | | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>144.1 (s, C-4);<br>136.2 (s, C-9);<br>124.9 (d, C-5);<br>17.3 (q, C-7-$C_2H_5$); | 137.4 (s, C-10);<br>135.6 (s, C-7);<br>112.8 (d, C-3);<br>12.8 (q, C-1-$CH_3$) ppm | 136.2 (d, C-6);<br>134.5 (d, C-8);<br>33.8 (t, C-7-$C_2H_5$); | 136.2 (d, C-2);<br>125.0 (s, C-1);<br>24.0 (q, C-4-CH3); |
| Frequency | 20 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | – °C |
| Spectrometer type and manufacturer | | CFT 20 Varian | |

| UV | see ref. [2] | | |
|---|---|---|---|
| $I_{max}$ | 370, 605.8 nm | log $\varepsilon_{max}$ | 3.7, 2.64 |
| Solvent | Cyclohexane | | |
| Sample conc. | $1.64 \times 10^{-3}$ mol/l | Cell thickness | – cm |
| Spectrometer type and manufacturer | | Lambda 5 UV/VIS, Perkin Elmer | |

8. Chromatographic Data

| TLC | see ref. [5] | | |
|---|---|---|---|
| Rf Value | 0.78 | | |
| Solvent | $CH_2Cl_2$ | Solvent system | $CH_2Cl_2$, Ethylacetate (98 : 2; v : v) |
| Saturated atmosphere | – | Detection/Color | deep blue |
| Plate manufacturer | Merck | Plate type/Product no. | Silicagel |

| GLC | see ref. [6] | | |
|---|---|---|---|
| Retention time | – | | |
| Column type | Chromosorb W | Column length | 2 m |
| Column packing | 3% OV 17 | Column temp. | 100–200 °C, 8 °C/min |
| Injector port temp. | – | Carrier gas | Helium 30 ml/min |
| Detector temp. | – | Sample solvent | Cyclohexane |
| Sample size | 5 µl | Sample conc. | – |
| Chromatograph type and manufacturer | | Hewlett Packard HP 5880 | |

| HPLC | | | |
|---|---|---|---|
| Retention time | 6.8 min | | |
| Column | Li-Chrosorb RP-8 | Stationary phase | RP-8, particle size 5 μm |
| Mobile phase | MeOH/$H_2O$ (9 : 1) | Flow rate | not given |
| Column temp. | room temperature | Pressure | not given |
| Detector | – | Sample solvent | – |
| Sample size | – | Sample conc. | – |
| Chromatograph type and manufacturer | | not given | |

9. Remarks

Degradation product obtained from steam distillation of matricin [8–11]

10. References


[1] Ruzicka, L., Rudolph, E.A., *Helv. Chim. Acta 9* (1926) 118–140 *(Name, occurrence)*

[2] Windholz, M. (Ed.); *The Merck Index*, 9th Ed., Merck & Co., Rahway 1976 *(Name, occurrence)*

[3] Sorm, F., Novak, J., Herout, V., *Coll. Czechoslov. Chem. Commun.* 18 (1953) 527–529 *(Occurrence)*

[4] Jakovlev, V., Isaac, 0., Flaskamp, E., *Planta Med. 49* (1983) 67–73 *(IR-MassSpectrometry, Proton-, Carbon-13-NMR)*

[5] Reichling, J., Becker, H., *Dtsch. Apoth. Ztg. 117* (1977) 275–277 *(TLC)*

[6] Carle, R., Fleischhauer, I., Fehr, D., *Dtsch. Apoth. Ztg. 127* (1987) 2451–245 *(GLC)*

[7] Schilcher, H. in: Kubeczka, H.-H. (Ed.), *Ätherische Öle, Analytik, Physiologie, Zusammensetzung*, Georg Thieme Verlag, Stuttgart, New York 1982, pp. 104115 *(HPLC)*

[8] Cekan, Z., Herout, V., Sorm, F., *Chem. and Ind.* (1954) 604–605

[9] Stahl, E., Chem. *Ber. 87* (1954) 202–205 *(Occurrence, chemical properties)*

[10] Stahl, E., *Chem. Ber. 87* (1954) 505–507

[11] Stahl, E., *Chem. Ber. 87* (1954) 1626–1628

# Chlorophyll a

*M. Senge*

## 1. Name of Compound

Common name

**Chlorophyll a**

Synonyms

Systematic name

Magnesium, 3-[(17S)-8-ethyl-13$^2$t-methoxycarbonyl-2,7,12,18t-tetramethyl-13$^1$-oxo-2-vinyl-13,13$^2$,17,18-tetrahydro-cyclopenta[at]porphyrin-17r-yl]-propionato-[(7R,11R)-trans-phytylester]
(13$^2$R)-13$^2$-Methoxycarbonyl-3$^1$,3$^2$-didehydro-phytochlorin-[(7R,11R)-trans-phytylester]

| Substance | Subgroup |
|---|---|
| Tetrapyrrole | Chlorin |

CAS registry number and other numbers

[479-61-8]

## 2. Formulas and Molecular Weight

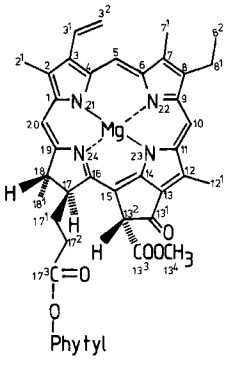
Molecular formular

$C_{55}H_{72}MgN_4O_5$

Molecular weight

892.5350

Structural formula



## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>1.079 g/cm$^3$ | Iodine value<br>not available |
| Melting Point mp<br>150–153 °C<br>corr., dec. | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_{720}^{25}$ = -262 °<br>(38.2 mg/100ml acetone) | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>ether, chloroform, alcohol, acetone | |
| Color<br>blue green | Odor<br>odorless | |

## 4. Occurrence

All organisms with oxygenic photosynthesis [2]

## 5. Health Hazard Data

| Toxicology | Hazard labeling |
|---|---|
| not known | $LD_{50}$ not available |

Waste disposal procedures:
By combustion

## 6. Transportation and Storage Instructions

Store under nitrogen at −20 °C.
Light sensitive compound, handle under green safety light.

## 7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS | FAB spectrum see ref. [3] | | |
| Base peak | 614 | Molecular ion | 892 |
| Ionization energy | 7–8 keV Ar-Atoms | Ion source temp. | – |
| Acceleration voltage | 8 kV | Emission current | – |
| Resolution | 2000 | Scan rate | – |
| Spectrometer type and manufacturer | | MS 50; Kratos | |

| IR | see ref. [4] |
|---|---|

Characteristic peaks
2952; 2925; 2865 (CH,st); 1736; 1690 (C=O ester); 1600m; 1550st; 1485w; 1447m; 1378m; 1345m; 1327m; 1301vw; 1286m; 1236w; 1180m; 1159m; 1128m; 1102w; 1071w; 1036m; 985w; 916m; 848vw; 797m; 765w; 742m; 703 vw; 639vw $cm^{-1}$

| Sample preparation | KBr pellet | Resolution | – |
|---|---|---|---|
| Spectrometer type and manufacturer | | JFS 88 | |

| NMR | see ref. [5] | Nucleus | $^1H$ |
|---|---|---|---|

Chemical Shifts

| | | |
|---|---|---|
| 9.66 (s; 10-H); | 9.35 (s; 5-H); | 8.50 (s; 20-H); |
| 8.09 (dd; J= 11, 17 Hz; $3^1$-H); | 6.22 (dd; J=2, 17 Hz; $3^2$-$H_B$); | 6.18 (s; $13^2$-H); |
| 5.99 (dd; J=2,17 Hz; $3^2$-$H_A$); | 5.04 (t; J=7 Hz; $P_2$-H); | 4.52 (q; J=7 Hz; H-17); |
| 4.3 (m; 18-H); | 3.83 (s; $13^4$-$CH_3$); | 3.80 (q; J=7 Hz; $8^1$-$CH_2$); |
| 3.58 (s; $12^1$-$CH_3$); | 3.31 (s; $2^1$-$CH_3$); | 3.26 (s; $7^1$-$CH_3$); |
| 2.48 (m; $17^1$-$CH_2$); | 2.21 (m; $17^2$-$CH_2$); | 1.76 (d; J=7Hz; $18^1$-$CH_3$); |
| 1.69 (t; J=7 Hz; $8^2$-$CH_3$); | 1.54 (s; $P_{3a}$-$CH_3$); | |

1.2–1.0 (m; $P_7$-$P_{15}$-CH; $P_4$-$P_{14}$-$CH_2$);
0.85; 0.83; 0.80; 0.78 (s,s,s,s; $P_{7a}$-$P_{16a}$-$CH_3$) ppm

| Frequency | 360 MHz | Solvent | $d_6$-acetone |
|---|---|---|---|
| Standard | acetone | Sample temp. | 20 °C |
| Spectrometer type and manufacturer | | Nicolet NT-360 | |

| NMR | see ref. [5] | Nucleus | $^{13}C$ |
|---|---|---|---|

Chemical Shifts

| | | | | |
|---|---|---|---|---|
| 155.4 (1-C), | 135.9 (2-C), | 139.5 (3-C), | 131.0 ($3^1$-C), | 119.8 ($3^2$-C), |
| 148.9 (4-C), | 100.3 (5-C), | 152.7 (6-C), | 134.2 (7-C), | 144.6 (8-C), |
| 146.6 (9-C), | 108.2 (10-C), | 148.1 (11-C), | 134.1 (12-C), | 130.9 (13-C), |
| 190.0 ($13^1$-C), | 170.7 ($13^3$-C), | 162.4 (14-C), | 105.8 (15-C), | 156.3 (16-C), |
| 173.1 ($17^3$-C), | 169.4 (19-C), | 93.4 (20-C), | 118.9 ($P_2$), | 142.2 ($P_3$) ppm |

| Frequency | 15.04 MHz | Solvent | $d_6$-acetone (c = 0.3 M) |
|---|---|---|---|
| Standard | TMS | Sample temp. | 20 °C |
| Spectrometer type and manufacturer | | JEOL FX-60 PFT | |

| | | | |
|---|---|---|---|
| UV | see ref. [7] | | |
| $\lambda_{max}$ | 429 nm | $\varepsilon_{max}$ | $11.54 \times 10^4\ M^{-1}\ cm^{-1}$ |
| Solvent | diethylether | | |
| Sample conc. | $8.67 \times 10^{-6}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Shimadzu-260 UV/vis spectrometer | |

8. Chromatographic Data

| | | | |
|---|---|---|---|
| TLC | | | |
| Rf Value | 0.66 | | |
| Solvent | – | Solvent system | petroleum ether (100–140 °C): 2-propanol : $H_2O$ (100 : 12 : 0.25) |
| Saturated atmosphere | – | Detection/Color | green color, red fluorescence |
| Plate manufacturer | self prepared | Plate type/Product no. | Hager, Meyer-Bertenrath System A (8) |

| | |
|---|---|
| GLC | not available |

| | | | |
|---|---|---|---|
| HPLC | see ref. [9] | | |
| Retention time | 32 min | | |
| Column | 4.6 × 250 mm | Stationary phase | Spherisorb RP 18, 5 μm |
| Mobile phase | $CH_3CN$(3) : $CH_3OH$ (1), superimposed by a multilinear gradient of water | Flow rate | 1 ml/min |
| Column temp. | 25 °C | Pressure | 80 bar |
| Detector | Uvikon 720 LC spectrometer VIS 430 nm | Sample solvent | acetone |
| Sample size | 20 μl | Sample conc. | $1.12 \times 10^{-3}$ mol/l |
| Chromatograph type and manufacture | | LC 210 high pressure pumps, programmer 200, HP 3390 A integrator (Kontron, Munich) | |

9. Remarks

The crystal structure of the corresponding chlorophyllide has been solved [10], the chemical synthesis was developed by Woodward [11] and a recent review on the biosynthesis has been published by Rüdiger and Schoch [12].

## 10. References


[1] Beilstein Handbook of Organic Chemistry, E III/IV, Vol. 26, Springer Verlag, Heidelberg 1978, pp. 3243-3246; Stoll, A., Wiedemann, E. *Fortschr. Chem. Org. Naturst. I*, 1938, 159-253; Fischer, H., Stern, A. *Die Chemie des Pyrrols*, Akad. Verlagsges., Leipzig, 1940; Smith, K.M. (Ed.) *Porphyrins and Metalloporphyrins*, Elsevier, Amsterdam, 1975 *(physical and chemical properties)*

[2] Svec, W.A., in: Dolphin, D., (Ed.), The *Porphyrins*, Vol. V, Academic Press, New York 1978, pp. 341-399 *(occurrence, structure, isolation, determination)*

[3] Budzikiewicz, H., in: Dolphin, D., (Ed.), *The Porphyrins*, Vol. III, Academic Press, New York 1978, pp. 395-461; Dougherty, R.C. *Biochem. Appl. Mass Spectrom.* 1980, (Ist suppl. Vol.), 693-701; Grese, R.P., Cerny, R.L., Gross, M.L., Senge, M. *J. Am. Soc. Mass, Spectrom.* 1990, *1*, in press *(mass spectrometry)*

[4] Sherman, G., Wang, S.-F. *Photochem. Photobiol.* 1967, *6*, 239-245; Katz, J.J., Dougherty, R.C., Boucher, L.J., in: Vernon, L.P.,Seely, G.R., (Eds.), *The Chlorophylls*, Academic Press, New York 1966, Ch. 7; Burger, H., in: Smith, K.M., (Ed.) *Porphyrins and Metalloporphyrins*, Elsevier, Amsterdam 1975, Ch. 11; Fujiwara, M., Tasumi, M. *J. Phys. Chem.* 1986, *90*,5646-5650; Chapados, C. *Photochem. Photobiol.* 1988, 47, 115-132; Andersson, L.A., Loehr, T.M., Cotton, T.M., Simpson, D.J., Smith, K.M. *Biochim. Biophys. Acta* 1989, 974, 163-179 *(IR spectroscopy of chlorophylls and related compounds)*

[5] Hynninen, P.H., Lötjönen, S., *Magn. Res. Chem.* 1985, *23*, 605-615; Scheer, H., Katz, J.J., in: Smith, K.M., (Ed.), *Porphyrins and Metalloporphyrins*, Elsevier, Amsterdam 1975, pp. 399-524; Katz, J.J., Brown, C.E., *Bull. Magn. Res.* 1983, *5*, 3-49 *($^1$H-NMR spectroscopy)*

[6] Lötjönen, S., Hynninen, P.H., *Org. Magn. Res.* 1981, *16*, 304-308; Lötjönen, S., Hynninen, P.H., *Org. Magn. Res.* 1983, *21*, 757-765 *($^{13}$C-NMR spectroscopy)*

[7] Holden, M., in: Goodwin, T.W., (Ed.), *Chemistry and Biochemistry of Plant Pigments*, Academic Press, New York, 1965, pp. 461-488; Hynninen, P.H., Sievers, G., *Z. Naturforsch.* 1981, *36b*, 1000-1009; Shipman, L.L., in: Govindjee, (Ed.), *Photosynthesis*, Vol. I, Academic Press, New York, 1982, pp. 251-291; Watanabe, T., Hongu, A., Honda, K., Nakazato, M., Konno, M., Saitoh, S., *Anal. Chem.* 1984, *56*, 251-256 *(UV/vis spectroscopy)*

[8] Hager, A., Meyer-Bertenrath, M., *Planta* 1966, *69*, 198-217 *(TLC, see also ref. [2])*

[9] Cavaleiro, J.A.S., Smith, K.M., *Talanta* 1986, *33*, 963-971 *(HPLC, see also ref. [12])*

[10] Kratky, C., Dunitz, J. D., J. Mol. Biol. 1977, 113, 431-442 *(Crystal structure of chlorophyllide a)*

[11] Woodward, R.B., *Angew. Chem.* 1960, *72*, 651-662 *(Chemical synthesis)*

[12] Rüdiger, W., Schoch, S., in: Goodwin, T.W., (Ed.), *Chemistry and Biochemistry of Plant Pigments*, Academic Press, New York 1989, pp- 1-59 *(Biosynthesis)*

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# Chlorophyll b

*M. Senge*

## 1. Name of Compound

Common name

**Chlorophyll b**

Synonyms

Systematic name

Magnesium, 3-[(17S)-7-formyl-8-ethyl-13$^2$-methoxycarbonyl-2,12,18t-trimethyl-13$^1$-oxo-3-vinyl-13$^1$,13$^2$,17,18-tetrahydro-cyclopenta[at] porphyrin-17r-yl]-propionato-[(7R,11R)-trans-phytylester]

Substance

Tetrapyrrole

Subgroup

Chlorin

CAS registry number and other numbers

[519-62-0]

## 2. Formulas and Molecular Weight

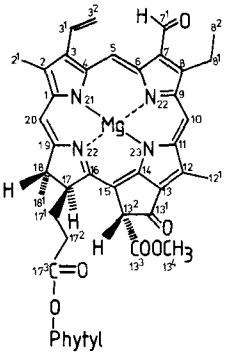
Molecular formular

$C_{55}H_{70}MgN_4O_6$

Molecular weight

906.5145

Structural formula



## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| solid | not available | not available |
| Melting Point mp | $n_D^{20}$ | Acid value |
| 183–185 (corr.) °C | not applicable | not applicable |
| Boiling point bp | $[\alpha]_{720}^{25} = -267$ ° | Saponification value |
| not available | (37.5 mg/100 ml MeOH/ acetone = 9 : 1) | not applicable |
| Flash point | Soluble in | |
| not available | ether, chloroform, alcohol, acetone | |
| Color | Odor | |
| dark green | odorless | |

## 4. Occurence

Higher plants, green algae, Euglenophyta, Prochlorophyta

## 5. Health Hazard Data

| Toxicology | Hazard labeling | |
|---|---|---|
| not known | $LD_{50}$ | not available |
| Waste disposal procedures<br>Combustion | | |

## 6. Transportation and Storage Instructions

Store under nitrogen at −20 °C. Light sensitive compound, handle under green safety light.

## 7. Spectroscopic Data

| MS | FAB spectrum | see ref. [3] | |
|---|---|---|---|
| Base peak | 628 | Molecular ion | 906 |
| Ionization energy | 7–8 keV Ar-Atoms | Ion source temp. | – |
| Acceleration voltage | 8 kV | Emission current | – |
| Resolution | 2000 | Scan rate | – |
| Spectrometer type and manufacturer | | MS 50; Kratos | |

| IR | see ref. [4] |
|---|---|

Characteristic peaks
2952; 2925; 2857 (CH,st); 1737; 1698; 1668; 1603; 1548; 1465; 1445; 1378; 1346; 1324; 1287; 1209; 1185; 1166; 1145; 1126; 1065; 1038; 982; 924; 867; 853; 840; 796; 757; 731; 700 $cm^{-1}$

| Sample preparation | KBr pellet | Resolution | – |
|---|---|---|---|
| Spectrometer type and manufacturer | | JFS 88 | |

| NMR * | see ref. [5] | Nucleus | $^1H$ |
|---|---|---|---|

Chemical Shifts

| | | |
|---|---|---|
| 11.19 (s; 71-CHO); | 10.09 (s; 10-H); | 9.84 (s; 5-H); |
| 8.44 (s; 20-H); | 8.00 (dd; J = 12, 18 Hz; $3^1$-H); | |
| 6.26 (dd; J = 2, 12 Hz; $3^2$-$H_B$); | 6.02 (dd; J = 2, 12 Hz; $3^2$-$H_A$); | |
| 6.13 (s; $13^2$-H); | 5.05 (t; J = 7 Hz; $P_2$-H); | |
| 4.30 (d; J = 7 Hz; $P_1$-$CH_2$); | 4.32 (m; 17-H; 18-H); | 3.83 (s; $13^4$-$CH_3$); |
| 3.80 (q; J = 7 Hz; $8^1$-$CH_2$); | 3.57 (s; $12^1$-$CH_3$); | 3.27 (s; $2^1$-$CH_3$); |
| 2.51 (m; $17^1$-$CH_2$); | 2.35 (m; $17^2$-$CH_2$); | 1.76 (d; J = 7 Hz; $18^1$-$CH_3$); |
| 1.77 (t; J = 7 Hz; $8^2$-$CH_3$); | 1.53 (s; $P_{3a}$-$CH_3$); | |
| 1.18 (s, broad; $P_7$-$P_{15}$-CH; $P_4$-$P_{14}$-$CH_2$); | | 0.89–0.72 (m; $P_{7a}$-$P_{16}$-$CH_3$) ppm |

| Frequency | 360 MHz | Solvent | $d_6$-acetone |
|---|---|---|---|
| Standard | acetone | Sample temp. | 20 °C |
| Spectrometer type and manufacturer | | Nicolet NT-360 | |

| NMR | see ref. [6] | Nucleus | $^{13}C$ |
|---|---|---|---|

Chemical Shifts

| | | | | |
|---|---|---|---|---|
| 157.5 (1-C), | 136.24 (2-C), | 12.6 ($2^1$-C), | 140.72 (3-C), | 129.79 ($3^1$-C), |
| 120.8 ($3^2$-C), | 149.85 (4-C), | 102.96 (5-C), | 149.23 (6-C), | 129.71 (7-C), |
| 187.66 ($7^1$-C), | 155.87 (8-C), | 19.18 ($8^1$-C), | 18.89 ($8^2$-C), | 142.64 (9-C), |
| 111.06 (10-C), | 148.76 (11-C), | 139.28 (12-C), | 12.22 ($12^1$-C), | 131.27 (13-C), |
| 190.26 ($13^1$-C), | 64.69 ($13^2$-C), | 170.79 ($13^3$-C), | 52.76 ($13^4$-C), | 163.9 (14-C), |
| 104.2 (15-C), | 158.71 (16-C), | 50.46 (17-C), | 31.06 ($17^1$-C), | 29.74 ($17^2$-C), |
| 173.55 ($17^3$-C), | 49.22 (18-C), | 23.34 ($18^1$-C), | 170.79 (19-C), | 92.96 (20-C) ppm |

| Frequency | – | Solvent | $CDCl_3$ ($H_2O$) |
|---|---|---|---|
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | Bruker WP 80 DS | |

| | | | |
|---|---|---|---|
| UV | see ref. [7] | | |
| $\lambda_{max}$ | 455 nm | $\varepsilon_{max}$ | $13.65 \times 10^4\ M^{-1}\ cm^{-1}$ |
| Solvent | acetone | | |
| Sample conc. | $7.33 \times 10^{-6}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Uvikon 820 (Kontron, Munich) | |

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.6 | | |
| Solvent | – | Solvent system | petroleum ether (100–140 °C): 2-propanol : $H_2O$ (100 : 12 : 0.25) |
| Saturated atmosphere | 90 min, 50 °C | Detection/Color | green color, red fluorescence |
| Plate manufacturer | self prepared | Plate type/Product no. | Hager, Meyer-Bertenrath System A (8) |

| GLC | not available |
|---|---|

| HPLC | see ref. [9] | | |
|---|---|---|---|
| Retention time | 27 min | | |
| Column | 4.6 × 250 mm | Stationary phase | Spherisorb RP 18, 5 µm material |
| Mobile phase | $CH_3CN$(3) : $CH_3OH$ (1), superimposed by a multilinear gradient of water | Flow rate | 1 ml/min |
| Column temp. | 25 °C | Pressure | 80 bar |
| Detector | Uvikon 720 LC spectrometer VIS 430 nm | Sample solvent | acetone |
| Sample size | 20 µl | Sample conc. | $8 \times 10^{-4}$ mol/l |
| Chromatograph type and manufacturer | LC 210 high pressure pumps, programmer 200, HP 3390 A integrator (Kontron, Munich) | | |

## 9. Remarks

10. References


[1] Stoll, A., Wiedemann, E. *Fortschr. Chem. Org. Naturst.* 1938, 1, 159–253; Fischer, H., Stern, A., *Die Chemie des Pyrrols*, Akad. Verlagsges., Leipzig, 1940; Beilstein Handbook of Organic Chemistry, EIII/IV, Vol. 26, Springer Verlag, Heidelberg 1978, pp. 3287–3290; Smith, K.M. (Ed.), *Porphyrins and Metalloporphyrins*, Elsevier, Amsterdam 1975 *(Physical and chemical properties)*

[2] Svec, W.A., in: Dolphin, D., (Ed.), *The Porphyrins*, Vol V, Academic Press, New York, 1978, pp. 341–399 *(also structure, isolation, determination)*; Rüdiger, W., Schoch, S. in: Goodwin, T.W.,(Ed.), *Chemistry and Biochemistry of Plant Pigments*, Academic Press, New York 1989, pp. 1–59 *(Occurrence, biosynthesis)*

[3] Budzikiewicz, H., in: Dolphin, D., (Ed.), *The Porphyrins*, Vol. III, Academic Press, New York 1978, pp. 395–461; Dougherty, R.C., *Biochem. Appl. Mass Spectrom.* 1980, (1st suppl. Vol.), 693–701; Grese, R.P., Cerny, R.L., Gross, M.L., Senge, M., *J. Am. Soc. Mass Spectrom.* 1990, *1*, in press *(Mass spectrometry)*

[4] Holt, A.S., Jacobs, E.E., *Plant Physiol.* 1955, *30*, 553; Katz, J.J., Dougherty, R.C., Boucher, L.J., in: Vernon, L.P., Seely, G.R., (Eds.), *The Chlorophylls*, Academic Press, New York 1966, Ch.7; Andersson, L.A., Loehr, T.M., Cotton, T.M., Simpson, D.J., Smith, K.M., *Biochim. Biophys. Acta* 1989, 974, 163–179 *(IR spectroscopy)*

[5] Katz, J.J., Norman, G.D., Svec, W.R., Strain, H.H., *J. Am. Chem. Soc.* 1968, *90*, 6841–6845; Hynninen, P.H., Lötjönen, S., *Synthesis* 1983, 705–708; Katz, J.J., Brown, C.E., *Bull. Magn. Res.* 1983, *5*, 3–49 *($^1$H-NMR)*

[6] Risch, N., Brockmann, H., *Tetrahedron Lett.* 1983, *24*, 173–176 *($^{13}$C-NMR)*

[7] Holden, M., in: Goodwin, T.W., (Ed.), *Chemistry and Biochemistry of Plant Pigments*, Academic Press, New York 1965, pp. 461–488; Shipmann, L.L. in: Govindjee, (Ed.), *Photosynthesis*, Vol. I, Academic Press, New York, 1982, pp. 275–291; Watanabe, T., Hongu, A., Honda, K., Nakazato, M., Konno, M., Saitoh, S., *Anal. Chem.* 1984, *56*, 251–256 *(UV/VIS spectrometric determination)*

[8] Hager, A., Meyer-Bertenrath, M., Planta 1966, *69*, 198–217 *(TLC, see also ref. [2])*

[9] Cavaleiro, J.A.S., Smith, K.M., *Talanta* 1986, *33*, 963–971 *(HPLC, see also ref. [2b])*

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# Deacetoxymatricarin

*Wu Chongming*

## 1. Name of Compound

Common name
**Deacetoxymatricarin**

Synonyms
Axillin; (+)-Leukodin

Systematic name
3,3a,4,5,9a,9b-Hexahydro-3,6,9-trimethyl-azuleno-[4,5-b]-furan-2,7-dione

| Substance | Subgroup |
|---|---|
| Sesquiterpene lactone | Guaianolides lactone |

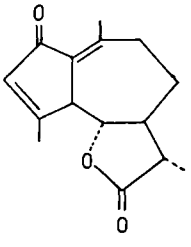
| CAS registry number and other numbers | |
|---|---|
| [17946-87-1] | BRN 1624200 |

## 2. Formulas and Molecular Weight

Molecular formular
$C_{15}H_{18}O_3$

Molecular weight
246.31

Structural formula



## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| solid | not available | not available |
| **Melting Point mp** | $n_D^{20}$ | **Acid value** |
| 203.5–204.5 °C | not applicable | not applicable |
| **Boiling point bp** | $[\alpha]_D^{20} = +49.4°$ | **Saponification value** |
| not available | (c=1.02, chloroform) | not applicable |
| **Flash point** | **Soluble in** | |
| not available | chloroform, ethyl acetate | |
| **Color** | **Odor** | |
| colorless | odorless | |

4. Occurrence

| |
|---|
| Several species of the genus Artemisia (Compositae) |

5. Health Hazard Data

| | |
|---|---|
| Toxicology | Hazard labeling |
| not available | $LD_{50}$ not available |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 246 | Molecular ion | 246 |
| Ionization energy | 70 eV | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | MM 7070H | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>1765, 1675, 1630, 1610, 1235, 1175, 1025, 980 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | PE-599B | |

| NMR | | Nucleus | $^1H$-$^1H$-correlation |
|---|---|---|---|
| Chemical Shifts<br>1.28 (d, J=6 Hz,3H, $C_{11}$-$CH_3$);<br>2.22 (s, 3H, $C_4$-$CH_3$);<br>3.45 (m, 2H, $C_5$-H and $C_6$-H); | | 1.60–2.40 (m,6H, H-$C_8$-H, H-$C_9$-H, $C_7$-H and $C_{11}$-H);<br>2.35 (s, 3H, $C_{10}$-$CH_3$);<br>6.05 (m, 1H, $C_3$-H) ppm | |
| Frequency | 90 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | – |
| Spectrometer type and manufacturer | | EM-390 | |

| UV * | | | |
|---|---|---|---|
| $I_{max}$ | 221, 257 nm | $\varepsilon_{max}$ | 5.000, 12.300 |
| Solvent | methanol | | |
| Sample conc. | – | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | SPECORD UV-VIS | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.44 | | |
| Solvent | methanol | Solvent system | petroleum ether : ethyl acetate (6 : 4) |
| Saturated atmosphere | – | Detection/Color | UV 254 (quenching) |
| Plate manufacturer | hand made | Plate type/Product no. | Silica gel-$F_{254}$, 0.25 mm |

| GLC | not available |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

10. References


[1] Geissman, T.A. et al.: Phytochem. *6*(6), 1967, 901
[2] Yoshioka, H. et al.: Sesqueterpene lactones 1973, 348
[3] Wu, C.M. et al.: Chinese Bulletin of Botany *3*(3), 1985, 34

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# Dehydrocorybulbine

*Wu Chongming*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Dehydrocorybulbine** | |
| Synonyms | |
| Systematic name<br>5,6-Dihydro-3-hydroxy-2,9,10-trimethoxy-13-methyl-dibenzo-[a,g]-quinolizinium | |
| Substance<br>Alkaloid | Subgroup<br>Protoberberine alkaloid |
| CAS registry number and other numbers<br>[59870-72-3] | BRN 1556229 |

## 2. Formulas and Molecular Weight

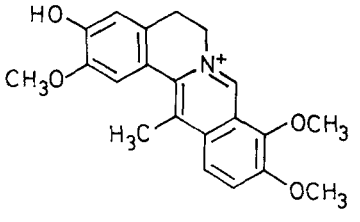
Molecular formular

$C_{21}H_{21}O_4N^+$

Molecular weight

351.403

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>188-190 °C<br>(decomposition) | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{20}$<br>not available | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>methanol | |
| Color<br>yellow | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Corydalis turtschaninovii Bess. f. yanhusuo Y.H. Chou et C.C. Hsu (Papaveraceae) |

5. Health Hazard Data

| Toxicology | Hazard labeling |
|---|---|
| not available | $LD_{50}$ not available |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| |

7. Spectroscopic Data

| MS | (hydrogenated derivative product) | | |
|---|---|---|---|
| Base peak | 178 | Molecular ion | 355 (32%) |
| Ionization energy | 70 eV | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | MM 7070H | |

| IR | | |
|---|---|---|
| Characteristic peaks<br>1590, 1500, 1275, 1100, 1010, 940 $cm^{-1}$ | | |
| Sample preparation | KBr | Resolution – |
| Spectrometer type and manufacturer | | PE-599B |

| NMR | | Nucleus | $^1H$-$^1H$-correlation |
|---|---|---|---|
| Chemical Shifts<br>2.94 (s, 3H, $C_{13}$-$CH_3$);<br>4.04 (s, 3H, -$OCH_3$);<br>6.90 (s, 1H, $C_4$-H);<br>9.82 (s, 1H, $C_8$-H); | 3.02 (br., 2H, -$CH_2$);<br>4.07 (s, 3H, -$OCH_3$);<br>7.29 (s, 1H, $C_1$-H);<br>9.88 (s, 1H, Ar-OH; disappear after $D_2O$) ppm | | 3.82 (s, 3H, -$OCH_3$);<br>4.78 (t, J=5 Hz, 2H, -$CH_2$);<br>8.11 (s, 2H, $C_{11}$-H and $C_{12}$-H); |
| Frequency | 90 MHz | Solvent | DMSO-$d_6$ |
| Standard | TMS | Sample temp. | – |
| Spectrometer type and manufacturer | | EM-390 | |

| UV * | | | |
|---|---|---|---|
| $I_{max}$ | 230, 265, 340, 420 nm | $\varepsilon_{max}$ | – |
| Solvent | methanol | | |
| Sample conc. | – | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | SPECORD UV-VIS | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.52 | | |
| Solvent | methanol | Solvent system | BuOH-AcOH-$H_2O$ (4 : 1 : 5, upper phase) |
| Saturated atmosphere | – | Detection/Color | UV 366 nm |
| Plate manufacturer | hand made | Plate type/Product no. | Silica gel, 0.25 mm |

| GLC | not available |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

10. References

[1] Chiaki Tani et al.: J. Pharm. Soc. Japan (YAKUGAKU ZASSHI) *90*(3), 1970, 407

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# II-2,3-Dihydroamentoflavone

*G. Skopp*

## 1. Name of Compound

Common name

**II-2,3-Dihydroamentoflavone** [1]

Synonyms

–

Systematic name

8-[5-(5,7-Dihydroxy-4-oxo-4*H*-1-benzopyran-2-yl)-2-hydroxyphenyl]-2 ,3-dihydro-5,7-dihydroxy-2-(4-hydroxyphenyl)-4*H*-1-benzopyran-4-one

| Substance | Subgroup |
|---|---|
| Flavonoid | Biflavonoid |

CAS registry number and other numbers

[106577-42-8] BRN 4627107

## 2. Formulas and Molecular Weight

Molecular formular

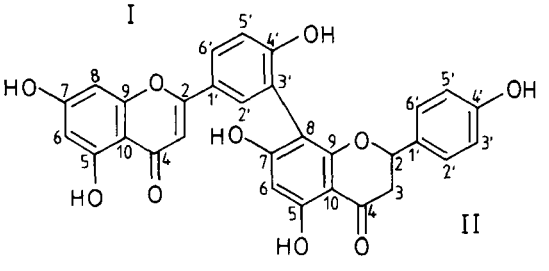
$C_{30}H_{20}O_{10}$

Molecular weight

540.486

Structural formula

I




II

## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| crystalline | not applicable | not applicable |
| Melting Point mp | $n_D^{20}$ | Acid value |
| 212 °C (decomposition) | not applicable | not applicable |
| Boiling point bp | $[\alpha]_D^{20}$ | Saponification value |
| not applicable | not measurable | not applicable |
| Flash point | Soluble in | |
| not applicable | acetone, methanol | |
| Color | Odor | |
| light yellow | odorless | |

4. Occurrence

| |
|---|
| Schinus terebinthifolius RADDI (Anacardiaceae) |

5. Health Hazard Data

| | |
|---|---|
| Toxicology | Hazard labeling – |
| unknown | $LD_{50}$ unknown |
| Waste disposal procedures<br>Combustion | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder; preferably in the dark |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 362 mu | Molecular ion | 540 mu |
| Ionization energy | 70 eV | Ion source temp. | 400 °C |
| Acceleration voltage | 3 kV | Emission current | 0.25 mA |
| Resolution | 1000 | Scan rate | – |
| Spectrometer type and manufacturer | | Dupont 21/492 | |

| | |
|---|---|
| IR | not available, see remarks |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>13.08 (s, I5-OH);<br>10.33 (br, I,II7-OH, I,II4'-OH);<br>7.40 (m, I2',6'-H);<br>6.78 (d, J=9 Hz, II3',5'-H);<br>6.32 (s, I-3H);<br>5.50 (dd, J=10.7, 3.0 Hz, II-2H); | | 12.15 (s, II5-OH);<br>7.58 (d, J=9 Hz, II2',6'-H);<br>6.98 (d, J=9 Hz, I5'-H);<br>6.77 (s, I8-H);<br>5.88 (s, I,II-6H);<br>3.4–2.6 (m, II-3H) ppm | |
| Frequency | 250 MHz | Solvent | DMSO-$d_6$ |
| Standard | TMS | Sample temp. | 21 °C |
| Spectrometer type and manufacturer | | Bruker WM 250 | |

| NMR | | | Nucleus | $^{13}C$ |
|---|---|---|---|---|
| Chemical Shifts | | | | |
| 196.3 (II4); | 181.3 (I4); | 166.5 (II7); | 163.0 (I2, II7); | 162.6 (II5); |
| 160.5 (I5); | 160.2 (I4′); | 159.8 (II9); | 156.9 (I9, II4′); | 131.2 (I6′); |
| 128.0 (II1′); | 127.8 (II2′); | 126.7 (II6′, I2′); | 121.6 (I3′); | 121.3 (I1′); |
| 117.0 (I5′); | 115.6 (II3′, 5′); | 106.1 (II8); | 103.6 (I10); | 101.8 (I3); |
| 101.6 (II10); | 100.2 (I6); | 95.6 (II6); | 94.9 (I8); | 78.6 (II2); |
| – (II3) ppm | | | | |
| Frequency | 62.9 MHz | | Solvent | DMSO-$d_6$ |
| Standard | TMS | | Sample temp. | 21 °C |
| Spectrometer type and manufacturer | | | Bruker WM 250 | |

| UV | additional UV-spectra with shift reagents see ref. [2] | | |
|---|---|---|---|
| $I_{max}$ | 287, 325 sh nm | $\varepsilon_{max}$ | |
| Solvent | Methanol (Uvasol) | | |
| Sample conc. | about $1.9 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Zeiss UV-Spectrophotometer DMR 10, double beam | | |

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.38 | | |
| Solvent | – | Solvent system | dichloromethane : ethyl acetate 1 : 1 (v : v) |
| Saturated atmosphere | yes | Detection/Color | UV : mauve<br>fast blue salt B 0.5%: light red |
| Plate manufacturer | Merck | Plate type/Product no. | Silica Gel 60, $F_{254}$ |

| GLC | underivatized not possible |
|---|---|

| HPLC | not available |
|---|---|

## 9. Remarks

The substance could be isolated just in small amounts. For structure elucidation NMR spectroscopic techniques were preferred.

10. References


[1] Skopp G.: Über phenolische Inhaltsstoffe aus Schinus terebinthifolius RADDI – n-Alkylphenole und Biflavonoide. Diss., 1986, *(name, MS, $^{1}$H-NMR, $^{13}$C-NMR, UV spectroscopy)*

[2] Markham, K.R.: Techniques of Flavonoid Identification. Academic Press, London, New York 1982 *(UV spectroscopy)*

# 2,3-Dihydro-5′,3‴-dihydroxyamentoflavone

*S. Anhut, H. Geiger, H.D. Zinsmeister*

## 1. Name of Compound

Common name
**2,3-Dihydro-5′,3‴-dihydroxyamentoflavone [1]**

Synonyms

Systematic name
8-[5-(2,3-Dihydro-5,7-dihydroxy-4-oxo-4*H*-1-benzopyran-2-yl)-2,3-dihydroxyphenyl]-5,7-dihydroxy-2-(3,4-dihydroxyphenyl)-4*H*-1-benzopyran-4-one

| Substance | Subgroup |
|---|---|
| Flavonoid | Biflavone |

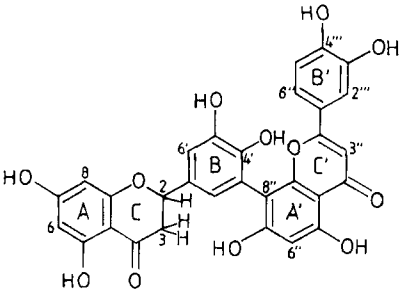
CAS registry number and other numbers
[122475-59-6]

## 2. Formulas and Molecular Weight

Molecular formular
$C_{30}H_{20}O_{12}$

Molecular weight
572.47

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline | $d_4^{20}$<br>not applicable | Iodine value<br>not applicable |
| Melting Point mp<br>225-227 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20} = -1.5°$<br>(c=0.0107 g/ml, acetone)<br>see remarks | Saponification value<br>not applicable |
| Flash point<br>not applicable | Soluble in<br>acetone, methanol | |
| Color<br>yellow | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Plagiomnium cuspidatum (Musci) [1, 2] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| not known | $LD_{50}$ | not known |
| Waste disposal procedures | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder; preferably in the dark |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS * | FAB technique – negative mode- Xenon gas -glycerol matrix | | |
| Base peak | | Molecular ion | 571 $[M-H]^-$ |
| Ionization energy | 4–6 keV | Ion source temp. | 40–50 °C |
| Acceleration voltage | – | Emission current | – |
| Resolution | >1000 | Scan rate | – |
| Spectrometer type and manufacturer | | Finnigan MAT 90 | |

| | |
|---|---|
| IR | see remarks |

| | | | |
|---|---|---|---|
| NMR | | Nucleus | $^1H$ |
| Chemical Shifts<br>7.12 (d, J = 2 Hz, H-6′);<br>6.84 (d, J = 2 Hz, H-2′);<br>6.38 (s, H-6″);<br>5.44 (dd, J = 3, 12 Hz, H-2);<br>2.79 (dd, J = 3, 17 Hz, H-3) ppm | 7.03 (dd, J = 2, 8 Hz, H-6‴);<br>6.77 (d, J = 8 Hz, H-5‴);<br>5.92 (d, J = 2 Hz, H-8);<br>3.16 (dd, J = 12, 17 Hz, H-3); | | 7.02 (d, J = 2 Hz, H-2‴);<br>6.53 (s, H-3″);<br>5.89 (d, J = 2 Hz, H-6); |
| Frequency | 400 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | 100 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | see ref. [1, 3] | Nucleus | $^{13}C$ |
|---|---|---|---|

Chemical Shifts

| | | | | |
|---|---|---|---|---|
| 195.4 (C-4); | 181.5 (C-4″); | 166.2 (C-7); | 163.6 (C-2″); | 163.1 (C-5); |
| 162.6 (C-9); | 161.5 (C-7″); | 160.0 (C-5″); | 154.2 (C-9″); | 149.1 (C-4‴); |
| 145.2 (C-3‴); | 145.1 (C-4′); | 143.9 (C-5′); | 128.5 (C-1′); | 121.8 (C-1‴); |
| 120.8 (C-2′); | 119.2 (C-3′); | 118.4 (C-6‴); | 115.4 (C-5‴); | 113.5 (C-2‴); |
| 112.8 (C-6′); | 104.6 (C-8″); | 103.5 (C-10″); | 102.1 (C-3″); | 101.6 (C-10); |
| 98.4 (C-6″); | 95.5 (C-6); | 94.7 (C-8); | 78.2 (C-2); | 42.1 (C-3) ppm |

| Frequency | 100 MHz | Solvent | DMSO-$d_6$ |
|---|---|---|---|
| Standard | DMSO-$d_6$ | Sample temp. | 100 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 278 sh, 288, 339 nm | $\varepsilon_{max}$ | – |
| Solvent | Methanol (Uvasol)<br>additional UV-spectra with shift reagents according to ref. [4] | | |
| Sample conc. | about $2 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Super Scan 3 (Varian); UV-VIS-spectrophotometer, double beam | | |

8. Chromatographic Data

| TLC | I-II on Cellulose (Avicel); III on Polyamide-6 | | |
|---|---|---|---|
| Rf Value | System I: 0.94, II: 0.43, III: 0.53 | | |
| Solvent system | I: BuOH(1) – Acetic Acid – Water (4 : 1 : 5) upper layer<br>II: Acetic Acid – Water (4 : 6)<br>III : Ethylacetate-Butanone(2)-Formic Acid-Water (5 : 3 : 1:1) | | |
| Saturated atmosphere | yes | Detection/Color | UV : deep purple;<br>UV, sprayed with Naturstoffreagenz A (NA): yellowish, turning brick red |
| Plate manufacturer | Schleicher & Schüll<br>Macherey & Nagel | Plate type/Product no. | Cellulose, Avicel F 1440<br>Polygram; 0.1 mm; Polyamide-TLC 6 |

| GLC | underivatized not possible |
|---|---|

| HPLC | | | |
|---|---|---|---|
| Retention time | 11.5 min | | |
| Column | Macherey & Nagel ET 250/8/4 with Nucleosil 5C18 guard column Nucleosil 5C18; 30 × 4 mm I.D. | Stationary phase | Nucleosil RP-18, 5 µm |
| Mobile phase | A: MeOH<br>B: Water – Acetic Acid (95 : 5) | Flow rate | 0.7 ml/min<br>isocratic 65A – 35B |
| Column temp. | ≈25 °C | Pressure | – |
| Detector | UV-Detector Waters 450<br>Detection: 330 nm | Sample solvent | MeOH |
| Sample size | 10 µl | Sample conc. | – |
| Chromatograph type and manufacturer | | Waters M 45 | |

9. Remarks

For structure elucidation only NMR spectroscopic techniques were used, since they allow a subsequent recovery. To get good resolved NMR-spectra these had to be recovered at 100 °C to overcome the rotation barrier around the interflavonylic linkage. The $[\alpha]_D^{20}$-value had been determined from the recovered sample.

10. References


[1] Anhut, S., Seeger, T., Zinsmeister, H.D. and Geiger, H.: New Dihydrobiflavones from the Moss Plagiomnium cuspidatum. *Z. Naturforsch. 44c*, 1989, pp 189–92 *(name, occurrence, $^{13}$C-NMR)*

[2] Geiger, H.: Biflavonoids in Bryophytes in: ‚Chemistry and Chemical Taxonomy of Bryophytes'. Zinsmeister, H.D. and Mues, R. (Eds.), Ann. Proc. Phytochem. Soc. Europe, Vol. 29, Oxfort UP, 1989 *(occurrence)*

[3] Markham, K.R. and Chari, V.M.: Carbon-13 NMR Spectroscopy of Flavonoids.in ‚The Flavonoids', Harborne, J.B. and Mabry (Eds.), Chapman and Hall London, 1982 *($^{13}$C-NMR)*

[4] Mabry, T.J., Markham, K.R. and Thomas, M.B.: The Systematic Identification of Flavonoids. Springer Verlag, 1970 *(UV spectroscopy)*

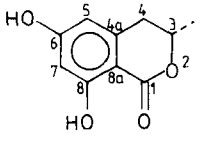
# (-)-3,4-Dihydro-6,8-dihydroxy-3-methylisocoumarin

*C. Franke, K. Krohn*

## 1. Name of Compound

| |
|---|
| Common name<br>**(-)-3,4-Dihydro-6,8-dihydroxy-3-methylisocoumarin** |
| Synonyms<br>(-)-6,8-Dihydroxy-3-methylisochroman-1-one |
| Systematic name<br>(-)-3,4-Dihydro-6,8-dihydroxy-3-methyl-1*H*-2-benzopyran-1-one |
| Substance: Polyketid — Subgroup: Isocoumarin |
| CAS registry number and other numbers<br>[19314-92-2] — BRN 3544385 |

## 2. Formulas and Molecular Weight

| | |
|---|---|
| Molecular formular<br>$C_{10}H_{10}O_4$<br><br>Molecular weight<br>194.19 | Structural formula<br>  |

## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not applicable |
| Melting Point mp<br>214–215 °C [1] | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{20} = -63°$<br>(c=0.6 g/l, EtOH) [1]<br>$[\alpha]_D^{25} = -56°$<br>(c=0.6 g/l, EtOH) | Saponification value<br>not available |
| Flash point<br>not available | Soluble in<br>methanol, acetone | |
| Color<br>colorless | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Isolated from the fungi Ceratocystis [2], Pyricularia oryzae [5], Plectophomella. |

5. Health Hazard Data

| Toxicology | Hazard labeling |
|---|---|
| unkown | $LD_{50}$ |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 194 | Molecular ion | 194 |
| Ionization energy | 70 eV | Ion source temp. | 80 °C |
| Acceleration voltage | 3 kV | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | 3 s/decade |
| Spectrometer type and manufacturer | | Finnigan MAT 8430 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3220; 3217; 3103; 3098; 3082; 1653; 1633; 1588; 1503; 1478; 1386; 1290; 1257; 1221; 1195; 1170; 1121; 1068; 736; 714; 589 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | 4.0 $cm^{-1}$ |
| Spectrometer type and manufacturer | | Nicolet 320 FT-IR-spectrometer | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>1.32 (d; J = 6 Hz; 3H, $CH_3$);<br>2.83 (ABq; J = 16, 4 Hz; 1H, CH);<br>6.13 (d; J = 2 Hz; 1H, Ar-H);<br>9.35 (s; 1H, OH); | | 2.72 (ABq; J = 16, 11 Hz; 1H, CH);<br>4.57 (m; 1H, CH);<br>6.16 (d; J = 2 Hz; 1H, Ar-H);<br>11.16 (s; 1H, OH) ppm | |
| Frequency | 400 MHz | Solvent | acetone-$d_6$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | $^1H$ decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>20.8 (q, $CH_3$),<br>101.9 (d, C-7),<br>165.2 (s, C-6), | 35.0 (t, C-4),<br>107.4 (d, C-5),<br>170.7 (s, C-1) ppm | 76.3 (d, C-3),<br>143.2 (s, C-8a), | 101.7 (s, C-4a),<br>165.1 (s, C-8), |
| Frequency | 100 MHz | Solvent | acetone-$d_6$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 216, 268, 302 nm | $\varepsilon_{max}$ | 20254, 12942, 5821 |
| Solvent | methanol | | |
| Sample conc. | $6.231 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Beckman UV 5230 | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.5 | | |
| Solvent | – | Solvent system | 7% MeOH/93% $CH_2Cl_2$ |
| Saturated atmosphere | – | Detection/Color | 254 nm/violet |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel $60F_{254}$/Merck 5562 |

| | |
|---|---|
| GLC | not available |

| | |
|---|---|
| HPLC | not available |

9. Remarks

Sublimation: 140 °C, 0.06 mm

10. References


[1] Curtis, R.F., Harries, P.C., Hassall, C.H., Levi, J.D., Phillips, D.M., *J. Chem. Soc. (C)*, 1966, pp. 168–174 *(biosynthesis)*

[2] Fisher, H. et al., *J. Org. Chem. 54*, 1989, pp. 4218–20 *(synthesis, $^{1}$H-NMR, $^{13}$C-NMR, UV)*

[3] Ayer, W.A., Attah-Poku, S.K., Browne, L.M., Orszanska, H., *Can. J. Chem. 65*, 1987, 765 *pp. (isolation)*

[4] Hill, R.A., Carter, R.H., Stannton, J., *J. Chem. Soc., Perkin Trans. 1*, 1981, 2570 *pp. (synthesis)*

[5] Iwasaki, S., Muro, H., Sasaki, K., Nozoe, S., Okuda, S., *Tetrahedron Lett. 37*, 1973, pp. 3537–42 *(isolation)*

[6] Webster, B.R., Rix, M.J., *J. Chem. Soc. (B)*, 1968, pp. 254–58 *(MS)*

# 2,3-Dihydro-5′-hydroxyamentoflavone

*S. Anhut, H. Geiger, H.D. Zinsmeister*

## 1. Name of Compound

Common name
**2,3-Dihydro-5′-hydroxyamentoflavone** [1]

Synonyms

Systematic name
8-[5-(2,3-Dihydro-5,7-dihydroxy-4-oxo-4H-1-benzopyran-2-yl)-2,3-dihydroxyphenyl]-5,7-dihydroxy-2-(4-hydroxyphenyl)-4*H*-1-benzopyran-4-one

Substance
Flavonoid

Subgroup
Biflavone

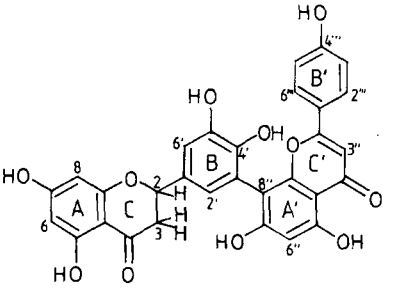
CAS registry number and other numbers
[122475-58-5]

## 2. Formulas and Molecular Weight

Molecular formular
$C_{30}H_{20}O_{11}$

Molecular weight
556.47

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline | $d_4^{20}$<br>not applicable | Iodine value<br>not applicable |
| Melting Point mp<br>188–190 °C (decomp.) | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20} = -7°$<br>(c = 0.01096 g/ml, acetone)<br>see remarks | Saponification value<br>not applicable |
| Flash point<br>not applicable | Soluble in<br>acetone, methanol | |
| Color<br>yellow | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Plagiomnium cuspidatum (Musci) [1, 2] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| not known | $LD_{50}$ | not known |
| Waste disposal procedures | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder; preferably in the dark. |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS * | FAB technique – negative mode – Xenon gas – glycerol matrix | | |
| Base peak | – | Molecular ion | 555 m/e $[M-H]^-$ |
| Ionization energy | 4–6 keV | Ion source temp. | 40–50 °C |
| Acceleration voltage | V | Emission current | – |
| Resolution | >1000 | Scan rate | – |
| Spectrometer type and manufacturer | | Finnigan MAT 90 | |

| | |
|---|---|
| IR | see remarks |

| | | | |
|---|---|---|---|
| NMR | | Nucleus | $^1H$ (see remarks) |
| Chemical Shifts<br>7.56 (d, J = 9 Hz, H-2‴, H-6‴);<br>6.87 (d, J = 2 Hz, H-2′);<br>6.65 (s, H-3″);<br>5.92 (d, J = 2 Hz, H-8);<br>5.46 (dd, J = 3, 12 Hz, H-2);<br>2.78 (dd, J = 3, 17 Hz, H-3) ppm | | 7.04 (d, J = 2 Hz, H-6′);<br>6.81 (d, J = 9 Hz, H-3‴, H-5‴);<br>6.40 (s, H-6″);<br>5.91 (d, J = 2 Hz, H-6);<br>3.18 (dd, J = 12, 17 Hz, H-3); | |
| Frequency | 400 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | 100 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | see ref. [1, 3] | Nucleus | $^{13}C$ |
|---|---|---|---|

| Chemical Shifts | | | | |
|---|---|---|---|---|
| 195.3 (C-4); | 181.6 (C-4″); | 166.2 (C-7); | 163.4 (C-2″); | 163.1 (C-5); |
| 162.6 (C-9); | 161.4 (C-7″); | 160.5 (C-5″); | 160.0 (C-4‴); | 154.2 (C-9″); |
| 145.1 (C-4′); | 143.9 (C-5′); | 128.6 (C-1′); | 127.7 (C-2‴, C-6‴); | |
| 121.3 (C-1‴); | 120.9 (C-2′); | 119.2 (C-3′); | 115.4 (C-3‴, C-5‴); | |
| 112.8 (C-6′); | 104.5 (C-8″); | 103.5 (C-10″); | 102.1 (C-3″); | 101.6 (C-10); |
| 98.4 (C-6″); | 95.5 (C-6); | 94.7 (C-8); | 78.2 (C-2); | 42.1 (C-3) ppm |

| Frequency | 100 MHz | Solvent | DMSO-$d_6$ |
|---|---|---|---|
| Standard | DMSO-$d_6$ | Sample temp. | 100 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 277sh, 288, 326 nm | $\varepsilon_{max}$ | – |
| Solvent | Methanol (Uvasol)<br>additional UV spectra with shift reagents according to ref. [4] | | |
| Sample conc. | about $2 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Super Scan 3 (Varian); UV-VIS-spectrophotometer, double beam | | |

8. Chromatographic Data

| TLC | I, II on Cellulose (Avicel); III on Polyamide-6 | | |
|---|---|---|---|
| Rf Value | System I: 0.95, II: 0.55, III: 0.67 | | |
| Solvent system | I: Butanol-(1) : Acetic Acid : Water (4 : 1 : 5) upper layer<br>II: Acetic Acid : Water (40 : 60)<br>III: Ethylacetate : Butanone(2) : Formic Acid : Water (5 : 3 :1 : 1) | | |
| Saturated atmosphere | yes | Detection/Color | UV deep purple; UV, sprayed with Naturstoffreagenz A (NA): yellowish, turning bright red |
| Plate manufacturer | Schleicher & Schüll<br>Macherey & Nagel | Plate type/Product no. | Cellulose, Avicel F 1440<br>Polygram; 0.1 mm; Polyamide-TLC 6 |

| GLC | underivatized not possible |
|---|---|

| HPLC | | | |
|---|---|---|---|
| Retention time | 15.5 min | | |
| Column | Macherey & Nagel<br>ET 250/8/4 with Nucleosil 5C18 guard column<br>Nucleosil 5C18; 30 × 4 mm I.D. | Stationary phase | Nucleosil RP-18, 5 µm |
| Mobile phase | A: MeOH<br>B: Water – Acetic Acid (95 : 5) | Flow rate | 0.7 ml/min<br>isocratic 65A – 35B |
| Column temp. | ambient temp., ≈25 °C | Pressure | – |
| Detector | UV-Detector Waters 450<br>Detection 330 nm | Sample solvent | MeOH |
| Sample size | 10 µl | Sample conc. | – |
| Chromatograph type and manufacturer | | Waters M 45 | |

9. Remarks

For structure elucidation only NMR spectroscopic techniques were used, since they allow a subsequent recovery. To get good resolved NMR-spectra these had to be recorded at 100 °C to overcome the rotation barrier around the interflavonylic linkage. The $[\alpha]_D^{20}$-value had been determined from the recovered sample. By $^1$H-NMR studies it was obvious that this compound contains additionally a small amount of 2,3-Dihydro-5′-hydroxyrobustaflavone [1] which is new, too.

10. References


[1] Anhut, S., Seeger, T., Zinsmeister, H.D. and Geiger, H.: New Dihydrobiflavones from the Moss Plagiomnium cuspidatum. *Z. Naturforsch. 44c*, 1989, pp 189–92 *(name, occurrence, $^{13}$C-NMR)*

[2] Geiger, H.: Biflavonoids in: Bryophytes. In:: ‚Chemixtry and Chemical Taxonomy of Bryophytes'. Zinsmeister, H.D. and Mues, R. (Eds.), Ann. Proc. Phytochem. Soc. Europe, Vol. 29, Oxfort UP, 1989 *(occurrence)*

[3] Markham, K.R. and Chari, V.M.: Carbon-13 NMR Spectroscopy of Flavonoids. In: ‚The Flavonoids', Harborne, J.B. and Mabry (Eds.), Chapman and Hall London, 1982 *($^{13}$C-NMR)*

[4] Mabry, T.J., Markham, K.R. and Thomas, M.B.: The Systematic Identification of Flavonoids. Springer Verlag, 1970 *(UV spectroscopy)*

# 5′,3‴-Dihydroxyrobustaflavone

*H.D. Zinsmeister, R. Mues, S. Anhut*

## 1. Name of Compound

Common name

**5′,3‴-Dihydroxyrobustaflavone** [1]

Synonyms

5′,6″-Biluteolin

Systematic name

6-[5-(5,7-Dihydroxy-4-oxo-4*H*-1-benzopyran-2-yl)-2,3-dihydroxyphenyl]-2-(3,4-dihydroxyphenyl)-5,7-dihydroxy-4H-1-benzopyran-4-one

| Substance | Subgroup |
|---|---|
| Flavonoid | Biflavone |

CAS registry number and other numbers

[104056-04-4] [1] [114865-40-6] [5]

## 2. Formulas and Molecular Weight

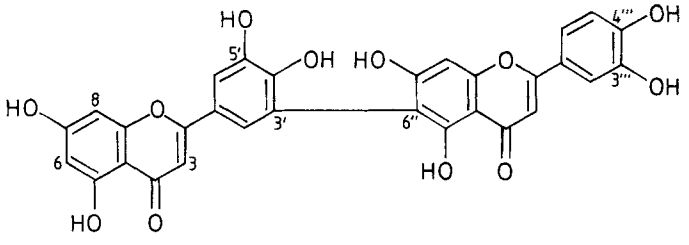
Molecular formular

$C_{30}H_{18}O_{12}$

Molecular weight

570.46

Structural formula




## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| crystalline | not applicable | not applicable |
| Melting Point mp | $n_D^{20}$ | Acid value |
| >255 °C (decomp.) | not applicable | not applicable |
| Boiling point bp | $[\alpha]_D^{20}$ | Saponification value |
| not applicable | not measurable | not applicable |
| Flash point | Soluble in | |
| not applicable | acetone (heated) | |
| Color | Odor | |
| yellow | odorless | |

4. Occurrence

| |
|---|
| Plagiomnium cuspidatum, Dicranoloma robustum, Hylocomium splendens (Musci), Racomitrium lanuginosum (Musci), Antitrichia curtipendula (Musci) [1, 2; 5] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| not known | $LD_{50}$ | not known |
| Waste disposal procedures | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder; preferably in the dark. |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS | FD – Mass spectrum | | |
| Base peak | – | Molecular ion | 570 m/e |
| Ionization energy | – | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | Varian MAT 311 with FD source | |

| | |
|---|---|
| IR | not available |

| | | | |
|---|---|---|---|
| NMR | | Nucleus | $^1H$ |
| Chemical Shifts<br>7.46 (dd, J=8, 2 Hz, H-6‴);<br>7.35 (d, J=2 Hz, H-2′);<br>6.65 (s, H-3);<br>6.20 (d, J=2 Hz, H-6) ppm | 7.45 (d, J=2 Hz, H-2‴);<br>6.92 (d, J=8 Hz, H-5‴);<br>6.59 (s, H-8″); | 7.40 (d, J=2 Hz, H-6′);<br>6.72 (s, H-3″);<br>6.45 (d, J=2 Hz, H-8); | |
| Frequency | 400 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | see ref. [3] | Nucleus | $^{13}C$ | see also remarks |
|---|---|---|---|---|

| Chemical Shifts | | | | |
|---|---|---|---|---|
| 181.6 (C-4); | 181.5 (C-4″); | 164.0 (C-2); | 164.0 (C-2″); | 163.6 (C-7); |
| 162.3 (C-7″); | 161.4 (C-5); | 159.0 (C-5″); | 157.2 (C-9); | 156.2 (C-9″); |
| 149.6 (C-4‴); | 148.6 (C-4′); | 145.7 (C-5′); | 145.7 (C-5‴); | 121.9 (C-2′); |
| 121.5 (C-1′); | 120.9 (C-1‴); | 120.2 (C-3′); | 118.9 (C-6‴); | 116.0 (C-5‴); |
| 113.3 (C-2‴); | 111.7 (C-6′); | 108.9 (C-6″); | 103.6 (C-10); | 103.4 (C-10″); |
| 102.8 (C-3); | 102.7 (C-3″); | 98.7 (C-6); | 93.8 (C-8″); | 93.4 (C-8) ppm |

| Frequency | 100 MHz | Solvent | DMSO-$d_6$ |
|---|---|---|---|
| Standard | DMSO-$d_6$ | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 249, 254, 295sh, 354 nm | $\varepsilon_{max}$ | – |
| Solvent | Methanol (Uvasol)<br>additional UV-spectra with shift reagents according to ref. [4] | | |
| Sample conc. | about $2 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Super Scan 3 (Varian); UV-VIS-spectrophotometer, double beam | | |

8. Chromatographic Data

| TLC | I-II on Cellulose (Avicel); III on Polyamide-6 | | |
|---|---|---|---|
| Rf Value | System I: 0.92, II: 0.29, III: 0.40 | | |
| Solvent system | I: BuOH(1) – Acetic Acid – Water (4 : 1 : 5) upper layer<br>II: Acetic Acid – Water (40 : 60)<br>III: Ethylacetate – Butanone(2) – Formic Acid – Water (5 : 3 : 1 : 1) | | |
| Saturated atmosphere | yes | Detection/Color | UV : deep purple;<br>UV, sprayed with Diphenylboric acid - β-aminoethylester (NA): yellow |
| Plate manufacturer | Schleicher & Schüll<br>Macherey & Nagel | Plate type/Product no. | Cellulose, Avicel F 1440<br>Polygram; 0.1 mm; Polyamide-TLC 6 |

| GLC | underivatized not possible |
|---|---|

| HPLC | | | |
|---|---|---|---|
| Retention time | 10.75 min | | |
| Column | Macherey & Nagel ET 250/8/4 with Nucleosil 5C18 guard column Nucleosil 5C18; 30 × 4 mm I.D. | Stationary phase | Nucleosil RP-18, 5 µm |
| Mobile phase | MeOH – 5% Acetic Acid (65:35) | Flow rate | 0.8 ml/min |
| Column temp. | ambient temp., ≈ 25 °C | Pressure | – |
| Detector | UV-Detector Waters 450 Detection: 330 nm | Sample solvent | MeOH |
| Sample size | 10–15 µl | Sample conc. | – |
| Chromatograph type and manufacturer | | Waters M 45 | |

9. Remarks

The substance could be isolated just in small amounts. For structure elucidation only NMR spectroscopic techniques were used, since they allow a subsequent recovery. The $^{13}$C-NMR-values for C-5 and C-5″ in [1] have to be altered.

10. References


[1] Becker, R., Mues, R., Zinsmeister, H.D., Herzog, F. and Geiger, H.: A new Biflavone and further Flavonoids from the Moss hylocomium splendens. *Z. Naturforsch. 41c*, 1986, pp 507–10 *(name, occurrence)*

[2] Geiger, H. and Quinn, C.: Biflavonoids. In: ‚The Flavonoids', Harborne, J.B. (Ed.), Chapman and Hall, London, 1988 *(occurrence)*

[3] Markham, K.R. and Chari, V.M.: Carbon-13 NMR Spectroscopy of Flavonoids. In: ‚The Flavonoids', Harborne, J.B. and Mabry (Eds.), Chapman and Hall, London, 1982 *($^{13}$C-NMR)*

[4] Mabry, T.J., Markham, K.R. and Thomas, M.B.: The Systematic Identification of Flavonoids. Springer Verlag, 1970 *(UV spectroscopy)*

[5] Geiger, H., Anhut, S. and Zinsmeister, H.D.: Biflavones from some Mosses. *Z. Naturforsch. 43c*, 1988, pp 1–4 *(occurrence)*

# Epicorazine A

*C. Franke, K. Krohn*

## 1. Name of Compound

Common name
**Epicorazine A**

Synonyms

Systematic name
4,4a,7,7a,11,11a,14,14a-Octahydro-4,11-dihydroxy-[4S-(4α,4aα,6aß,7aß,11α,11aα,13aß,14aß)]-8H,13H-6a,13a-epidithio-1H,6H-pyrazino[1,2-a:4,5-a']-diindole-1,6,8,13-tetrone

| Substance | Subgroup |
|---|---|
| Piperazine | Epidithiodiketopiperazine |

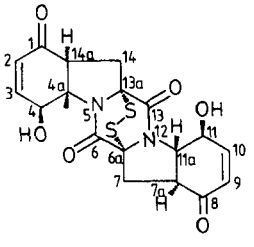
| CAS registry number and other numbers | |
|---|---|
| [62256-05-7] | BRN 1095850 |

## 2. Formulas and Molecular Weight

Molecular formular
$C_{18}H_{16}N_2O_6S_2$

Molecular weight
420.45

Structural formula



## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| solid | not applicable | not available |
| **Melting Point mp** | $n_D^{20}$ | **Acid value** |
| 198 °C | not applicable | not applicable |
| **Boiling point bp** | $[\alpha]_D^{22} = -293°$ | **Saponification value** |
| not available | (1,7 mg/ml, $CHCl_3$) [1] | not applicable |
| **Flash point** | **Soluble in** | |
| not available | methanol, acetone, chloroform, ... | |
| **Color** | **Odor** | |
| colorless | odorless | |

4. Occurrence

| |
|---|
| Isolated from the fungus Epicoccum nigrum. |

5. Health Hazard Data

| Toxicology | Hazard labeling |
|---|---|
| cytotoxic activity | $LD_{50}$ unknown |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| Storage temperature |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 356 | Molecular ion | 420 |
| Ionization energy | 70 eV | Ion source temp. | 300 °C |
| Acceleration voltage | 3 kV | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | 3 s/decade |
| Spectrometer type and manufacturer | | Finnigan MAT 8430 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3350, 1690, 1670, 1405, 1375, 1360, 1245, 1190, 1180, 1135, 1090, 1030, 810, 670 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | 2.5 $cm^{-1}$ |
| Spectrometer type and manufacturer | | Perkin Elmer 1420 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>2.60 (dd, $J_{14ß\text{-}14a}$ = 5.5 Hz, $J_{14ß\text{-}14α}$ = 14.8 Hz, 1H, H-14ß);<br>3.04 (dd, $J_{14α\text{-}14ß}$ = 14.8 Hz, $J_{14α\text{-}14a}$ = 12.7 Hz, 1H, H-14α);<br>3.27 (ddd, $J_{14a\text{-}4a}$ = 12.7 Hz, $J_{14a\text{-}14α}$ = 12.3 Hz, $J_{14a\text{-}14ß}$ = 5.6 Hz, 1H, H-14a);<br>3.83 (dd, $J_{4a\text{-}14a}$ = 13 Hz, $J_{4\text{-}4a}$ = 8.2 Hz, 1H, H-4a);<br>4.81 (d, $J_{4\text{-}4a}$ = 7.8 Hz, 1H, H-4);<br>5.86 (s, 1H, OH);<br>6.16 (dd, $J_{2\text{-}3}$ = 10.2 Hz, $J$ = 2 Hz, 1H, H-2);<br>7.12 (d, $J_{3\text{-}2}$ = 10.2 Hz, 1H, H-3) ppm | | | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | $^1H$ decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>31.0 (t; C-14), 48.9 (d; C-14a), 69.4 (d; C-4a), 71.1 (d; C-4),<br>75.7 (s; C-13a), 129.2 (d; C-2), 150.7 (d; C-3), 164.2 (s; C-13),<br>193.7 (s; C-1) ppm | | | |
| Frequency | 100 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 212 nm | $\varepsilon_{max}$ | 11481 |
| Solvent | methanol | | |
| Sample conc. | $2.4732 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Beckmann UV 5230 | |

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.65 | | |
| Solvent | – | Solvent system | 7% MeOH/93% $CH_2Cl_2$ |
| Saturated atmosphere | | Detection/Color | 254 nm/violet |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel $60F_{254}$/Merck 5562 |

| | |
|---|---|
| GLC | not available |

| | |
|---|---|
| HPLC | not available |

9. Remarks

Active against gram positive bacteria.

10. References


[1] Baute, M.A. et al., *J. Antibiotics 31*, 1978, 1099 pp. *(fermentation, isolation, biologic activity)*
[2] Baute, M.A. et al., *Bull. Soc. Pharm. Bordeaux 119*, 1980, 213-10 *(isolation, biologic activity)*
[3] Baute, M.A. et al., *Bull. Soc. Pharm. Bordeaux 120*, 1981, pp. 23-32 *(structure, MS, NMR, IR)*
[4] Baute, M.A. et al., *Tetrahedron Lett. 44*, 1976, pp. 3943-4 *(MS, NMR, IR, UV)*
[5] Deffieux G., Gardet, M., Leger, J.-M., Acta Cryst. B33, 1977, pp. 1474-78 *(crystallization)*

# Fabiatrin

*A.A. Semenov, A.I. Syrchina*

## 1. Name of Compound

Common name

**Fabiatrin** [1]

Synonyms

Systematic name

6-Methoxy-7-[(6-0-ß-D-xylopyranosyl-ß-D-glucopyranosyl)oxy]-2*H*-1-benzopyran-2-one

| Substance | Subgroup |
|---|---|
| Coumarin | Coumarin glycoside |

CAS registry number and other numbers

[18309-73-4] BRN 71130

## 2. Formulas and Molecular Weight

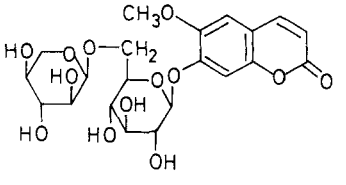
Molecular formular

$C_{21}H_{26}O_{13}$

Molecular weight

486.433

Structural formula



## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline | $d_4^{20}$<br>not applicable | Iodine value<br>not applicable |
| Melting Point mp<br>234-236 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20}$<br>see ref. [2] | Saponification value<br>not applicable |
| Flash point<br>not applicable | Soluble in<br>water, ethanol, methanol | |
| Color<br>white | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Physochlaina spp. [1, 3], other spp. [2] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| unknown | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder, preferably in the dark |

7. Spectroscopic Data

| | |
|---|---|
| MS | not available |

| | |
|---|---|
| IR | not available |

| | | | |
|---|---|---|---|
| NMR * | | Nucleus | $^1H$ |
| Chemical Shifts<br>7.95 (d, $J$=9.5 Hz, H-3);<br>6.31 (d, $J$=9.5 Hz, H-4);<br>3.81 (s, $OCH_3$-6, 3H); | 7.28 (s, H-8);<br>5.07 (br d, $J$=7 Hz, H-1′);<br>3.9–2.7 (m, glu, xyl) ppm | 7.18 (s, H-5);<br>4.13 (d, $J$=7 Hz, H-1″); | |
| Frequency | 89.55 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | 28 °C |
| Spectrometer type and manufacturer | | JEOL FX-90Q | |

| | | | | | | |
|---|---|---|---|---|---|---|
| NMR * | | | Nucleus | $^{13}C$ | | |
| Chemical Shifts<br>160.6 (2); | 149.9 (7); | 149.0 (9); | 146.1 (6); | 144.3 (4); | 113.4 (3) | 112.4 (10); |
| 109.9 (5); | 104.2 (1″); | 103.2 (8); | 99.6 (1′); | 76.7 (5′, 3″); | 75.5 (3′); | |
| 73.3/73.1 (2′, 2″); | 69.6 (4′); | 63.3 (4″); | 68.3 (6′); | 65.7 (5″); | 56.12 ($OCH_3$) ppm | |
| Frequency | 22.49 MHz | | Solvent | DMSO-$d_6$ | | |
| Standard | DMSO-$d_6$ | | Sample temp. | 28 °C | | |
| Spectrometer type and manufacturer | | | JEOL FX-90Q | | | |

| UV * | | | |
|---|---|---|---|
| $\lambda_{max}$ | 230, 283, 340 nm | $\varepsilon_{max}$ | |
| Solvent | methanol | | |
| Samp. conc. | about $2 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Specord UV-vis | |

8. Chromatographic Data

| TLC | not available |
|---|---|

| GLC | not available |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

10. References


[1] Daandai, G., Naran, R., Gantimur, D., Syrchina, A.I., Larin, M.F., Semenov, A.A., Khim. Prir. Soed. 1 (1988), 130 *(occurence, $^{13}$C-NMR)*

[2] Murray, R.D.H., Mendez, J., Brown, S.A., The natural coumarins, John Wiley, 1982 *(name, occurence)*

[3] Chen Zenai, Chang Xingruo et al., Zhongcaoyao, 12 (1981), 1 (CA 96: 11552, 1982) *(occurence)*

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# Heterobryoflavone

*S. Anhut, H.D. Zinsmeister, R. Mues*

## 1. Name of Compound

Common name
**Heterobryoflavone**

Synonyms

Systematic name
8-[5-(5,7-Dihydroxy-4-oxo-4*H*-1-benzopyran-3-yl)-2,3-dihydroxyphenyl]-2-(3,4-dihydroxyphenyl)-5,7-dihydroxy-4*H*-1-benzopyran-4-one

| Substance | Subgroup |
|---|---|
| Flavonoid | Biflavonoid |

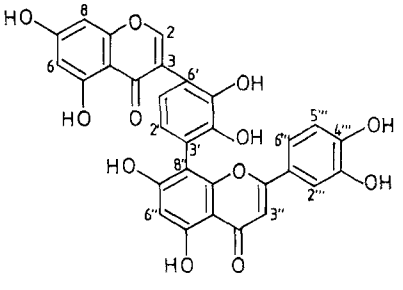
CAS registry number and other numbers
[111200-23-8]

## 2. Formulas and Molecular Weight

Molecular formular
$C_{30}H_{18}O_{12}$

Molecular weight
570.462

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline | $d_4^{20}$<br>not applicable | Iodine value<br>not applicable |
| Melting Point mp<br>240-245 °C (decomp.) | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20}$<br>not measurable | Saponification value<br>not applicable |
| Flash point<br>not applicable | Soluble in<br>acetone, methanol | |
| Color<br>yellow | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Bryum capillare (Musci) [1, 2] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| not known | $LD_{50}$ | not known |
| Waste disposal procedures | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder; preferably in the dark. |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS | FD-Mass spectrum | | |
| Base peak | | Molecular ion | 570 mu |
| Ionization energy | | Ion source temp. | |
| Acceleration voltage | | Emission current | |
| Resolution | | Scan rate | |
| Spectrometer type and manufacturer | | Varian MAT 311 with FD source | |

| | |
|---|---|
| IR | not available |

| | | | |
|---|---|---|---|
| NMR | | Nucleus | $^1H$ |
| Chemical Shifts<br>13.13 and 13.01 (s, OH-5″ and OH-5);<br>7.13 (d, J = 2 Hz, H-2‴);<br>7.06 (dd, J = 8, 2 Hz, H-6‴);<br>6.70 (d, J = 8 Hz, H-5‴);<br>6.37 (d, J = 2 Hz, H-8);<br>6.22 (d, J = 2 Hz, H-6) ppm | | 8.29 (s, Isoflav, H-2);<br>7.12 (d, J = 2 Hz, H-6′);<br>6.87 (d, J = 2 Hz, H-2′);<br>6.62 (s, Flavone, H-3″);<br>6.36 (s, H-6″); | |
| Frequency | 400 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | see ref. [1, 3, 4] | Nucleus | $^{13}C$ | |
|---|---|---|---|---|
| Chemical Shifts | | | | |
| 181.8 (C-4″); | 180.1 (C-4); | 164.0 (C-7); | 163.7 (C-2); | 161.9 (C-7‴); |
| 160.0 (C-5″); | 157.4 (C-9); | 154.3 (C-9″); | 153.5 (C-2); | 149.3 (C-4‴); |
| 145.3 (C-3‴); | 145.0 (C-5′); | 144.0 (C-4′); | 123.0 (C-2′); | 122.3 (C-3); |
| 121.8 (C-1′); | 120.6 (C-1‴); | 119.2 (C-3′); | 118.7 (C-6‴); | |
| 115.4/115.3 (C-6′, C-5‴); | 113.6 (C-2‴); | 104.8/104.4 (C-8″, C-10); | | |
| 103.4 (C-10″); | 102.2 (C-3″); | 98.8/98.5 (C-6, C-6″); | 93.4 (C-8) ppm | |
| Frequency | 100 MHz | Solvent | DMSO-$d_6$ | |
| Standard | DMSO-$d_6$ | Sample temp. | 24 °C | |
| Spectrometer type and manufacturer | | Bruker AM 400 | | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 261, 291sh, 343nm | $\varepsilon_{max}$ | – |
| Solvent | Methanol (Uvasol)<br>additional UV spectra with shift reagents according to ref. [5] | | |
| Sample conc. | about $2 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Super Scan 3(Varian); UV-VIS-spectrophotometer, double beam | | |

8. Chromatographic Data

| TLC | I,II on Cellulose (Avicel); III on Polyamide-6 | | |
|---|---|---|---|
| Rf Value | System I: 0.91 II: 0.42 III: 0.41 | | |
| Solvent system | I: Butanol-(1) : Acetic Acid : Water (4 : 1 : 5) upper layer<br>II: Acetic Acid : Water (4 : 6)<br>III: Ethylacetate : Butanone(2) : Formic Acid : Water (5 : 3 : 1 : 1) | | |
| Saturated atmosphere | yes | Detection/Color | UV deep purple;<br>UV, sprayed with Diphenylboric-acid-β-aminoethylester (NA): yellow |
| Plate manufacturer | Schleicher & Schüll<br>Macherey & Nagel | Plate type/Product no. | Cellulose, Avicel F 1440<br>Polygram; 0.1 mm; Polyamide-TLC 6 |

| GLC | underivatized not possible |
|---|---|

| HPLC | | | |
|---|---|---|---|
| Retention time | 35.6 min | | |
| Column | Knauer, Berlin<br>250 × 4 mm | Stationary phase | Spherisorb ODS II, 5 µm<br>(Octadecylsilan II) |
| Mobile phase | A: MeOH – Acetic Acid<br>(95 : 5)<br>B: Water – Acetic Acid<br>(95 : 5) | Flow rate | 1.0 ml/min<br>Gradient; linear<br>0- 5 min 30A – 70B<br>5-35 min 70A – 30B<br>35-40 min 70A – 30B |
| Column temp. | ≈ 25 °C | Pressure | – |
| Detector | Waters 990<br>Photodioden-Array-Detector | Sample solvent | MeOH |
| Sample size | 10 µl | Sample conc. | – |
| Chromatograph type and manufacturer | | Waters Multisolvent Delivery System | |

9. Remarks

The substance could be isolated just in small amounts. For structure elucidation only NMR spectroscopic techniques were used, since they allow a subsequent recovery.

10. References


[1] Geiger, H., Stein, W., Mues, R. and Zinsmeister, H.D.: Bryoflavone and Heterobryoflavone, Two New Isoflavone-Flavone Dimers from Bryum capillare. *Z. Naturforsch. 42c*, 1987, pp 863-7 *(name, $^{13}$C-NMR)*

[2] Geiger, H. and Quinn, C.: Biflavonoids. In ‚The Flavonoids', Harborne, J.B. (Ed.), Chapman and Hall London, 1988 *(name, occurrence)*

[3] Chari, V.M., Ilyas M., Wagner, H., Neszmély, A., Chen, F.-C., Chen, L.-K., Lin, Y.-C. and Lin, Y.-M.: $^{13}$C-NMR Spectroscopy of Biflavonoids. *Phytochemistry 16*, 1977, pp. 1273-8 *($^{13}$C-NMR)*

[4] Markham, K.R. and Chari, V.M.: Carbon-13 NMR Spectroscopy of Flavonoids. In ‚The Flavonoids', Harborne, J.B. and Mabry, T.J. (Eds.), Chapman and Hall London, 1982 *($^{13}$C-NMR)*

[5] Mabry, T.J., Markham, K.R. and Thomas, M.B.: The Systematic Identification of Flavonoids, Springer Verlag, 1970 *(UV spectroscopy)*

# Honokiol

*Wang Zhongdong*

## 1. Name of Compound

| |
|---|
| Common name<br>**Honokiol** |
| Synonyms |
| Systematic name<br>[1,1′-Biphenyl]-2,4′-diole, 3′,5-di-2-propenyl- |
| Substance<br>Lignan |
| CAS registry number and other numbers<br>[35354-74-6] BRN 1982052 |

## 2. Formulas and Molecular Weight

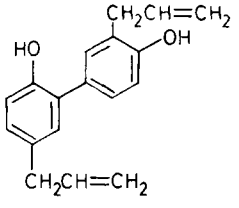
Molecular formular

$C_{18}H_{18}O_2$

Molecular weight

266.32

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>needle crystals | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>87.5 °C | $n_D^{20}$<br>not available | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{15} = \pm 0°$ | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>methanol, ethanol, ethyl acetate, aquatic alcaline | |
| Color<br>colorless | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Several species of Magnolia, Magnoliaceae, such as M. officinalis Rehd. et Wils., M. obvata Thunb. |

5. Health Hazard Data

| | |
|---|---|
| Toxicology | Hazard labeling |
| | $LD_{50}$ not available |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| |

7. Spectroscopic Data

| | |
|---|---|
| MS | not available |

| | | | |
|---|---|---|---|
| IR | | | |
| Characteristic peaks<br>3280, 1640, 1610, 1590, 1500, 1430, 1365, 1320, 1290, 1270, 1210, 1180, 1153, 1125, 1045, 1000, 980, 910, 900 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | IR27G (Shimadzu, Japan) | |

| | | | |
|---|---|---|---|
| NMR | | Nucleus | $^1H$-$^1H$-correlation |
| Chemical Shifts<br>7.29-6.82, 6.15-5.82, 5.32-4.95, 3.50-3.30 ppm | | | |
| Frequency | 80 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | – |
| Spectrometer type and manufacturer | | AC-80 (Bruker, Switzerland) | |

| UV * | | | |
|---|---|---|---|
| $\lambda_{max}$ | 294, 312 nm | $\varepsilon_{max}$ | 8.200, 1.280 |
| Solvent | EtOH + NaOH | | |
| Sample conc. | – | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | SP800 | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | about 0.6 | | |
| Solvent | EtOH | Solvent system | Benzene/MeOH (27 : 1) |
| Saturated atmosphere | – | Detection/Color | Iodine vapor;<br>UV 254 (quenching) |
| Plate manufacturer | hand made | Plate type/Product no. | Silica gel-G (China) |

| GLC | not applicable |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

10. References


[1] Mitiiti Fujita, Hideji Itokawa and Yutaka Sashida: Yakugakuzashi, 1973, *93*, 422

[2] Mitiiti Fujita, Hideji Itokawa and Yutaka Sashida: Chem. Pharm. Bull. *20*, 1972, 212

[3] CA 1972, 76:112819z

[4] CA 1973, 79:35031u

[5] Yan Wenmei: Chinese Herb Medicine Communication *12*, 1978, 1 (Chinese)

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# 5′-Hydroxyamentoflavone

*S. Anhut, H. D. Zinsmeister*

## 1. Name of Compound

Common name

**5′-Hydroxyamentoflavone** [1]

Synonyms

Systematic name

8-[5-(5,7-Dihydroxy-4-oxo-4*H*-1-benzopyran-2-yl)-2,3-dihydroxyphenyl]-5,7-dihydroxy-2-(4-hydroxyphenyl)4*H*-1-benzopyran-4-one

| Substance | Subgroup |
|---|---|
| Flavonoid | Biflavone |

CAS registry number and other numbers

[114865-39-3]

## 2. Formulas and Molecular Weight

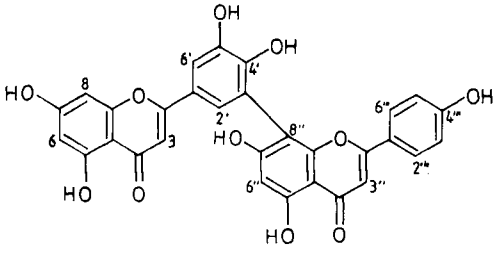
Molecular formular

$C_{30}H_{18}O_{11}$

Molecular weight

554.46

Structural formula




## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| crystalline | not applicable | not applicable |
| **Melting Point mp** | $n_D^{20}$ | **Acid value** |
| 288 °C | not applicable | not applicable |
| **Boiling point bp** | $[\alpha]_D^{20}$ | **Saponification value** |
| not applicable | not measurable | not applicable |
| **Flash point** | **Soluble in** | |
| not applicable | methanol, acetone | |
| **Color** | **Odor** | |
| pale yellow | odorless | |

4. Occurrence

| |
|---|
| Plagiomnium elatum (Musci) [1, 2] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| not known | $LD_{50}$ | not known |
| Waste disposal procedures | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder; preferably in the dark. |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS | FD-Mass spectrum | | |
| Base peak | | Molecular ion | 554 m/e |
| Ionization energy | | Ion source temp. | |
| Acceleration voltage | | Emission current | |
| Resolution | | Scan rate | |
| Spectrometer type and manufacturer | | Varian MAT 311 with FD source | |

| | |
|---|---|
| IR | not available |

| | | | |
|---|---|---|---|
| NMR | | Nucleus | $^1H$ |
| Chemical Shifts<br>7.58 (d, J = 9 Hz, H-2‴, H-6‴);<br>7.48 (d, J = 2 Hz, H-2′);<br>6.70 (d, J = 9 Hz, H-3‴, H-5‴);<br>6.18 (d, J = 2 Hz, H-6) ppm | | 7.52 (d, J = 2 Hz, H-6′);<br>6.78 (s, H-3″);<br>6.42 (d, J = 2 Hz, H-8); | 6.70 (s, H-3);<br>6.38 (s, H-6″); |
| Frequency | 400 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | see ref. [3] | Nucleus | $^{13}C$ |
|---|---|---|---|

| Chemical Shifts | | | | |
|---|---|---|---|---|
| 182.0 (C-4); | 181.5 (C-4″); | 164.0 (C-7); | 163.9 (C-2″); | 163.7 (C-2); |
| 161.9 (C-7″); | 161.4 (C-4‴); | 160.9 (C-5); | 160.4 (C-5″); | 157.3 (C-9); |
| 154.5 (C-9″); | 148.4 (C-4′); | 145.7 (C-5′); | 128.2 (C-2‴); | 128.2 (C-6‴); |
| 122.3 (C-2′); | 121.4 (C-1‴); | 120.5 (C-1′); | 120.1 (C-3′); | 115.7 (C-3‴); |
| 115.7 (C-5‴); | 112.0 (C-6′); | 104.1 (C-8″); | 103.6 (C-10); | 103.6 (C-10″); |
| 102.9 (C-3); | 102.5 (C-3″); | 98.8 (C-6); | 98.6 (C-6″); | 93.8 (C-8) ppm |

| Frequency | 100 MHz | Solvent | DMSO-$d_6$ |
|---|---|---|---|
| Standard | DMSO-$d_6$ | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV * | | | |
|---|---|---|---|
| $I_{max}$ | 265, 291sh, 343nm | $\varepsilon_{max}$ | – |
| Solvent | Methanol (Uvasol)<br>additional UV spectra with shift reagents according to ref. [4] | | |
| Sample conc. | about $2 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Super Scan 3 (Varian); UV-VIS-spectrophotometer, double beam | | |

8. Chromatographic Data

| TLC | I, II on Cellulose (Avicel); III on Polyamide-6 | | |
|---|---|---|---|
| Rf Value | System I: 0.91 II: 0.58 III: 0.40 | | |
| Solvent system | I: tert. Butanol : Acetic Acid : Water (3 : 1 : 1)<br>II: Acetic Acid : Water (4 : 6)<br>III: Ethylacetate : Butanone(2) : Formic Acid : Water (5 : 3 : 1 : 1) | | |
| Saturated atmosphere | yes | Detection/Color | UV deep purple;<br>UV, sprayed with Diphenylboric-acid-β-aminoethylester (NA): yellow |
| Plate manufacturer | Schleicher & Schüll | Plate type/Product no. | Cellulose, Avicel F 1440<br>(Mikro-) Polyamide F 1700 |

| GLC | underivatized not possible |
|---|---|

| HPLC | | | |
|---|---|---|---|
| Retention time | 17 min | | |
| Column | Macherey & Nagel<br>ET 250/8/4 Nucleosil 5C18 with guard column<br>Nucleosil 5C18; 30 x 4 mm I.D. | Stationary phase | Nucleosil RP-18, 5 µm |
| Mobile phase | A: MeOH-5% Acetic Acid (65:35) | Flow rate | 0.8 ml/min |
| Column temp. | ambient temp., ≈25 °C | Pressure | – |
| Detector | UV-Detector Waters 450<br>Detection: 330 nm | Sample solvent | methanol |
| Sample size | 10–15 µl | Sample conc. | – |
| Chromatograph type and manufacturer | | Waters M 45 | |

9. Remarks

The substance could be isolated just in small amounts. For structure elucidation only NMR spectroscopic techniques were used, since they allow a subsequent recovery.

10. References


[1] Geiger, H., Anhut, S. and Zinsmeister, H.D.: Biflavones from Some Mosses. *Z. Naturforsch. 43c*, 1988, pp 1–4 *(name, occurrence)*

[2] Geiger, H. and Quinn, C.: Biflavonoids. In ‚The Flavonoids', Harborne, J.B. (Ed.), Chapman and Hall, London, 1988 *(occurrence)*

[3] Markham, K.R. and Chari, V.M.: Carbon-13 NMR Spectroscopy of Flavonoids. In ‚The Flavonoids', Harborne, J.B. and Mabry (Eds.), Chapman and Hall London, 1982 *($^{13}$C-NMR)*

[4] Mabry, T.J., Markham, K.R. and Thomas, M.B.: The Systematic Identification of Flavonoids. Springer Verlag, 1970.*(UV spectroscopy)*

# Hydroxybutenolide

*J. Wilschke, B. Sprengel, H. Rudolph*

## 1. Name of Compound

Common name
**Hydroxybutenolide** [1]

Synonyms

Systematic name
(-) 2,5-Dihydro-5-hydroxy-4-[4′-hydroxyphenyl]-furan-2-one

Substance
Butenolide

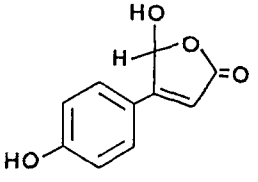
CAS registry number and other numbers
[123564-56-7]

## 2. Formulas and Molecular Weight

Molecular formular
$C_{10}H_8O_4$

Molecular weight
192

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>228 °C | $n_D^{20}$<br>not available | Acid value<br>not applicable |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20} = -3.5°$<br>(c=0.5537, $CH_3OH$) | Saponification value<br>not available |
| Flash point<br>not available | Soluble in<br>water, methanol, ethanol | |
| Color<br>colorless | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Moss Sphagnum species [2] |

5. Health Hazard Data

| | |
|---|---|
| Toxicology | Hazard labeling not usual |
| unknown | $LD_{50}$ |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder. |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS * | EIMS | | |
| Base peak | – | Molecular ion | 192 |
| Ionization energy | 70 eV | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | MAT 8230 Finnigan | |

| | |
|---|---|
| IR | not available |

| | | | |
|---|---|---|---|
| NMR * | | Nucleus | $^1H$ |
| Chemical Shifts<br>10.28 (1H, s, OH);<br>6.94 (2H, m, H-3′ and H-5′); | 7.96 (1H, d, J = 7 Hz, OH);<br>6.58 (1H, d, J = 9 Hz, H-5); | 7.72 (2H, m, H-2′ and H-6′);<br>6.57 (1H, s, H-3) ppm | |
| Frequency | 360.13 MHz | Solvent | DMSO-$D_6$ |
| Standard | DMSO-$D_6$ | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR * | | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>171.38 (s, C-2); 163.57 (s, C-4); 160.56 (s, C-4′);<br>130.37 (d, C-2′ and C-6′); 120.81 (s, C-1′); 115.87 (d, C-3′ and C-5′);<br>111.19 (d, C-3); 97.97 (d, C-5) ppm | | | |
| Frequency | 90.56 MHz | Solvent | DMSO-$D_6$ |
| Standard | DMSO-$D_6$ | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV * | | | |
|---|---|---|---|
| $\lambda_{max}$ | a) 312 b) 352 nm | $\varepsilon_{max}$ | b) $\varepsilon_{280} = 4661$ mole $^{-1}$/cm $^{-1}$ |
| Solvent | a) Methanol (Uvasol) b) HPLC-gradient: $t_R$ = 17.9 min<br>3.47% HCOOH, 65.93% $H_2O$, 30.6% MeOH, v/v/v | | |
| Sample conc. | – | Cell thickness | a) 1 b) 0.6 cm |
| Spectrometer type and manufacturer | | a) Hitachi Spectrophotometer 100-60<br>b) Uvikon 720 LC | |

8. Chromatographic Data

| TLC | Color reactions with Millon's and Pauly's reagents | | |
|---|---|---|---|
| Rf Value | 0.55 | | |
| Solvent | EtOH | Solvent system | BuOH-HAc-$H_2O$ (3 : 2 : 95), v/v/v) |
| Saturated atmosphere | yes | Detection/Color | bright blue fluorescence |
| Plate manufacturer | – | Plate type/Product no. | Cellulose $UV_{254}$ layers |

| GLC | underivatized not possible |
|---|---|

| HPLC | | | |
|---|---|---|---|
| Retention time | 17.9 min | | |
| Column | Kontron steel column | Stationary phase | ODS II, 5 μm, 250 × 4.6 mm |
| Mobile phase | a) $HCOH/H_2O$ (5 : 95, v/v)<br>b) $CH_3OH$ | Flow rate | – |
| Column temp. | ca. 25 °C | Pressure | – |
| Detector | Uvikon 720 LC micro | Sample solvent | $CH_3OH$ |
| Sample size | 20 μl | Sample conc. | – |
| Chromatograph type and manufacturer | KONTRON INSTRUMENTS:Anacomp 220, LC-pumps T-414, Uvikon 720 LC | | |

9. Remarks

The compound could be isolated just in small amounts.

10. References


[1] Wilschke, J., Sprengel, B., Wolff, C. and Rudolph H., *Phytochemistry 28*, 1989, pp. 25–127 *(name, peroxidatic preparation, NMR)*

[2] Rudolph, H. and Samland J., *Phytochemistry 24*, 1985, pp. 745–49 *(occurrence, TLC)*

# L-Hyoscyamine

*F. Oprach*

## 1. Name of Compound

| | | |
|---|---|---|
| Common name<br>**L-Hyoscyamine** | | |
| Synonyms<br>Daturine; Duboisine | | |
| Systematic name<br>1αH,5αH-Tropan-3α-ol(-)-tropate<br>3α-Tropanyl-S-(-)-tropate | | |
| Substance<br>Alkaloid | Subgroup<br>Tropane Alkaloid | |
| CAS registry number and other numbers<br>[101-31-5] | Merck Index *10*, 4782 | BRN 91259 |

## 2. Formulas and Molecular Weight

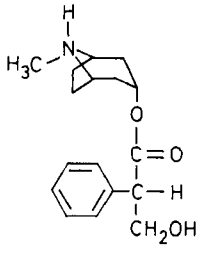
Molecular formular

$C_{17}H_{23}NO_3$

Molecular weight

289.4

Structural formula



## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>108.5 °C | $n_D^{20}$<br>not available | Acid value<br>not available |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20} = -21.0°$<br>(alc) [1] | Saponification value<br>not available |
| Flash point<br>not applicable | Soluble in<br>chloroform, ethanol, ether, acetone | |
| Color<br>white | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Main alkaloid of Atropa belladonna; Hyoscyamus niger; several Datura species like suaveolens, sanguinea, candida, metel, stramonium. |

5. Health Hazard Data

| | |
|---|---|
| Toxicology<br><br>anticholinergic substance | Hazard labeling avoid contact with skin<br>very toxic by inhalation and if swallowed |
| | $LD_{50}$ not available |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| Keep well closed and protect from light and heat. |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 124 | Molecular ion | 289 |
| Ionization energy | 24 eV | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | AEI MS 30 | |

| | | | |
|---|---|---|---|
| IR * | see ref. [2] | | |
| Characteristic peaks<br>1715, 1705, 1020, 1145, 1050 $cm^{-1}$ | | | |
| Sample preparation | KBr (1 mg/200 mg KBr) | Resolution | – |
| Spectrometer type and manufacturer | Acculab 2-Spektrophotometer Beckman | | |

| | |
|---|---|
| NMR | not available |

| | | | |
|---|---|---|---|
| UV * | see ref. [3] | | |
| $\lambda_{max}$ | not available | $\varepsilon_{max}$ | $E^{1\%}_{1\,cm}$ = 5.95 at 257 nm (as sulfate) |
| Solvent | 0.01 N HCl | | |
| Sample conc. | – | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Pye Unicam SP8-200 UV-Vis-spectrophotometer | |

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.6 | | |
| Solvent | MeOH | Solvent system | $CHCl_3$-MeOH-$NH_3$ 25% (60 : 40 : 3) |
| Saturated atmosphere | yes | Detection/Color | orange color after detection with Sodiumbismuthiodide solution |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel $F_{254}$ |

| GLC | | | |
|---|---|---|---|
| Retention time | Retention indices $RI_{DB1}$ 2175, $RI_{DB17}$ 2774 | | |
| Column type | fused silica (id 0.25 mm) | Column length | 30 m |
| Column packing | SE 30 | Column temp. | 70–300 °C; 6 °C/min |
| Injector port temp. | 300 °C | Carrier gas | Helium, 2 ml/min |
| Detector temp. | 300 °C | Sample solvent | MeOH |
| Sample size | 1 µl | Sample conc. | 50 mg/50 ml |
| Chromatograph type and manufacturer | | Perkin Elmer Sigma 2b | |

| HPLC | | | |
|---|---|---|---|
| Retention time | 3.9 min | | |
| Column | Waters PN 27324 | Stationary phase | μ Bondapak C18, 10 μm 250 mm × 4 mm ID |
| Mobile phase | methanol-acetate buffer 50:50, (pH 5.5; 0.1 mol) | Flow rate | 1 ml/min |
| Column temp. | room temperature | Pressure | 120 bar |
| Detector | UV detection at 230 nm | Sample solvent | mobile phase |
| Sample size | 20 μl | Sample conc. | 20 μg/20 μl |
| Chromatograph type and manufacturer | | Perkin Elmer Series 2 | |

9. Remarks

Used as anticholinergic substance in pharmacy

10. References


[1] Merck Index, 10, 4782

[2] Auterhoff, Kovar, in: Identifizierung von Arzneistoffen, WV GmbH, Stuttgart, 1977, 157–8

[3] Böhme, H. and Hartke, K., in: Europäisches Arzneibuch, Bd. I/II, Komm., 546–51, Wiss. Verlagsges. mbH, Stuttgart, Govi-Verlag GmbH, Frankfurt, 1978

# Isoalantolactone

*H. Häberlein*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Isoalantolactone** | |
| Synonyms<br>Isohelenin [1] | |
| Systematic name<br>Eudesma-4(14),11(13)-dien-12-oic acid,8β-hydroxy-,γ-lactone | |
| Substance<br>Terpene | Subgroup<br>Sesquiterpene lactone |
| CAS registry number and other numbers<br>[470-17-7] | BRN 13402 |

## 2. Formulas and Molecular Weight

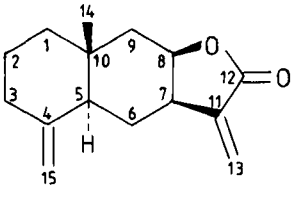
Molecular formular

$C_{15}H_{20}O_2$

Molecular weight

232.33

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline solid | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>112 °C [2] | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{20}$:<br>186.1° (1, MeOH) [3] | Saponification value<br>not applicable |
| Flash point<br>not applicable | Soluble in<br>chloroform, dichloromethane, methanol, diethyl ether, acetone | |
| Color<br>white | Odor<br>indifferent | |

## 4. Occurrence

| |
|---|
| Main constituent from Inula species [4] |

## 5. Health Hazard Data

| | |
|---|---|
| Toxicology<br><br>Irritating substance | Hazard labeling Xn<br>Harmful by inhalation, in contact with skin and if swallowed<br>Prolonged or repeated exposure may cause allergie<br>reactions in certain sensitive individuals. |
| | $ID_{50}$: 15 µg/ml [5] (ID = inhibition dose);<br>in vitro cytotoxicity on Human lung Carcinoma<br>Cell Line |
| Waste disposal procedures<br>Dissolve or mix the material with a combustible solvent and burn in a chemical incinerator equipped with an afterburner and scrubber. | |

## 6. Transportation and Storage Instructions

| |
|---|
| Storage temperature: 0 °C |

## 7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | m/z 190 | Molecular ion | m/z 232.32545 |
| Ionization energy | 70 eV | Ion source temp. | 120 °C |
| Acceleration voltage | 4000 V | Emission current | 0.2 mA |
| Resolution | 1000 | Scan rate | 2.6 s/decade |
| Spectrometer type and manufacturer | | Vacuum Generators Micromass 7070 H | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>2960, 2950, 2810, 1740, 1655, 1635, 1255, 1130, 945, 890, 820 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | IR Spectrometer 398, Perkin Elmer | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts | | | |
| 6.11 (d, 1H, *H*C-13); | 2.31 (m, 1H, $H_\beta$C-3); | 1.48 (dd, 1H, $H_\alpha$C-9); | |
| 5.57 (d, 1H, *H*C-13); | 2.17 (dd, 1H, $H_\beta$C-9); | 1.35 (ddd, 1H, $H_\beta$C-6); | |
| 4.75 (d, 1H, *H*C-15); | 1.97 (dt, 1H, $H_\alpha$C-3); | 1.22 (m, 1H, $H_\alpha$C-3); | |
| 4.48 (dt, 1H, $H_\alpha$C-8); | 1.81 (dd, 1H, $H_\alpha$C-5); | 0.81 (s, 3H, $H_{3\beta}$C-14) | |
| 4.42 (d, 1H, *H*C-15); | 1,71 (ddd, 1H, $H_\alpha$C-6) | | |
| 2.95 (ddd, 1H, $H_\alpha$C-7); | 1.51–1.60 (m, 3H, $H_\beta$C-1, $H_2$C-2) ppm | | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 23 °C |
| Spectrometer type and manufacturer | | Bruker WH 400 | |

| NMR * | $^1H$-decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts | | | |
| 170.3 (s, C-12); | 148.8 (s, C-4); | 142.3 (s, C-11); | 119.7 (t, C-13); |
| 106.5 (t, C-15); | 76.7 (d, C-8); | 46.0 (d, C-5); | 42.1 (t, C-9); |
| 41.3 (t, C-1); | 40.3 (d, C-7); | 36.8 (t, C-3); | 34.2 (s, C-10); |
| 27.5 (t, C-6); | 22.7 (t, C-2); | 17.6 (qu, C-14) ppm | |
| Frequency | 100 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 23 °C |
| Spectrometer type and manufacturer | | Bruker WH 400 | |

| UV | not applicable |
|---|---|

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.35 | | |
| Solvent | acetone | Solvent system | toluene/ethyl acetate (97 : 3) |
| Saturated atmosphere | yes | Detection/Color | 5% solution of $AlCl_3$ in ethanol, 10 min at 120 °C, brown under UV (366 nm) |
| Plate manufacturer | Riedel de Haën | Plate type/Product no. | DC-Mikrokarten Si $F_{254nm}$ Art. 37341 |

| GLC | For application of GLC for the separation of sesquiterpene lactones see ref. [6] and [7]. |
|---|---|

| | | | |
|---|---|---|---|
| HPLC | see ref. [3] | | |
| Retention time | k′ = 8.37 | | |
| Column | 4.0 mm × 250 mm | Stationary phase | silica gel (5 m) |
| Mobile phase | n-pentane/diethyl ether (90 : 10) | Flow rate | 1 ml/min |
| Column temp. | room temperature | Pressure | 150 bar |
| Detector | UV 210 nm | Sample solvent | n-pentane/diethyl ether (90 : 10) |
| Sample size | 10 µl | Sample conc. | $8.62 \times 10^{-4}$ mol/l |
| Chromatograph type and manufacturer | Pump L-6200, UV-Detector 655A-23<br>Chromato-Integrator D-2500, Merck-Hitachi | | |

9. Remarks

Used in the chemotaxonomy of higher plants and in several methods of chromatography as reference substance

10. References


[1] Karrer, W., *Konstitution und Vorkommen der organischen Pflanzenstoffe*, Birkhäuser Verlag, Basel 1958, No. 1901

[2] Glasby, J.S., *Encyclopaedia of the Terpenoids*, John Whiley & Sons (1982) pp. 1348

[3] Häberlein, H., unpublished data

[4] Bohlmann, F., Mahanta, P.K., Jakupovic, J., Rastogi, R.C., Natu, A., *Phytochemistry 17* (1978) 1165–1172

[5] Woerdenbag, H.J., Meijer, C., Mulder, N.H., de Vries, E.G.E., Hendriks, H., Malingre, Th.M., *Planta Med. 2* (1986) 112–114

[6] Zinchenko, V.V., Khvorost, P.P., Bakai, S.I., Biryuk, V.A., Tarusin, A.D., Kravchina, T.S., *Rastit Resur. 19(4)*, (1983) 544–548

[7] Rosik, G.G., Zinchenko, V.V., Reznichenko, A.A., Koralev, I.P., *Khim.-Farm Zh. 21(5)*, (1987) 632–634

# Isofurcatain-7-O-β-D-glucoside

*S. Anhut, H. D. Zinsmeister, R. Mues*

## 1. Name of Compound

Common name

**Isofurcatain-7-O-β-D-glucoside** [1]

Synonyms

6-C-α-L-Rhamnopyranosyl-apigenin-7-O-β-glucopyranoside

Systematic name

6-(6-Deoxy-α-L-mannopyranosyl)-7-(β-D-glucopyranosyloxy)-5-hydroxy-2-(4-hydroxyphenyl)4*H*-1-benzopyran-4-one

| Substance | Subgroup |
|---|---|
| Flavonoid | Flavone Glycoside<br>Flavone C/O-Glycoside |

CAS registry number and other numbers

[83463-97-2]

## 2. Formulas and Molecular Weight

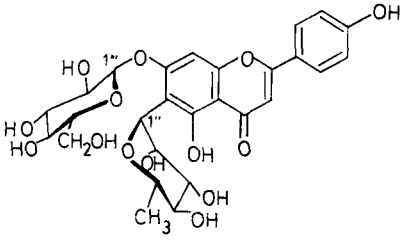
Molecular formular

$C_{27}H_{30}O_{14}$

Molecular weight

578

Structural formula




## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| crystalline, needles | not applicable | not applicable |
| Melting Point mp | $n_D^{20}$ | Acid value |
| 223-225 °C [1] | not applicable | not applicable |
| Boiling point bp | $[\alpha]_D^{20}$ | Saponification value |
| not applicable | not available | not applicable |
| Flash point | Soluble in | |
| not applicable | methanol | |
| Color | Odor | |
| pale yellow | odorless | |

4. Occurrence

| |
|---|
| Metzgeria furcata (Hepaticae; Bryophytes) [1, 2]<br>(only known from this species!) |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| not known | $LD_{50}$ | not known |
| Waste disposal procedures | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder; preferably in the dark. |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS | as Permethylether [1] | | |
| Base peak | m/e = 673 | Molecular ion | $M^+ = 704$ |
| Ionization energy | 90 eV | Ion source temp. | 150 °C |
| Acceleration voltage | 1.8 kV | Emission current | 2 mA |
| Resolution | 2000 | Scan rate | – |
| Spectrometer type and manufacturer | | Varian MAT 311A – 100 MS | |

| |
|---|
| IR for structure elucidation of Flavone-C-glycosides not practicable |

| | | | |
|---|---|---|---|
| NMR | (see remarks) | Nucleus | $^1H$ |
| Chemical Shifts<br>Aglycone:<br><br>Sugars: rha:<br>glu: | 7.95 (d, J=8.8 Hz, H-2′, H-6′);<br>6.93 (d, J=8.8 Hz, H-3′, H-5′);<br>5.26 (d, J=9.9 Hz, H-1″);<br>4.86 (d, J=7.6 Hz, H-1‴); | 6.96 (s, H-3);<br>6.87 (s, H-8)<br>1.36 (d, J=7 Hz, rhamnose $CH_3$)<br>5.1–3 (mult., sugar protons) ppm | |
| Frequency | 400 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | ambient temp. ≈25 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR * | see ref. [1, 3] | Nucleus | $^{13}C$ (see remarks) | |
|---|---|---|---|---|
| Chemical Shifts | | | | |
| 182.1 (C-4); | 164.1 (C-2); | 163.3 (C-7); | 161.3 (C-4'); | 159.7 (C-5); |
| 156.2 (C-9); | 128.5 (C-2' and C-6'); | 120.9 (C-1'); | 116.0 (C-3' and C-5'); | |
| 111.9 (C-6); | 105.1 (C-10); | 103.2 (C-3); | 94.6 (C-8); | |
| 77.4 (C-1''); | 75.4 (C-2'); | 73.6 (C-3''); | 72.2 (C-4''); | |
| 71.5 (C-5''); | 16.3 (C-6''); | 102.3 (C-1'''); | 73.6 (C-2'''); | |
| 77.4 (C-3'''); | 69.7 (C-4'''); | 75.4 (C-5'''); | 60.7 (C-6''') ppm | |
| Frequency | 20 MHz | Solvent | DMSO-$d_6$ | |
| Standard | TMS | Sample temp. | 30 °C | |
| Spectrometer type and manufacturer | | Varian | | |

| UV * | | | |
|---|---|---|---|
| $I_{max}$ | 273, 332 nm | $\varepsilon_{max}$ | – |
| Solvent | Methanol (Uvasol)<br>(additional UV spectra with shift reagents were reported in ref. [1]) | | |
| Sample conc. | about $2 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Super Scan 3 (Varian); UV-VIS-spectrophotometer, double beam | | |

8. Chromatographic Data

| TLC | I – III on Cellulose (Avicel); IV on Polyamide-6 [1] | | |
|---|---|---|---|
| Rf Value | System I: 0.50<br>System II: 0.76 | System III: 0.66<br>System IV: 0.56 | |
| Solvent system | I: Acetic Acid : Water (15 : 85);<br>II: Acetic Acid : Water (40 : 60)<br>III: Butanol-(1) : Acetic Acid : Water (4 : 1 : 5) upper layer<br>IV: Water : Butanone(2): MeOH : Pentanedione (65 : 15 : 15 : 5) | | |
| Saturated atmosphere | yes | Detection/Color | UV dark; UV, sprayed with Diphenylboric-acid-β-aminoethylester (NA): green |
| Plate manufacturer | Schleicher & Schüll<br>Macherey & Nagel | Plate type/Product no. | Cellulose, Avicel F 1440<br>Polygram; 0.1 mm; Polyamide-TLC 6 |

| GLC | underivatized not possible |
|---|---|

| HPLC | | | |
|---|---|---|---|
| Retention time | 12.8 min | | |
| Column | Macherey & Nagel<br>ET 250/8/4, Nucleosil 5C18 | Stationary phase | Nucleosil RP-18, 5 µm |
| Mobile phase | A: MeOH;<br>B: Water – Acetic Acid (95 : 5), isocratic 35A-65B | Flow rate | 1.0 ml/min |
| Column temp. | ambient temp., ≈25 °C | Pressure | – |
| Detector | UV-Detector Waters 450<br>Detection: 254 nm | Sample solvent | DMSO-$d_6$ |
| Sample size | 5–10 µl | Sample conc. | – |
| Chromatograph type and manufacturer | | Waters M 45 | |

9. Remarks

The assignments for the anomeric sugar protons have been reversed in comparison to ref. [1] basing on recent NMR studies.
For assignment of the sugar carbon-atoms the revised data of ref. [3], p. 331, have been used.

10. References


[1] Markham, K.R., Theodor, R., Mues, R. and Zinsmeister, H.D.: 6-C-α-L-Rhamno-pyranosyl-apigenin-7-O-β-D-glycopyranoside (isofurcatain-7-O-β-D-glycoside), a New Flavone Glycoside from Metzgeria furcata. *Z. Naturforsch. 37c*, 1982, pp 562–4

[2] Chopin, J. and Dellamonica, G.: C-Glycosylflavonoids. In ‚The Flavonoids', Harborne, J.B. (Ed.), Chapman and Hall, London, 1988

[3] Agrawal, P.K. and Bansal, M.C.: Flavonoid Glycosides. In ‚Carbon-13 NMR of Flavonoids', Agrawall, P.K. (Ed.), Studies in Organic Chemistry 39, Elsevier Sci. Pub., 1989, p. 331

# Isosclerone

*R. Bahramsari, K. Krohn*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Isosclerone** | |
| Synonyms<br>4,8-Dihydroxy-1-tetralone | |
| Systematic name<br>3,4-Dihydro-4,8-dihydroxy-1(2H)-naphthalenone (DDN) | |
| Substance<br>Naphthalene | Subgroup<br>Tetralone |
| CAS registry number and other numbers<br>[62332-73-4] | BRN 4743566 |

## 2. Formulas and Molecular Weight

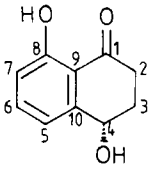
Molecular formular

$C_{10}H_{10}O_3$

Molecular weight

178

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid, needles | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>74-76 °C [1] | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D$ = -3.3°<br>(0.077 g/l in EtOH) [1] | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>$CH_2Cl_2$, benzene, chloroform, ethyl acetate | |
| Color<br>colorless | Odor<br>odorless | |

| NMR | see ref. [1] | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>6.91 (1H, dd, $J_{7-6}=7.7$ Hz, $J_{7-5}=1.3$ Hz, H-7);<br>7.01 (1H, dd, $J_{5-6}=7.7$ Hz, $J_{5-7}=1.3$ Hz, $J_{5-4}=1.0$ Hz, H-5);<br>7.48 (1H, t, $J_{6-5}=J_{6-7}=7.7$ Hz, H-6); 12.35 (1H, s, 8-OH); 4.92 (1H, m, H-4);<br>2.91 and 2.63 (1H, ddd, J=18.3); 8.7 and 4.8 Hz, H-2d and H-2-3);<br>2.29-2.37 (1H, dddd m, H-3β); 2.13-2.22 (1H, dddd m, H-3 α) ppm | | | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | see ref. [1] | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>31.24 (C-3), 34.55 (C-2), 67.77 (C-4), 115.30 (C-8a), 117.35 (C-7), 117.77 (C-5), 136.93 (C-6), 145.93 (C-4a), 162.78 (C-8), 204.21 (C-1) ppm | | | |
| Frequency | 75.47 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV * | see ref. [1] | | |
|---|---|---|---|
| $I_{max}$ | 216, 259, 333 nm | $\varepsilon_{max}$ | 23988, 14791, 5888 |
| Solvent | Ethanol | | |
| Sample conc. | – | Cell thickness | – |
| Spectrometer type and manufacturer | | – | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.32 | | |
| Solvent | – | Solvent system | 1% MeOH/99% $CH_2Cl_2$ |
| Saturated atmosphere | – | Detection/Color | 254 nm/violet |
| Plate manufacturer | Merck, Darmstadt | Plate type/Product no. | Kieselgel 60 $F_{254}$, Art. 5562 |

4. Occurrence

| |
|---|
| Isolated from the fungi Cryptosporiopsis, Juglans regia [1] and Sclerotinia sclerotiorum [2] |

5. Health Hazard Data

| Toxicology | Hazard labeling |
|---|---|
| antibiotic activity | $LD_{50}$ |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| |

7. Spectroscopic Data

| MS | see ref. [2], [3] | | |
|---|---|---|---|
| Base peak | 178 | Molecular ion | 178 |
| Ionization energy | 70 eV | Ion source temp. | 20 °C |
| Acceleration voltage | 3 kV | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | 3 s/decade |
| Spectrometer type and manufacturer | | Finnigan MAT 8430 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>1638.8 (H bonded CO), 3249.3–3278.3 (br, chelated OH) and<br>746.74 (1,2,3-trisubstituted benzene) $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | 2.5 $cm^{-1}$ |
| Spectrometer type and manufacturer | | Perkin Elmer 1420 | |

| | |
|---|---|
| GLC | not available |

| | |
|---|---|
| HPLC | not available |

9. Remarks

10. References


[1] Sunil K. Talapatra*, Bimala Karamacharya, Shambhu C. DE and Bani Talapatra*, *Phytochemistry*, Vol. 27,12 (1988), 3929-3932 *(structure, stereochemistry, conformation)*

[2] Toshiaki Morita and Hiroo Aoki*, *Agr. Biol. Chem. 38(8)*, 1501-1505,1974 *(structure, $^{1}H$-, $^{13}C$-NMR, biological activity)*

[3] Kunio Suzuki*, Takeshi Sassa, Hiroshi Tanka, Hiroo Aoki and Mitsuo Namiki, *Agr. Bio-chem. 32*, 1471-1475, 1968 *(IR, NMR, MS)*

# Kingiside

*L. F. Tietze, C. Bärtels*

## 1. Name of Compound

| |
|---|
| Common name<br>**Kingiside** [1] |
| Synonyms<br>– |
| Systematic name<br>Methyl-(1S,5S,8S,9S)-1-(β-D-glucopyranosyloxy)-1,5,6,7,8,9-hexahydro-8-methyl-7-one-4-pyrano[3,4-c]-pyran-4-carboxylate |
| Substance<br>Secoiridoide |
| CAS registry number and other numbers<br>[25406-67-1] BRN 1408042 |

## 2. Formulas and Molecular Weight

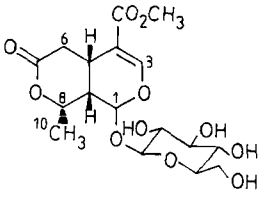
Molecular formular

$C_{17}H_{24}O_{11}$

Molecular weight

404.36

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>106 °C | $n_D^{20}$<br>not applicable | Acid value<br>not available |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20} = -119°$<br>(c = 1, MeOH) | Saponification value<br>not available |
| Flash point<br>not applicable | Soluble in<br>water, methanol, acetone, chloroform, etc. | |
| Color<br>white | Odor<br>not applicable | |

4. Occurrence

| |
|---|
| Minor constituent of lonicera morroii A. Gray [2] and of strychnos spinosa |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not available |
| not available | $LD_{50}$ | not available |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage temperature: +4°C |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | m/z = 43 | Molecular ion | not available |
| Ionization energy | 70 eV | Ion source temp. | 150°C |
| Acceleration voltage | 3000 V | Emission current | 0.3 mA |
| Resolution | 1000 | Scan rate | 2 s/decade |
| Spectrometer type and manufacturer | | Varian MAT 311 A | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3426 (OH), 1708 (C=O), 1640 (C=C), 1076, 1042 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | Bruker IFS 25 | |

| NMR | assigned by 2D-spectrum | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>1.51 (d, J = 3.5 Hz, 3H, $H_3C$-10),<br>2.62 (dd, J = 17, 6 Hz, 1H, $H_2C$-$6_{ax}$),<br>3.16–3.40 (m, 5H, *H*C-2′, *H*C-3′, *H*C-4′, *H*C-5′, *H*C-5),<br>3.71 (s, 3H, $H_3CO_2C$),<br>4.66 (d, J = 8 Hz, 1H, *H*C-1′),<br>5.65 (d, J = 4.5 Hz, 1H, *H*C-1), | | 2.42 (ddd, J = 8, 6, 4.5 Hz, 1H, *H*C-9),<br>3.00 (dd, J = 17, 7.5 Hz, 1H, $H_2C$-$6_{eq}$),<br>3.58–3.69 (m, 1H, $H_2C$-$6_a$′),<br>3.89 (dd, J = 12, 2 Hz, 1H, $H_2C$-$6_b$′),<br>4.74 (dd, J = 8.35 Hz, 1H, *H*C-8),<br>7.50 (d, J = 0.5 Hz, 1H, *H*C-3) ppm | |
| Frequency | 500 MHz | Solvent | $CD_3OD$ |
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | | |

| NMR | $^1H$-decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>18.4 (C-10), 27.9 (C-5), 34.3 (C-6), 39.8 (C-9), 51.9 ($CO_2CH_3$),<br>62.8 (C-6′), 71.6 (C-4′), 74.6 (C-2′), 76.7 (C-8), 77.9 (C-3′),<br>78.4 (C-5′), 94.4 (C-1), 100.1 (C-1′), 111.4 (C-4), 154.3 (C-3),<br>168.2 ($CO_2CH_3$), 174.3 (C-7) ppm | | | |
| Frequency | 50.3 MHz | Solvent | $CD_3OD$ |
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | Varian XL 200 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | $2.35 \times 10^{-5}$ nm | $\varepsilon_{max}$ | 10764.05 |
| Solvent | methanol | | |
| Sample conc. | $4.55 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Varian Cary 219 | |

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.49 | | |
| Solvent | methanol | Solvent system | chloroform/methanol (4/1) |
| Saturated atmosphere | – | Detection/Color | UV 254 nm |
| Plate manufacturer | Macherey-Nagel | Plate type/Product no. | SIL G/$UV_{254}$ |

| | |
|---|---|
| GLC | not applicable |

| | |
|---|---|
| HPLC | not available |

9. Remarks

10. References


[1] Inouye, H., Yoshida, T., Tobita, S., Tanaka, T., Nishioka T., *Tetrahedron Lett. 30*, 1974, 201 *(biosynthesis)*

[2] Souzu, I., Mitsuhashi, W., *Tetrahedron Lett. 32*, 1969, 2725 *(isolation)*

# Leiocarposide

*K. Hiller*

## 1. Name of Compound

Common name
**Leiocarposide**

Synonyms

Systematic name
3-β-D-Glucopyranosyloxy-2-methoxy-6-hydroxy-benzoic acid-2′-β-D-glucopyranosyl-oxybenzylester

| Substance | Subgroup |
|---|---|
| Glycoside | Phenolic glycoside |

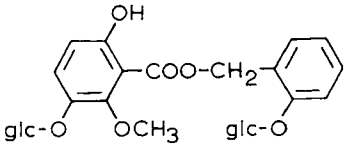
CAS registry number and other numbers
[71953-77-0]

## 2. Formulas and Molecular Weight

Molecular formular
$C_{27}H_{34}O_{16}$

Molecular weight
614.5420

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>190-192 °C | $n_D^{20}$<br>not applicable | Acid value<br>not available |
| Boiling point bp<br>not available | $[\alpha]_D^{20}$<br>-61.28° (c=0.5, MeOH) | Saponification value<br>not available |
| Flash point<br>not available | Soluble in<br>water, methanol | |
| Color<br>colorless | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Up to date only in the species Solidago virgaurea L. (Asteraceae, Compositae) |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | |
| | $LD_{50}$ | Extract of the above mentioned plant has no toxic effect. |
| Waste disposal procedures | | |

6. Transportation and Storage Instructions

| |
|---|
| |

7. Spectroscopic Data

| MS * | | | |
|---|---|---|---|
| Base peak | m/e 166 ($C_8H_6O_4$) | Molecular ion | – |
| Ionization energy | 70 eV | Ion source temp. | 180 °C |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | MS 902, AEI (Manchester) | |

| IR * | | | |
|---|---|---|---|
| Characteristic peaks<br>3440–3340 (assoc. OH groups); 1670 (CO-group in o-position to OH-group); 1610; 1500; 1475; 1075–1055; 770 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | UR-10, VEB Carl Zeiss, Jena | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>see reproduction of the measured spectrum | | | |
| Frequency | – | Solvent | $CDCl_3$ |
| Standard | – | Sample temp. | ambient temperature |
| Spectrometer type and manufacturer | | Bruker AM 300 | |

| NMR | | | | | | | |
|---|---|---|---|---|---|---|---|
| Nucleus | $^{13}C$ | | | | | | |
| Chemical Shifts | | | | | | | |
| 168.8 s; | 155.7 s; | 152.7 s; | 143.3 s; | 143.9 s; | 131.2 dd; | 125.4 s; | 123.9d; |
| 123.7d; | 116.4d; | 114.2 s; | 113.3d; | 102.6d; | 101.5d; | 76.9 dd; | 76.6 dd; |
| 74.0d; | 70.3 dd; | 64.0 t; | 63.1 q; | 61.6 t; | 61.5 t ppm | | |
| Frequency | – | | | Solvent | DMSO-$d_6$ | | |
| Standard | Hexamethyldisiloxan ($\alpha = 1.92$) | | | Sample temp. | | | |
| Spectrometer type and manufacturer | | | | CFT 20 Varian USA | | | |

| UV * | | | |
|---|---|---|---|
| $I_{max}$ | 274, 295, 323 nm | $\varepsilon_{max}$ | 3.14; 3.11; 3.05 |
| Solvent | MeOH | | |
| Sample conc. | – | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Specord M 40, VEB Carl Zeiss, Jena | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.57 | | |
| Solvent | – | Solvent system | $CHCl_3(65):CH_3OH(35):H_2O(7)$ |
| Saturated atmosphere | 20–23 °C | Detection/Color | 2,6-dichlorquinonechlorimid + $NH_3$, blue-violet |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel G |

| GLC | not applicable |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

10. References


[1] Hiller, K., Gil-Rjong, R., Franke, P., Gründemann, E., Pharmazie 34 (1979) 360
Gründemann, E., Gil-Rjong, R., Hiller, K., Pharmazie 34 (1979) 430
Fötsch, G., Gründemann, E., Pfeifer, S., Hiller, K., Salzwedel, D.,
Pharmazie 43 (1988) 278 *(Structure)*

[2] Hiller, K., Fötsch, G., Pharmazie 41 (1986) 415 *(Quantitative determination)*

[3] Metzner, J., Hirschelmann, R., Hiller, K., Pharmazie 39 (1984) 869
*(antiphlogistic and analgesic effects)*

[4] Chodera, A., Dabrowska, K., Skrzypczak, L., Budzianowski, J.,
Acta Polon. Pharm. 43 (1986) 499 *(Diuretic effects)*

[5] Chodera, A., Dabrowska, K., Bobkiewicz-Kozlowska, T., Tkaczyk, J.,
Skrzypczak, L., Budzianowski, J., Acta Polon. Pharm. 45 (1988) 181
*(Effects on experimental urolithiasis)*

[6] Fötsch, G., Pfeifer, S., Bartoszek, M., Hiller, K., Pharmazie 44 (1989) 555 *(Biotransformation)*

# Limonene

*H. J. Bestmann, O. Vostrowsky*

## 1. Name of Compound

Common name

**Limonene**
(+)-, (−)- and (±)-Limonene

Synonyms

(+): Carvene, Citrene, Hesperidene [1]
(±): Dipentene, Cynene, Cinene, Cajeputene, Kautschine, Isoterebenthene, Diisoprene [1]

Systematic name

1-Methyl-4-(1-methylethenyl)-cyclohexene;
4-Isopropenyl-1-methylcyclohexene; *p*-Mentha-1,8-diene

| Substance | Subgroup |
|---|---|
| Terpene | Monoterpene hydrocarbon |

CAS registry number and other numbers

| | | |
|---|---|---|
| (+): [5989-27-5], | Merck Index 10, 5321 | BRN (+): 2204754 |
| (−): [5989-54-8], | EG: [601-029-00-7] | (−): 2323991 |
| (±): [7705-14-8] | | (±): 3195091 |

## 2. Formulas and Molecular Weight

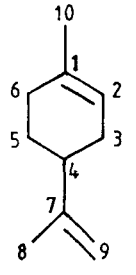
Molecular formular

$C_{10}H_{16}$

Molecular weight

136.24

Structural formula



## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| liquid | (+): 0.8419 [2]<br>(−): 0.8433 [2] | not available |
| **Melting Point mp** | $n_D^{20}$ | **Acid value** |
| (±): −89.03 °C [2] | (+): 1.4750 [2]<br>(−): 1.4730 [2] | not applicable |
| **Boiling point bp** | $[\alpha]_D^{20}$ | **Saponification value** |
| 176–178 °C [1]<br>(+): 60–61 °C/13 | (+): +126.6° [2]<br>+113° (10, EtOH)<br>(−): −126.3° [2]<br>−94° (10, EtOH) | not applicable |
| **Flash point** | **Soluble in** | |
| (+): 53<br>(−): 48 °C | ethanol, ether, hydrocarbons, chloroform, acetone etc. | |
| **Color** | **Odor** | |
| colorless | (+): orange, (−): lemon | |

## 4. Occurrence

Main constituent of citrus fruit peels oils, common in many other essential oils.
(+)-limonene: orange, lemon, grape oils;
(−)- and (±)-limonene: obtained from a variety of turpentine oils.

## 5. Health Hazard Data

| Toxicology | Hazard labeling | flammable, irritating to skin |
|---|---|---|
| Irritating substance | $LD_{50}$ | not available |
| **Waste disposal procedures**<br>Combustion | | |

## 6. Transportation and Storage Instructions

Storage temperature: +4 °C.
GGVE/GGVS: 3/31 c; UN 2052

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | m/z 68 | Molecular ion | m/z 136 (136.12520) |
| Ionization energy | 70 eV | Ion source temp. | 200 °C |
| Acceleration voltage | 4000 V | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | – |
| Spectrometer type and manufacturer | | a) Varian MAT 311A; b) Finnigan MAT 90 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3080, 3000, 3960, 2910, 1635, 1445, 1430, 1370, 1150, 1140, 910, 880, 790 $cm^{-1}$ | | | |
| Sample preparation | liquid film (100%) | Resolution | – |
| Spectrometer type and manufacturer | | Beckmann Acculab A8 | |

| NMR | | Nucleus | $^{1}H$ |
|---|---|---|---|
| Chemical Shifts<br>1.42–1.54 and 1.72–1.84 (AB, 2H, $H_2C$-5);<br>1.73 (s, 3H, $H_3C$-9);<br>2.02–2.19 (m, 1H, $HC$-4);<br>5.39–5.40 (mc, 1H, $HC$=-2) ppm | | 1.65 (s, 3H, $H_3C$-10);<br>1.94–2.09 (m, 4H, $H_2C$-3 and $H_2C$-6);<br>4.70 (s, 2H, $H_2C$=-8); | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | JEOL JNM-GX 400 | |

| NMR | see ref. [3] | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>20.73 (qu, C-9); 23.40 (qu, C-10); 27.91 (t, C-5); 30.58 (t, C-3);<br>30.80 (t, C-6); 41.09 (d, C-4); 108.38 (t, C-8); 120.66 (d, C-2);<br>133.75 (s, C-1); 150.06 (s, C-7) ppm | | | |
| Frequency | 100 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | Jeol JNM-GC 400 | |

| UV | not applicable |
|---|---|

8. Chromatographic Data

| | |
|---|---|
| TLC | not available |

| | | | |
|---|---|---|---|
| GLC | see ref. [3] | | |
| Retention time | Kovats retention indices a) $RI_{SE54}$ 1029, b) $RI_{SP2100}$ 1020 | | |
| Column type | fused silica capillary (int. diameter 0.25 mm) | Column length | a) 50 m<br>b) 25 m |
| Column coating | a) SE54 b) SP2100 | Column temp. | a) 4 min 60 °C, 60–260 °C, 3°/min<br>b) 50–250 °C, 4°/min |
| Injector port temp. | 220 °C | Carrier gas | $N_2$, 1 ml/min (22 cm/s lin. gas velocity) |
| Detector temp. | 260 °C | Sample solvent | n-$C_6H_{14}$ |
| Sample size | 0.5 µl | Sample conc. | 5% |
| Chromatograph type and manufacturer | | a) Perkin Elmer Sigma 1 b) Varian 1400 | |

| | |
|---|---|
| HPLC | For application for the separation of flavour compounds see ref. [4] |

9. Remarks

Used in the reconstitution of natural flavours and perfumes, its principal value lies in its use as an intermediate of other organoleptic materials.

10. References


[1] Karrer, W., *Konstitution und Vorkommen der organischen Pflanzenstoffe*, Birkhäuser-Verlag, Basel 1958, No. 53 and 54

[2] *Beilsteins Handbuch der organischen Chemie* (Beilstein Institut), Vol. V, 4th Ed., H pp. 133–137 (1922), EI pp. 70–72 (1930), EII pp. 88–89 (1943), EII pp. 348 (1963), EIV pp. 438–441 (1976)

[3] Craveiro, A.A., Alencar, J.W., Vostrowsky, 0., *J. Nat. Prod.* 1986, *49*, 1146.

[4] Kubeczka, K.-H. in: Schreier, P., (Ed.), *Flavour '81*, Walter de Gruyter & Co, Berlin-New York 1981, pp. 345–359; Schwanbeck, J., Kubeczka, K.-H.in Kubeczka, K.-H. (Ed.), *Vorkommen und Analytik ätherischer Öle*, G. Thieme Verlag, Stuttgart, 1979

[5] Racemization, isomerization studies and intramolecular disproportionation reactions are reported in:
Bates, R.B., Caldwell, E.S., Klein, H.P., *J. Org. Chem.* 1969, *34*, 2615;
Pines, H., Eschinazi, H.E., *J. Am. Chem. Soc.* 1955, *77*, 6314;
Pines, H., Schaap, L., *J. Am. Chem. Soc.* 1957, *79*, 2956;
Bank, S., Rowe, C.A., jr., Schriesheim, A.S., Noslund, L.A., *J. Org. Chem.* 1968, *33*, 221
Verghese, J., *Perfum. Essent. Oil Rec. 1969*, 439

[6] Total synthesis of racemic limonene by Diels-Alder reaction of isoprene with methyl vinyl ketone and subsequent Wittig reaction of the resulting ketone with methylene triphenylphosphorane
Vig, 0.P., Matta, R.L. Lal, A., Raj, I., *J. Ind. Chem. Soc.* 1964, *41*, 142

[7] Additional analytical data are found in:
a) *Retention indices (OV 101 and CW 20M):* Jennings, W., Shibamoto, T. (Ed.), *Qualitative Analysis of Flavor and Fragrance Volatiles by Glas Capillary Gas Chromatography*, Academic Press, New York 1980

b) *Mass spectra:* Jennings, W., Shibamoto, T., ibidem; Stenhagen, E., Abrahamsson, S., McLafferty, W.F. (Eds.), *Registry of Mass Spectral Data*, J. Wiley & Sons, New York 1974; Masada, Y. (Ed.), Analysis of Essential Oils by Gas Chromtography and Mass Spectrometry, J. Wiley & Sons, New York 1967

c) *$^{13}$C-NMR spectra*: Bremser, W., Ernst, L., Franke, B. (Eds.), Carbon-13 NMR Spectral Data, Verlag Chemie, Weinheim, New York 1978; Formacek, V., Kubeczka, K.-H. (Eds.), *Essential Oil Analysis by Capillary Gas Chromatography and Carbon-13 NMR Spectroscopy*, John Wiley & Sons, 1982

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# Linalool

*H.-P. Hanssen, W.-R. Abraham*

## 1. Name of Compound

Common name

**Linalool**

Synonyms

Coriandrol (S-Linalool)
l-Licareol (R-Linalool)

Systematic name

3,7-Dimethyl-1,6-octadien-3-ol

| Substance | Subgroup |
|---|---|
| Terpene | Acrylic monoterpene alcohol |

CAS registry number and other numbers

| | |
|---|---|
| racemic: [78-70-6] | BRN 1721488 |
| S-(+)-: [126-90-9] | BRN 1721486 |
| R-(−)-: [126-91-0] | BRN 1721487 |

## 2. Formulas and Molecular Weight

Molecular formular

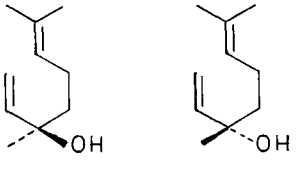
$C_{10}H_{18}O$

Molecular weight

154.1358

Structural formula

(-) (+)



## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| liquid | 0.861 g/cm$^3$ | not available |
| Melting Point mp | $n_D^{20}$ | Acid value |
| not available | 1.462 | not applicable |
| Boiling point bp | $[\alpha]_D^{20}$ | Saponification value |
| 198 °C (1013 mbar)<br>87–90 °C ($kp_{15}$)C | -19.10° (heat) | not more than 1.5 |
| Flash point | Soluble in | |
| 75 (78) °C | methylene chloride, methanol, diethylether | |
| Color | Odor | |
| colorless | flowery-fresh | |

## 4. Occurrence

Yeast, fungi, insects; widespread among higher plants.
(−)-linalool: >80% in Shiu oils (Cinnamomum camphora)
(+)-linalool: 60–70% in coriander oil.

## 5. Health Hazard Data

| Toxicology | Hazard labeling | |
|---|---|---|
| no specific data available | $LD_{50}$ | oral rat: 2.790 mg |

Waste disposal procedures
By combustion; according to Merck instructions, collect laboratory waste materials together with other unreactive, halogen-free organic substances

## 6. Transportation and Storage Instructions

GGVE/GGVS: 3/32c; RID 3/32c; ADR 3/32 c

## 7. Spectroscopic Data

| MS | see attached spectrum and remarks | | |
|---|---|---|---|
| Base peak | 71 | Molecular ion | – |
| Ionization energy | 70 eV | Ion source temp. | 200 °C |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | GC-MS coupling: GC: Hewlett-Packard 5840 A;<br>MS: Hewlett-Packard-Quadrupol 5985 A | | |

| IR | see attached spectrum | | |
|---|---|---|---|
| Sample preparation | 2% in $CCl_4$ | Resolution | – |
| Spectrometer type and manufacturer | | Perkin Elmer PE 841 | |

| NMR | | Nucleus | $^1H$-$^1H$ correlation |
|---|---|---|---|
| Chemical Shifts<br>H-1: 5.06 ppm dd, J=10.8 Hz (H-2), 1.2 Hz (H-1');<br>H-1': 5.21 dd, J=17.3 Hz (H-2), 1.2 Hz (H-1);<br>H-2: 5.91 dd, J=17.3 Hz (H-1'), 10.8 (H-1);<br>H-4: 1.59 m;<br>H-5: 2.03 m;<br>H-6: 5.12 tqq, J=7.2 Hz (H-5), 1.4 (H-8, H-9);<br>H-8: 1.68d, J=1.4 Hz, (H-6));<br>H-9: 1.60d, J=01.4 Hz, (H-6);<br>H-10: 1.28 s | | | |
| Frequency | – | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | EM 300 Bruker | |

| NMR | | Nucleus | $^1H$-$^{13}C$ correlation |
|---|---|---|---|
| Chemical Shifts<br>111.3 (C-1); 145.0 (C-2); 72.7 (C-3); 41.2 (C-4); 22.7 (C-5); 124.6 (C-6);<br>130.4 (C-7); 25.3 (C-8); 17.5 (C-9); 27.2 (C-10) ppm | | | |
| Frequency | – | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | EM 300 Bruker | |

| UV | see ref. [5] |
|---|---|

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.53 | | |
| Solvent | – | Solvent system | n-hexane/EtOAc 3:2 |
| Saturated atmosphere | 20–25 °C | Detection/Color | anisaldehyde/$H_2SO_4$<br>develop 1 min at 140 °C: blue |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel 60 $F_{254}$, 0.25 mm |

| GLC | | | |
|---|---|---|---|
| Retention time | 19.97 min | | |
| Column type | quartz-capillary column | Column length | 22 m<br>(int. diameter 0.4 mm) |
| Column packing | WG 11 (FFAP) | Column temp. | isothermic 100 °C |
| Injector port temp. | 210 °C | Carrier gas | $N_2$, 1 ml/min |
| Detector temp. | 210 °C | Sample solvent | pentane |
| Sample size | 1.0 µl | Sample conc. | about 0.01 % |
| Chromatograph type and manufacturer | | Perkin-Elmer PE F22 with FID | |

| HPLC | | | |
|---|---|---|---|
| Retention time | 22.5 min | | |
| Column | Merck LiChrosorb<br>(25 × 0.7 cm) | Stationary phase | RP-18<br>p.s. 7 µm |
| Mobile phase | linear gradient 30–95 %<br>methanol in 0.1 % acetic acid | Flow rate | 2 ml/min |
| Column temp. | 23 °C | Pressure | 1000 psig |
| Detector | UV 254 nm | Sample solvent | 30 % methanol<br>in 0.1 % acetic acid |
| Sample size | 15 µl/min | Sample conc. | – |
| Chromatograph type and manufacturer | | Waters M 660/M 6000 A | |

9. Remarks

Mass spectrometry: under certain MS conditions, other fragmentation patterns can be obtained, e.g. 41 as base peak and 93 > 71.

10. References


[1] NIOSH, Registry of Toxic Effects of Chemical Substances, National Institute of Occupational Safety and Health, Rockville, MD, 1975 *(Toxicity)*

[2] Strickler, H., Ohloff, G., Kovats, E., *Tetrahedron Lett.* (1964) 649; Vlad, P., Soucek, M., *Coll. Czech. Chem. Commun. 27* (1962) 1726; Gaydou, E.M., Randriamiharisoa, R.P., *J. Chromatogr. 396* (1987) 378-381; Rudman, A.A., Aldrich, J.A., *J. Chromatogr. 407* (1987) 324-329; Ravid, U., Putievsky, E., Bassat, M., Ikan, R., Weinstein, V., in: Bioflavour ,87 (Schreier, P., Ed.), de Gruyter, Berlin, New York, 1988, pp. 97-104 *(Structure)*

[3] Ohwa, M., Kogure, T., Eliel, E.L., *J. Org. Chem. 51* (1986) 2599-2601 *(Synthesis)*

[4] v. Sydow, E., *Acta Chem. Scand. 17* (1963) 2504 *(Mass spectrometry)*

[5] Naves, Y.R., Frei, Ch., *Helv. Chim. Acta 46* (1963) 2551 *(UV absorbance in heptane in comparison with other terpene alcohols)*

[6] Gildemeister, E., Hoffmann, F., Die Ätherischen Öle, Vol. IIIa, Akademie Verlag, Berlin, 1960, pp. 510-539 *(Occurrence in more than 200 essential oils from herbs, leaves, flowers, and wood, review)*;
Liu, C., Chu, S., Lu, H., Nian, S., *Huaxue Xuebao* (1981) 248-255 *(Aglaia odorata)*;
Rao, P.S., Sood, V.K., *Parfüm. Kosmet. 45* (1964) 63 *(Arteinisia vulgaris)*;
Prasad, Y.R., *Parfüm. Kosmet. 69* (1988) 418-419 *(Atalantia monophylla)*;
Nahrstedt, A., Economou, D., Hammerschmidt, F.J., *Planta med.* (1985) 247-249 *(Bystropogon plumosus)*;
Fujita, Y., Fujita, S., Nishida, S., *Osaka Kogyo Gijutsu Shikensho Kiho 25* (1974) 244-246 *(Cinnamomum camphora var. linaloolifera)*;
Banerjee, A.K., Kaul, V.K., Nigam, S.S., *Essenze Deriv. Agrum. 54* (1984) 117-121 *(Curcuma caesia)*;
Agbakwuru, E.O.P., Osisiogu, I.U.W., Rücker,G., *Niger. J. Pharm. 10* (1979) 203-205 *(Dennettia tripetala)*;
McFadden, W.H., Teranishi, R., Corse, J., Black, D.R., Mon, T.R., *J. Chromatogr. 18* (1965) 10 *(Fragaria vesca)*;
Fujita, S., Ishimatsu, Y., FujiLa, Y., *Yakugaku Zasshi 97* (1977) 1216-1218 *(Magnolia denudata)*;
Bang, B.T., Nikolaev, A.G., *Izv. Akad. Nauk Mold. SSR, Ser. Biol.Khim. Nauk* (1975) 89, *(Mentha longifolia)*;
Gora, J., Kalemba, D., *Herba Pol. 25* (1979) 269-275 *(M. spicata)*;
Shimizu, S., *Kiyo-Iida Joshi Tanki Daigaku* (1988) 24-33 *(M. spp.)*;
Murugesan, M., Damodaran, N.P., *Indian Perfum. 31* (1987) 163-165 *(Ocimum basilicum)*;
Bestmann, H.-J., Classen, B., Kobold, U., Vostrowsky, 0., Klingauf, F., Stein, U., *Phytochemistry 27* (1988) 85-90 *(Rhus typhina)*;
Zhen, S., Shen, X., Chen, H., Lu, R., Fu, Z., *Youji Huaxue 8* (1988) 143-146 *(Rodgersia aesculifolia)*;
Kovats, E.S., *J. Chromatogr. 406* (1987) 185-222 (Rosa damascena)
Yoshida, T., Sawasaki, T., *Nettai Nogyo 21* (1978) 145-149 *(Salvia sclarea)*;
Garnero, J., Buil, P., Pellecuer, J., *Riv. Ital. EPPOS 63* (1981) 344-349 *(Satureia montana)*;
Gupta, Y.N., Bhandari, K.S., *Indian Perfum. 18, Pt.2* (1974) 29-33 *(Tagetes erecta)*;
Gupta, Y.N., Bhandari, K.S., *Indian Perfum. 19, Pt.1* (1975) 29-32 *(T. minuta)*;
Strauss, C.R., Wilson, B., Williams, P.J., *J. Agric. Food Chem. 36* (1988) 569-572 *(Vitis vinifera)*;
Collins, R.P., Halim, A.F., *Lloydia 34* (1970) 481-483; Klingenberg,A., Sprecher,E., *Planta med.* (1985) 264-265; Hanssen, H.P., Sinnwell, V., Abraham, W.-R., *Z. Naturforsch. 41c*, (1986) 825-829 *(Fungi/yeasts)*;
Birch, M.C., Wood, D.L., *J. Chem. Ecol. 1* (1975) 101 *(insects)*

[7] Moore, J.W., Lamparsky, D., *Helv. Chim. Acta 46* (1963) 185 *(R-(-)-Linalool in pimenta racemosa)*;
Ravid, U., Putievsky, E., Bassat, M., Ikan, R., Weinstein, V. in
Bioflavour ,87 (Schreier, P., Ed.), de Gruyter, Berlin, New York, 1988, pp. 97-104 *(R-(-)-Linalool in ocimum basilicum)*

[8] Naves, Y.R., Alves, H.M., Arndt, V.H., Gottlieb, O.R., Magalhaes, M.T., *Helv. Chim. Acta 46* (1963) 1056 *(S-(+)-Linalool in cryptocarya aschersoniana)*;
Ravid, U., Putievsky, E., Bassat, M., Ikan, R., Weinstein, V. in
Bioflavour ,87 (Schreier, P., Ed.), de Gruyter, Berlin, New York, 1988, pp. 97-104 *(S-(+)-Linalool in coriandrum sativum)*

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# Loganin

*L. F. Tietze, C. Bärtels*

## 1. Name of Compound

Common name
**Loganin** [1, 2]

Synonyms
Loganoside

Systematic name
Methyl-(1S,5S,7S,8R,9R)-1-(β-D-glucopyranosyloxy)-1,5,6,7,8,9-hexahydro-7-hydroxy-8-methylcylopenta(c)pyran-4-carboxylate

Substance
Iridoide [3]

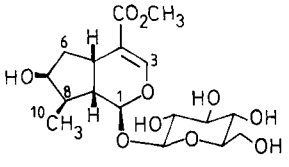
CAS registry number and other numbers
[18524-94-2] BRN 55724

## 2. Formulas and Molecular Weight

Molecular formular
$C_{17}H_{26}O_{10}$

Molecular weight
390.38

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>222 °C | $n_D^{20}$<br>not applicable | Acid value<br>not available |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20} = -85.1°$<br>(c=1.2, MeOH) | Saponification value<br>not available |
| Flash point<br>not applicable | Soluble in<br>water, methanol, acetone etc. | |
| Color<br>white | Odor<br>not applicable | |

4. Occurrence

| |
|---|
| Constituent of symphoricarpos albus (L.) and of several lonicera and strychnos species [3] |

5. Health Hazard Data

| | |
|---|---|
| Toxicology | Hazard labeling not available |
| not available | $LD_{50}$ not available |
| Waste disposal procedures<br>Combustion | |

6. Transportation and Storage Instructions

| |
|---|
| Storage temperature: +4 °C |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | m/z = 179 | Molecular ion | not available |
| Ionization energy | 70 eV | Ion source temp. | 150 °C |
| Acceleration voltage | 3000 V | Emission current | 0.3 mA |
| Resolution | 1000 | Scan rate | 2 s/decade |
| Spectrometer type and manufacturer | | Varian MAT 311 A | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3506, 3410 (OH), 1712 ($CO_2CH_3$), 1648 (C=C), 1100, 1074, 1038, 1000 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | Bruker IFS 25 | |

| NMR | assigned by 2D-spectrum | Nucleus | $^1H$ |
|---|---|---|---|

Chemical Shifts

1.08 (d, J = 6.5 Hz, 1H, $H_3$C-10),
1.60 (ddd, J = 14, 6.5, 5 Hz, 1H, $H_2$C-$6_{ax}$),
1.86 (m, 1H, *H*C-8),
2.00 (ddd, J = 18, 9, 4.5 Hz, 1H, *H*C-9),
2.21 (ddd, J = 14, 7.5, 1.5 Hz, 1H, $H_2$C-$6_{eq}$),
3.04–3.16 (m, 1H, *H*C-5),
3.21–3.43 (m, 4H, *H*C-2′,*H*C-3′,*H*C-4′,*H*C-5′),
3.53–3.68 (m, 1H, $H_2$C-$6'_a$),
3.70 (s, 3H, $H_3CCO_2$),
3.89 (dd, J = 12, 2 Hz, 1H, $H_2$C-$6'_b$),
4.03 (ddd, J = 5, 4.5, 1.5 Hz, 1H, *H*C-7),
4.65 (d, J = 8 Hz, 1H, *H*C-1′),
5.28 (d, J = 4.5 Hz, 1H, *H*C-1),
7.41 (d, J = 1.5 Hz, 1H, *H*C-3) ppm

| Frequency | 200 MHz | Solvent | $CD_3OD$ |
|---|---|---|---|
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | Varian VXR 200 | |

| NMR | $^1H$-decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|

Chemical Shifts

13.4 (C-10), 32.0 (C-5), 42.0 (C-8), 42.6 (C-6), 51.7 ($H_3CCO_2$), 62.6 (C-6′),
71.4 (C-4′), 74.5 (C-2′), 74.9 (C-7), 77.8 Cc-3′), 78.1 (C-5′), 97.6 (C-1),
99.9 (C-1′), 113.8 (C-4), 152.0 (C-3), 169.4 ($H_3CCO_2$) ppm

| Frequency | 50.3 MHz | Solvent | $CD_3OD$ |
|---|---|---|---|
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | Varian VXR 200 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | $2.34 \times 10^{-4}$ nm | $\varepsilon_{max}$ | 11 127.78 |
| Solvent | methanol | | |
| Sample conc. | $5.12 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Varian Cary 219 | |

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.30 | | |
| Solvent | methanol | Solvent system | chloroform/methanol (4/1) |
| Saturated atmosphere | – | Detection/Color | UV 254 nm |
| Plate manufacturer | Macherey-Nagel | Plate type/Product no. | SIL G/$UV_{254}$ |

| | |
|---|---|
| GLC | not applicable |

| | |
|---|---|
| HPLC | not available |

9. Remarks

Loganin is the biogenetic precursor of secologanin, which is a key intermediate in the biosynthesis of a multitude of alkaloids and secoiridoids [5].

10. References


[1] Tietze, L.F., *Angew. Chem. 95*, 1983, 840; *Angew. Chem. Int. Ed. Engl. 22*, 1983, 828 *(review)*

[2] Partridge, J.J., Chadha, N.K., Uskokovic, M.R., *J. Am. Chem Soc. 24*, 1973, 532; Büchi, G., Carlson, J.A., Powell, J.E., Tietze, L.F., *J. Am. Chem. Soc. 24*, 1973, 540; Tietze, L.F., Fischer, R., Remberg, G., Holdgrün X., Meier, H., *Liebigs Ann. Chem. 1987*, 971 *(synthesis)*

[3] Bobbit, J.M., Segebarth, K.P., in Cyclopentanoid Terpene Derivatives. Taylor, W.I., Battersby, A.R. (Eds.), M. Dekker, New York 1969; Inouye, H., Uesato, S., *Prog. Chem. Org. Nat. Prod. 50*, 1986, 169 *(biosynthesis)*

[4] Dunstan, W.R., Short, F.W., *Pharm. J. Trans. 14*, 1984, 1025; Partridge, J.J., Chadha, N.K., Uskokovic, M.R., *J. Am. Chem. Soc. 95*, 1973, 532 *(biosynthesis)*

[5] Battersby A.R., *Chem. Soc. Spec. Period. Rep. 1*, 1971, 31; Scott, A.I., *Accounts Chem. Res. 3*, 1970, 151; Gross, D., *Fortschr. Chem. Org Naturst. 28*, 1970, 140; Leete, E., *Accounts Chem. Res. 2*, 1969, 59 *(biosynthesis)*

# Loxylostosidine A

*L. F. Tietze, C. Bärtels*

## 1. Name of Compound

Common name

**Loxylostosidine A**

Synonyms

—

Systematic name

(1S,8S,9R,9aS,10aS)-9-Ethenyl-8-(β-D-glucopyranosyloxy)-2,3,9,9a,10,10a-hexahydro-5H,8H-pyrano[4,3-d]-thiazolo-[3,2-a] pyridine-5-one-1-oxide

| Substance | Subgroup |
|---|---|
| Alkaloid | Monoterpene Alkaloid |

CAS registry number and other numbers

[78119-20-7]

## 2. Formulas and Molecular Weight

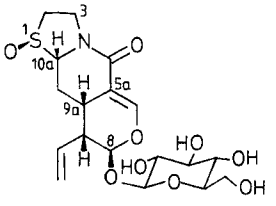
Molecular formular

$C_{18}H_{25}NO_9S$

Molecular weight

431.46

Structural formula




## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| solid | not applicable | not available |
| Melting Point mp | $n_D^{20}$ | Acid value |
| 162 °C | not applicable | not available |
| Boiling point bp | $[\alpha]_D^{20}$ = -248.32° | Saponification value |
| not applicable | (c=0.65, MeOH) | not available |
| Flash point | Soluble in | |
| not applicable | water, methanol, acetone, chloroform, etc. | |
| Color | Odor | |
| white | not applicable | |

4. Occurrence

| |
|---|
| Minor constituent of lonicera xylosteum L. |

5. Health Hazard Data

| | |
|---|---|
| Toxicology<br>not available | Hazard labeling not available<br>$LD_{50}$ not available |
| Waste disposal procedures<br>Combustion | |

6. Transportation and Storage Instructions

| |
|---|
| Storage temperature: +4 °C |

7. Spectroscopic Data

| MS | loxylostosidine A tetra acetate | | |
|---|---|---|---|
| Base peak | m/z = 43 | Molecular ion | m/z = 600 |
| Ionization energy | 70 eV | Ion source temp. | 150 °C |
| Acceleration voltage | 3000 V | Emission current | 0.3 mA |
| Resolution | 1000 | Scan rate | 2 s/decade |
| Spectrometer type and manufacturer | | Varian MAT 311 A | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3418 (OH), 1660 (C=O), 1600 (C=C), 1426, 1072, 1042, 996 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | Bruker IFS 25 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>1.65 (ddd, J=12.5, 12.5, 12.5 Hz, 1H, $H_2C$-$10_{ax}$)<br>2.52 (ddd, J=12.5, 4,4 Hz, 1H, $H_2C$-$10_{eq}$)<br>2.79 (ddd, J=9.5, 5.5, 2 Hz, 1H, *H*C-9)<br>2.99 (ddd, J=13, 7, 4.5 Hz, 1H, $H_2C$-$2_{ax}$)<br>3.11–3.44 (m, 6H, $H_2C$-$2_{eq}$, *H*C-2′, *H*C-3′, *H*C-4′, *H*C-5′, *H*C-9a)<br>3.56–3.86 (m, 2H, $H_2C$-$3_{ax}$, $H_2C$-6′a)<br>3.91 (dd, J=12, 2 Hz, 1H, $H_2C$-$6'_b$)<br>4.32 (dd, J=12.5, 4 Hz, 1H, *H*C-10a)<br>4.68 (d, J=8 Hz, 1H, *H*C-1′)<br>4.69 (ddd, J=12, 7, 5 Hz, 1H, $H_2C$-$3_{eq}$)<br>5.24 (dd, J 10, 2.5 Hz, 1H, $H_2C$=-2)<br>5.36 (dd, j=17.5, 2.5 Hz, 1H, $H_2C$=-E)<br>5.55 (d, J=2 Hz, 1H, *H*C-8)<br>5.56 (ddd, J=17.5, 10, 9.5 Hz, 1H, *H*C=$CH_2$)<br>7.48 (d, J=2.5 Hz, 1H, *H*C-7) ppm | | | |
| Frequency | 200 MHz | Solvent | $CD_3OD$ |
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | Varian XL 200 | |

| NMR | $^1H$-decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>27.3 (C-10), 28.0 (C-9a), 44.0 (C-3), 49.7 (C-2), 62.6 (C-6′),<br>71.5 (C-4′), 74.6 (C-2′), 77.8 (C-3), 78.3 (C-5′), 83.7 (C-10a),<br>97.3 (C-8), 99.5 (C-1′), 107.5 (C-6), 121.2 (CH=*C*$H_2$), 133.4 (*C*H=$CH_2$),<br>149.9 (C-7), 164.4 (C-5) ppm | | | |
| Frequency | 50.3 MHz | Solvent | $CD_3OD$ |
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | Varian VXR 200 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | $2.40 \times 10^{-4}$ nm | $\varepsilon_{max}$ | 18749.95 |
| Solvent | methanol | | |
| Sample conc. | $3.13 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Varian Cary 219 | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.33 | | |
| Solvent | methanol | Solvent system | chloroform/methanol (4/1) |
| Saturated atmosphere | – | Detection/Color | UV 254 nm |
| Plate manufacturer | Macherey-Nagel | Plate type/Product no. | SIL G/$UV_{254}$ |

| GLC | not applicable |
|---|---|

| HPLC | not applicable |
|---|---|

9. Remarks

10. References


[1] Chaudhuri, R.K., Sticher, O., Winkler, T., *Tetrahedron Lett. 22*, 1981, 559 *(isolation)*

[2] Tietze, L.F., Bärtels, C., Fennen, J., *Liebigs Ann. Chem. 12*, 1989, 1241 *(synthesis)*

# Lucenin-2

*R. Mues, H. D. Zinsmeister, S. Anhut*

## 1. Name of Compound

Common name

**Lucenin-2**

Synonyms

Luteolin-6,8-di-C-glucoside
Luteolin-6,8-di-C-β-D-glucopyranoside
6,8-Di-C-β-D-glucopyranosyl-luteolin

Systematic name

2-(3,4-Dihydroxyphenyl)-6,8-di-β-D-glucopyranosyl-5,7-dihydroxy-4*H*-1-benzopyran-4-one

| Substance | Subgroup |
|---|---|
| Flavonoid | Flavone Glycoside<br>Flavone C-Glycoside |

CAS registry number and other numbers

[29428-58-8]

BRN 1338702

## 2. Formulas and Molecular Weight

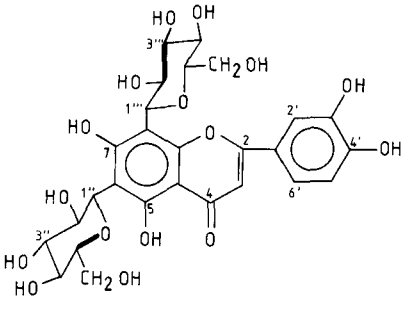
Molecular formular

$C_{27}H_{30}O_{16}$

Molecular weight

610

Structural formula



## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline | $d_4^{20}$<br>not applicable | Iodine value<br>not applicable |
| Melting Point mp<br>265–267 °C (decomp.) [2] | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20}$:<br>+36° (c=0.544, pyridine)<br>+41° (c=0.468, methanol) | Saponification value<br>not applicable |
| Flash point<br>not applicable | Soluble in<br>water, ethanol, methanol | |
| Color<br>yellow | Odor<br>odorless | |

## 4. Occurrence

Many sources [3] – e.g. for Bryophytes
Musci: Hedwigia ciliata [2]; Plagiomnium elatum (unpubl.)
Hepaticae: Mylia anomala; Mylia taylorii [4]

## 5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology<br>not known | Hazard labeling | not usual |
| | $LD_{50}$ | not known |
| Waste disposal procedures | | |

## 6. Transportation and Storage Instructions

Storage as powder; preferably in the dark and in a drying box (silica gel)

## 7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS | as Perdeuteromethylether (PDM)<br>as Permethylether (PM) | | |
| Base peak<br>range >400: | PDM: m/e = 94<br>PM: m/e = 747 | Molecular ion | PDM: 814<br>PM: 778 |
| Ionization energy | 70 eV | Ion source temp. | PDM: 170 °C<br>PM: 220 °C |
| Acceleration voltage | PDM: 3 kV<br>PM: 5 kV | Emission current | – |
| Resolution | >1000 | Scan rate | – |
| Spectrometer type and manufacturer | PDM: Varian MAT 311 spectrometersystem 188<br>PM: Finnigan MAT 90 – mass spectrometer | | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3360 ±50 (s), 1625 (s), 1570 (m), 1510 (w), 1475 (w), 1430 (m), 1350 (m), 1285 (s), 1220 (m), 1080 (s), 1045–1030 (m), 890 (w), 830 (w), 810 (w), 760 (w) $cm^{-1}$ | | | |
| Sample preparation | pellet,<br>2 mg in 200 mg KBr | Resolution | 4000–2000 $cm^{-1}$: 1.4 $cm^{-1}$<br>2000– 600 $cm^{-1}$: 0.5 $cm^{-1}$ |
| Spectrometer type and manufacturer | Beckmann IR-Spectrophotometer 4210 | | |

| NMR | | Nucleus | $^{1}H$ |
|---|---|---|---|
| Chemical Shifts<br>Aglycone: 6.59 (s, H-3); 6.90 (d, J=8 Hz, H-5′); 7.44 (d, J=2 Hz, H-2′); 7.46 (dd, J=8, 2 Hz, H-6′)<br>Sugars: 4.8 (d, J=10 Hz, H-1″); 4.9 (H-1‴); 3–4 (sugar protons) ppm | | | |
| Frequency | 400 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | 80 °C |
| Spectrometer type and manufacturer | Bruker AM 400 | | |

| NMR | see ref. [2, 5] | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>181.7 (C-4); 166.8 (C-2); 163.8 (C-7); 161.0 (C-9); 158.9 (C-5);<br>149.4 (C-4′); 145.4 (C-3′); 121.7 (C-1′); 119.0 (C-6′); 115.7 (C-5′);<br>113.7 (C-2′); 108.4 (C-6); 103.4 (C-8); 102.3 (C-3); 81.4 (C-5‴);<br>80.9 (C-5″); 78.3 (C-3″, C-3‴); 73.6 (C-1″, C-1‴);<br>71.2 (C-2″,C-2‴); 69.7 (C-4‴, 4‴); 60.7 (C-6″, 6‴) ppm | | | |
| Frequency | 100 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | 80 °C |
| Spectrometer type and manufacturer | Bruker AM 400 | | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 257, 270, 291sh, 345 nm | $\varepsilon_{max}$ | – |
| Solvent | Methanol (Uvasol)<br>(additional UV spectra with shift reagents according to ref. [6]) | | |
| Sample conc. | 15–35 × $10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Super Scan 3(Varian); UV-VIS-spectrophotometer, double beam | | |

8. Chromatographic Data

| | | | |
|---|---|---|---|
| TLC | I,II on Cellulose (Avicel); III on Polyamide-6 | | |
| Rf Value | System I: 0.23; II: 0.32; III: 0.52 | | |
| Solvent system | I: Butanol-(1) : Acetic Acid : Water (4 : 1 : 5)<br>II: Acetic Acid : Water (15 : 85)<br>III: Butanone-(2): Methanol : Pentanedione- (2,4) (65 : 15 : 15 : 5) | | |
| Saturated atmosphere | yes | Detection/Color | UV deep purple;<br>UV, sprayed with Diphenylboric-acid-β-aminoethylester (NA): yellow |
| Plate manufacturer | Schleicher & Schüll<br>Macherey & Nagel | Plate type/Product no. | Cellulose, Avicel F 1440<br>Polygram; 0.1 mm; Polyamide-TLC 6 |

| | |
|---|---|
| GLC | underivatized not possible |

| HPLC | | | |
|---|---|---|---|
| Retention time | 11 min | | |
| Column | L = 250 mm, 4 mm I.D. | Stationary phase | Lichrosorb RP 18, 10 µm |
| Mobile phase | MeOH-Acetic Acid-Water (17 : 5 : 78) | Flow rate | 1.5 ml/min |
| Column temp. | ambient temp., ≈25 °C | Pressure | about 70 bar |
| Detector | UV-variable wave length Detector Waters 450, Detection 254 nm | Sample solvent | MeOH |
| Sample size | 5–10 µl | Sample conc. | $150–350 \times 10^{-6}$ mol/l |
| Chromatograph type and manufacturer | | Waters M 45 | |

9. Remarks

10. References


[1] Seikel, M.K. and Mabry, T.J.: A new Type of Glycoflavonoid from Vitex lucens. *Tetrahedron Lett. 16*, 1965, pp. 1105–9 *(name, occurrence)*

[2] Österdahl, B.-G.: Chemical Studies on Bryophytes. 19th Application of $^{13}C$ NMR. In ‚Structural Elucidation of Flavonoid C-Glycosides from Hedwigia ciliata', *Acta Chemica Scandinavica B32*, 1978, pp. 93–97 *(occurrence, $^{13}C$-NMR)*

[3] Chopin, J. and Dellamonica, G.: C-Glycosylflavonoids. In: ‚The Flavonoids', Harborne, J.B. (Ed.), Chapman and Hall, London, 1988 *(occurrence)*

[4] Mues, R., Müller, C., Pröbsting, U. and Zinsmeister, H.D.: Phytochemical Studies on the Genus Mylia S. Gray (Jungermanniaceae Hepaticae). *Bull. Natn. Sci. Mus., Tokyo, Ser. B, 14(4)*, 1988, pp. 149–55 *(occurrence)*

[5] Markham, K.R. and Chari, V.M.: Carbon-13 NMR Spectroscopy of Flavonoids. In: ‚The Flavonoids', Harborne, J.B. and Mabry T.J. (Eds.), Chapman and Hall London, 1982 *($^{13}C$-NMR)*

[6] Mabry, T.J., Markham, K.R. and Thomas, M.B.: The Systematic Identification of Flavonoids. Springer Verlag, 1970 *(UV spectroscopy)*

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# Lucidin

*J. Westendorf, B. Poginsky*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Lucidin** | |
| Synonyms | |
| Systematic name<br>1,3-Dihydroxy-2-hydroxymethyl-9,10-anthraquinone | |
| Substance<br>Anthracene derivative | Subgroup<br>Hydroxyanthraquinone |
| CAS registry number and other numbers<br>[478-08-0] | BRN 1888954 |

## 2. Formulas and Molecular Weight

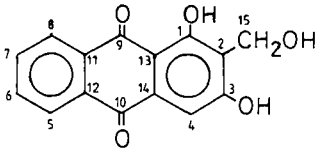
Molecular formular

$C_{15}H_{10}O_5$

Molecular weight

270.2425

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>$>330\,°C$ | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{20}$<br>not applicable | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>DMSO, slightly soluble in methanol, acetone, ethyl acetate | |
| Color<br>yellow | Odor<br>odorless | |

## 4. Occurrence

| |
|---|
| Several species of the family Rubiceae: Rubia, Coprosma, Galium, Morinda, Asperula |

## 5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology<br><br>Mutagenic,<br>possible carcinogen | Hazard labeling | May cause cancer.<br>Avoid contact with skin and eyes.<br>Do not breathe dust |
| | $LD_{50}$ | not available |
| Waste disposal procedures<br>By combustion | | |

## 6. Transportation and Storage Instructions

| |
|---|
| Sensitive to light |

## 7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 252.2272 ($M^+$-18) | Molecular ion | 270.2425 (13%) |
| Ionization energy | 70 eV | Ion source temp. | 250 °C |
| Acceleration voltage | 8000 V | Emission current | 1 mA |
| Resolution | low | Scan rate | – |
| Spectrometer type and manufacturer | | 70-2505 VG Analytical Manchester | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3418, 1659, 1617, 1589, 1335, 1283 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | 10 $cm^{-1}$ |
| Spectrometer type and manufacturer | | 001 Perkin Elmer | |

| NMR * | see ref. [3] | Nucleus | $^{1}H$ |
|---|---|---|---|
| Chemical Shifts<br>4.54 (s, 2H, benzyl $H_2C$-); 7.2 (s, 1H, H-4); 7.88, 7.94 (m, 2H, H-6, 7);<br>8.12, 8.23 (m, 2H, H-5, 8); 13.20 (s, 1H, OH) ppm | | | |
| Frequency | 400 MHz | Solvent | DMSO-$d_6$ |
| Standard | TMS | Sample temp. | ambient temperature |
| Spectrometer type and manufacturer | | JEOL-FX-400 | |

| NMR * | see ref. [3] | | Nucleus | $^{13}C$ | |
|---|---|---|---|---|---|
| Chemical Shifts | | | | | |
| 163.4 (s); | 120.1 (s); | 163.0 (s); | 107.7 (d); | 126.5 (d); | 134.3 (d); |
| 133.1 (d); | 126.2 (d); | 185.9 (s); | 181.5 (s); | 110.6 (s); | 133.9 (s); |
| 132.7 (s); | 131.7 (s, C1-14); | 51.2 (t, C-15) ppm | | | |
| Frequency | 50.1 MHz | | Solvent | $CDCl_3$ | |
| Standard | – | | Sample temp. | ambient temperature | |
| Spectrometer type and manufacturer | | | not given | | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 242, 246, 280,<br>330, 415 nm | $\varepsilon_{max}$ | 4.38, 4.39, 4.35,<br>3.54, 3.75 |
| Solvent | Ethanol | | |
| Sample conc. | – | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Beckman, DB | |

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.54 | | |
| Solvent | – | Solvent system | Toluol(80):Ethyl acetate<br>(20):Formic acid(10) |
| Saturated atmosphere | 20–25 °C | Detection/Color | day light or UV 265/yellow |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel 60, 0.25 mm |

| | |
|---|---|
| GLC | not available |

| HPLC | | | |
|---|---|---|---|
| Retention time | 16 min | | |
| Column | Kontron | Stationary phase | RP 18, 10 µm |
| Mobile phase | 1% acetic acid (A)<br>acetonitril (B) | Flow rate | 2 ml/min |
| Column temp. | ambient temp. | Pressure | 70 bar |
| Detector | UV, 254 nm | Sample solvent | methanol |
| Sample size | 5 µg | Sample conc. | 0.4 mol/l |
| Chromatograph type and manufacturer | | Beckman, System Gold | |

9. Remarks

Addendum to mobile phase: Gradient: 0–5 min (80% A), 5–15 min (80–55% A), 15–20 min (55% A), 20–30 min (55–0% A), 30–40 min (100% B)

10. References


[1] Burnett, A.R., Thomson, R.H., *J. Chem.Soc. (c):* 2437 (1968) (*melting point*)

[2] Westendorf, J., Poginsky, B., Marquardt, H., Groth, G., Marquardt, H., *Cell Biol.Toxicol.* 4: 225–239 (1988) (*mutation*)

[3] Spectroscopic Data:
Leistner, E., *Planta med. Suppl. 28:* 214 (1975) (*UV*)
Yasui, Y. and Takeda, K., *Mutat.Res.* 121: 185 (1983) ($^{1}$*H-NMR*)
Inoue, K. et al., *J. Chem. Soc. Chem. Commun. 957* (1979) ($^{13}$*C-NMR*)

[4] Occurrence:
Borisov, M.I., *Rastit.Resur 11* (3): 362 (1975) (*Asperula besseriana Klock*)
Burnett, A.R., Thomson, R.H., *J. Chem. Soc. (c):* 854 (1968) (*A. odorata*)
Hocquemiller, R.A., Fournet, A., Bouquet, A., Bruneton, J., Cavé, A., *Plant. Med. Phytothér. 10*: 110 (1976) (*Commitheca liebrechtsiana Brem.*)
Briggs, L.H., Beachen, J.F., Cambie, R.C., Dudman, N.P.B., Steggles, A.W., Ruttedge, P.S., *J. Chem. Soc. Perk. Trans.* 1: 1789 (1976) (*Coprosma lucida J.R. et G. Forst C. and rotundifolia A. Cunn)*
Burnett, A.R., Thomson, R.H., *J. Chem. Soc. (c)*: 854 (1968) (*Gallum aparinel., G. pumilum L., G. saxatile L., G. saxatile xG. sternerie, G. sterneri Ehrend., G. verum L.*)
Zhuravlev, N.S., Borisov, M.I., *Khim. Prir. Soedin. 5* (2): 118 (1969) und *Farm. Zh. (Kiev) 25* (1): 76 (1970) (*G. dasypodum Klock.)*
Bauch, H.-J., Leistner, E., *Plant.Med. 33*: 105 und 124 (1978)
Burnett, A.R., Thomson, R.H., *J. Chem. Soc. (c)*: 854 (1968)
Inoue, K.Y., Shiobara, Y., Nayeshiro, H., Inouye, H., Wilson, G., Zenk, M.H., *J. Chem. Soc. Chem. Commun*: 957 (1979) und *Phytochemistry 23*(2): 307 (1984) *(G. mollugo L.)*
Borisov, M.I., *Rastit. Resur. 11* (3): 362 (1975) (*G. ruthenicum Willd.*)
Zhuravlev, N.S., *Khim. Prir. Soedin. 10* (5): 656 (1974) (*G. semiamictum Klock)*
Brew, E.J.C., Thomson, R.H., *J. Chem. Soc. (c):* 2001 (1971) (*Hymenodictyon excelsum Wall.*)
Inoue, K.H., Nayeshiro, H., Inouye, H., Zenk, M.H., *Phytochemistry 20* (7): 1693 (1981) (*Morinda citrifolia L.*)
Leistner, E., *Planta Med. Suppl. 28*: 214 (1975) (*Morinda citrifolia L.*)
Demagos, G.P., Baltus, W., Höfle, G., *Z. Naturforsch. 36* (b): 1180 (*M. lucida Benth.*)
Park, Y.H., Dissertation, Univ. of Mississippi (1977) (*M. roioc L*).
Burnett, A.R., Thomson, R.H., *Phytochemistry 7:* 1421 (1968) (*M. umbellata L.*)
Itokawa, H.K., Mihara, K., Takeya, K., *Chem. Pharm. Bull* (Tokio) *31* (7): 2353 (1983) (*Rubia cordifolia L.*)
Murti, V.V.S., Seshadri, T.R., Sivakumaran, S., *Phytochemistrv 11*: 1524 (1972) (*R. iberica C. Koch)*
Burnett, A.R., Thomson, R.H., *J. Chem. Soc. (c):* 2437 (1968) (*R. tinctorum L.*)
Murti, V.V.S., Seshadri, T.R., Sivakumaran, S., *Indian J. Chem. 8:* 779 (1970) (*R. tinctorum L.*)

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# Magnolol

*Wang Zhongdong*

## 1. Name of Compound

| |
|---|
| Common name<br>**Magnolol** |
| Synonyms |
| Systematic name<br>[1,1′-Biphenyl]-2,2′-diol, 5,5′-di-2-propenyl- |
| Substance<br>Lignan |
| CAS registry number and other numbers<br>[528-43-8] BRN 1982224 |

## 2. Formulas and Molecular Weight

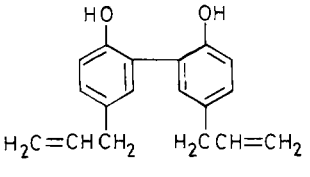
Molecular formular

$C_{18}H_{18}O_2$

Molecular weight

266.32

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>102 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{20}$<br>not available | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>methanol, ethanol, ethyl acetate, chloroform, alkali | |
| Color<br>colorless | Odor<br>odorless | |

## 4. Occurrence

| |
|---|
| Several species of Magnolia, Magnoliaceae, such as M. officinale Rehd. et Wils., M. obvata Thunb., M. rostrata W.W.Smith, M. grandiflora L. |

## 5. Health Hazard Data

| | |
|---|---|
| Toxicology | Hazard labeling |
| | $LD_{50}$ not available |
| Waste disposal procedures | |

## 6. Transportation and Storage Instructions

| |
|---|
| |

## 7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 266 | Molecular ion | 266 (M+, 100) |
| Ionization energy | 70 eV | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | – | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3125, 1640, 1610, 1495, 1440, 1409, 1365, 1337, 1280, 1260, 1220, 1150, 1130, 1110, 1085, 985, 945, 925, 897, 876 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | IR-27G (Shimadzu, Japan) | |

| NMR | | Nucleus | $^1H$-$^1H$-correlation |
|---|---|---|---|
| Chemical Shifts<br>3.36 (d, 4H); 5.00 (m, 2H); 5.15 (m, 2H); 5.73–6.15 (m, 2H); 6.89–7.25 (m, 6H) ppm | | | |
| Frequency | 80 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | ambiente temperature |
| Spectrometer type and manufacturer | | Bruker AC-80 (Switzerland) | |

| UV * | | | |
|---|---|---|---|
| $\lambda_{max}$ | 294 nm | $\varepsilon_{max}$ | – |
| Solvent | ethanol | | |
| Sample conc. | – | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | SP-800 | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | ca. 0.70 | | |
| Solvent | ethanol | Solvent system | benzene:methanol (27 : 1) |
| Saturated atmosphere | – | Detection/Color | iodine vapor |
| Plate manufacturer | hand made | Plate type/Product no. | silica gel-G (China) |

| GLC | not available |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

10. References


[1] Yan Wenmei, Zhongzaoyao (Chinese Herb Medicine) *12* (1978) 1

[2] Mitiiti Fujita,Hideji Itokawa & Yutaka Sashida, Yakugakuzashi *93*(4)(1973) 429

[3] Jiang Jiwu, Shang Qingxiang, Handbook of Active Constituents of Herb Medicine (Chinese) 1986, p. 693

[4] Li Anjuan, Guo Xinfang, Wang Xiaomin, Chen Changbiao, Shi Yunhua, Sui Ninghai & Du Jiqing, Chinese Journal of Pharmaceutical Analysis (Chinese)

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# Matricine

*R. Carle*

## 1. Name of Compound

Common name

**Matricine**

Synonyms

1-Hydroxy-6-acetoxyguaj-2,4-(10)-dien-8,12-olide

Systematic name

(-)-(3S, 3aR, 4S, 9aS, 9bS)-4-Acetoxy-2,3,3a,4,5,9,9a,9b-octahydro-9-hydroxy-3,6,9-trimethyl-azuleno[4,5-b]-furan-2-one

| Substance | Subgroup |
|---|---|
| Sesquiterpene lactone | Proazulene |

CAS registry number and other numbers

[29041-35-8] BRN 4265755

## 2. Formulas and Molecular Weight

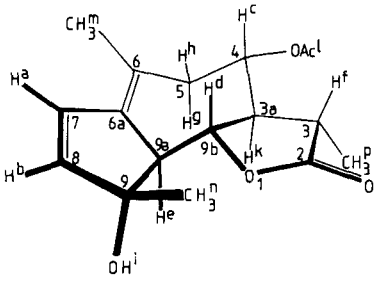
Molecular formular

$C_{17}H_{22}O_5$

Molecular weight

306.36

Structural formula



## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline [2] | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>158–160 °C [1]<br>(rapid heating) | $n_D^{20}$<br>not available | Acid value<br>not available |
| Boiling point bp<br>not available | $[\alpha]_D^{20}$<br>−122 [2] | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>ethanol, methanol | |
| Color<br>colorless | Odor<br>odorless | |

## 4. Occurrence

| |
|---|
| Chamomile |

## 5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| unknown | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

## 6. Transportation and Storage Instructions

| |
|---|
| Storage in closed containers, protected from light |

## 7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS * | EI see ref. [3] | | |
| Base peak | 228 m/e | Molecular ion | 288 m/e |
| Ionization energy | 70 eV | Ion source temp. | 130 °C |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | Finnigan 4023 | |

| IR * | see ref. [2] |
|---|---|
| Characteristic peaks<br>3420, 3070, 3050, 3000–2860, 1750, 1725, 1450, 1410, 1385, 1375, 1360, 1255, 1240, 1200, 1190, 1075, 780 $cm^{-1}$ | |
| Sample preparation | KBr |
| Resolution | – |
| Spectrometer type and manufacturer | Perkin Elmer |

| NMR * | see ref. [3] | Nucleus | $^{1}H$ |
|---|---|---|---|
| Chemical Shifts<br>1.35 (d, $CH_3$, C-3); 1.41 (s, $CH_3$ C-9); 1.85 (m, $CH_3$, C-6); 2.11 (s, Ac-$CH_3$);<br>2.21 (m, H-3a); 2.2 (br s, OH); 2.39 (m, H-5β); 2.48 (m, H-5α);<br>2.57 (dq, H-3); 2.91 (m, H-9a); 4.09 (dd, H-9b); 4.94 (ddd, H-4);<br>5.93 (br d, H-8); 6.32 (d, H-7) ppm | | | |
| Frequency | 270 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 27 °C |
| Spectrometer type and manufacturer | | Bruker WH 270 | |

| NMR * | see ref. [3] | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>15.4 (C-3); 21.1 (C-9); 23.5 (C-6); 25.1 (Ac-$CH_3$); 40.4 (C-3a); 42.5 (C-5);<br>56.7 (C-3); 58.8 (C-9a); 71.8 (C-9b); 79.3 (C-4); 84.2 (C-9); 123.7 (C-6);<br>129.8 (C-8); 136.8 (C-6a); 140.7 (C-7); 169.9 (C=O); 177.6 (C-2) ppm | | | |
| Frequency | 20 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | not given |
| Spectrometer type and manufacturer | | Varian CFT-20 | |

| UV | see ref. [1] | | |
|---|---|---|---|
| $\lambda_{max}$ | 243 nm | log $\varepsilon_{max}$ | 4.32 |
| Solvent | Methanol | | |
| Sample conc. | $4.9 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Perkin Elmer LAMBDA 5 UV/VIS | |

8. Chromatographic Data

| | | | |
|---|---|---|---|
| TLC | see ref. [2] | | |
| Rf Value | 0.20–0.25 | | |
| Solvent | $CH_2Cl_2$ | Solvent system | $CH_2Cl_2$/EtOH (98 : 2; v : v) |
| Saturated atmosphere | yes | Detection/Color | anisaldehyde/sulfuric acid: violett |
| Plate manufacturer | not given | Plate type/Product no. | silica gel |

| | |
|---|---|
| GLC | underivatized not possible |

| | | | |
|---|---|---|---|
| HPLC | see ref. [4] | | |
| Retention time | 21 min | | |
| Column | RP-18, 250 × 4.6 mm | Stationary phase | RP-18 |
| Mobile phase | MeOH/$H_2O$ (52 : 48) | Flow rate | not given |
| Column temp. | room temp. | Pressure | not given |
| Detector | 254 nm | Sample solvent | $CH_2Cl_2$ |
| Sample size | not given | Sample conc. | not given |
| Chromatograph type and manufacturer | | Eyela PLE-10 Zinsser Analytica | |

9. Remarks

Upon steam distillation chamazulene is obtained [5]

10. References


[1] Cekan, Z., Herout, V., Sorm, F.,
a) *Coll. Czech. Chem. Commun. 19* (1954) 798–804
b) *Coll. Czech. Chem. Commun. 22* (1957) 1921–1929
c) *Chem. Ind.* (1956) 1234–1235 *(UV-spectroscopy, name, occurence)*

[2] Stahl, E., Schild, W. in: *Pharmazeutische Biologie 4, Drogenanalyse II: Inhaltsstoffe und Isolierungen*, Gustav Fischer Verlag, Stuttgart, New York 1981 *(Isolation, IR-Spectroscopy, TLC)*

[3] Flaskamp, E., Zimmermann, G., Nonnenmacher, G., Isaac, 0., *Z. Naturforsch. 37b* (1982) 508–511 *(Carbon-13-NMR, Mass Spectrometry)*

[4] Ghassemi-Dehkordi, N., *Analytik, Radioisotopenmarkierung und Pharmakokinetik von Matricin und Spiroäthern aus Matricaria recutita L.*, Doctoral thesis, Universität Marburg 1988 *(HPLC)*

[5] Cekan, Z., Herout, V., Sorm, F., *Chem. Ind.* (1954) 604–605 *(Decomposition)*

# Mellein

*K. Ludewig, K. Krohn*

## 1. Name of Compound

Common name
**Mellein**

Synonyms
Ochracin

Systematic name
3,4-Dihydro-8-hydroxy-3-methyl-[1H]-2-benzopyran-1-one

Substance
Benzopyran

CAS registry number and other numbers

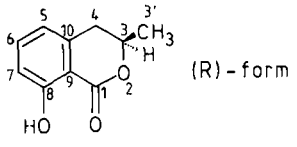
| | | | |
|---|---|---|---|
| [17397-85-2] | (R)-form | [480-33-1] | BRN 83702 |
| | (S) | [62623-84-1] | BRN 1284678 |
| BRN 150795 | (±) | [1200-93-7] | BRN 83703 |

## 2. Formulas and Molecular Weight

Molecular formular
$C_{10}H_{10}O_3$

Molecular weight
178.18

Structural formula



(R)-form

## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not applicable |
| Melting Point mp<br>(R, S): 51-52 °C<br>(±): 39 °C | $n_D^{20}$<br>not applicable | Acid value<br>not available |
| Boiling point bp<br>not available | $[\alpha]_D^{22} = -100$ (R)<br>(c = 1.01, $CHCl_3$)<br>$[\alpha]_D^{25} = +102$ (S)<br>(c = 1.07, $CHCl_3$) | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>$CH_2Cl_2$, $CHCl_3$, $Et_2O$ | |
| Color<br>colorless | Odor<br>odorless | |

4. Occurrence

| |
|---|
| (R)-form: isolated from Aspergillus spp. and Pezicula Lioida [4]<br>(S)-form: metabolite of an unidentified fungus [3] |

5. Health Hazard Data

| Toxicology | Hazard labeling |
|---|---|
| antifungal activity | $LD_{50}$ not available |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 178 | Molecular ion | 178 |
| Ionization energy | 70 eV | Ion source temp. | room temperature |
| Acceleration voltage | 3 kV | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | 3 s/decade |
| Spectrometer type and manufacturer | | Finnigan MAT 8430 | |

| IR * | see ref. [2] | | |
|---|---|---|---|
| Characteristic peaks<br>3450, 3000, 1665, 1615, 1580, 1460, 1410, 1380, 1365, 1325, 1300, 1235, 1220, 1200, 1165, 1115, 1060, 1050, 955, 900, 810, 785, 740 $cm^{-1}$ | | | |
| Sample preparation | – | Resolution | – |
| Spectrometer type and manufacturer | | JASCO A-102 spectrometer | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>1.53 (d, $J=6.3$ Hz, 3H, 3'-H); 2.93 (d, $J=7$ Hz, 2H, 4-H); 4.73 (m, 1H, 3-H);<br>6.69 (dd, $J=7.4$, 0.7 Hz, 1H); 6.89 (d, $J=8.4$ Hz, 1H); 7.41 (dd, 1H);<br>11.04 (s, 1H, OH) ppm | | | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | – |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>20.76 (p, C-3'), 34.60 (s, C-4), 76.10 (t, C-3), 108.29 (q),<br>116.23 (t), 117.68 (t), 136.14 (t, C-7), 139.39 (q),<br>162.18 (q, C-8), 169.96 (q, C-1) ppm | | | |
| Frequency | 25 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | – |
| Spectrometer type and manufacturer | | Hitachi R-24 A spectrometer | |

| UV * | see ref. [1] | | |
|---|---|---|---|
| $I_{max}$ | 246 and 314nm | $\varepsilon_{max}$ | 6457 und 3981 |
| Solvent | Ethanol | | |
| Sample conc. | – | Cell thickness | – |
| Spectrometer type and manufacturer | | Unicam SP 800 spectrometer | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.9 | | |
| Solvent | $CH_2Cl_2$ | Solvent system | 100% $CH_2Cl_2$ |
| Saturated atmosphere | – | Detection/Color | UV |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel $60F_{254}$/Merck 5749 |

| | |
|---|---|
| GLC | not available |

| | |
|---|---|
| HPLC | not available |

9. Remarks

10. References


[1] Anderson, J.R. et al. *J. Chem. Soc. Perkin Trans I*, 1983, 2185 *(UV)*

[2] Mori, K. et al. *Tetrahedron Lett. 41,22*, 1985, pp. 5295-5299, *($^{13}C$, $^{1}H$, IR)*

[3] Bycroft, B.W. Dictionary of Antibiotics and Related Substances *(occurrence)*

[4] Krohn, K., Ludewig K., Aust, H.J., Draeger S., Schulz B. (to be published)

# Methylmellein

*K. Ludewig, K. Krohn*

## 1. Name of Compound

| |
|---|
| Common name<br>**Methylmellein** (R-form) |
| Synonyms<br>5-Methylochracin |
| Systematic name<br>3,4-Dihydro-8-hydroxy-3,5-dimethyl-1H-2-benzopyran-1-one |
| Substance<br>Isocoumarin |
| CAS registry number and other numbers<br>[7734-92-1] BRN 4248173 |

## 2. Formulas and Molecular Weight

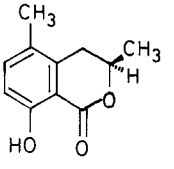
Molecular formular

$C_{11}H_{12}O_3$

Molecular weight

192.214

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not applicable |
| Melting Point mp<br>126 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{24}$: −112° | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>methylene chloride, chloroform | |
| Color<br>white | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Minor metabolite of Fusicoccum amygdali, also obtained from Semecarpus spp., Hypoxylon spp., Nummularia spp. and Idriella bambusae [4]. |

5. Health Hazard Data

| | |
|---|---|
| Toxicology<br>antibiotic activity [1]<br>antifungal activity [4] | Hazard labeling<br>$LD_{50}$ |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 192 | Molecular ion | 192 |
| Ionization energy | 70 eV | Ion source temp. | room temp. |
| Acceleration voltage | 3 kV | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | 3 s/decade |
| Spectrometer type and manufacturer | | Finnigan MAT 8430 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>2982 (OH), 1672 (C=O), 1661, 1481, 1392, 1230 (C−O), 1214, 1129, 1053, 1038, 843, 803 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | Nicolet 320 FT-IR Spectrometer | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>1.55 (d, J=6.3 Hz, 3H, 3′-H);<br>2.1 (s, 3H, 5′-H);<br>2.71 (dd, J=11.6 and 16.7 Hz, 1H, 4-$H_{a,b}$);<br>2.95 (dd, J=3.3 and 16.7 Hz, 1H, 4-$H_{a/b}$); | | 4.69 (m, 1H, 3-H);<br>6.82 (d, J=7.5 Hz, 1H, 6-H);<br>7.26 (d, J=7.5 Hz, 1H, 7-H);<br>11.0 (s, OH) ppm | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temp. |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | $^1H$-decoupled | | Nucleus | $^{13}C$ |
|---|---|---|---|---|
| Chemical Shifts<br>18.10 (p, C-5′);<br>108.12 (q);<br>137.95 (t, C-7); | 20.95 (p, C-3′);<br>115.73 (t, C-6);<br>160.55 (q, C-8); | 31.95 (s, C-4);<br>124.93 (q);<br>170.36 (q, C-1) ppm | | 75.44 (t, C-3);<br>137.06 (q); |
| Frequency | 100 MHz | | Solvent | $CDCl_3$ |
| Standard | TMS | | Sample temp. | room temp. |
| Spectrometer type and manufacturer | | Bruker AM 400 | | |

| UV * | see ref. [3] | | |
|---|---|---|---|
| $I_{max}$ | 247, 323 nm | $\varepsilon_{max}$ | 4365, 2691 |
| Solvent | Ethanol | | |
| Sample conc. | – | Cell thickness | – |
| Spectrometer type and manufacturer | | Unicam SP 800 spectrometer | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.85 | | |
| Solvent | $CH_2Cl_2$ | Solvent system | 100% $CH_2Cl_2$ |
| Saturated atmosphere | – | Detection/Color | UV |
| Plate manufacturer | Merck, Darmstadt | Plate type/Product no. | Kieselgel $60F_{254}$, Art. 5749 |

| | |
|---|---|
| GLC | not available |

| | |
|---|---|
| HPLC | not available |

9. Remarks

Phytotoxin
Inhibits the growth of Ustilago oiolacea and Chlorella pyrenoidosa [4].

10. References


[1] Bycroft, B.W., Dictionary of Antibiotics and Related Substances *(Occurrence)*

[2] Ballio, A. et al., *Tetrahedron Lett.* 1966, 3723 *(Isolation, Structure)*

[3] Anderson, J.R. et al., *J. Chem. Soc. Perkin Trans. 1* (1983) 2185

[4] Krohn, K., Ludewig, K., Aust, H.J., Draeger, S., Schulz, B. (to be published)

# Mollisin

*R. Bahramsari, K. Krohn*

## 1. Name of Compound

| |
|---|
| Common name<br>**Mollisin** [1] |
| Synonyms<br>8-Dichloroacetyl-2,7-dimethyl-5-hydroxy-1,4-naphthoquinone |
| Systematic name<br>1,4-Naphthoquinone, 8-(dichloroacetyl)-5-hydroxy-2,7-dimethyl |
| Substance<br>Naphthoquinone |
| CAS registry number and other numbers<br>[667-92-5] BRN 2290360 |

## 2. Formulas and Molecular Weight

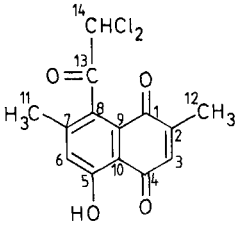
Molecular formular

$C_{14}H_{10}O_4Cl_2$

Molecular weight

313.15

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid, needles | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>194 °C<br>Lit.: 202-203 °C [4] | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{20}$<br>not applicable | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>$CH_2Cl_2$ and $CHCl_3$ | |
| Color<br>yellow | Odor | |

## 4. Occurrence

| |
|---|
| Isolated from the fungus Mollisia caesia [1-4]. |

## 5. Health Hazard Data

| Toxicology | Hazard labeling |
|---|---|
| antibiotic activity | $LD_{50}$ |
| Waste disposal procedures | |

## 6. Transportation and Storage Instructions

| |
|---|
| |

## 7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 244 | Molecular ion | 312 |
| Ionization energy | 70 eV | Ion source temp. | 60 °C |
| Acceleration voltage | 3 kV | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | 3 s/decade |
| Spectrometer type and manufacturer | | Finnigan MAT 8430 | |

| IR | see also ref. [2] | | |
|---|---|---|---|
| Characteristic peaks<br>1720 (acetonyl, carbonyl), 1645, 1614.7, 1567.3 (carbonyls), 3266.5-3449.9 (OH- band) $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | 2.5 $cm^{-1}$ |
| Spectrometer type and manufacturer | | Perkin Elmer 1420 | |

| NMR | | | Nucleus | $^1H$ |
|---|---|---|---|---|
| Chemical Shifts<br>2.17 (d, $J = 1.41$ Hz, 11-$CH_3$); 2.44 (s, 3H, 12-$CH_3$); 6.32 (s, 1H, 14-$CH_3$);<br>6.85 (d, $J = 1.44$ Hz, 3-CH); 7.20 (s, 1H, 6-CH); 11.98 (s, 1H, OH) ppm | | | | |
| Frequency | 400 MHz | | Solvent | $CDCl_3$ |
| Standard | TMS | | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | | Bruker AM 400 | |

| NMR | $^{1}H$ decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>16.44 (C-11),<br>130.062 and<br>162.16 (C-5), | 20.58 (C-12), 70.89 (C-14),<br>130.25 (C-9 and C-10),<br>186.04 (C-1), 189.30 (C-4), | 112.80 (C-7),<br>136.12 (C-3),<br>191.91 (C-13) ppm | 126.08 (C-6),<br>146.37 (C-2), 148.70 (C-8), |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV | not available |
|---|---|

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.6 (0.74 in $CH_2Cl_2$) | | |
| Solvent | – | Solvent system | 1% MeOH/99% $CH_2Cl_2$ |
| Saturated atmosphere | – | Detection/Color | 254 and 366 nm |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel $60F_{254}$/Merck 5562 |

| GLC | not available |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

10. References


[1] Bentley, R. and Gatenbeck, S., *Biochem. 4*, 1965, pp. 1150-1156 *(biosynthesis)*

[2] Overeem, J.C. and Van der Kerk, G.J.M., *Recuel 83*, 1964, 995-1004 *(structure, IR)*

[3] Casery M.L., Paulick, R.C., Whitlock H.W. Jr.*, *J. Am. Chem. Soc. 98*, pp. 2636-2640 *($^{13}C$-NMR, biosynthesis)*

[4] Tanabe, M. and Seto, H., *Biochem. 9*, 1970, pp. 4851-4853 *(occurrence, physical properties)*

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# Mycorrhizin A

*K. Ludewig, K. Krohn*

## 1. Name of Compound

Common name
**Mycorrhizin A**

Synonyms

Systematic name
1(S),6(S),9(R)-6-Hydroxy-8,8-dimethyl-4-(1-chloro-prop-enyl)-tricyclo-[4.4.0.0$^{1,9}$]-7-oxa-dec-3-ene-2,5-dione

Substance
Pentaketid

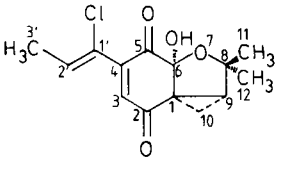
CAS registry number and other numbers
[64356-85-0] BRN 1256313

## 2. Formulas and Molecular Weight

Molecular formular
$C_{14}H_{15}O_4Cl$

Molecular weight
282.72

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>162 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{25} = +3.33°$<br>(1.89 mg/ml, EtOH) [1] | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>methanol, ethanol | |
| Color<br>yellow | Odor<br>odorless | |

4. Occurence

| |
|---|
| Isolated from the fungus Monotropa hypopitys and of Gilmaniella hunucola [1], also obtained from the fungus Peziaila Livida [4] |

5. Health Hazard Data

| Toxicology | Hazard labeling |
|---|---|
| antifungal activity | $LD_{50}$ |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 264 | Molecular ion | 284 [$M^+ + 2$], 282 [$M^+$] |
| Ionization energy | 70 eV | Ion source temp. | 110 °C |
| Acceleration voltage | 3 kV | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | 3 s/decade |
| Spectrometer type and manufacturer | | Finnigan MAT 8430 | |

| IR * | see ref. [1] | | |
|---|---|---|---|
| Characteristic peaks<br>3400 ($OH^-$), 1720 (C=O), 1672 (C=O), 1612 (C=C), 1574, 1384, 1376 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | Perkin Elmer 257 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>1.24 (s, 3H); 1.34 (s, 3H);<br>1.91 (dd, $J = 4.8$ and 8.3 Hz, 1H, 10-H);<br>2.23 (dd, $J = 5.9$ and 8.3 Hz, 1H, 10-H);<br>7.11 (s, 1H, 3-H) ppm | | 1.60 (dd, $J = 4.9$ and 5.8 Hz, 1H, 9-H);<br>2.03 (d, $J = 6.9$ Hz, 3H, 3′-H);<br>7.0 (q, $J = 6.9$ Hz, 1H, 2′-H), | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | $^1H$ decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>14.95 (s, C-10),<br>43.10 (q, C-1),<br>127.26 (q, C-1′),<br>192.24 (q, C-5), | 16.47 (p, C-3′),<br>44.68 (t, C-9),<br>135.73 (t, C-2′),<br>192.56 (q, C-4) ppm | 25.00 (p, C-11/12),<br>82.86 (q, C-8),<br>137.35 (t, C-3), | 29.06 (, C-11/12),<br>101.22 (q, C-6),<br>144.88 (q, C-4), |
| Frequency | 100 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV * | see ref. [1] | | |
|---|---|---|---|
| $I_{max}$ | 246, 305 nm | $\varepsilon_{max}$ | 7900, 1730 |
| Solvent | Ethanol | | |
| Sample conc. | – | Cell thickness | – |
| Spectrometer type and manufacturer | | Bausch & Lomb Spectronic 505 | |

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.7 | | |
| Solvent | $CH_2Cl_2$ | Solvent system | 2% MeOH/98% $CH_2Cl_2$ |
| Saturated atmosphere | – | Detection/Color | UV |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel $60F_{254}$/Merck 5749 |

| | |
|---|---|
| GLC | not available |

| | |
|---|---|
| HPLC | not available |

9. Remarks

Inhibits growth of the tree root rot fungus Fomes annosus.
Inhibits growth of fungus Eurotium repeus [4].

10. References


[1] Trofast, J. et al. *Tetrahedron Lett. 33*, 1977, 875 pp *(isolation, structure, IR, UV, PMR, CMR)*

[2] Chexal, K.K. et al. *Helv. Chim. Acta 62*, 1979, 1129pp *(isolation)*

[3] Bycroft, B.W. Dictionary of Antibiotics and Related Substances *(occurrence)*

[4] Krohn, K., Ludewig K., Aust, H.J., Draeger S., Schulz B. (to be published)

# Narcissin

*A. A. Semenov, A. I. Syrchina*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Narcissin** | |
| Synonyms<br>Narcisoside [2)] | |
| Systematic name<br>3-[6-0-(6-Deoxy-α-L-mannopyranosyl)-≈β-D-glucopyranosyl]oxy-5,7-dihydroxy-2-≈(4-hydroxy-3-methoxyphenyl)-4*H*-1-benzopyran-4-one | |
| Substance<br>Flavonoid | Subgroup<br>Flavonol glycoside |
| CAS registry number and other numbers<br>[604-80-8] | BRN 75621 |

## 2. Formulas and Molecular Weight

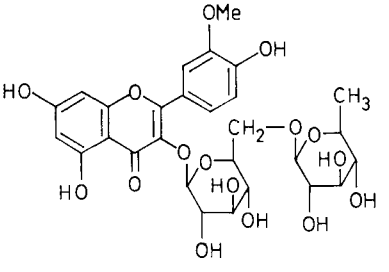
Molecular formular

$C_{28}H_{32}O_{16}$

Molecular weight

624.557

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline | $a_4^{20}$<br>not applicable | Iodine value<br>not applicable |
| Melting Point mp<br>176-178 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20} = +112°$<br>(c = 0.82; methanol) | Saponification value<br>not applicable |
| Flash point<br>not applicable | Soluble in<br>methanol, ethanol | |
| Color<br>yellow | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Salsola spp. [1, 3, 4], other spp. [2, 5, 6] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| unknown | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS * | FAB-Mass spectrum | | |
| Base peak | – | Molecular ion | 647 (M+Na) |
| Ionization energy | – | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | LKB-2091 | |

| | |
|---|---|
| IR | not available |

| | | | |
|---|---|---|---|
| NMR | | Nucleus | $^1H$ |
| Chemical Shifts<br>12.56 (s, OH-5);<br>9.81 (s, OH-4);<br>7.51 (dd, $J = 2.5$, 8.5 Hz, H-6′);<br>6.44 (d, $J = 2$ Hz, H-8);<br>5.44 (d, $J = 7$ Hz, H-1″);<br>3.84 (s, $OCH_3$-3′);<br>0.95 (d, $J = 6$ Hz, $CH_3$-6″) ppm | | 10.91 (s, OH-7);<br>7.86 (d, $J = 2.5$ Hz, H-2′);<br>6.91 (d, $J = 8.5$ Hz, H-5′);<br>6.21 (d, $J = 2$ Hz, H-6);<br>4.42 (s, H-1‴);<br>3.06–3.7 (m, glu, rhe); | |
| Frequency | not given | Solvent | not given |
| Standard | DMSO-$d_6$ | Sample temp. | not given |
| Spectrometer type and manufacturer | | Bruker WP-200sy | |

| NMR | | Nucleus | $^{13}C$ | |
|---|---|---|---|---|
| Chemical Shifts | | | | |
| 177.4 (C-2); | 164.2 (C-7); | 161.4 (C-5); | 156.8 (C-2, C-8); | 149.6 (C-3′); |
| 147.2 (C-4′); | 133.3 (C-3); | 122.6 (C-6′); | 121.4 (C-1′); | 115.6, (C-5′); |
| 113.6 (C-2′); | 104.3 (C-10); | 101.4 (C-1″); | 101.2 (C-1‴); | 99.0 (C-6); |
| 94.1 (C-8); | 76.7 (C-3′); | 76.2 (C-5″); | 72.1 (C-4‴); | |
| 70.9/70.6 (C-2‴, C-3‴); | | 68.6 (C-5‴); | 67.2 (6″); | 56.0 ($OCH_3$); |
| 17.9 (C-6‴) ppm | | | | |
| Frequency | 22.49 MHz | Solvent | DMSO-$d_6$ | |
| Standard | DMSO-$d_6$ | Sample temp. | 28 °C | |
| Spectrometer type and manufacturer | | JEOL FX-90Q | | |

| UV * | additional UV-spectra with shift reagents see ref. [8] | | |
|---|---|---|---|
| $I_{max}$ | 254, 268sh, 358 nm | $\varepsilon_{max}$ | – |
| Solvent | methanol | | |
| Samp. conc. | about $2 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Specord UV-vis | |

8. Chromatographic Data

| TLC | not available |
|---|---|

| GLC | not available |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

10. References


[1] Syrchina, A.I., Vereshchagin, A.L., Larin M.F., Semenov A.A., *Khim. Prir. Soedin 5*, 1989, 725-26 *(occurrence)*

[2] Grigorescu E., Contz, O., *Pharmazie 21(2)*, 1966, 116-120 *(occurrence)*

[3] Thomas, F., Morenilla A., Barberan F.A.T., *Fitoterapia 56(6)*, 1985, p. 365 *(occurrence)*

[4] Mnacakanyan, V.A., Agababian E.Yu., Aratjunian, L.S., *Khim. Prir. Soedin. 5*, 1981, 660 *(occurrence)*

[5] Geissmann, T.A., The Chemistry of Flavonoid Compounds, London, New, York, Paris. 1962. *(name, occurrence)*

[6] Hörhammer, L., *Ber. 99*, 1966, p. 1384 *(occurrence)*

[7] Markham, K.R., Ternai B., Stanley R. et al., *Tetrahedrou 14*, 1978, 1389-1397 *($^{13}C$ NMR)*

[8] Mabry, T.I., Markham, K.R., Thomas M.B., The Systematic Identification of Flavonoids. Springer Verlag, Berlin, Heidelberg, New York, 1970 *(UV-spectroscopy)*

# trans-Nerolidol

*H.-P. Hanssen, W.-R. Abraham*

## 1. Name of Compound

Common name
**trans-Nerolidol**

Synonyms
Melaleucol

Systematic name
3,7,11-Trimethyl-1,6,10-dodecatrien-3-ol

| Substance | Subgroup |
|---|---|
| Terpene | Acyclic sesquiterpene alcohol |

CAS registry number and other numbers

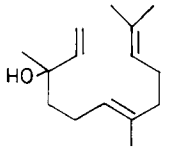
| | |
|---|---|
| racemic [40716-66-3] | BRN 1724138 |
| S-(+) [1119-38-6] | BRN 1724136 |
| R-(−) [77551-75-8] | BRN 4307250 |

## 2. Formulas and Molecular Weight

Molecular formular
$C_{15}H_{26}O$

Molecular weight
222.1984

Structural formula




## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| liquid | 0.8783 g/cm$^3$ | not available |
| **Melting Point mp** | $n_D^{20}$ | **Acid value** |
| −75 °C (rac. mixture) | 1.4801 | not applicable |
| **Boiling point bp** | $[\alpha]_D^{20}$ | **Saponification value** |
| 78 °C (0,15 mm Hg) | +14.2° ($CHCl_3$) | not applicable |
| **Flash point** | **Soluble in** | |
| 96 °C | hexane, methylene chloride, methanol | |
| **Color** | **Odor** | |
| colorless | weakly floral | |

4. Occurrence

| |
|---|
| Widespread in yeasts/fungi and higher plants, main constituent (80%) of Cabreuva (Myrocarpus fastigiatus, M. frondosus; Fabales) oil |

5. Health Hazard Data

| Toxicology | Hazard labeling |
|---|---|
| no data available | $LD_{50}$ not available |
| Waste disposal procedures<br>By combustion; according to Merck instructions, collect laboratory waste materials together with other relatively unreactive, halogen-free organic substances | |

6. Transportation and Storage Instructions

| |
|---|
| GGVE/GGVS: 3/32c; RID 3/32c; ADR 3/32 c |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 69 | Molecular ion | – |
| Ionization energy | 70 eV | Ion source temp. | 200 °C |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | GC-MS coupling: GC: Hewlett-Packard 5840 A;<br>MS: Hewlett-Packard-Quadrupol 5985 A | | |

| IR | see attached spectrum | | |
|---|---|---|---|
| Sample preparation | 2% in $CCl_4$ | Resolution | – |
| Spectrometer type and manufacturer | | Perkin Elmer PE 841 | |

| NMR | | Nucleus | $^1H$-$^1H$ correlation |
|---|---|---|---|
| Chemical Shifts<br>H-1: 5.06 ppm dd, J = 10.7 Hz (H-2), 1.2 Hz (H-1′);<br>H-1′: 5.22 ppm dd, J = 17.0 Hz (H-2), 1.2 Hz (H-1);<br>H-2: 5.92 ppm dd, J = 17.0 Hz (H-1′), 10.7 (H-1);<br>H-4: 1.59 ppm m; H-5: 2.03 ppm m; H-6: 5.14 ppm t, J = 7.0 Hz (H-5);<br>H-8: 2.00 ppm m; H-9: 2.05 ppm m; H-10: 5.07 ppm t, J = 7.0 Hz (H-9);<br>H-12: 1.69 ppm br s; H-13, H-14: 1.61 ppm br s; H-15: 1.29 ppm s | | | |
| Frequency | 300 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | EM 300 Bruker | |

| NMR | | Nucleus | $^1H$-$^{13}C$ correlation |
|---|---|---|---|
| Chemical Shifts<br>C-1: 111.7; C-2: 145.2; C-3: 73.5; C-4: 42.2; C-5: 22.8;<br>C-6: 124.3; C-7: 135.6; C-8: 39.7; C-9: 26.7; C-10: 124.3;<br>C-11: 131.4; C-12: 25.7; C-13: 17.7; C-14: 16.0; C-15: 27.9 ppm | | | |
| Frequency | 300 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | EM 300 Bruker | |

| UV | not applicable |
|---|---|

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.55 | | |
| Solvent | – | Solvent system | n-hexane/EtOAc 3 : 2 |
| Saturated atmosphere | 20–25 °C | Detection/Color | anisaldehyde/$H_2SO_4$<br>develop 1 min at 140 °C:<br>violet |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel 60 $F_{254}$, 0.25 mm |

| GLC | | | |
|---|---|---|---|
| Retention time | 27.65 min | | |
| Column type | quartz-capillary column | Column length | 22 m<br>(int. diameter 0.4 mm) |
| Column packing | WG 11 (FFAP) | Column temp. | isothermic 150 °C |
| Injector port temp. | 210 °C | Carrier gas | $N_2$ at 1 ml/min |
| Detector temp. | 210 °C | Sample solvent | pentane |
| Sample size | 1.0 µl | Sample conc. | about 0.01 % |
| Chromatograph type and manufacturer | | Perkin-Elmer PE F22 with FID | |

| HPLC | not available |
|---|---|

9. Remarks

10. References


[1] Structure: Hesse, A., Zeitschel, O., *J. pr. Chem. 66* (1902), 481

[2] Synthesis: Ruzicka, L., *Helv. Chim. Acta 6* (1923), 483 and 492

[3] Occurrence: Gildemeister, E., Hoffmann, F., Die Ätherischen Öle, Vol. IIIB, Akademie Verlag, Berlin, 1962, pp. 239-244
Cinnamomum platyphyllum: Huang, Y., Wen, M., Xiao, S., Zhao, H., Ren, W., *Yunnan Zhiwu Yanjiu 8* (1986), 359-362
C. sieboldii: Fujita, S., *Yakugaku Zasshi 106* (1986), 17-21
Citrus bergamia: Huang, Y., Wen, M., Xiao, S, Zhao, H., Ren, W., Chen, Q. Liu, X., Gui, T., *Yunnan Zhiwu Yanjiu 8* (1986), 471-476
Comptonia peregrina: Collin, G.J., Hachey, J.M., Simard, S., Vernin, G., Fraisse, D., *Flavour Fragrance J. 3* (1988), 65-68
Elleteria cardamomum: Pieribattesti, J.C., Smadja, J., Mondon, J.M., *Dev. Food Sci. 18* (1988), 697-706
Epimedium grandiflorum var. thunbergianum: Miyase, T., Ueno, A., Takizawa, N., Kobayashi, H., Karasawa, H., *Chem. Pharm. Bull. 35* (1987) 1109-1117
Heracleum dissectum: Montanarella, L., Bos, R., Fischer, F.C., *Planta med.* (1986), 332-334
Matricaria chamomilla: Graciela Malinskas, G.A., Santi, M.N., Retamar, J.A., *Essenze Deriv. Agrum. 55* (1985), 52-61
Piper guineense: Ekundayo, O., Laakso, I., Adegbola, R.M., Oguntimein, B., Sofowora, A., Hiltunen, R., *J. Agric. Food Chem. 36* (1988), 880-882
Ribes nigrum: Marriott, R.J., *Dev. Food Chem. 36* (1988), 387-403
Schisandra chinensis: Qin, B., Tian, Z., Lou, Z., *Yaoxue Tongbao 23* (1988), 338-339
Srobilanthes auriculatus: Weyerstahl, P., Marschall-Weyerstahl, H., Manteuffel, E., Kaul, V.K., *Dev. Food Sci. 18* (1988), 567-571
Zingiber officinale: Ekundayo, O., Laakso, I., Hiltunen, R., *Flavour Fragrance J. 3* (1988), 85-90
Yeasts/Fungi: Klingenberg, A., Sprecher, E., *Planta med.* (1985), 264-265
Berger, R.G., Neuhäuser, K., Drawert, F., *Flavour Fragrance J. 1* (1986), 181-185
Hanssen, H.-P., Abraham, W.-R., *Z. Naturforsch. 41c* (1986), 959-962

# 9,12-Octadecadien-6-ynoic acid

*P. Beutelmann, G. Kohn*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**9,12-Octadecadien-6-ynoic acid** | |
| Synonyms | |
| Systematic name<br>9,12-Octadecadien-6-ynoic acid | |
| Substance<br>Fatty acid | Subgroup<br>Acetylenic fatty acid |
| CAS registry number and other numbers<br>[56795-52-9] | BRN 1912699 |

## 2. Formulas and Molecular Weight

| | |
|---|---|
| Molecular formular<br>$C_{18}H_{28}O_2$ | Structural formula<br>$HOOC-(CH_2)_4-C\equiv C-CH_2-CH=CH-CH_2-CH=CH-(CH_2)_4-CH_3$ |
| Molecular weight<br>276.41 | |

## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>liquid | $d_4^{20}$<br>unknown | Iodine value<br>unknown |
| Melting Point mp<br>unknown | $n_D^{20}$<br>unknown | Acid value<br>unknown |
| Boiling point bp<br>unknown | $[\alpha]_D^{30}$<br>unknown | Saponification value<br>not applicable |
| Flash point<br>unknown | Soluble in<br>hexane, diethylether, chloroform | |
| Color<br>unknown | Odor<br>unknown | |

4. Occurrence

| |
|---|
| In several moss and liverwort species [1, 2] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| unknown | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage in chloroform solution, below 0 °C |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS | GC-MS as methylester | | |
| Base peak | 91 | Molecular ion | not detectable |
| Ionization energy | 70 eV | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | Finnigan 3200 | |

| | |
|---|---|
| IR | not available |

| | |
|---|---|
| NMR | not available |

| | |
|---|---|
| UV | not available |

8. Chromatographic Data

| | | | |
|---|---|---|---|
| TLC | only as glycerol triester (see remarks) | | |
| Rf Value | 0.40 | | |
| Solvent | – | Solvent system | n-heptane, diethylether, acetic acid (75 : 25 : 4) |
| Saturated atmosphere | yes | Detection/Color | iodine vapor/brown |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel 60 |

| GLC | as methylester | | |
|---|---|---|---|
| Retention time | 9.99 min | | |
| Column type | WCOT, 0.32 mm ID | Column length | 50 m |
| Column packing | CP Sil 88 Chrompack | Column temp. | 200 °C |
| Injector port temp. | 250 °C | Carrier gas | $N_2$ |
| Detector temp. | 250 °C | Sample solvent | petroleum ether (bp 140 °C) |
| Sample size | 1 µl | Sample conc. | 50–100 µmol/ml |
| Chromatograph type and manufacturer | | Dani 3800 | |

| HPLC | | | |
|---|---|---|---|
| Retention time | 7.0 min | | |
| Column | 4 × 250 mm | Stationary phase | Lichrospher RP 18 5 µ |
| Mobile phase | Acetonitril/MeOH/$H_2O$ (28/64/8) | Flow rate | 1 ml/min |
| Column temp. | 25 °C | Pressure | 55 bar |
| Detector | UV 205 nm | Sample solvent | Acetonitril |
| Sample size | 20 µl | Sample conc. | $10^{-4}$ mol/l |
| Chromatograph type and manufacturer | | KONTRON 420 | |

## 9. Remarks

In the plant material the substance is found as fatty acid component of the trigyceride fraction

## 10. References


[1] Kohn, G., Vierengel, A., Vandekerkhove, O., Hartmann E. *Phytochemistry 26*, 1987, pp. 2100–02 *(occurrence, analytic data)*

[2] Kohn, G., Vandekerkhove, O., Hartmann E., Beutelmann, P. *Phytochemistry 27*, 1988, pp. 1049–51 *(occurrence)*

[3] Kohn, G., Demmerle, S., Vandekerkhove, O., Hartmann E. *Phytochemistry 26*, 1987, pp. 2271–75 *(occurrence)*

[4] Anderson, W.H., Gellerman, J.L., Schlenk, H. *Lipids 10*, 1975, 501–2 *(occurrence)*

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# 9,12,15-Octadecatrien-6-ynoic acid

*P. Beutelmann, G. Kohn*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**9,12,15-Octadecatrien-6-ynoic acid** | |
| Synonyms | |
| Systematic name<br>9,12,15-Octadecatrien-6-ynoic acid | |
| Substance<br>Fatty acid | Subgroup<br>Acetylenic fatty acid |
| CAS registry number and other numbers<br>[53766-42-0] | BRN 4432637 |

## 2. Formulas and Molecular Weight

| | |
|---|---|
| Molecular formular<br>$C_{18}H_{26}O_2$ | Structural formula<br>$HOOC-(CH_2)_4-C\equiv C-CH_2-CH=CH-CH_2-CH=CH-CH_2-CH=CH-CH_2-CH_3$ |
| Molecular weight<br>274.39 | |

## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>liquid | $d_4^{20}$<br>unknown | Iodine value<br>unknown |
| Melting Point mp<br>unknown | $n_D^{20}$<br>unknown | Acid value<br>unknown |
| Boiling point bp<br>unknown | $[\alpha]_D^{30}$<br>unknown | Saponification value<br>not applicable |
| Flash point<br>unknown | Soluble in<br>hexane, diethylether, chloroform | |
| Color<br>unknown | Odor<br>unknown | |

4. Occurrence

| |
|---|
| In several moss and liverwort species [1, 2] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| unknown | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage in chloroform solution, below 0 °C |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS | GC-MS as methylester | | |
| Base peak | 91 | Molecular ion | not detectable |
| Ionization energy | 70 eV | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | Finnigan 3200 | |

| | |
|---|---|
| IR | not available |

| | | | |
|---|---|---|---|
| NMR | as methyl ester | Nucleus | $^1H$ |
| Chemical Shifts<br>5.43–5.27 (6H, m, C-9,-10,-12,-13,-15,-16);<br>2.90 (2H, m, C-8);<br>2.31 (2H, t, J = 7.5 Hz, C-2);<br>2.05 (2H, m, C-17);<br>1.49 (2H, m, C-4); | | 3.65 (3H, s, Me at C-1);<br>2.80 (4H, m, C-11, 14);<br>2.15 (2H, tt, J = 2.5, 7.1 Hz, C-5);<br>1.70 (2H, m, C-3);<br>0.95 (3H, t, J = 7.5 Hz, C-18) ppm | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | as methyl ester | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>179.3 (C-1);<br>33.6 (C-2);<br>20.6 (C-17); | 132.1-125.4 (C-9,-10,-12,-13,-15,-16);<br>28.4 (C-4); 25.5 (C-11, 14);<br>18.5 (C-5); 17.2 (C-8); | | 51.4 (Me at C-1);<br>24.2 (C-3);<br>14.2 (C-18) ppm |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV not available |
|---|

8. Chromatographic Data

| TLC | only as glycerol triester (see remarks) | | |
|---|---|---|---|
| Rf Value | 0.40 | | |
| Solvent | – | Solvent system | n-heptane, diethylether, acetic acid (75 : 25 : 4) |
| Saturated atmosphere | yes | Detection/Color | iodine vapor/brown |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel 60 |

| GLC | as methylester | | |
|---|---|---|---|
| Retention time | 12.08 min | | |
| Column type | WCOT, 0.32 mm ID | Column length | 50 m |
| Column packing | CP Sil 88 Chrompack | Column temp. | 200 °C |
| Injector port temp. | 250 °C | Carrier gas | $N_2$ |
| Detector temp. | 250 °C | Sample solvent | petroleum ether (bp 140 °C) |
| Sample size | 1 µl | Sample conc | 50-100 µmol/ml |
| Chromatograph type and manufacturer | | Dani 3800 | |

| HPLC | | | |
|---|---|---|---|
| Retention time | 6.0 min | | |
| Column | 4 × 250 mm | Stationary phase | Lichrospher RP 18 5 μ |
| Mobile phase | Acetonitril/MeOH/$H_2O$ (28/64/8) | Flow rate | 1 ml/min |
| Column temp. | 25 °C | Pressure | 55 bar |
| Detector | UV 205 nm | Sample solvent | Acetonitril |
| Sample size | 20 μl | Sample conc. | $10^{-4}$ mol/l |
| Chromatograph type and manufacturer | | KONTRON 420 | |

9. Remarks

In the plant material the substance is found as fatty acid component of the trigyceride fraction

10. References


[1] Kohn, G., Vierengel, A., Vandekerkhove, O., Hartmann E., *Phytochemistry 26*, 1987, pp. 2100-02 *(occurrence, analytic data)*

[2] Kohn, G., Vandekerkhove, O., Hartmann E., Beutelmann, P. *Phytochemistry 27*, 1988, pp. 1049-51 *(occurrence)*

[3] Kohn, G., Demmerle, S., Vandekerkhove, O., Hartmann E., *Phytochemistry 26*, 1987, pp. 2271-75 *(occurrence)*

[4] Anderson, W.H. et al.: *Lipids 9*, 1974, 506-11 *(occurrence)*

# 9-Octadecen-6-ynoic acid

*P. Beutelmann, G. Kohn*

## 1. Name of Compound

| |
|---|
| Common name<br>**9-Octadecen-6-ynoic acid** |
| Synonyms |
| Systematic name<br>9-Octadecen-6-ynoic acid |
| Substance: Fatty acid<br>Subgroup: Acetylenic fatty acid |
| CAS registry number and other numbers<br>[115374-25-9] |

## 2. Formulas and Molecular Weight

| | |
|---|---|
| Molecular formular<br>$C_{18}H_{30}O_2$<br>Molecular weight<br>278.42 | Structural formula<br>$HOOC-(CH_2)_4-C\equiv C-CH_2-CH=CH-(CH_2)_7-CH_3$ |

## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>liquid | $d_4^{20}$<br>unknown | Iodine value<br>unknown |
| Melting Point mp<br>unknown | $n_D^{20}$<br>unknown | Acid value<br>unknown |
| Boiling point bp<br>unknown | $[\alpha]_D^{30}$<br>unknown | Saponification value<br>not applicable |
| Flash point<br>unknown | Soluble in<br>hexane, diethylether, chloroform | |
| Color<br>unknown | Odor<br>unknown | |

4. Occurrence

| |
|---|
| In several species of the genus Riccia (Hepaticae) [1, 2] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| unknown | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage in chloroform solution, below 0 °C |

7. Spectroscopic Data

| MS | GC-MS as methylester | | |
|---|---|---|---|
| Base peak | 91 | Molecular ion | $[M^+]$ 292 |
| Ionization energy | 70 eV | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | Finnigan 3200 | |

| IR | as methylester | | |
|---|---|---|---|
| Characteristic peaks<br>2910 ($CH_2$), 2840 (O-Me), 2310 (C≡C), 1740 (C=O), 1640 (C=C) $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | 5 $cm^{-1}$ |
| Spectrometer type and manufacturer | | Perkin-Elmer 299 | |

| NMR | as methylester | Nucleus | $^{1}H$ |
|---|---|---|---|
| Chemical Shifts<br>5.44 (m, 1H, C-1); 5.36 (dd, J = 11.6 Hz, 1H, CH-9);<br>3.65 (s, 3H, Me at C-1); 2.87 (m, 2H, H-8);<br>2.31 (t, J = 7.5 Hz, 2H, C-2); 2.16 (tt, J = 2.5, 7.0 Hz, 2H, C-5);<br>2.00 (q, J = 6.7 Hz, 2H); 1.70 (m, 2H, C-3);<br>1.49 (m, 2H, C-4); 1.24 (m, 12H, C-12 to C-17);<br>0.86 (t, J = 6.8 Hz, 3H, C-18) ppm | | | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | | | Nucleus | $^{13}C$ |
|---|---|---|---|---|
| Chemical Shifts<br>173.9 (C-1); 131.4 (C-9); 125.0 (C-10); 79.3 (C-6); 79.0 (C-7);<br>51.4 (Me at C-1); 33.7 (C-2); 31.9 (C-16); 29.5 (C-13); 29.4 (C-10);<br>29.3 (C-12, C-15); 28.5 (C-4); 27.2 (C-11); 24.2 (C-3); 22.7 (C-17);<br>18.5 (C-5); 17.2 (C-8); 14.0 (C-18) ppm | | | | |
| Frequency | 400 MHz | | Solvent | $CDCl_3$ |
| Standard | TMS | | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | | Bruker AM 400 | |

| UV | not available |
|---|---|

## 8. Chromatographic Data

| TLC | only as glycerol triester (see remarks) | | |
|---|---|---|---|
| Rf Value | 0.45 | | |
| Solvent system | n-heptane, diethylether, acetic acid (75 : 25 : 4) | | |
| Saturated atmosphere | yes | Detection/Color | iodine vapor/brown |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel 60 |

| GLC | as methylester | | |
|---|---|---|---|
| Retention time | 8.62 min | | |
| Column type | WCOT, 0.32 mm ID | Column length | 50 m |
| Column packing | CP Sil 88 Chrompack | Column temp. | 200 °C |
| Injector port temp. | 250 °C | Carrier gas | $N_2$ |
| Detector temp. | 250 °C | Sample solvent | petroleum ether (bp 140 °C) |
| Sample size | 1 µl | Sample conc | 50–100 µmol/ml |
| Chromatograph type and manufacturer | | Dani 3800 | |

| HPLC | not available |
|---|---|

9. Remarks

In the plant material the substance is found as fatty acid component of the trigyceride fraction, and in smaller amounts, of the glyco-lipid fraction.

10. References


[1] Kohn, G., Vierengel, A., Vandekerkhove, O., Hartmann E., *Phytochemistry 26*, 1987, pp. 2100–02 *(occurrence, analytic data)*

[2] Kohn, G., Vandekerkhove, O., Hartmann E., Beutelmann, P., *Phytochemistry 27*, 1988, pp. 1049–51 *(occurrence)*

# Oosporein

*C. Franke, K. Krohn*

## 1. Name of Compound

| |
|---|
| Common name<br>**Oosporein** |
| Synonyms<br>Chaetomidin, Iso-oosporein |
| Systematic name<br>3,3′,6,6′-Tetrahydroxy-5,5′-dimethyl-2,2′-bi-p-benzoquinone |
| Substance<br>Quinone |
| CAS registry number and other numbers<br>[475-54-7] BRN 1892735 |

## 2. Formulas and Molecular Weight

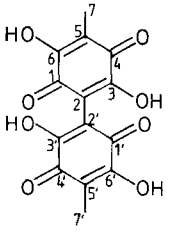
Molecular formular

$C_{14}H_{10}O_8$

Molecular weight

306.2

Structural formula



## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>decomp. at 220 °C | $n_D^{20}$<br>not applicable | Acid value<br>not available |
| Boiling point bp<br>not available | $[\alpha]_D^{20}$<br>not applicable | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>pyridine, dioxan, DMSO, methanol | |
| Color<br>pH 1: yellow<br>pH 4: red<br>pH 5: violet<br>pH 7: blue | Odor<br>odorless | |

4. Occurrence

| |
|---|
| isolated from Chaetomium aureum [1], Chaetomium trilaterale [2], Verticillium psalliotae [2], Acremonium sp. [2], Oospora colorans [3], Metarrhizium flavoviride |

5. Health Hazard Data

| Toxicology | Hazard labeling |
|---|---|
| growth inhibitory and phytotoxic effects in plants [4] | $LD_{50}$ (cockerel) 6.12 mg/kg oral [4] |
| Waste disposal procedures | |

6. Transportation and Storage Instructions

| |
|---|
| |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 306 | Molecular ion | 306 |
| Ionization energy | 70 eV | Ion source temp. | 135 °C |
| Acceleration voltage | 3 kV | Emission current | 1 mA |
| Resolution | – | Scan rate | 3 s/decade |
| Spectrometer type and manufacturer | | Finnigan MAT 8430 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3320, 1650, 1390, 1340, 1305, 1245, 1075, 920, 770, 715 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | 2.5 $cm^{-1}$ |
| Spectrometer type and manufacturer | | Perkin-Elmer 1420 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>1.80 (s, 6H, $CH_3$) ppm | | | |
| Frequency | 400 MHz | Solvent | $CD_3OD$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | $^{1}H$ decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>7.2 (q; C-7, C-7′); 108.6 (s; C-5, C-5′); 112.4 (s, C-2, C-2′);<br>170.2, 170.4 (4s, C-1, C-1′, C-3, C-3′, C-4, C-4′, C-6, C-6′) ppm | | | |
| Frequency | 50 MHz | Solvent | $d_5$-pyridine |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AC 200 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 208, 289 nm | $\varepsilon_{max}$ | 16520, 24831 |
| Solvent | methanol | | |
| Sample conc. | $3.4 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Beckman UV 5230 | |

8. Chromatographic Data

| TLC | not available |
|---|---|

| GLC | not available |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

Sublimation: 160 °C, 0.1 mm

10. References


[1] Lloyd, G., Robertson, A., Sankey, G.B., Whalley, W.B., *J. Chem. Soc.*, 1955, 2163-2165 *(isolation, derivation)*

[2] Smith, J., Thomson, R.H., *Tetrahedron 10*, 1960148-152 *(isolation, IR)*

[3] Kögl, F., Van Wessem; G.C., *Recls. Trav. Chim. Pays-Bas 63*, 1944, 5-12 *(synthesis, isolation, UV, derivation)*

[4] Cole, R.J., Kirksey, J.W., Cutler, H.G., Davis, E.E., *J. Agric. Food Chem. 22*, 1974, 517-520 *(isolation, toxicology)*

[5] Steiner, E. et al., *Helv. Chim. Acta 57*, 1974, 2377 pp *(biosynthesis)*

[6] Kalamar et al., *Helv. Chim. Acta 57*, 1974, 2368 pp *(synthesis)*

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# Patulin

*K. Ludewig, K. Krohn*

## 1. Name of Compound

Common name

**Patulin**

Synonyms

Clavacin, Clavatin, Claviformin, Penicidin, Expansin, Mycoin $C_3$

Systematic name

4-Hydroxy-4H-furo-[3,2-c]pyran-2[6H]-one

Substance

Tetraketid

CAS registry number and other numbers

[149-29-1] BRN 149675 Merck Index *10*, 6914
Merck Index *11*, 7002

## 2. Formulas and Molecular Weight

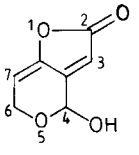
Molecular formular

$C_7H_6O_4$

Molecular weight

154.122

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>111 °C | $n_D^{20}$<br>not applicable | Acid value<br>not available |
| Boiling point bp<br>not available | $[\alpha]_D^{21} = -6.2$<br>(6.4 mmol/l in $CHCl_3$) [8] | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>methanol, ether, acetone, chloroform | |
| Color<br>colorless | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Antibiotic produced by several fungi, e.g. Aspergillus clavatus, Aspergillus terreus, Penicillium patulum [1]. |

5. Health Hazard Data

| Toxicology | Hazard labeling | |
|---|---|---|
| Antibiotic, very toxic | $LD_{50}$ | 35 mg/kg (rat, oral) [2] |
| Waste disposal procedures<br>Combustion with a flammable solvent [4] | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage temperature 0 °C [4] |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | m/z 55 | Molecular ion | m/z 154 |
| Ionization energy | 70 eV | Ion source temp. | room temperature |
| Acceleration voltage | 3 kV | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | 3 s/decade |
| Spectrometer type and manufacturer | | Finnigan MAT 8430 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3330–3430, 1780, 1740, 1680, 1410, 1220, 1180, 1100, 1070, 1000, 880, 790 $cm^{-1}$ | | | |
| Sample preparation | liquid film | Resolution | – |
| Spectrometer type and manufacturer | | Perkin Elmer 1420 | |

| NMR | see ref. [3] | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>3.86 (s, 1H, OH);<br>4.72 (dd, $J$ = 17.0 and 2.9 Hz, 1H, $H_2C$-6);<br>6.02 (d, $J$ = 0.9 Hz, 1H, HC = -3); | | 4.44 (dd, $J$ = 17.0 and 2.9 Hz, 1H, $H_2C$-6);<br>5.95 (m, 1H, HC = -7);<br>6.06 (s, 1H, HC-4) ppm | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | $^1H$ decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>59.55 (t, C-6); 88.84 (d, C-7); 107.79 (d, C-3);<br>111.1 (d, C-4); 146.25 (s, C-3a); 150.1 (s, C-7a), 168.88 (s, C-2) ppm | | | |
| Frequency | 100 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV * | see ref. [8] | | |
|---|---|---|---|
| $I_{max}$ | 276 nm | $\varepsilon_{max}$ | 1436 ± 4 |
| Solvent | methanol | | |
| Sample conc. | 0.449 mmol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | | |

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.6 | | |
| Solvent | $CH_2Cl_2$ | Solvent system | 4% $CH_2Cl_2$/96% MeOH |
| Saturated atmosphere | – | Detection/Color | UV |
| Plate manufacturer | Merck, Darmstadt | Plate type/Product no. | Kieselgel $60F_{254}$, Nr. 5749 |

| | |
|---|---|
| GLC | see ref. [6] and [6a] |

| | |
|---|---|
| HPLC | for application see ref. [7] |

9. Remarks

Occurrence in fruit-juice and rot fruits.

10. References


[1] Bycroft B.W., Dictionary of Antibiotics and Related Substances *(occurrence)*

[2] Belitz H.-D., Grosch W., Lehrbuch der Lebensmittelchemie, Springer 1982 *($LD_{50}$)*

[3] Hesse M.; Meier H.; Zeeh B., Spektroskopische Methoden in der organischen Chemie, Thieme, 1987, 3. Ed. *(information for spectral data)*

[4] SIGMA Chemie GmbH, Biochemikalien und organische Verbindungen für die Forschung und Diagnostika 1990

[5] Birkinshaw, H.J. et al., *Lancet* (1943) *245*, 625 *(isolation, structure)*

[6] Ogawa Hitoshi et al., Kenkyu Nenpo-Tokyo-tontsu Eisei Kenkyusho (1984),*35*, 224-230 *(GLC)*

[6a] Abstract 1985 102:219714u *(GLC)*

[7] Engstrom G.W. et al., *J. Agric. Food Chem.* 1977 *(25,4)* 8336 pp *(HPLC)*

[8] Engstrom G.W. et al., *Pure Appl. Chem.* (1982) *54*, 2220 *(UV)*

# Peimine

*Wang Zhongdong*

## 1. Name of Compound

Common name

**Peimine**

Synonyms

Verticine

Systematic name

5α, 14α-Cevanine-3β,6α,20β-triol

| Substance | Subgroup |
|---|---|
| Alkaloid | Steroidal alkaloid |

CAS registry number and other numbers

[23496-41-5] BRN 93323

## 2. Formulas and Molecular Weight

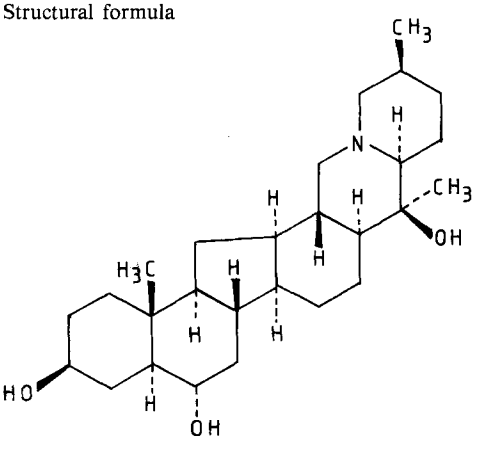
Molecular formular

$C_{27}H_{45}NO_3$

Molecular weight

431.64

Structural formula



## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>needle crystalline | $d_4^{20}$<br>not available | Iodine value<br>not applicable |
| Melting Point mp<br>223-224 °C | $n_D^{20}$<br>not available | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{16}$:<br>−19.4° (EtOH, c=0.55)<br>−20.0° ($CHCl_3$, c=0.69) | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>methanol, chloroform, ethyl acetate | |
| Color<br>colorless | Odor<br>not available | |

## 4. Occurrence

In spp. of Fritillaria, Liliaceae; such as F. verticillata Willd. var. thunbergii bak., F. imperialis, L. var. rubra Maxima & F. roylei Hook etc.

## 5. Health Hazard Data

| | |
|---|---|
| Toxicology | Hazard labeling |
| | $LD_{50}$ 9 mg/kg |
| Waste disposal procedures | |

## 6. Transportation and Storage Instructions

## 7. Spectroscopic Data

MS not available

IR

Characteristic peaks
3350, 2920, 2880, 2750, 1467, 1446, 1372, 1340, 1318, 1307, 1285, 1255, 1243, 1215, 1160, 1130, 1052, 1032, 966, 936, 900, 877 $cm^{-1}$

| | |
|---|---|
| Sample preparation KBr | Resolution – |
| Spectrometer type and manufacturer | IR-27G (Shimadzu, Japan) |

| NMR | | Nucleus | $^1H$-$^1H$-correlation |
|---|---|---|---|
| Chemical Shifts<br>4.27, 3.76, 3.68, 2.62, 1.55, 1.32, 1.26, 1.24, 1.15, 1.05, 0.88, 0.82, 0.74 ppm | | | |
| Frequency | 80 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | – |
| Spectrometer type and manufacturer | | AC-80 (Bruker, Switzerland) | |

| UV * | | | |
|---|---|---|---|
| $\lambda_{max}$ | 285, 260 nm | $\varepsilon_{max}$ | 1.350, 12.250 |
| Solvent | methanol | | |
| Sample conc. | – | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Hitachi 200-10 (Japan) | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | about 0.85 | | |
| Solvent | absolute alcohol | Solvent system | EtOAc/MeOH/$NH_4OH$<br>(17 : 2 : 1) |
| Saturated atmosphere | – | Detection/Color | Dragendorff's reagent |
| Plate manufacturer | hand made | Plate type/Product no. | Silica gel-G (China) |

| GLC | not available |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

| |
|---|

10. References


[1] Yamaguchi, K.: Spectral Data of Natural Products *1*, 1970, 711

[2] Wu Ji-zhou: Zhoucaoyao (Chinese Herb Medicine) *13*(8), 1982, 339

[3] CA 1978, 89:20324z

[4] CA 1961, 55:8449f

[5] Chu Tze-tsin, Loh Jen-yung: Acta Chimica Sinica *21*(3), 1955, 227

[6] Chu Tze-tsin: Bulletin of Chemistry *4*, 1979, 302

# Peiminine

*Wang Zhongdong*

## 1. Name of Compound

Common name

**Peiminine**

Synonyms

Fritillarine

Systematic name

5α,14α-Cevanine-3β,20β-dihydroxy-6-one

| Substance | Subgroup |
|---|---|
| Alkaloid | Steroidal alkaloid |

CAS registry number and other numbers

[18059-10-4] BRN 96785

## 2. Formulas and Molecular Weight

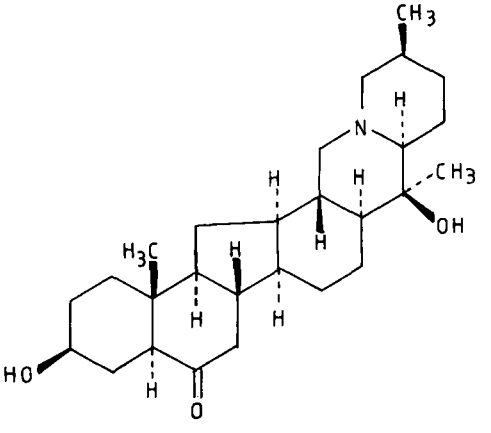
Molecular formular

$C_{27}H_{43}NO_3$

Molecular weight

429.62

Structural formula



## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>powder (amorphous) | $d_4^{20}$<br>not available | Iodine value<br>not applicable |
| Melting Point mp<br>212-213 °C | $n_D^{20}$<br>not available | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^9$:<br>-73.8° (EtOH, c=0.21) | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>methanol, ethanol, acetone, chloroform | |
| Color<br>colorless | Odor<br>not available | |

## 4. Occurrence

| |
|---|
| In Fritillaria thunbergii Miq., Liliaceae |

## 5. Health Hazard Data

| | |
|---|---|
| Toxicology | Hazard labeling |
| | $LD_{50}$ not available |
| Waste disposal procedures | |

## 6. Transportation and Storage Instructions

| |
|---|
| |

## 7. Spectroscopic Data

| |
|---|
| MS not available |

| | |
|---|---|
| IR | |
| Characteristic peaks<br>3350, 2920, 2750, 1705, 1467, 1446, 1372, 1340, 1318, 1307,1285,<br>1255-1243, 1215, 1160, 1130, 1052, 1032, 966, 936, 900 $cm^{-1}$ | |
| Sample preparation KBr | Resolution – |
| Spectrometer type and manufacturer | IR-27G (Shimadzu, Japan) |

| NMR | | Nucleus | $^1H$-$^1H$-correlation |
|---|---|---|---|
| Chemical Shifts<br>4.27, 2.62, 2.04, 1.77, 1.60, 1.51, 1.39, 1.26, 1.06, 0.95, 0.79, 0.74 ppm | | | |
| Frequency | 80 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | |
| Spectrometer type and manufacturer | | AC-80 (Bruker, Switzerland) | |

| UV * | | | |
|---|---|---|---|
| $I_{max}$ | 288 nm | $\varepsilon_{max}$ | 1.870 |
| Solvent | ethanol | | |
| Sample conc. | – | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | UV-200 (Shimadzu, Japan) | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | about 0.6 | | |
| Solvent | absolute alcohol | Solvent system | EtOAc/MeOH/$NH_4OH$<br>(17 : 2 : 1) |
| Saturated atmosphere | – | Detection/Color | Dragendorff's reagent |
| Plate manufacturer | hand made | Plate type/Product no. | Silica gel-G (China) |

| GLC | not available |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

10. References


[1] Wu Ji-zhou: Zhoucaoyao (Chinese Herb Medicine) *13*(8), 1982, 339

[2] Lin Quishou: Chinese Herb Medicinal Chemistry, Beijing 1977, 813

[3] Li Quinghua, Wu Zonghao: Acta Pharmaceutica Sinica *21*(8), 1986, 767

[4] CA 1981, 95:30629y

[5] Chu Tze-tsin, Loh Jen-yung: Acta Chimica Sinica *21*(3), 1955, 227

[6] Chu Tze-tsin: Bulletin of Chemistry *4*, 1979, 302

# Penicillic acid

*K. Ludewig, K. Krohn*

## 1. Name of Compound

| |
|---|
| Common name<br>**Penicillic acid** |
| Synonyms |
| Systematic name<br>3-Methoxy-5-methyl-4-oxo-2,5-hexadiene |
| Substance<br>Tetraketid |
| CAS registry number and other numbers<br>[90-65-3] BRN 1773464 Merck Index *11*, 7033 |

## 2. Formulas and Molecular Weight

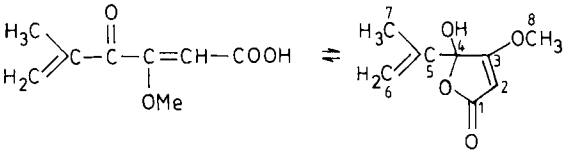
Molecular formular

$C_8H_{10}O_4$

Molecular weight

170.16

Structural formula



## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>64-65 °C (hydrat)<br>87 °C (anhyd.) | $n_D^{20}$<br>not applicable | Acid value<br>$pK_a = 5.9$ |
| Boiling point bp<br>not available | $[\alpha]_D^{20}$<br>not applicable | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>ether, acetone, methanol, chloroform | |
| Color<br>colorless | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Toxic metabolism of various Penicillium and Aspergillus spp. |

5. Health Hazard Data

| Toxicology | Hazard labeling | |
|---|---|---|
| very toxic | $LD_{50}$ | 70 mg/kg (mice i.p.) [7] |
| Waste disposal procedures<br>Combustion with a flammable solvent | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage temperature 0 °C |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 69 | Molecular ion | 178 |
| Ionization energy | 70 eV | Ion source temp. | 200 °C |
| Acceleration voltage | 3 kV | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | 3 s/decade |
| Spectrometer type and manufacturer | | Finnigan MAT 8430 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3300, 2950, 1730–1750, 1640, 1460, 1350, 1280, 1230, 1170, 1060, 1040, 1030, 910, 810, 780 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | Perkin Elmer 1420 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>1.77 (s, 3H, $H_3C$-7); 3.92 (s, 3H, $H_3CO$-8); 5.12 (s, 1H, H-2);<br>5.21 (s, 1H, $H_2C$-6); 5.48 (s, 1H, $H_2C$-6) ppm | | | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>19.15 (qu, C-7); 62.10 (qu, C-8); 91.48 (d, C-2); 117.73 (t, C-6);<br>143.22 (s, C-5); 174.86 (s, C-4); 183.34 (s, C-1) ppm | | | |
| Frequency | 100 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV * | see ref. [6] | | |
|---|---|---|---|
| $\lambda_{max}$ | 224 nm | $\varepsilon_{max}$ | 1063 |
| Solvent | Methanol | | |
| Sample conc. | 0.551 mmol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | not given | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.65 | | |
| Solvent | $CH_2Cl_2$ | Solvent system | 3.5% $CH_2Cl_2$/96.5% MeOH |
| Saturated atmosphere | – | Detection/Color | UV |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel $60F_{254}$/Merck 5749 |

| GLC | see ref. [4, 4a] |
|---|---|

| HPLC | for application see ref. [5] |
|---|---|

9. Remarks

| |
|---|

10. References


[1] Bycroft, B.W., Dictionary of Antibiotics and Related Substances *(occurrence)*

[2] SIGMA Chemie GmbH, Biochemikalien und organische Verbindungen für die Forschung und Diagnostika, 1990

[3] Pohland, A.E. et al., *Pure Appl. Chem 54*, 1982, 2220pp *(UV, IR, PMR, MS)*

[4] Ogawa, Hitoshi et al., *Kenkyu Nenpo-Tokyo-tantsu Eisei Kenkyusho* 1984, (35), 224-30

[4a] Abstract 1985 102:219714u *(GLC)*

[5] Engstrom, G.W. et al., *J. Agric. Food Chem.* 1977, *(25,4)*, 8336pp *(HPLC)*

[7] Lindenfilser, L.A. et al., *Dev. Ind. Microbiol.* 1973, *14*, 331-6 *($LD_{50}$)*

# Saponarin

*H. D. Zinsmeister, S. Anhut, R. Mues*

## 1. Name of Compound

Common name
**Saponarin**

Synonyms
Isovitexin-7-O-glucoside
6-C-β-D-Glucopyranosyl-apigenin-7-O-β-glucopyranoside

Systematic name
6-β-D-Glucopyranosyl-7-(β-D-glucopyranosyloxy)-5-hydroxy-2-(4-hydroxyphenyl)4*H*-1-benzopyran-4-one

| Substance | Subgroup |
|---|---|
| Flavonoid | Flavone Glycoside<br>Flavone C/O-Glycoside |

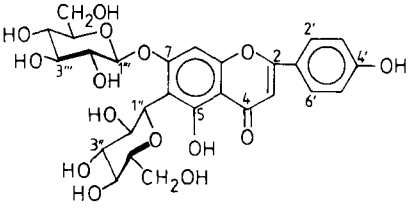
| CAS registry number and other numbers | |
|---|---|
| [20310-89-8] | BRN 75710 |

## 2. Formulas and Molecular Weight

Molecular formular
$C_{27}H_{30}O_{15}$

Molecular weight
594

Structural formula




## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| crystalline | not applicable | not applicable |
| **Melting Point mp** | $n_D^{20}$ | **Acid value** |
| 235-236 °C | not applicable | not applicable |
| **Boiling point bp** | $[\alpha]_D^{20} = -32°$ | **Saponification value** |
| not applicable | (c = 0.392, methanol) | not applicable |
| **Flash point** | **Soluble in** | |
| not applicable | water (heated), methanol (heated) | |
| **Color** | **Odor** | |
| pale yellowish | odorless | |

4. Occurrence

| |
|---|
| Many sources [1, 2] – e.g. for Bryophytes:<br>Musci: Plagiomnium undulatum [3]; Plagiomnium cuspidatum (unpubl.)<br>Hepaticae: Porella platyphylla [1]; Porella cordaeana (unpubl.) |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| not known | $LD_{50}$ | not known |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder; preferably in the dark |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS | FAB technique – negative mode – Xenon gas – glycerol matrix | | |
| Base peak | in range >200: m/e = 431 | Molecular ion | m/e = 593 $[M-H]^-$ |
| Ionization energy | 4.3 keV | Ion source temp. | 40–50 °C |
| Acceleration voltage | – | Emission current | – |
| Resolution | >1000 | Scan rate | – |
| Spectrometer type and manufacturer | | Finnigan MAT 90 | |

| | | | |
|---|---|---|---|
| IR | | | |
| Characteristic peaks<br>3400 ±50 (s), 2920–2870 (m), 1618 (s), 1581 (s), 1534 (s), 1470 (s), 1450 (s),<br>1418 (s), 1313 (s), 1265 (m), 1246 (m), 1215 (m), 1155 (s), 1145 (s),<br>1047–1010 (s), 870 (m), 850 (m), 791 (s), 767 (m) $cm^{-1}$ | | | |
| Sample preparation | pellet,<br>2 mg in 200 mg KBr | Resolution | 4000–2000 $cm^{-1}$: 1.4 $cm^{-1}$<br>2000– 600 $cm^{-1}$: 0.5 $cm^{-1}$ |
| Spectrometer type and manufacturer | | Beckmann IR-Spectrophotometer 4210 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>Aglycone: 7.88 (d, J=8 Hz, H-2′, H-6′);<br>6.89 (H-3,5);<br>Sugars: 4.99 (d, J=7 Hz, H-1‴);<br>3–4 (sugar protons) ppm | | 6.95 (d, J=9 Hz, H-3′, H-5′);<br>6.72 (H-8,0)<br>4.77 (d, J=10 Hz, H-1″) | |
| Frequency | 400 MHz | Solvent | DMSO-$d_6$ |
| Standard | TMS | Sample temp. | 100 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | see ref. [4] | Nucleus | $^{13}C$ |
|---|---|---|---|

Chemical Shifts

| | | | | |
|---|---|---|---|---|
| 181.7 (C-4); | 164.0 (C-2); | 161.9 (C-7); | 160.9 (C-4′); | 159.6 (C-5); |
| 156.1 (C-9); | 128.0 (C-2′, C-6′); | 120.9 (C-1′); | 115.7 (C-3′, C-5′); | |
| 110.7 (C-6); | 105.1 (C-10); | 103.0 (C-3); | 101.4 (C-1‴); | 93.8 (C-8); |
| 80.7 (C-5″); | 78.8 (C-3″); | 76.9(C-5‴); | 75.9 (C-3‴); | 73.4 (C-1″); |
| 72.7 (C-2‴); | 70.6 (C-2″); | 70.3 (C-4″); | 69.8 (C-4‴); | 60.8 (C-6″, C-6‴) ppm |

| | | | |
|---|---|---|---|
| Frequency | 100 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | 100 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 272, 332 nm | $\varepsilon_{max}$ | – |
| Solvent | Methanol (Uvasol)<br>additional UV spectra with shift reagents according to ref. [5] | | |
| Sample conc. | $15-35 \times 10^{-6}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Super Scan 3 (Varian); UV-VIS-spectrophotometer, double beam | | |

8. Chromatographic Data

| | | | |
|---|---|---|---|
| TLC | I, II on Cellulose (Avicel); III on Polyamide-6 | | |
| Rf Value | System I: 0.42; II: 0.56; III: 0.69 | | |
| Solvent system | I: Butanol-(1) : Acetic Acid : Water (4 : 1 : 5) upper layer<br>II: Acetic Acid : Water (15 : 85)<br>III: Water : Butanone(2) : MeOH : Pentanedione-(2,4) (65 : 15 : 15 : 5) | | |
| Saturated atmosphere | yes | Detection/Color | UV deep purple;<br>UV, sprayed with Diphenylboric-acid-β-aminoethylester (NA): dark green |
| Plate manufacturer | Schleicher & Schüll<br>Macherey & Nagel | Plate type/Product no. | Cellulose, Avicel F 1440<br>Polygram; 0.1 mm; Polyamide-TLC 6 |

| | |
|---|---|
| GLC | underivatized not possible |

| HPLC | | | |
|---|---|---|---|
| Retention time | 9.5 min | | |
| Column | Knauer Spherisorb ODS II<br>L = 250 mm, 4 mm I.D. | Stationary phase | RP-18, 5 µm |
| Mobile phase | MeOH – 5% Acetic Acid (25 : 75) | Flow rate | 1.0 ml/min |
| Column temp. | ambient temperature | Pressure | – |
| Detector | UV-Detector Waters 450<br>Detection 254 nm | Sample solvent | MeOH |
| Sample size | 5–10 µl | Sample conc. | $150\text{–}350 \times 10^{-6}$ mol/l |
| Chromatograph type and manufacturer | | Waters M 45 | |

9. Remarks

Easy to crystallize from methanol/water.

10. References


[1] Chopin, J. and Dellamonica, G.: C-Glycosylflavonoids. In ‚The Flavonoids', Harborne, J.B. (Ed.), Chapman and Hall, London, 1988 *(occurrence)*

[2] Chopin, J. and Bouillant, M.L.: C-Glycosylflavonoids. In ‚The Flavonoids', Harborne, J.B., Mabry, T.J. and Mabry, H. (Eds.), Chapman and Hall, London, 1975 *(occurrence)*

[3] Österdahl, B.-G.: Chemical Studies on Bryophytes. 19th Application of $^{13}C$ NMR. In Structural Elucidation of Flavonoid C-glycosides from Hedwigia ciliata, *Acta Chemica Scandinavica B32*, 1978, pp. 93–97 *(occurrence)*

[4] Markham, K.R. and Chari, V.M.: Carbon-13 NMR Spectroscopy of Flavonoids. In ‚The Flavonoids', Harborne, J.B. and Mabry T.J. (Eds.), Chapman and Hall London, 1982 *($^{13}C$-NMR)*

[4] Mabry, T.J., Markham, K.R. and Thomas, M.B.: The Systematic Identification of Flavonoids. Springer Verlag, 1970. *(UV spectroscopy)*

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# Secologanin

*L. F. Tietze, C. Bärtels*

## 1. Name of Compound

Common name

**Secologanin** [1, 2]

Synonyms

Loniceroside

Systematic name

Methyl(1S,5S,9R)-5-(formylmethyl)-1-(ß-D-glucopyranosyloxy)-9-vinyl-5,9-dihydro-1H-pyran-4-carboxylate

Substance

Secoiridoide

CAS registry number and other numbers

[19351-63-4] BRN 1441446

## 2. Formulas and Molecular Weight

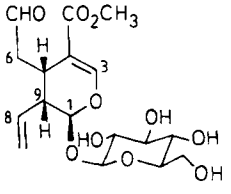
Molecular formular

$C_{17}H_{24}O_{10}$

Molecular weight

388.37

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>98 °C | $n_D^{20}$<br>not applicable | Acid value<br>not available |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20} = -128°$<br>(c=1, MeOH) | Saponification value<br>not available |
| Flash point<br>not applicable | Soluble in<br>water, methanol, acetone, etc. | |
| Color<br>white | Odor<br>not applicable | |

4. Occurrence

| |
|---|
| Constituent of symphoricarpos albus (L.) and nearly of all lonicera species [3] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not available |
| not available | $LD_{50}$ | not available |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage temperature: +4 °C |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | m/z = 157 | Molecular ion | not availavle |
| Ionization energy | 70 eV | Ion source temp. | 150 °C |
| Acceleration voltage | 3000 V | Emission current | 0.3 mA |
| Resolution | 1000 | Scan rate | 2 s/decade |
| Spectrometer type and manufacturer | | Varian MAT 311 A | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3400 (OH), 1710 (C=O), 1635 (C=C) $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | Bruker IFS 25 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>2.47(ddd, J = 12, 6, 1 Hz, 1H, $H_2C$-7α), 2.64–2.86 (m, 2H, $H_2C$-7β, *H*C-9),<br>2.94–3.02 (m, 1H, *H*C-5), 3.04–3.98 (m, 6H, *H*C-2′, *H*C-3′, *H*C-4′, *H*C-5′,$H_2C$-6′),<br>3.66 (s, 3H, $CH_3$), 4.66 (d, J = 8 Hz, 1H, C*H*-1′), 5.18–5.44 (m, 2H, $H_2C$-10),<br>5.49 (d, J = 4 Hz, 1H, *H*C-1), 5.46–5.80 (m, 1H, *H*C-8), 7.51 (d, J = 2 Hz, 1H,*H*C-3),<br>9.69 (d, J = 1 Hz, *H*C-7) ppm | | | |
| Frequency | 200 MHz | Solvent | $CD_3OD$ |
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | Varian XL 200 | |

| NMR | $^1H$-decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>28.8 (C-5), 46.3 (C-6, C-9), 53.7 ($CO_2CH_3$), 63.2 (C-6′), 70.9 (C-4′), 75.0 (C-2′),<br>78.2 (C-3′), 78.9 (C-5′), 99.2 (C-1), 101.0 (C-1′), 110.7 (C-4), 122.9 (C-10),<br>135.0 (C-8), 155.3 (C-3), 171.0 ($CO_2CH_3$), 197.1 (CHO) ppm | | | |
| Frequency | 50.3 MHz | Solvent | $CD_3OD$ |
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | Varian | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | $2.34 \times 10^{-4}$ nm | $\varepsilon_{max}$ | 10887.83 |
| Solvent | methanol | | |
| Sample conc. | $4.49 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Varian Cary 219 | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.39 | | |
| Solvent | methanol | Solvent system | chloroform/methanol (4/1) |
| Saturated atmosphere | | Detection/Color | UV 254 nm |
| Plate manufacturer | Macherey-Nagel | Plate type/Product no. | SIL G/$UV_{254}$ |

| | |
|---|---|
| GLC | not applicable |

| | |
|---|---|
| HPLC | not available |

9. Remarks

Secologanin is a key intermediate in the biosynthesis of the majority of the indole, the ipecacuanha, chinchona, pyrroloquinoline and of simple monoterpene alkaloids [4] and the parent compound of the secoiridoids [5].

10. References


[1] Tietze, L.F., *Angew. Chem. Int. Ed. Engl. 22*, 1983, 828 *(review)*

[2] Battersby, A.R., Burnett, A.R., Parsons, P.G., *J. Chem. Soc. (C), 1969*, 1187 *(first detection, secologanin as intermediate in the biosynthesis of alkaloids)*

[3] Tietze, L.F., Henke, S., Bärtels, C., *Tetrahedron 44*, 1988, 7145; Tietze, L.F., Bärtels, C., *Tetrahedron 45*, 1989, 681; Hermans-Lokkutsch, A., Verpoorte, R., *Planta Medica 1987*, 546; Kinast, G., Tietze, L.F., *Chem Ber. 109*, 1976, 3640 *(isolation)*

[4] Phillipson, I.D., Zenk, M.H., *Indole and Biogenetically Related Alkaloids*, Academic Press, London, 1980 *(biosynthesis of alkaloids)*

[5] Inouye, H., Uesato, S., *Prog. Chem. Org. Nat. Prod. 50*, 1986, 169 *(biosynthesis of secoiridoids)*

# Senkirkine

*E. Röder*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Senkirkine** | |
| Synonyms<br>Renardine | |
| Systematic name<br>8,12-Dihydroxy-4-methyl-11,16-dioxosenecionanium | |
| Substance<br>Alkaloid | Subgroup<br>Pyrrolizidine alkaloid |
| CAS registry number and other numbers<br>[2318-18-5] | BRN 50907 |

## 2. Formulas and Molecular Weight

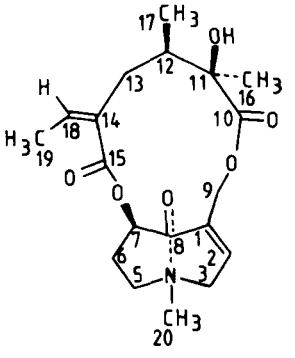
Molecular formular

$C_{19}H_{27}NO_6$

Molecular weight

365.1843

Structural formula




## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>197.5-198 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{15}$: −6.2 ($CHCl_3$)<br>$[\alpha]_D^{25}$: −1.88 (c=1, EtOH) | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>methylene chloride, methanol | |
| Color<br>colorless | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Several species of the tribe senecioneae (Compositae, Asteraceae)<br>Brachyglottis, Petasites, Farfugium, Tussilago, Emilia. |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology<br>hepatotoxic<br>probable carcinogen | Hazard labeling | T; R: 25-40; S: 1-22-45 |
| | $LD_{50}$ | not available |
| Waste disposal procedures:<br>By combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| GGVE/GGVS 6.1/12ab |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 43.0218 ($C_2H_3O$) | Molecular ion | 365.1843 (6.34%, $C_{19}H_{27}NO_6$) |
| Ionization energy | 70 eV | Ion source temp. | 180 °C |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | MS 50, AEI Brunner | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3440 (OH), 3025 (C=C), 1735, 1705 (α,β-unsat. ester), 1650 (α,β-unsat. ketone) $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | Beckman Acculab 2 | |

| NMR see Cheng, Röder 1986 | | Nucleus | $^{1}H$-$^{13}C$ correlation |
|---|---|---|---|
| Chemical Shifts<br>10.93 (C-17); 15.26 (C-19); 24.55 (C-16); 36.36 (C-6); 37.69 (C-13);<br>38.58 (C-12); 40.37 (C-20); 53.13 (C-5); 58.52 (C-3); 64.32 (C-9);<br>76.58 (C-11); 78.06 (C-7); 131.67 (C-14); 134.32 (C-1); 137.00 (C-18);<br>137.39 (C-2); 166.45 (C-15); 177.98 (C-10); 192.07 (C-8) ppm | | | |
| Frequency | – | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | AC 200 and WM 400 Bruker | |

| NMR see Cheng, Röder 1986 | | Nucleus | $^{1}H$-$^{1}H$ correlation |
|---|---|---|---|
| Chemical Shifts<br>0.91 (d, $J_{12\beta} = 6.0$ Hz, 12-$CH_3$); 1.33 (s, 11-$CH_3$);<br>1.68 (ddq, $J_{13\alpha} = 13.5$, $J_{13\beta} = 12.0$, $J_{17\text{-}CH3} = 6.0$ Hz, H-12β);<br>1.81 (dd, $J_{13\alpha} = 13.5$, $J_{12\beta} = 12.0$ Hz, H-13β);<br>1.99 (dd, $J_{H\text{-}18} = 7.5$, $J_{13\alpha} = 1.5$ Hz, 18-$CH_3$);<br>2.08 (s, N-$CH_3$); 2.31 (br d, $J_{13\beta} = 13.5$ Hz, H-13α);<br>2.36 (ddt, $J_{6\alpha} = 13.5$, $J_{5\beta} = 4.5$, $J_{5\alpha} = J_{7\alpha} = 3.0$ Hz, H-6β);<br>2.55 (ddt, $J_{6\beta} = J_{5\alpha} = 13.5$, $J_{5\beta} = 3.05$, $J_{7\alpha} = 3.0$ Hz, H-6α);<br>2.74 (dt, $J_{5\alpha} = J_{6\alpha} = 13.5$, $J_{6\beta} = 4.5$ Hz, H-5β);<br>2.87 (ddd, $J_{5\beta} = 13.5$, $J_{6\beta} = 4.5$, $J_{6\alpha} = 3.0$ Hz, H-5α);<br>3.25 (dt, $J_{3\alpha} = 19.5$, $J_{H\text{-}2} = J_{9\beta} = 2.0$ Hz, H-3β);<br>3.43 (br d, $J_{3\beta} = 19.5$, $J_{H\text{-}2} = 2.0$ Hz, H-3α); 4.35 (d, $J_{9\beta} = 11.0$ Hz, H-9β);<br>4.98 (t, $J_{6\alpha} = J_{6\beta} = 3.0$ Hz, H-7α); 5.42 (d, $J_{9\beta} = 11.0$ Hz, H-9α);<br>5.86 (dq, $J_{18\text{-}CH3} = 7.5$, $J_{13\alpha} = 1.5$ Hz, H-18); 6.14 (t, $J_{3\alpha} = J_{3\beta} = 2.0$ Hz, H-2) ppm | | | |
| Frequency | – | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | WM 400 Bruker | |

| UV | not applicable |
|---|---|

## 8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.23 | | |
| Solvent | – | Solvent system | $CH_2Cl_2$:$CH_3OH$:$NH_3$ 25%<br>(85 : 14 : 1) |
| Saturated atmosphere | 20–25 °C | Detection/Color | Dragendorff brown |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel $60F_{254}$ 0.25 mm |

| | | | |
|---|---|---|---|
| GLC | quartz-capillary column | | |
| Retention time | 13.87 min | | |
| Column type | Macherey & Nagel FS/SE-54-CB-0.25 | Column length | 50 m (int. diameter 0.32 mm) |
| Column packing | – | Column temp. | 214–280 °C (speed 10°/min) |
| Injector port temp. | 280 °C | Carrier gas | Helium |
| Detector temp. | 280 °C | Sample solvent | $CH_2Cl_2$ |
| Sample size | – | Sample conc. | 0.00625% |
| Chromatograph type and manufacturer | | Shimadzu GC-917 with FID | |

| | |
|---|---|
| HPLC | not available |

9. Remarks

The three-dimensional structure of Senkirkine was determined by x-ray cristallography (see ref. [5]).

10. References


[1] Hepatotoxicity: Schoental,R., *Nature* (London) *227* (1970) 401–2

[2] Mutation: Takanashi,H., Umeda,M., Hirono,J., *Mutat. Res. 78* (1980) 67–77
Candrian,U., Lüthy,J., Graf,U., Schlatter,C., *Food Chem. Toxicol. 22* (1984) 223–5
Kraus,C., Abel,G., Schimmer,O., *Planta Med. 1985*, 89–91

[3] Hepatocarcinogenicity: Hirono,J., Haga,M., Fujii,M., Matsuura,S., Matsubara,N.,Nakayama,M., Furuya,T., Hikichi,M., Takanashi,H. et al., *J. Natl. Cancer Inst. 63* (1979) 469

[4] Structure: Briggs,L.H., Cambie,R.C., Candy,B.J., O'Donovan,G.M., Russell,R.H., Seelye,R.N., *J. Chem. Soc. 1965*, 2492–98

[5] Crystal Structure: Birnbaum,G.J., *J. Am. Chem. Soc. 96* (1974) 6165–68

[6] Occurrence:
Brachyglottis repanda: Mortimer,P.H., White,E.P., *Nature 214* (1967) 1255–56
Crotalaria laburnifolia, ssp. eldomac: Crout,D.H.G., *J. Chem. Soc. Perkin Trans. I, 1972*, 1602–7
Emilia sonchifolia: Cheng,D., Röder,E., *Planta Med. 1986*, 484–6
Farfugium japonicum: Furuya,T., Murakami,K.,Hikichi,M., *Phytochemistry 10* (1971) 3306–7
Petasites japonicus: Yamada,K., Tatimatsu,H., Kyotani,Y., Hirata,Y., Haga,M., Hirono,J., *Phytochemistry 17* (1978) 1667–68
Senecio antieuphorbium: Rodriguez,D.F., Gonzales,G.A., *Farm.Nueva 36* (1971) 810
Senecio desfantainei: Rizk,A.M., Hammouda,F.M., Ismail,S.J., Ghaleb,H.A., Madkaur,M.K., Pohland,A.E., Wood,G., *Fitoterapia 54* (1983) 115–21
Senecio illinitus: Gonzalez,A.G., de la Fuente,G.,Reina,M., Loyola,L.A., *Planta Med. 1986*, 160
Senecio kirkii: see ref. [4]
Senecio kleinia: Diaz,R.M., Gonzalez,G., Morales,M.A., *Farm. Nueva 36* (1971) 803–10
Senecio pierotii: Asada,Y., Furuya,T., *Planta Med. 44* (1982) 182

| | |
|---|---|
| Senecio procerus: | Jovceva,R.J., Boeva,A., Potesilova,H., Klasek,A., Santavy,F., *Collect. Czech. Chem. Commun. 43* (1978) 2312 |
| Senecio tenuifolius: | Bhakuni,D.S., Gupta,S., *Planta Med. 1982*, 251 |
| Senecio vernalis: | Röder,E., Wiedenfeld,H., Pastewka,U., *Planta Med. 37* (1979), 131-6 |
| Tussilago farfara: | Borka,L., Onshuus,J., *Medd. Nor. Farm. Selsk. 41* (1979) 165-8 |
| | Culvenor,C.C.J., Edgar,J.A., Smith,L.W., Hirono,J., *Aust. J. Chem. 29* (1976) 229-30 |
| | Jost,E.J., Dissertation, Bonn 1983, 1-103 |
| | Lüthy,J., Zweifel,U., Schlatter,C., Benn,M.H., *Mitt. Geb. Lebensmittelunters. Hyg. 71* (1980) 73-80 |

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# Sphagnum acid

*H. Rudolph, J. Wilschke, R. Tutschek*

## 1. Name of Compound

Common name

**Sphagnum acid** [2]

Synonyms

p-Hydroxy-β-[carboxymethyl]cinnamic acid

Systematic name

3-[4-Hydroxy-phenyl]pentenedioic acid

| Substance | Subgroup |
|---|---|
| Phenolic | Cinnamic acid derivative |

| CAS registry number and other numbers | |
|---|---|
| [57100-28-4] | BRN 3304997 |

## 2. Formulas and Molecular Weight

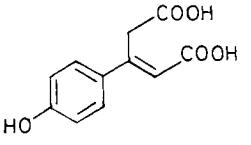
Molecular formular

$C_{11}H_{10}O_5$ [2]

Molecular weight

222

Structural formula




## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| crystalline | | |
| Melting Point mp<br>$mp_1$ = 178-180 °C<br>$mp_2$ = 202-204 °C | $n_D^{20}$ | Acid value<br>$pK_{a1}$ = 3.98<br>$pK_{a2}$ = 5.53<br>$pK_{a3}$ = 9.88 |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20}$ | Saponification value |
| Flash point | Soluble in<br>water ($2 \times 10^{-3}$ M), methanol, ethanol | |
| Color | Odor<br>odorless | |

4. Occurrence

| |
|---|
| Moss Sphagnum [3] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| unknown | $LD_{50}$ | unknown |
| Waste disposal procedures | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder, preferably in the dark |

7. Spectroscopic Data

| | |
|---|---|
| MS | not available |

| | | | |
|---|---|---|---|
| IR * | see ref. [1, 2] | | |
| Characteristic peaks<br>1735, 1680, 1605, 1590, 1501, 1420, 1390, 835 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | Beckmann | |

| | | | |
|---|---|---|---|
| NMR * | see ref. [1] | Nucleus | |

| | | | |
|---|---|---|---|
| UV * | see ref. [2] | | |
| $I_{max}$ | 288 nm | $\varepsilon_{max}$ | – |
| Solvent | MeOH (Uvasol)/$HCl_{conc}$ (99:1, v/v) | | |
| Sample conc. | – | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Hitachi spectrometer 100-40 | |

8. Chromatographic Data

| | | | |
|---|---|---|---|
| TLC | see ref. [3, 4] | | |
| Rf Value | 0.71–0.72 | | |
| Solvent | EtOH | Solvent system | Butanol/Acetic Acid/Water (3 : 2 : 95, v/v/v) |
| Saturated atmosphere | yes | Detection/Color | UV-spectrum<br>Millon's/Pauly's reagent |
| Plate manufacturer | not given | Plate type/Product no. | not given |

| | |
|---|---|
| GLC | underivatized not possible |

| | | | |
|---|---|---|---|
| HPLC | | | |
| Retention time | 20.3 min | | |
| Column | Kontron steel column | Stationary phase | ODS II, 5 µm,<br>250 × 4.6 mm |
| Mobile phase | a) HCCOH/$H_2O$ (5 : 95, v/v)<br>b) $CH_3OH$ | Flow rate | 1 ml/min |
| Column temp. | ca. 25 °C | Pressure | – |
| Detector | Uvikon 720 LC micro | Sample solvent | $CH_3OH$ |
| Sample size | 20 µl | Sample conc. | – |
| Chromatograph type and manufacturer | | KONTRON INSTRUMENTS: Anacomp 220, LC-pumps T-414, Uvikon 720 LC | |

9. Remarks

A convenient synthesis of sphagnum acid has been described [5].

10. References


[1] Tutschek, R. *Z. Pflanzenphysiol. 76*, 1975, pp. 353–365 *(isolation, characterization)*

[2] Tutschek, R., Rudolph, H., Wagner, P.H., Kreher, R. *Biochem. Physiol. Pflanz. 164*, 1973 *(structure, physical properties)*

[3] Rudolph, H. and Samland J. *Phytochemistry 24*, 1985, pp. 745–49 *(occurrence, metabolism)*

[4] Engmann, B. *Biochem. Physiol. Pflanz. 163*, 1972, pp. 200–15 *(TLC-data)*

[5] Wächter, D. and Rudolph H. *Z. Naturforsch. 39c*, 1984, pp. 311–12 *(synthesis)*

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# Squarrogenin 1

*A. S. Gromova, V. I. Lutsky, A. A. Semenov*

## 1. Name of Compound

Common name

**Squarrogenin 1** [1]

Synonyms

Systematic name

21(R),22(S),23(R),3β,22β,30-Trihydroxy-21-methoxy-21,23-epoxycycloart-24-ene

| Substance | Subgroup |
|---|---|
| Triterpenoid | Lanostane (cycloartane) |

| CAS registry number and other numbers | |
|---|---|
| [125445-28-5] | BRN 4239601 |

## 2. Formulas and Molecular Weight

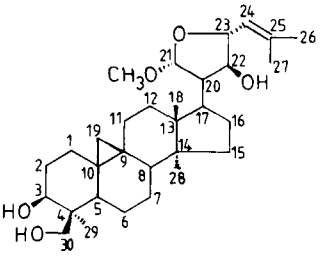
Molecular formular

$C_{31}H_{50}O_5$

Molecular weight

502.733

Structural formula



## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| crystalline | not applicable | not applicable |
| **Melting Point mp** | $n_D^{20}$ | **Acid value** |
| 169-171 °C | not applicable | not applicable |
| **Boiling point bp** | $[\alpha]_{546}^{20} = -11.06°$ | **Saponification value** |
| not applicable | (c = 4.52; pyridin) | not applicable |
| **Flash point** | **Soluble in** | |
| not applicable | acetone, methanol, chloroform | |
| **Color** | **Odor** | |
| white | odorless | |

4. Occurrence

| |
|---|
| Thalictrum squarrosum Stephan ex Willd [1] |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | unknown |
| unknown | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder; preferably in the dark |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS * | EI – mass spectrum | | |
| Base peak | 484 | Molecular ion | 502 |
| Ionization energy | 70 eV | Ion source temp. | 310 °C |
| Acceleration voltage | 2330 V | Emission current | 50 mA |
| Resolution | 1000 | Scan rate | 8 s/decade |
| Spectrometer type and manufacturer | | LKB- 2091, Sweden, LKB | |

| | |
|---|---|
| IR | not available, see remarks |

| | | | |
|---|---|---|---|
| NMR | | Nucleus | $^1H$ |
| Chemical Shifts<br>5.40 (H-24, dt, $J = 9.16$, 1.22 Hz); 4.83 (H-21, d, $J = 3.97$ Hz);<br>4.65 (H-23, dd, $J = 9.16$, 3.66 Hz); 4.41; 3,40 (2H-30, d, $J = 11.0$ Hz);<br>3.88 (H-22, t, $J = 3.97$); 3.50 (H-3, m); 3.33 ($CH_3O$, s);<br>2.20 (1H-20, m); 1.80 ($CH_3$, d, $J = 1.22$ Hz);<br>1.78 ($CH_3$, d, $J = 1.22$ Hz);<br>1.22; 1.01; 0.94 (3 $CH_3$, s); 0.41; 0.37; (2H-19, d, $J = 4.0$ Hz) ppm | | | |
| Frequency | 200.13 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker WP-200 | |

| NMR | | | Nucleus | $^{13}C$ | | |
|---|---|---|---|---|---|---|
| Chemical Shifts | | | | | | |
| 137.4 (25); | 121.4 (24); | 104.9 (21); | 80.7 (23); | 80.1 (3); | 75.0 (22); | 64.5 (30); |
| 54.5 ($OCH_3$); | 52.5 (20); | 48.6 (5); | 48.4 (14); | 47.7 (8); | 45.4 (13); | 43.8 (4); |
| 40.7 (17); | 36.1 (12); | 32.4 (1); | 31.7 (2); | 31.3 (19); | 30.6 (15); | 30.0 (16); |
| 27.0 (7); | 26.7 (11); | 26.3 (18); | 26.0 (10); | 21.8 (6); | 21.7 (9); | 21.3 (26); |
| 19.8 (27, 28); | 18.4 (29) ppm | | | | | |
| Frequency | 22.49 MHz | | Solvent | $C_5D_5N$ | | |
| Standard | TMS | | Sample temp. | 24 °C | | |
| Spectrometer type and manufacturer | | | JEOL FX – 90 Q | | | |

| UV | not informative |
|---|---|

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.30 | | |
| Solvent | – | Solvent system | chloroform/benzene/acetone (3 : 2 : 1) |
| Saturated atmosphere | yes | Detection/Color | violet-pink 0.5% vanillin in 50% $H_3PO_4$ |
| Plate manufacturer | Lachema Czechoslovakia | Plate type/Product no. | Silicagel LS 5/40 |

| GLC | underivatized not possible |
|---|---|

| HPLC | unknown |
|---|---|

9. Remarks

The substance could be isolated just in small amounts.
IR-spectrum has little information.

10. References

[1] Lutsky V.I., Xamidullina, E.A., Gromova, A.S., Semenov, A.A., *Khim. Prir. Soedin.*, (1989), N4 510–516 *(name, occurrence, $^1H$- and $^{13}C$-NMR, MS)*

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# Squarrogenin 2

*A. S. Gromova, V. I. Lutsky, A. A. Semenov*

## 1. Name of Compound

Common name

**Squarrogenin 2** [1]

Synonyms

Systematic name

21(S),22(S),23(R),3β,22β,30-Trihydroxy-21-methoxy-21,23-epoxycycloart-24-ene

| Substance | Subgroup |
|---|---|
| Triterpenoid | Lanostane (cycloartane) |

CAS registry number and other numbers

[125445-29-6] BRN 4239602

## 2. Formulas and Molecular Weight

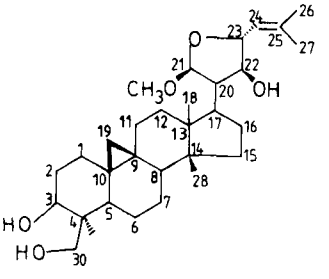
Molecular formular

$C_{31}H_{50}O_5$

Molecular weight

502.733

Structural formula



## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| crystalline | not applicable | not applicable |
| Melting Point mp | $n_D^{20}$ | Acid value |
| 190–193 °C | not applicable | not applicable |
| Boiling point bp | $[\alpha]_{546}^{20} = +106.6°$ | Saponification value |
| not applicable | (c = 0.30; pyridin) | not applicable |
| Flash point | Soluble in | |
| not applicable | acetone, methanol, chloroform | |
| Color | Odor | |
| white | odorless | |

4. Occurrence

| |
|---|
| Thalictrum squarrosum Stephan ex Willd [1]. |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | unknown |
| unknown | $LD_{50}$ | unknown |
| Waste disposal procedures<br>Combustion. | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage as powder; preferably in the dark. |

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS * | EI- mass spectrum | | |
| Base peak | 452 | Molecular ion | 502 |
| Ionization energy | 70 eV | Ion source temp. | 310 °C |
| Acceleration voltage | 2330 V | Emission current | 50 mA |
| Resolution | 1000 | Scan rate | 8 s/decade |
| Spectrometer type and manufacturer | | LKB- 2091, Sweden, LKB | |

| | |
|---|---|
| IR | not available, see remarks |

| | | | |
|---|---|---|---|
| NMR | | Nucleus | $^1H$ |
| Chemical Shifts<br>5.35 (H-24, dt, $J = 7.93$, 1.53 Hz);<br>4.66 (H-23, dd, $J = 7.93$, 2.14 Hz);<br>3.98 (H-22, q, $J = 3.97$, 2.14);<br>3.39 ($CH_3O$, s);<br>1.8O ($CH_3$, d, $J = 1.53$ Hz);<br>1.22; 1.06; 0.92 (3 $CH_3$, s); 0.37; | | 4.78 (H-21, d, $J = 3.97$);<br>4.40; 3,47 (2H-30, d, $J = 11.0$ Hz);<br>3.51 (H-3, m);<br>2.20(H-20, m);<br>1.78 ($CH_3$, d, $J = 1.53$ Hz);<br>0.34 (2H-19, d, $J = 4.0$ Hz) ppm | |
| Frequency | 200.13 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | 24 °C |
| Spectrometer type and manufacturer | | Bruker WP-200 | |

| NMR | | | Nucleus | $^{13}C$ | | |
|---|---|---|---|---|---|---|
| Chemical Shifts | | | | | | |
| 139.4 (25); | 119.1 (24); | 108.7 (21); | 80.2 (3); | 79.0 (23); | 76.7 (22); | 64.6 (30); |
| 55.6 (20); | 54.8 ($OCH_3$); | 48.7 (5); | 48.5 (14); | 47.8 (8); | 45.6 (13); | 44.8 (17); |
| 43.8 (4); | 35.9 (12); | 32.4 (1); | 31.7 (2); | 30.7 (19); | 30.5 (15); | 27.8 (16); |
| 26.9 (7); | 26.5 (11); | 26.4 (18); | 26.0 (10); | 21.9 (6); | 21.8 (9); | 21.3 (26); |
| 19.8 (27); | 18.9 (28); | 18.6 (29) ppm | | | | |
| Frequency | 22.49 MHz | | Solvent | $C_5D_5N$ | | |
| Standard | TMS | | Sample temp. | 24 °C | | |
| Spectrometer type and manufacturer | | | JEOL FX – 90 Q | | | |

| UV | not informative |
|---|---|

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.26 | | |
| Solvent | – | Solvent system | chloroform/benzene/acetone (3 : 2 : 1) |
| Saturated atmosphere | yes | Detection/Color | violet-pink 0.5% vanillin in 50% $H_3PO_4$ |
| Plate manufacturer | Lachema Czechoslovakia | Plate type/Product no. | Silicagel LS 5/40 |

| GLC | underivatized not possible |
|---|---|

| HPLC | unknown |
|---|---|

9. Remarks

The substance could be isolated just in small amounts.
IR-spectrum has little information

10. References

[1] Lutsky V.I., Xamidullina, E.A., Gromova, A.S., Semenov, A.A., *Khim. Prir. Soedin.*, (1989), N4 510–516 *(name, occurrence, $^1H$- and $^{13}C$-NMR, MS)*

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# Tricetin 6-C-arabinoside-8-C-glucoside

*R. Mues, S. Anhut, H. D. Zinsmeister*

## 1. Name of Compound

Common name

**Tricetin 6-C-arabinoside-8-C-glucoside** [2]

Synonyms

Tricetin-6-C-α-L-arabinopyranoside-8-C-β-D-glucopyranoside
6-C-α-L-Arabinopyranosyl-8-C-β-D-glucopyranosyl-tricetin

Systematic name

6-Arabinopyranosyl-8-β-D-glucopyranosyl-5,7-dihydroxy-2-(3,4,5-trihydroxyphenyl)-4*H*-1-benzopyran-4-one

Substance

Flavonoid

Subgroup

Flavone Glycoside
Flavone-C-glycoside

CAS registry number and other numbers

[86022-78-8]

## 2. Formulas and Molecular Weight

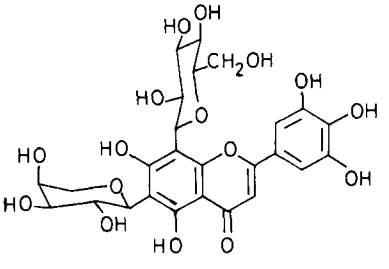
Molecular formular

$C_{26}H_{28}O_{16}$

Molecular weight

596.50

Structural formula



## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| crystalline | not applicable | not applicable |
| Melting Point mp | $n_D^{20}$ | Acid value |
| >250 °C (decomp.) | not applicable | not applicable |
| Boiling point bp | $[\alpha]_D^{20}$ | Saponification value |
| not applicable | not available | not applicable |
| Flash point | Soluble in | |
| not applicable | methanol | |
| Color | Odor | |
| yellow | odorless | |

## 4. Occurrence

Apometzgeria pubescens
Metzgeria furcata and Radula complanata (Hepaticae; Bryophytes) [1, 2, 3, 5].

## 5. Health Hazard Data

| Toxicology | Hazard labeling | not usual |
|---|---|---|
| not known | $LD_{50}$ | not known |
| Waste disposal procedures | | |

## 6. Transportation and Storage Instructions

Storage as powder; preferably in the dark.

## 7. Spectroscopic Data

| MS | see ref. [1] | as Perdeuteromethylether (PDM)<br>as Permethylether (PM) | |
|---|---|---|---|
| Base peak | PM: 733<br>PDM: 766 | Molecular ion | PM: 764<br>PDM: 800 |
| Ionization energy | 90 eV | Ion source temp. | 150 °C |
| Acceleration voltage | 1.8 kV | Emission current | 2 mA |
| Resolution | 2000 | Scan rate | – |
| Spectrometer type and manufacturer | | Varian MAT 311A – 100 MS | |

| IR | for structure elucidation of Flavone-C-glycosides not practicable |
|---|---|

| NMR | see ref. [5] | Nucleus | $^{1}H$ (see remarks) |
|---|---|---|---|
| Chemical Shifts<br>Aglycone:<br>Sugars: | 7.07; 6.93 (s, H-2',6'); 6.59 (s, H-3); 6.55 (s, H-3)<br>4.87 (d, J=9.2 Hz, H-1''); 4.73 (d, J=9.2, 10.0 Hz, H-1'');<br>4.69 and 4.62 (d, J=9.6 Hz, H-1'''); 13.89 and 13.63 (s, 5-OH); ppm | | |
| Frequency | 400 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | see ref. [4, 5] | Nucleus | $^{13}C$ (see remarks) |
|---|---|---|---|
| Chemical Shifts (Assignments bearing the same superscript are interchangeable.)<br>183.7 (C-4); 165.9/165.7 (C-2); 163.1/162.4 (C-7); 160.5/159.6 (C-5);<br>156.7/156.0 (C-9); 147.2 (C-3' and C-5'); 139.1 (C-4'); 122.4/122.1 (C-1');<br>109.7/108.5 (C-6); 107.9/107.3 (C-2' and C-6'); 105.1 (C-10)[a];<br>105.5/104.2 (C-8)[a]; 104.6/104.2 (C-3)[a];<br>6-C-Arabinose: 75.6 (C-3''); 75.2 (C-1''); 69.9 (C-4'')[b];<br>69.8 (C-2'')[b]; 71.8 (C-5'')<br>8-C-Glucose: 82.8/82.3 (C-5'''); 79.6/79.4/79.2 (C-3'''); 74.9/74.5 (C-1''');<br>72.5/72.4 (C-2'''); 70.9/70.7 (C-4'''); 62.9/61.9 (C-6''') ppm | | | |
| Frequency | 20 MHz | Solvent | DMSO-$d_6$ |
| Standard | TMS | Sample temp. | 30 °C |
| Spectrometer type and manufacturer | | Varian | |

| UV | see ref. [1] | | |
|---|---|---|---|
| $I_{max}$ | 274–356 nm | $\varepsilon_{max}$ | – |
| Solvent | Methanol (Uvasol)<br>additional UV-spectra with shift reagents were reported in [1] | | |
| Sample conc. | about $2 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Super Scan 3 (Varian); UV-VIS-spectrophotometer, double beam | | |

8. Chromatographic Data

| | | | |
|---|---|---|---|
| TLC | I-III on Cellulose (Avicel); IV on Polyamide-6 [1] | | |
| Rf Value | System I: 0.16<br>System II: 0.37 | System III: 0.09<br>System IV: 0.43 | |
| Solvent system | I: Acetic Acid – Water (15 : 85)<br>II: Acetic Acid – Water (40 : 60)<br>III: BuOH(1) – Acetic Acid – Water (4 : 1 : 5) upper layer<br>IV: Water – Butanone(2) – MeOH – Pentanedione(2,4) (65 : 15 : 15 : 5) | | |
| Saturated atmosphere | yes | Detection/Color | UV : dark; UV, sprayed with Naturstoffreagenz A: orange |
| Plate manufacturer | Schleicher & Schüll<br>Macherey & Nagel | Plate type/Product no. | Cellulose, Avicel F 1440<br>Polygram; 0.1 mm; Polyamide-TLC 6 |

| | |
|---|---|
| GLC | underivatized not possible |

| | | | |
|---|---|---|---|
| HPLC | | | |
| Retention time | 9.0 min | | |
| Column | Macherey & Nagel<br>ET 250/8/4 Nucleosil 5C18 | Stationary phase | Nucleosil RP-18, 5 µm |
| Mobile phase | A: MeOH; B: $H_2O$ : AcOH (95 : 5), isocratic 25A – 75B | Flow rate | 1.0 ml/min |
| Column temp. | ambient temp., ≈25 °C | Pressure | – |
| Detector | UV Detector Waters 450<br>UV at 254 nm | Sample solvent | MeOH |
| Sample size | 10 µl | Sample conc. | – |
| Chromatograph type and manufacturer | | Waters M 45 | |

9. Remarks

The two rotamers by interaction of 8-C-linked hexose with B-ring are NMR-distinguishable; see [5]. Underlined values for this compound are refered to in [4].

10. References


[1] Theodor, R., Zinsmeister, H.D., Mues, R. and Markham, K.R.: Flavone C-glycosides of Apometzgeria pubescens. *Phytochemistry 19*, 1980, pp. 1695–1700

[2] Theodor, R., Markham, K.R., Mues, R. and Zinsmeister, H.D.: The structures of new Flavone-di-C-glycosides from Apometgeria pubescens. *Phytochemistry 20/6*, 1981, pp. 1457–8

[3] Chopin, J. and Dellamonica, G.: C-Glycosylflavonoids. In ‚The Flavonoids', Harborne, J.B. (Ed.), Chapman and Hall London, 1988

[4] Agrawal, P.K. and Bansal, M.C.: Flavonoid Glycosides in ‚Carbon-13 NMR of Flavonoids', Agrawal, P.K. (Ed.), Studies in Organic Chemsitry 39, pp. 333–4, Elsevier Science Pub., 1989

[5] Markham, K.R., Mues, R., Stoll, M. and Zinsmeister, H.D.: NMR Spectra of Flavone Di-C-glycosides from Apometzgeria pubescens and the Detection of Rotational Isomerism in 8-C-Hexosylflavones. *Z. Naturforsch. 42c*, 1987, pp 1039–42

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# Tricetin 6,8-di-C-glucoside

*R. Mues, S. Anhut, H. D. Zinsmeister*

## 1. Name of Compound

Common name

**Tricetin 6,8-di-C-glucoside** [1, 2]

Synonyms

Tricetin-6,8-di-C-β-D-glucopyranoside
6,8-Di-C-β-D-glucopyranosyl-tricetin

Systematic name

6,8-Di-β-D-glucopyranosyl-5,7-dihydroxy-2-(3,4,5-trihydroxyphenyl)-4*H*-1-benzopyran-4-one

Substance

Flavonoid

Subgroup

Flavone Glycoside
Flavone-C-glycoside

CAS registry number and other numbers

[76491-11-7]

## 2. Formulas and Molecular Weight

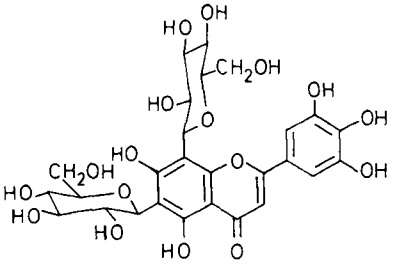
Molecular formular

$C_{27}H_{30}O_{17}$

Molecular weight

626.53

Structural formula



## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline | $d_4^{20}$<br>not applicable | Iodine value<br>not applicable |
| Melting Point mp<br>>240 °C<br>(decomp.) | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20}$<br>not available | Saponification value<br>not applicable |
| Flash point<br>not applicable | Soluble in<br>methanol | |
| Color<br>yellow | Odor<br>odorless | |

## 4. Occurrence

Plagiochila asplenioides, Metzgeria furcata, Apometzgeria pubescens
Radula spp.: Hepaticae; Bryophytes [1, 2, 3, 5]

## 5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| not known | $LD_{50}$ | not known |
| Waste disposal procedures | | |

## 6. Transportation and Storage Instructions

Storage as powder; preferably in the dark.

## 7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS | see ref. [1] | as Permethylether (PM)<br>as Perdeuteromethylether (PDM) | |
| Base peak | PM derivative: 777;<br>PDM derivative: 813 | Molecular ion | PM: 808;<br>PDM: 847 |
| Ionization energy | 90 eV | Ion source temp. | 150 °C |
| Acceleration voltage | 1.8 kV | Emission current | 2 mA |
| Resolution | 2000 | Scan rate | – |
| Spectrometer type and manufacturer | | Varian MAT 311A – 100 MS | |

| IR | for structure elucidation of Flavone-C-glycosides not practicable |
|---|---|

| NMR | see ref. [5] | Nucleus | $^{1}H$ (see remarks) |
|---|---|---|---|
| Chemical Shifts<br>Aglycone:<br><br>Sugars: | 7.08 (s); 6.99 (s, H-2′, H-6′); 6.57 (s, H-3);<br>6.54 (s, H-3); 13.77 and 13.69 (s, 5-OH);<br>4.97 and 4.79 (d, J=9.7, 10.1 Hz, H-1″);<br>4.74 and 4.65 (d, J=9.8, 9.6 Hz, H-1‴) ppm | | |
| Frequency | 400 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | ambient temp., ≈ 25 °C |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | see ref. [4, 5] | Nucleus | $^{13}C$ (see remarks) |
|---|---|---|---|
| Chemical Shifts (Assignments bearing the same superscript are interchangeable.)<br>182.5 (C-4); 164.9/164.3 (C-2); 162.0/161.2 (C-7); 160.2/158.9 (C-5);<br>155.4/154.3 (C-9); 146.7 (C-3′ and C-5′); 138.3/138.2 (C-4′);<br>121.4/120.9 (C-1′); 109.4/107.8 (C-6); 106.8/106.3 (C-2′ and C-6′)[a];<br>105.5/104.2 (C-8)[b]; 103.6/103.4 (C-10)[b]; 102.9/102.8 (C-3);<br>6-C-Glucose: 82.5/82.1 (C-5″); 79.5/79.3 (C-3″); 75.4/74.5 (C-1″)[b];<br>72.3/72.1 (C-2″)[c]; 70.8/69.7 (C-4″)[c]; 62.2/61.7 (C-6″);<br>8-C-Glucose: 81.8/81.3 (C-5‴); 78.2/78.2 (C-3‴); 73.8/73.6 (C-1‴);<br>71.1 (C-2‴)[c]; 69.5 (C-4‴)[c]; 60.6/60.2 (C-6‴) ppm | | | |
| Frequency | 100 MHz | Solvent | DMSO-$d_6$ |
| Standard | DMSO-$d_6$ | Sample temp. | ambient temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 275–358 nm | $\varepsilon_{max}$ | – |
| Solvent | Methanol (Uvasol)<br>additional UV-spectra with shift reagents were reported in [1] | | |
| Sample conc. | about $2 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Super Scan 3 (Varian); UV-VIS-spectrophotometer, double beam | | |

8. Chromatographic Data

| | | | |
|---|---|---|---|
| TLC | I-III on Cellulose (Avicel); IV on Polyamide-6 [1] | | |
| Rf Value | System I: 0.22<br>System II: 0.44 | System III: 0.11<br>System IV: 0.55 | |
| Solvent system | I: Acetic Acid – Water (15 : 85)<br>II: Acetic Acid – Water (40 : 60)<br>III: BuOH(1) – Acetic Acid – Water (4 : 1 : 5) upper layer<br>IV: Water – Butanone(2) – MeOH – Pentanedione(2,4) (65 : 15 : 15 : 5) | | |
| Saturated atmosphere | yes | Detection/Color | UV : dark; UV, sprayed with Naturstoffreagenz A: orange |
| Plate manufacturer | Schleicher & Schüll<br>Macherey & Nagel | Plate type/Product no. | Cellulose, Avicel F 1440<br>Polygram; 0.1 mm; Polyamide-TLC 6 |

| | |
|---|---|
| GLC | underivatized not possible |

| | | | |
|---|---|---|---|
| HPLC | | | |
| Retention time | 5.6 min | | |
| Column | Macherey & Nagel<br>ET 250/8/4 Nucleosil 5C18 | Stationary phase | Nucleosil RP-18, 5 µm |
| Mobile phase | A: MeOH B = $H_2O$ : AcOH<br>(95 : 5), isocratic 25A – 75B | Flow rate | 1.0 ml/min |
| Column temp. | ambient temp., ≈ 25 °C | Pressure | – |
| Detector | UV Detector Waters 450<br>UV at 254 nm | Sample solvent | MeOH |
| Sample size | 10 µl | Sample conc. | – |
| Chromatograph type and manufacturer | | Waters M 45 | |

9. Remarks

The two rotamers by interaction of 8-C-linked hexose with B-ring are NMR-distinguishable; see [5]. Underlined values for this compound are refered to in [4].

10. References


[1] Theodor, R., Zinsmeister, H.D., Mues, R. and Markham, K.R.: Flavone C-glycosides of Apometzgeria pubescens. *Phytochemistry 19*, 1980, pp. 1695–1700

[2] Theodor, R., Markham, K.R., Mues, R. and Zinsmeister, H.D.: The structures of new Flavone-di-C-glycosides from Apometgeria pubescens. *Phytochemistry 20/6*, 1981, pp. 1457–8

[3] Chopin, J. and Dellamonica, G.: C-Glycosylflavonoids. In ‚The Flavonoids', Harborne, J.B. (Ed.), Chapman and Hall London, 1988

[4] Agrawal, P.K. and Bansal, M.C.: Flavonoid Glycosides. In ‚Carbon-13 NMR of Flavonoids', Agrawal, P.K. (Ed.), Studies in Organic Chemistry 39, pp. 333–4, Elsevier Sci. Pub., 1989

[5] Markham, K.R., Mues, R., Stoll, M. and Zinsmeister, H.D.: NMR Spectra of Flavone Di-C-glycosides from Apometzgeria pubescens and the Detection of Rotational Isomerism in 8-C-Hexosylflavones. *Z. Naturforsch. 42c*,1987,pp 1039–42

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# Tricin 6,8-di-C-arabinoside

*H. D. Zinsmeister, S. Anhut, R. Mues*

## 1. Name of Compound

Common name

**Tricin 6,8-di-C-arabinoside** [2]

Synonyms

Tricin-6,8-di-C-α-L-arabinopyranoside
6,8-Di-C-α-L-arabinopyranosyltricin

Systematic name

6,8-Diarabinopyranosyl-5,7-dihydroxy-2-(4-hydroxy-3,5-dimethoxyphenyl)-4*H*-1-benzopyran-4-one

Substance

Flavonoid

Subgroup

Flavone Glycoside
Flavone-C-glycoside

CAS registry number and other numbers

[89886-36-2]

## 2. Formulas and Molecular Weight

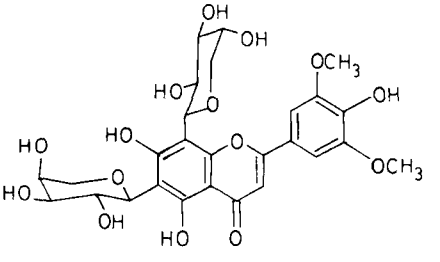
Molecular formular

$C_{27}H_{30}O_{15}$

Molecular weight

594.53

Structural formula



3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>crystalline,<br>small needles | $d_4^{20}$<br>not applicable | Iodine value<br>not applicable |
| Melting Point mp<br>228-230 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not applicable | $[\alpha]_D^{20}$<br>not available | Saponification value<br>not applicable |
| Flash point<br>not applicable | Soluble in<br>methanol, methanol/water mixtures | |
| Color<br>yellow | Odor<br>odorless | |

4. Occurence

Apometzgerin pubescens (Hepaticae; Bryophytes) [1, 2, 3, 5]
(only known from this species!)

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not usual |
| not known | $LD_{50}$ | not known |
| Waste disposal procedures | | |

6. Transportation and Storage Instructions

Storage as powder; preferably in the dark.

7. Spectroscopic Data

| | | | |
|---|---|---|---|
| MS | see ref. [1] | as Permethylether (PM)<br>as Perdeuteromethylether (PDM) | |
| Base peak | PM: 689; PDM: 713 | Molecular ion | PM: 720; PDM: 747 |
| Ionization energy | 90 eV | Ion source temp. | 150 °C |
| Acceleration voltage | 1.8 kV | Emission current | 2 mA |
| Resolution | 2000 | Scan rate | – |
| Spectrometer type and manufacturer | | Varian MAT 311A – 100 MS | |

| IR | for structure elucidation of Flavone-C-glycosides not practicable |
|---|---|

| NMR | see ref. [5] | Nucleus | $^{1}H$ |
|---|---|---|---|
| Chemical Shifts<br>Aglycone:<br>Sugars: | 7.38 (s, H-2′,6′);<br>4.83 (d, J=9.5 Hz, H-1″);<br>3.89 (s, Methoxyl); | 6.94 (s, H-3);<br>4.58 (d, J=9.5 Hz, H-1‴);<br>5–3 (mult., sugar protons) ppm | |
| Frequency | 80 MHz | Solvent | DMSO-$d_6$ |
| Standard | TMS | Sample temp. | 30 °C |
| Spectrometer type and manufacturer | | Varian | |

| NMR | see ref. [4, 5] | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts (Assignements bearing the same superscript are interchangeable.) | | | |
| 182.5 (C-4); | 163.9 (C-2); 162.6 (C-7); | 159.5 (C-5); | 154.2 (C-9); |
| 148.3 (C-3′ and C-5′); | 139.8 (C-4′); | 120.9 (C-1′); | 109.2 (C-6); |
| 104.8 (C-2′ and C-6′); | 104.1 (C-8[a]); | 103.6 (C-10[a]); | 103.3 (C-3[a]); |
| 6-C-Arabinose: | 75.5 (C-3″); | 74.9 (C-1″); | 70.8 (C-5″); |
| | 69.3 (C-4″); | 68.8 (C-2″) | |
| 8-C-Arabinose: | 74.9 (C-3‴); | 74.1 (C-1‴); | 70.5 (C-5‴); |
| | 69.1 (C-4‴); | 68.5 (C-2‴); | 56.4 ($OCH_3$) ppm |
| Frequency | 20 MHz | Solvent | DMSO-$d_6$ |
| Standard | TMS | Sample temp. | 30 °C |
| Spectrometer type and manufacturer | | Varian | |

| UV | additional UV-spectra with shift reagents were reported in [1] | | |
|---|---|---|---|
| $I_{max}$ | 274–350 nm | $\varepsilon_{max}$ | – |
| Solvent | Methanol (Uvasol) | | |
| Sample conc. | about $2 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | Super Scan 3 (Varian); UV-VIS-spectrophotometer, double beam | | |

8. Chromatographic Data

| | | | |
|---|---|---|---|
| TLC | I-III on Cellulose (Avicel);<br>IV on Polyamide-6 [1] | | |
| Rf Value | System I: 0.26<br>System II: 0.65 | System III: 0.28<br>System IV: 0.65 | |
| Solvent system | I: Acetic Acid – Water (15 : 85)<br>II: Acetic Acid – Water (40 : 60)<br>III: BuOH(1) – Acetic Acid – Water (4 : 1 : 5) upper layer<br>IV: Water – Butanone(2) – MeOH – Pentanedione(2,4) (65 : 15 : 15 : 5) | | |
| Saturated atmosphere | yes | Detection/Color | UV dark; sprayed with Naturstoffreagenz A: yellow |
| Plate manufacturer | Schleicher & Schüll<br>Macherey & Nagel | Plate type/Product no. | Cellulose, Avicel F 1440<br>Polygram; 0.1 mm; Polyamide-TLC 6 |

| | |
|---|---|
| GLC | underivatized not possible |

| | | | |
|---|---|---|---|
| HPLC | | | |
| Retention time | 7.7 min | | |
| Column | Macherey & Nagel<br>ET 250/8/4 Nucleosil 5C18 | Stationary phase | Nucleosil RP-18, 5 µm |
| Mobile phase | A: MeOH, B: $H_2O$ : AcOH<br>(95:5), isocratic 40A – 60B | Flow rate | 1.0 ml/min |
| Column temp. | ambient temp., ≈ 25 °C | Pressure | – |
| Detector | UV Detector Waters 450<br>UV at 254 nm | Sample solvent | MeOH |
| Sample size | 10 µl | Sample conc. | – |
| Chromatograph type and manufacturer | | Waters M 45 | |

9. Remarks

10. References


[1] Theodor, R., Zinsmeister, H.D., Mues, R. and Markham, K.R.: Flavone C-glycosides of Apometzgeria pubescens. *Phytochemistry 19*, 1980, pp. 1695–1700

[2] Theodor, R., Markham, K.R., Mues, R. and Zinsmeister, H.D.: The structures of new Flavone-di-C-glycosides from Apometgeria pubescens. *Phytochemistry 20/6*, 1981, pp. 1457–8

[3] Chopin, J. and Dellamonica, G.: C-Glycosylflavonoids. In ‚The Flavonoids', Harborne, J.B. (Ed.), Chapman and Hall London, 1988

[4] Agrawal, P.K. and Bansal, M.C.: Flavonoid Glycosides. In ‚Carbon-13 NMR of Flavonoids', Agrawal, P.K. (Ed.), Studies in Organic Chemsitry 39, pp. 333–4, Elsevier Science Pub., 1989

[5] Markham, K.R., Mues, R., Stoll, M. and Zinsmeister, H.D.: NMR Spectra of Flavone Di-C-glycosides from Apometzgeria pubescens and the Detection of Rotational Isomerism in 8-C-Hexosylflavones. *Z. Naturforsch. 42c*, 1987, pp 1039–42

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# Verrucarin A

*C. Franke, K. Krohn*

## 1. Name of Compound

Common name

**Verrucarin A**

Synonyms

Muconomycin A

Systematic name

4,5,6,7,16,16a,19a,22-Octahydro-4-hydroxy-5,16a,21-trimethyl-spiro[16,18-methano-1H,3H,23H-[1,6,12]trioxacyclooctadecino-[3,4-d]-[1]benzopyran-17(18H),2′-oxirane]-3,9,14-trione

Substance

Makrolid

CAS registry number and other numbers

[3148-09-2] BRN 1337118

## 2. Formulas and Molecular Weight

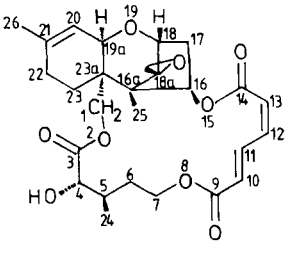
Molecular formular

$C_{27}H_{34}O_9$

Molecular weight

502.56

Structural formula



## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>>250 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{25} = +199°$<br>(c=0.386 g/l in $CHCl_3$)<br>$[\alpha]_D^{22} = 206 \pm 2°$<br>(c=1.012 g/l in $CHCl_3$) | Saponification value<br>not available |
| Flash point<br>not available | Soluble in<br>chloroform, methanol | |
| Color<br>colorless | Odor<br>odorless | |

## 4. Occurrence

Isolated from the fungus Myrothecium.

## 5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology<br>antifungal and cytostatic activity [2, 3] | Hazard labeling | extremely hazardous |
| | $LD_{50}$ | (mice): 1.5 mg/kg intravenous [2] |
| Waste disposal procedures | | |

## 6. Transportation and Storage Instructions

Storage at 0 °C

## 7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 105 | Molecular ion | 502 |
| Ionization energy | 70 eV | Ion source temp. | 300 °C |
| Acceleration voltage | 3 kV | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | 3 s/decade |
| Spectrometer type and manufacturer | | Finnigan MAT 8430 | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3480, 2850, 1715, 1630, 1585, 1475, 1412, 1385, 1350, 1335, 1275, 1210,<br>1195, 1150, 1125, 1105, 1085, 1050, 1025, 1000, 970, 930, 885, 825 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | 2.5 $cm^{-1}$ |
| Spectrometer type and manufacturer | | Perkin Elmer 1420 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>8.04 (ddd, $J_{11-10}=15$ Hz, $J_{11-12}=12$ Hz, $J_{11-13}\leq 1$ Hz, 1H, H-11);<br>6.68 (t, $J_{12-11,12-13}=11$ Hz, 1H, H-12); 6.16 (d, $J_{13-12}=11$ Hz, 1H, H-12);<br>6.05 (d, $J_{10-11}=16$ Hz, 1H, H-10);<br>5.81 (dd, $J_{16-17\alpha}=8$ Hz, $J_{16-17\beta}=4$ Hz, 1H, H-16);<br>5.44 (d, $J_{20-19a}=5$ Hz, 1H, H-20); 4.80 (d, $J_{gem}=12$ Hz, 1H, H-1α);<br>4.51 (ddd, $J_{7\alpha-6\alpha}=11$ Hz, $J_{7\alpha-6\beta}=5$ Hz, $J_{gem}=2$ Hz, 1H, H-7α);<br>4.22 (d, $J_{gem}=12$ Hz, 1H, H-1β); 4.15 (dd, $J_{4-OH}=7$ Hz, $J_{4-5}=2$ Hz, 1H, H-4);<br>3.98 (dt, $j_{7\beta-6}=12$ Hz, $J_{gem}=3$ Hz, 1H, H-7β); 3.87 (d, $J_{18-17}=5$ Hz, 1H, H-18);<br>3.57 (d, $J_{19a-20}=5$ Hz, 1H, H-19a); 3.12 (d, $J_{gem}=4$ Hz, 1H, H-2'α);<br>2.81 (d, $J_{gem}=4$ Hz, 1H, H-2'β); 2.67 (d, $J_{OH-4}=7$ Hz; 1H, OH);<br>2.48 (dd, $J_{gem}=15$ Hz, $J_{17\alpha-16}=8$ Hz, 1H, H-17α); 2.36 (mc, 1H, H-5);<br>2.23 (dt, $J_{gem}=15$ Hz, $J_{17\beta-16,17\beta-18}=5$ Hz, 1H, H-17β);<br>1.97 (AB, $J_{AB}=20$ Hz, 1H, H-22α); 1.93 (AB, $J_{AB}=21$ Hz, 1H, H-6α);<br>1.91 (AB, $J_{AB}=20$ Hz, 1H, H-22β); 1.90 (AB, $J_{AB}=21$ Hz, 1H, H-23α);<br>1.78 (AB, $J_{AB}=21$ Hz, 1H, H-6β); 1.75 (s, 3H, $CH_3$-26);<br>1.69 (AB, $J_{AB}=21$ Hz, 1H, H-23β); 0.87 (d, $J_{-5}=7$ Hz, 3H, $CH_3$-24);<br>0.84 (s, 3H, $CH_3$-25) ppm | | | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | | |

| NMR | $^1H$ decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>174.7 (s, C-3), 166.1 (s, C-9), 165.4 (s, C-14), 141.2 (s, C-21),<br>138.9 (d, C-12), 138.8 (d, C-11), 127.5 (d, C-10), 125.8 (d, C-13),<br>117.9 (d, C-20), 78.9 (d, C-18), 75.5 (d, C-16), 74.2 (d, C-4),<br>66.9 (d, C-19a), 65.2 (s, C-18a), 63.5 (t, C-1), 61.1 (t, C-7),<br>49.5 (s, C-16a), 47.8 (t, C-2'), 44.2 (s, C-23a), 34.9 (t, C-17),<br>33.2 (d, C-)5, 32.2 (t, C-6), 27.5 (t, C-22), 23.3 (q, C-26),<br>20.0 (t, C-23), 10.0 (q, C-24), 7.3 (q, C-25) ppm | | | |
| Frequency | 100 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV | | | |
|---|---|---|---|
| $\lambda_{max}$ | 1) 207; 2) 259nm | $\varepsilon_{max}$ | 1) 5662; 2) 23362 |
| Solvent | methanol | | |
| Sample conc. | $2.945 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Beckman UV 5230 | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.28 | | |
| Solvent | – | Solvent system | 1% MeOH/99% $CH_2Cl_2$ |
| Saturated atmosphere | $I_2$ → yellow/brown | Detection/Color | 254 nm/violet |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel $60F_{254}$/Merck 5562 |

| GLC | not available |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

Contact with eyes causes cerato conjunctivitis.
0.0006 γ/ml cause 50% inhibition of Mastocytom P-815 [2].

10. References


[1] Tamm, C., Breitenstein W., *Helv. Chim. Acta 58*, 1975, 1172 pp. *($^{13}$C-NMR)*

[2] Tamm, C. et al., *Helv. Chim. Acta 45*, 1962, 839–53 *(isolation, UV, IR, biologic activity)*

[3] Tamm, C. et al., *Helv. Chim. Acta 48*, 1965, 157 pp. *(UV, IR, $^{1}$H-NMR, synthesis)*

# Verrucarin B

*C. Franke, K. Krohn*

## 1. Name of Compound

Common name

**Verrucarin B**

Synonyms

Muconomycin B

Systematic name

1a,2,12,12a,15a,18-Hexahydro-1a,12a,17-trimethyl-spiro[12,14-methano-3H,19H,20H-oxireno[8,9][1,6,12]trioxacyclooctadecino-[3,4-d]-[1] benzopyran-13(14H),2′-oxirane]-5,10,22,(22aH)trione

Substance

Makrolid

CAS registry number and other numbers

[2290-11-1] BRN 951589

## 2. Formulas and Molecular Weight

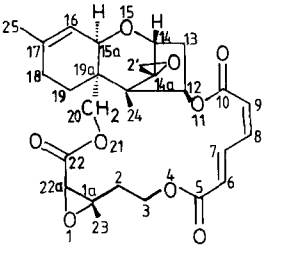
Molecular formular

$C_{27}H_{32}O_9$

Molecular weight

500.54

Structural formula



## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not applicable | Iodine value<br>not available |
| Melting Point mp<br>>330 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{23} = +94°$<br>(c=0.99 g/l in $CHCl_3$) | Saponification value<br>not available |
| Flash point<br>not available | Soluble in<br>methanol, acetone, chloroform | |
| Color<br>colorless | Odor<br>odorless | |

## 4. Occurrence

| |
|---|
| Isolated from the fungus Myrothecium |

## 5. Health Hazard Data

| | |
|---|---|
| Toxicology<br>antifungal and cytostatic activity | Hazard labeling<br>$LD_{50}$ (mice): 7.0 mg/kg intravenous [1] |
| Waste disposal procedures | |

## 6. Transportation and Storage Instructions

| |
|---|
| |

## 7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 500 | Molecular ion | 500 |
| Ionization energy | 70 eV | Ion source temp. | 300 °C |
| Acceleration voltage | 3 kV | Emission current | 1 mA |
| Resolution | 1000 | Scan rate | 3 s/decade |
| Spectrometer type and manufacturer | | Finnigan MAT 8430 | |

| IR * | see ref. [1] and [2] | | |
|---|---|---|---|
| Characteristic peaks<br>1750, 1710, 1635, 1590, 1190, 1080, 1040, 965, 880 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | 2.5 $cm^{-1}$ |
| Spectrometer type and manufacturer | | Perkin Elmer 1420 | |

| NMR | | Nucleus | $^1H$ |
|---|---|---|---|
| Chemical Shifts<br>7.89 (ddd, $J_{7-6}=15$ Hz, $J_{7-8}=12$ Hz, $J_{7-9}<1$ Hz, 1H, H-7);<br>6.63 (t, $J_{8-7,8-9}=11$ Hz, 1H, H-8); 6.17 (d, $J_{9-8}=11$ Hz, 1H, H-9);<br>6.07 (d, $J_{6-7}=16$ Hz, 1H, H-6); 5.85 (dd, $J_{12-13\alpha}=8$ Hz, $J_{12-13\beta}=4$ Hz, 1H, H-12);<br>5.44 (d, $J_{16-15a}=5$ Hz, 1H, H-16); 4.50 (d, $J_{gem}=12$ Hz, 1H, H-20α);<br>4.34 (d, $J_{gem}=12$ Hz, 1H, H-20β); 3.59 (d, $J_{15a-16}=5$ Hz, 1H, H-15a);<br>3.40 (s, 1H, H-22a); 3.14 (d, $J_{gem}=4$ Hz, 1H, H-2′α);<br>2.83 (d, $J_{gem}=4$ Hz; 1H, H-2′β); 1.74 (s, 3H, $CH_3$-25);<br>1.56 (s, 3H, $CH_3$-23); 0.89 (s, 3H, $CH_3$-24) ppm | | | |
| Frequency | 400 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| NMR | $^1H$ decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>167.8 (s, C-22), 166.3 (s, C-5), 165.4 (s, C-10), 141.2 (s, C-17),<br>138.6 (d, C-8), 138.3 (d, C-7), 127.6 (d, C-6), 126.0 (d, C-9),<br>118.0 (d, C-16), 79.0 (d, C-14), 75.6 (d, C-12), 67.2 (d, C-15a),<br>65.3 (s, C-14a), 63.9 (t, C-20), 61.7 (s, C-1a), 60.8 (t, C-3),<br>58.4 (d, C-22a), 49.3 (s, C-12a), 48.0 (t, C-2′), 43.9 (s, C-19a),<br>37.0 (t, C-2), 35.1 (t, C-13), 27.6 (t, C-18), 23.3 (q, C-25),<br>20.1 (t, C-19), 16.0 (q, C-23), 7.7 (q, C-24) ppm | | | |
| Frequency | 100 MHz | Solvent | $CDCl_3$ |
| Standard | TMS | Sample temp. | room temperature |
| Spectrometer type and manufacturer | | Bruker AM 400 | |

| UV * | see ref. [2] | | |
|---|---|---|---|
| $I_{max}$ | 285.5 nm | $\varepsilon_{max}$ | 23442 |
| Solvent | – | | |
| Sample conc. | – | Cell thickness | – |
| Spectrometer type and manufacturer | | Beckman DK 2 | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.30 | | |
| Solvent | – | Solvent system | 1% MeOH/99% $CH_2Cl_2$ |
| Saturated atmosphere | $I_2$ → yellow/brown | Detection/Color | 254 nm/violet |
| Plate manufacturer | Merck | Plate type/Product no. | Kieselgel $60F_{254}$/Merck 5562 |

| GLC | not available |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

0.003 γ/ml cause 50% inhibition of Mastocytom P-815

10. References


[1] Tamm, C. et al., *Helv. Chim. Acta 45*, 1962, pp. 839–53 *(isolation, UV, IR, biological activity)*

[2] Tamm, C., Gutzwiller, J., *Helv. Chim. Acta 48*, 1965, pp. 177–82 *($^1$H-NMR, UV, IR)*

[3] Tamm, C., Breitenstein, W., *Helv. Chim. Acta 58*, 1975, pp. 1172–80 *($^{13}$C-NMR)*

# Xylostosidine

*L. F. Tietze, C. Bärtels*

## 1. Name of Compound

Common name

**Xylostosidine**

Synonyms

–

Systematic name

(8S,9R,9aS,10aS)-9-Ethenyl-8-(β-D-glucopyranosyloxy)-2,3,9,9a,10,10a-hexahydro-5H,8H-pyrano[4,3-d]thiazolo-[3,2-a]pyridine-5-one

| Substance | Subgroup |
|---|---|
| Alkaloid | Monoterpene Alkaloid |

CAS registry number and other numbers

[74518-57-3]

## 2. Formulas and Molecular Weight

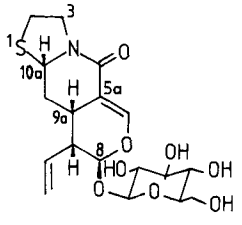
Molecular formular

$C_{18}H_{25}NO_8S$

Molecular weight

415.47

Structural formula




## 3. Physical and Chemical Properties

| State of matter | $d_4^{20}$ | Iodine value |
|---|---|---|
| solid | not applicable | not available |
| **Melting Point mp** | $n_D^{20}$ | **Acid value** |
| 190 °C | not applicable | not available |
| **Boiling point bp** | $[\alpha]_D^{20} = -289.37°$ | **Saponification value** |
| not applicable | (c = 0.56, MeOH) | not available |
| **Flash point** | **Soluble in** | |
| not applicable | water, methanol, acetone, chloroform, etc. | |
| **Color** | **Odor** | |
| white | not applicable | |

4. Occurrence

| |
|---|
| Minor constituent of lonicera xylosteum L. |

5. Health Hazard Data

| | | |
|---|---|---|
| Toxicology | Hazard labeling | not available |
| not available | $LD_{50}$ | not available |
| Waste disposal procedures<br>Combustion | | |

6. Transportation and Storage Instructions

| |
|---|
| Storage temperature: +4 °C |

7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | m/z = 184 | Molecular ion | m/z = 415 |
| Ionization energy | 70 eV | Ion source temp. | 150 °C |
| Acceleration voltage | 3000 V | Emission current | 0.3 mA |
| Resolution | 1000 | Scan rate | 2 s/decade |
| Spectrometer type and manufacturer | | Varian MAT 311 A | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>3416 (OH), 1656 (C=O), 1590 (C=C), 1438, 1074, 1046, 1018, 996 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | Bruker IFS 25 | |

| NMR | | Nucleus | $^{1}H$ |
|---|---|---|---|
| Chemical Shifts<br>1.51 (ddd, J=12.5, 12, 11 Hz, 1H, $H_2C$-$10_{ax}$),<br>2.21 (ddd, J=12.5, 3.5, 4 Hz, 1H, $H_2C$-$10_{eq}$),<br>2.72 (ddd, J=9.5, 5.5, 2 Hz, 1H, *HC*-9),<br>3.04–3.46 (m, 7H, *HC*-2′, *HC*-3′, *HC*-4′, *HC*-6′, $H_2C$-2, *HC*-9a),<br>3.56–3.86 (m, 2H, $H_2C$-$3_{ax}$, $H_2C$-6′a),<br>4.16 (ddd, J=12, 7.5, 7.5 Hz, 1H, $H_2C$-$3_{eq}$),<br>4.89 (dd, J=11, 3.5 Hz, 1H, *HC*-10a),<br>5.34 (dd, J=17.5, 2 Hz, 1H, $H_2C$=-E),<br>5.58 (ddd, J=17.5, 10, 9.5 Hz, 1H, $H_2C$=C*H*),<br>3.93 (dd, J=12, 2 Hz, 1H, $H_2C$-6′b),<br>4.70 (d, J=8 Hz, 1H, *HC*-1′),<br>5.29 (dd, J=10, 2 Hz, 1H, $H_2C$=-Z),<br>5.55 (d, J=2 Hz, 1H, *HC*-8),<br>7.46 (d, J=2.5 Hz, 1H, *HC*-7) ppm | | | |
| Frequency | 200 MHz | Solvent | $CD_3OD$ |
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | Varian XL 200 | |

| NMR | $^{1}H$-decoupled | Nucleus | $^{13}C$ |
|---|---|---|---|
| Chemical Shifts<br>28.5 (C-9a), 28.7 (C-2), 32.6 (C-10), 44.4 (C-9), 49.8 (C-3),<br>62.4 (C-10a), 62.6 (C-6′), 71.4 (C-4′), 74.6 (C-2′), 77.8 (C-3′),<br>78.2 (C-5′), 97.3 (C-8), 99.5 (C-1′), 108.0 (C-6), 120.7 ($CH=CH_2$),<br>133.6 ($CH=CH_2$), 148.9 (C-7), 164.7 (C-5) ppm | | | |
| Frequency | 50.3 MHz | Solvent | $CD_3OD$ |
| Standard | TMS | Sample temp. | 25 °C |
| Spectrometer type and manufacturer | | Varian VXR 200 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | $2.39 \times 10^{-4}$ nm | $\varepsilon_{max}$ | 24322.04 |
| Solvent | methanol | | |
| Sample conc. | $3.71 \times 10^{-5}$ mol/l | Cell thickness | 1 cm |
| Spectrometer type and manufacturer | | Varian Cary 219 | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.46 | | |
| Solvent | methanol | Solvent system | chloroform/methanol (4/1) |
| Saturated atmosphere | – | Detection/Color | UV 254 nm |
| Plate manufacturer | Macherey-Nagel | Plate type/Product no. | SIL G/$UV_{254}$ |

| GLC | not applicable |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

10. References


[1] Chaudhuri, R.K., Sticher, O., Winkler, T., *Helvetia Chim. Acta 63*, 1980, 1045 *(isolation)*

[2] Tietze, L.F., Bärtels, C., Fennen, J., *Liebigs Ann. Chem. 12*, 1989, 1241 *(synthesis)*

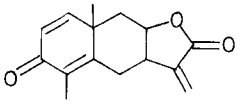
# Yomogin

*Wu Chongming*

## 1. Name of Compound

| | |
|---|---|
| Common name<br>**Yomogin** | |
| Synonyms | |
| Systematic name<br>3a,8a,9,9a-Tetrahydro-5,8a-dimethyl-3-methylene-naphtho-[2,3-b]-furan-2,6(3H,4H)-dione | |
| Substance<br>Sesquiterpene lactone | Subgroup<br>Eudesmanolides lactone |
| CAS registry number and other numbers<br>[10067-18-2] | BRN 1581704 |

## 2. Formulas and Molecular Weight

| | |
|---|---|
| Molecular formular<br>$C_{15}H_{16}O_3$<br><br>Molecular weight<br>244.29 | Structural formula<br>  |

## 3. Physical and Chemical Properties

| | | |
|---|---|---|
| State of matter<br>solid | $d_4^{20}$<br>not available | Iodine value<br>not available |
| Melting Point mp<br>207-209 °C | $n_D^{20}$<br>not applicable | Acid value<br>not applicable |
| Boiling point bp<br>not available | $[\alpha]_D^{20} = -109°$<br>(c=0.10, chloroform) | Saponification value<br>not applicable |
| Flash point<br>not available | Soluble in<br>chloroform, methanol | |
| Color<br>colorless | Odor<br>odorless | |

## 4. Occurrence

| |
|---|
| Several species of the genus Artemisia (Compositae) |

## 5. Health Hazard Data

| Toxicology | Hazard labeling | |
|---|---|---|
| not available | $LD_{50}$ | not available |
| Waste disposal procedures | | |

## 6. Transportation and Storage Instructions

| |
|---|
| |

## 7. Spectroscopic Data

| MS | | | |
|---|---|---|---|
| Base peak | 105 | Molecular ion | 244 (66%) |
| Ionization energy | 70 eV | Ion source temp. | – |
| Acceleration voltage | – | Emission current | – |
| Resolution | – | Scan rate | – |
| Spectrometer type and manufacturer | | MM 7070H | |

| IR | | | |
|---|---|---|---|
| Characteristic peaks<br>1758, 1650, 1616, 1601, 1132, 1018, 950, 830 $cm^{-1}$ | | | |
| Sample preparation | KBr | Resolution | – |
| Spectrometer type and manufacturer | | PE-599B | |

| NMR | | | Nucleus | $^1H$-$^1H$-correlation |
|---|---|---|---|---|
| Chemical Shifts<br>1.34 (s, 3H, $C_{10}$-$CH_3$);<br>2.40 (d, 1H, $C_8$-H);<br>5.71 (s, 1H, $C_{13}$-$H_a$);<br>6.27 (d, J = 10 Hz, 1H, $C_1$-H) ppm | | 1.97 (s, 3H, $C_4$-$CH_3$);<br>2.40 (q, 1H, $C_9$-$H_B$);<br>6.24 (s, 1H, $C_{13}$-$H_b$); | | 1.80 (q, 1H, $C_9$-$H_A$)<br>3.00 (m, 2H, $C_6$-$CH_2$);<br>6.22 (d, J = 10 Hz, 1H, $C_2$-H); |
| Frequency | 90 MHz | | Solvent | $CDCl_3$ |
| Standard | TMS | | Sample temp. | – |
| Spectrometer type and manufacturer | | | EM-390 | |

| UV | | | |
|---|---|---|---|
| $I_{max}$ | 239, 263 nm | $\varepsilon_{max}$ | 11.870, 8.309 |
| Solvent | methanol | | |
| Sample conc. | – | Cell thickness | – |
| Spectrometer type and manufacturer | | SPECORD UV-VIS | |

8. Chromatographic Data

| TLC | | | |
|---|---|---|---|
| Rf Value | 0.32 | | |
| Solvent | ethanol | Solvent system | petroleum ether : ethyl acetate (6 : 4) |
| Saturated atmosphere | – | Detection/Color | UV 254 (quenching) |
| Plate manufacturer | hand made | Plate type/Product no. | Silica gel-$F_{254}$, 0.25 mm |

| GLC | not applicable |
|---|---|

| HPLC | not available |
|---|---|

9. Remarks

10. References

[1] Geissman, T.A.: J. Org. Chem. *31*(3), 1966, 2523
[2] Wu, C.M. et al.: Chinese Bulletin of Botany *3*(3), 1985, 34

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# Part II

# Structural Formulas and Spectra

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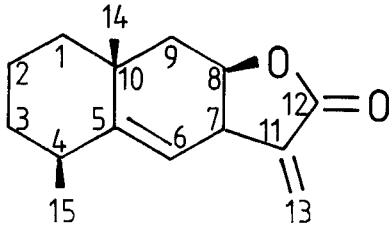
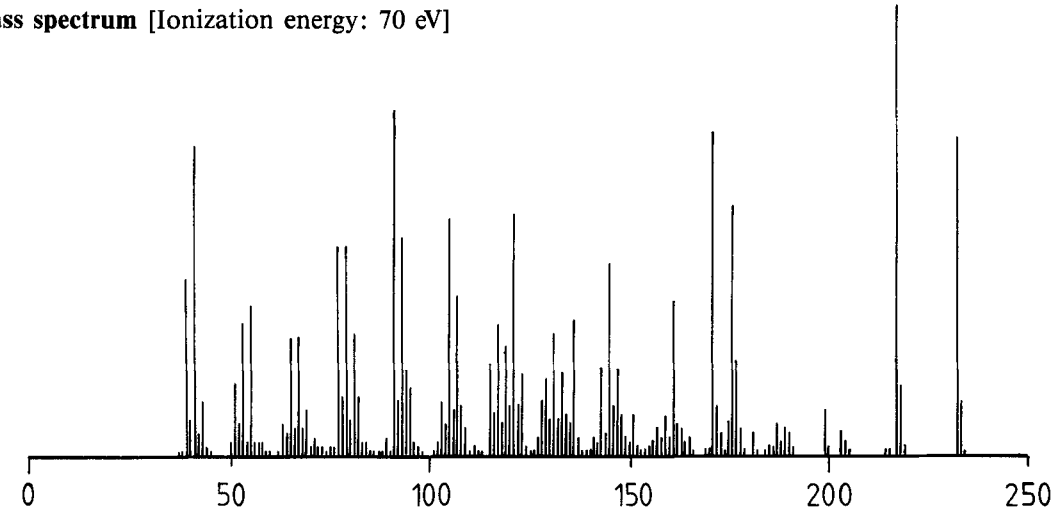
# Alantolactone

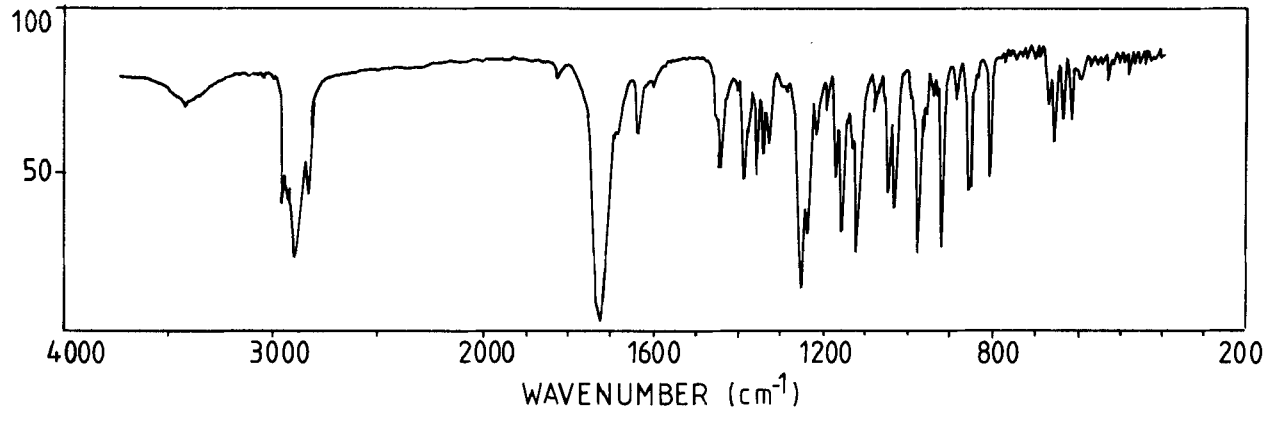
$C_{15}H_{20}O_2$



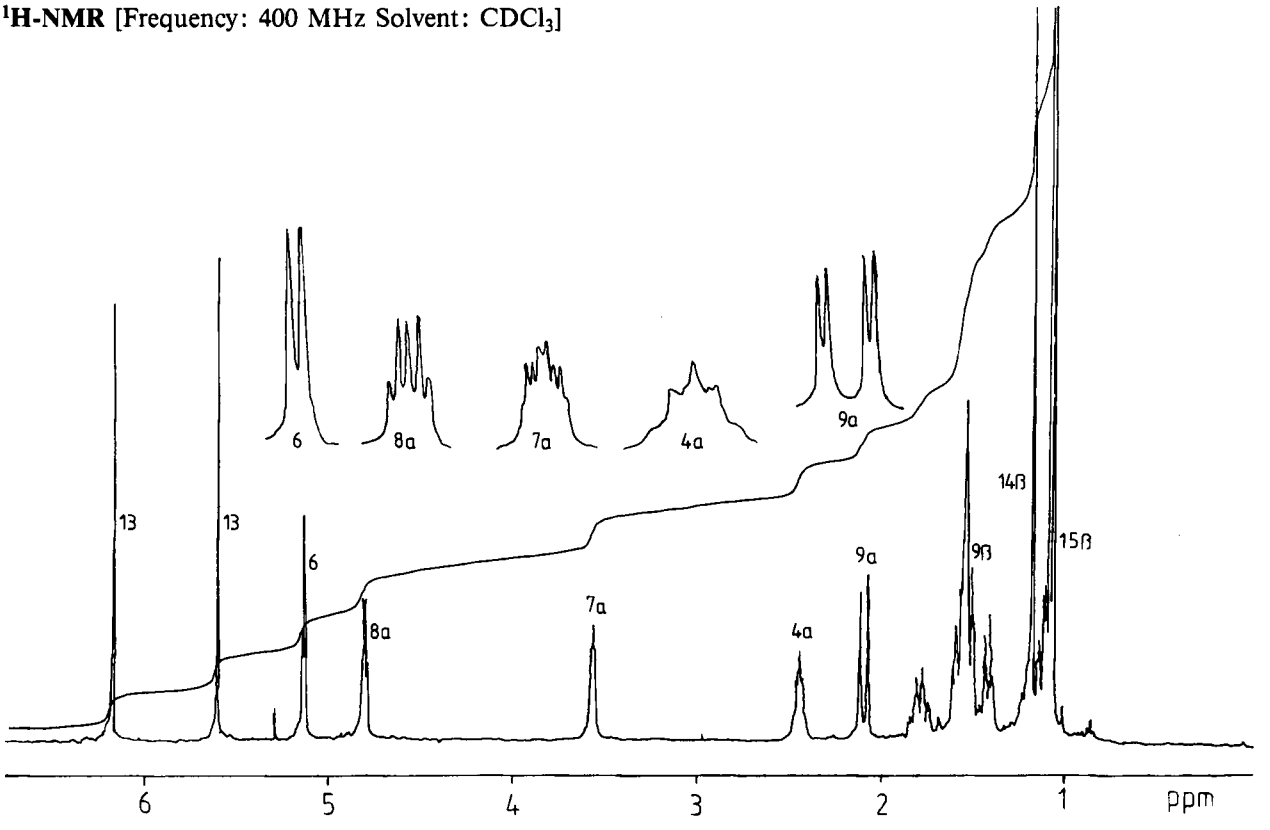

**Mass spectrum** [Ionization energy: 70 eV]



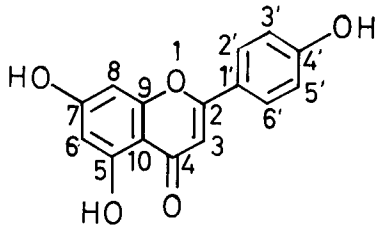

**IR spectrum** [Preparation: KBr]



**$^1$H-NMR** [Frequency: 400 MHz Solvent: $CDCl_3$]

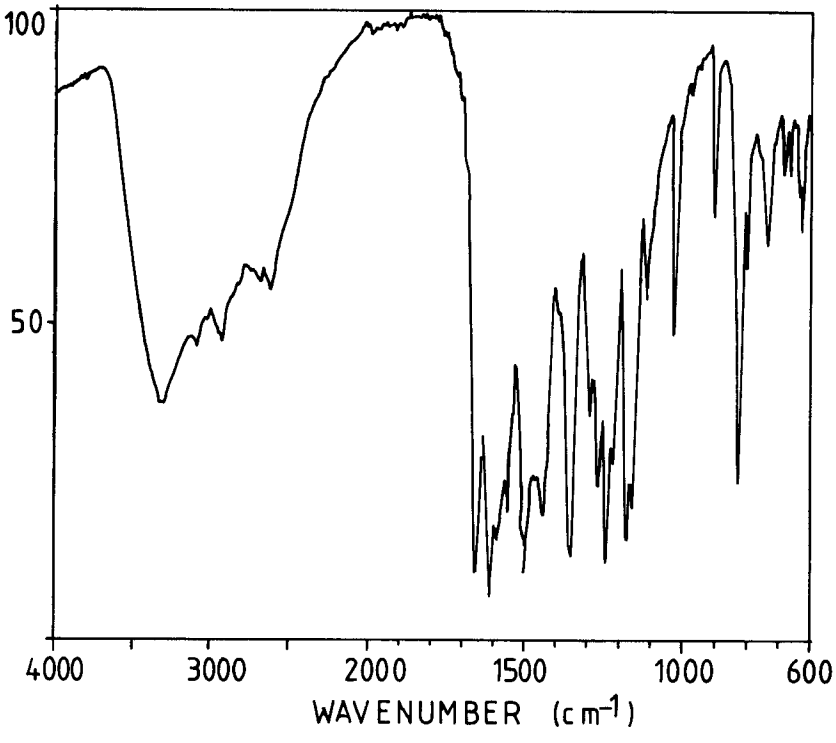


# Apigenin

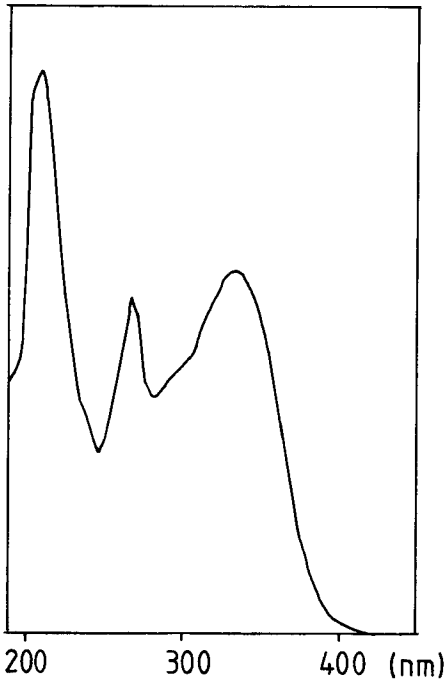



$C_{15}H_{10}O_5$

**IR spectrum** [KBr]




**UV spectrum** [Solvent: methanol]



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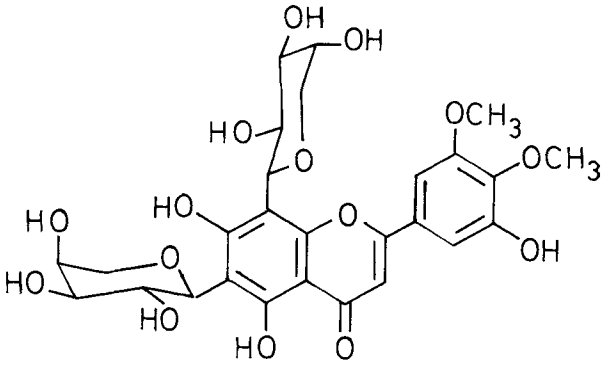
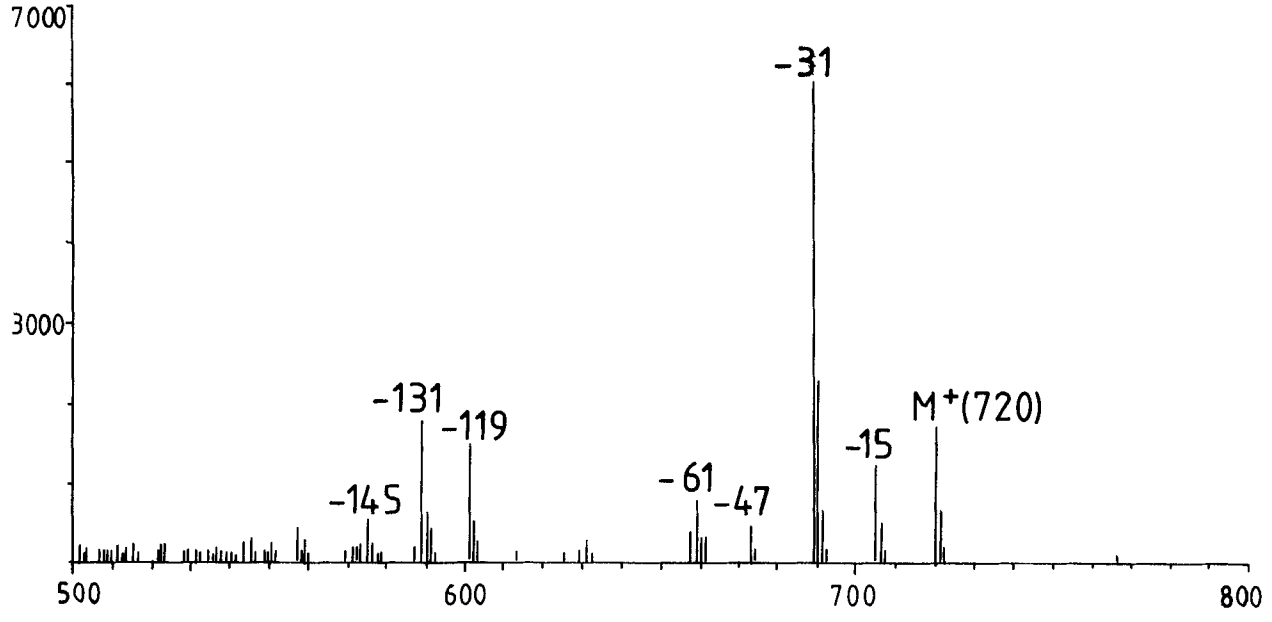
# Apometzgerin-6,8-di-C-arabinoside

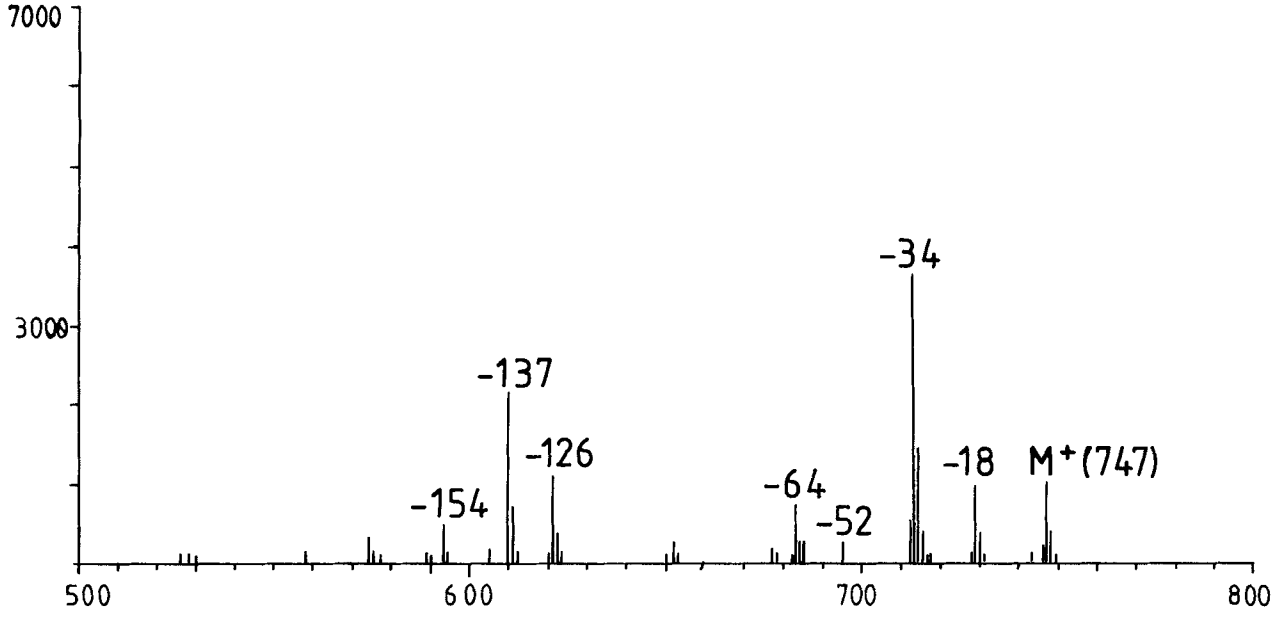
$C_{27}H_{30}O_{15}$



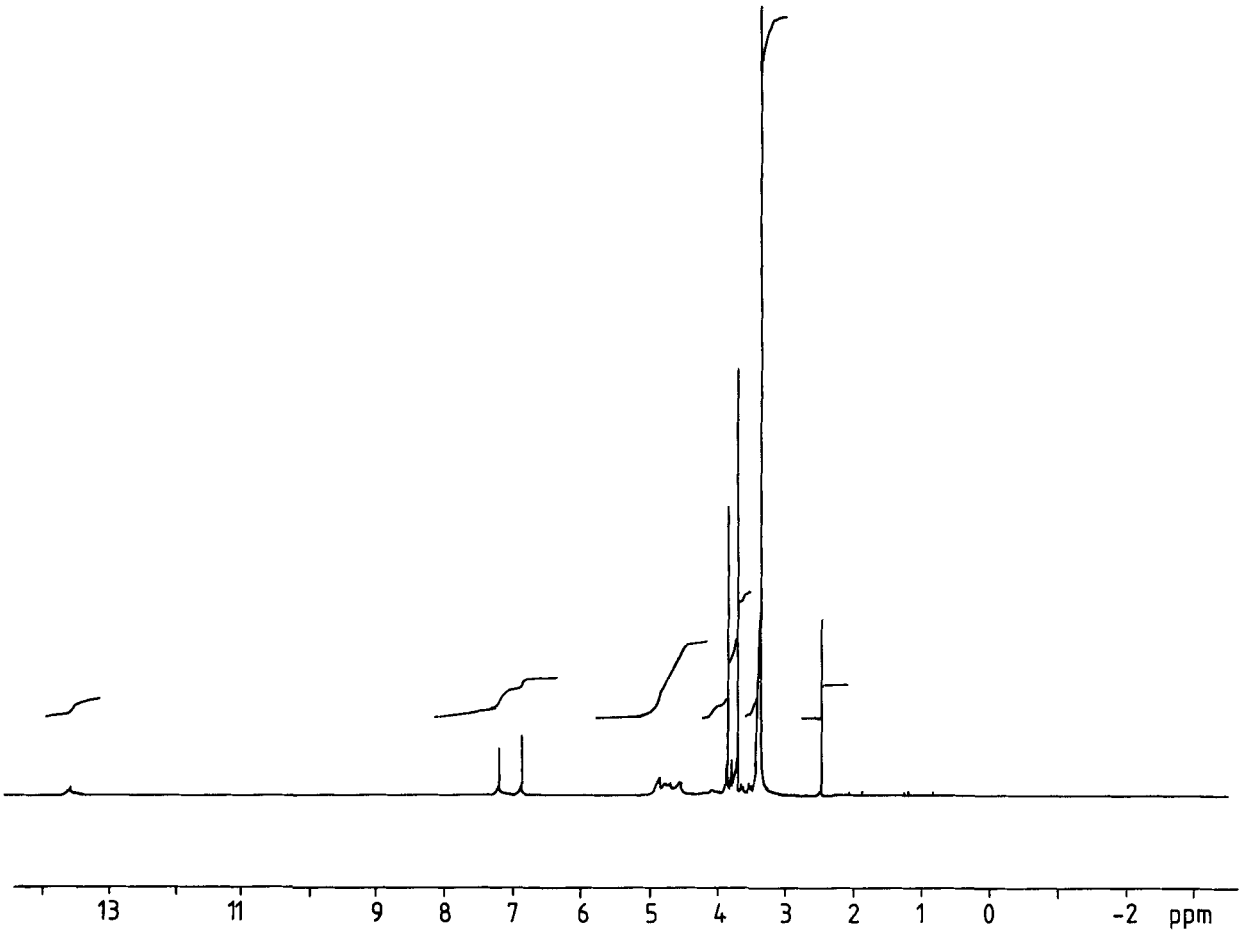

**Mass spectrum** of PM-derivative [90 eV]



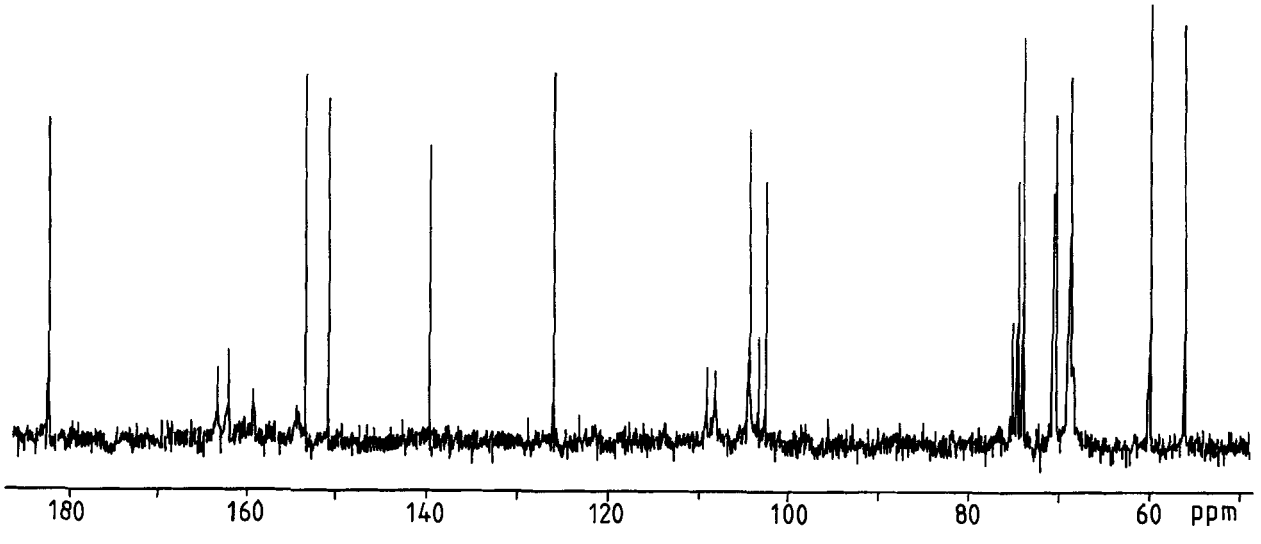

**Mass spectrum** of PDM-derivative [90 eV]



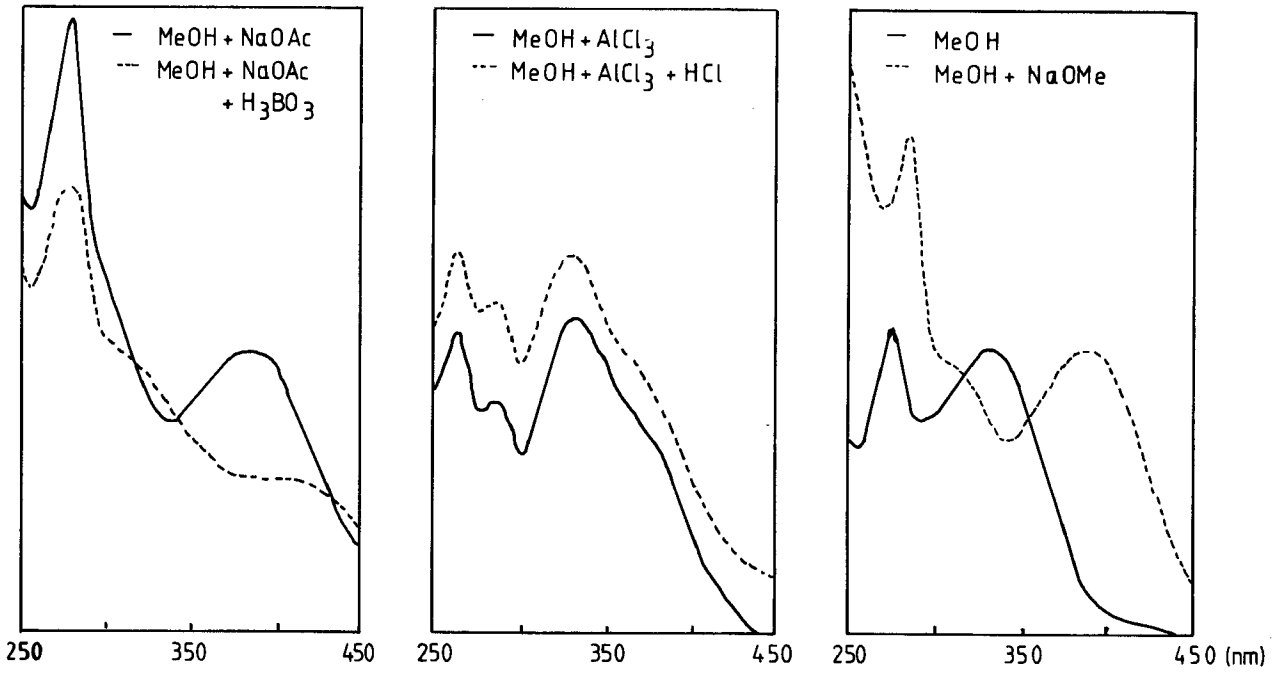
**$^1$H-NMR** [400 MHz, DMSO-$d_6$]




**$^{13}$C-NMR** [100 MHz, DMSO-$d_6$]



**UV spectrum** [methanol]



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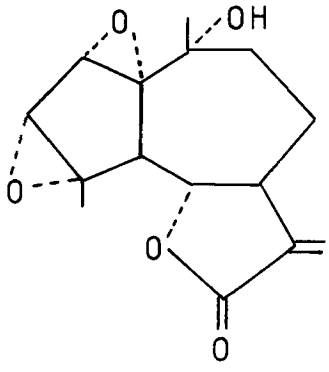
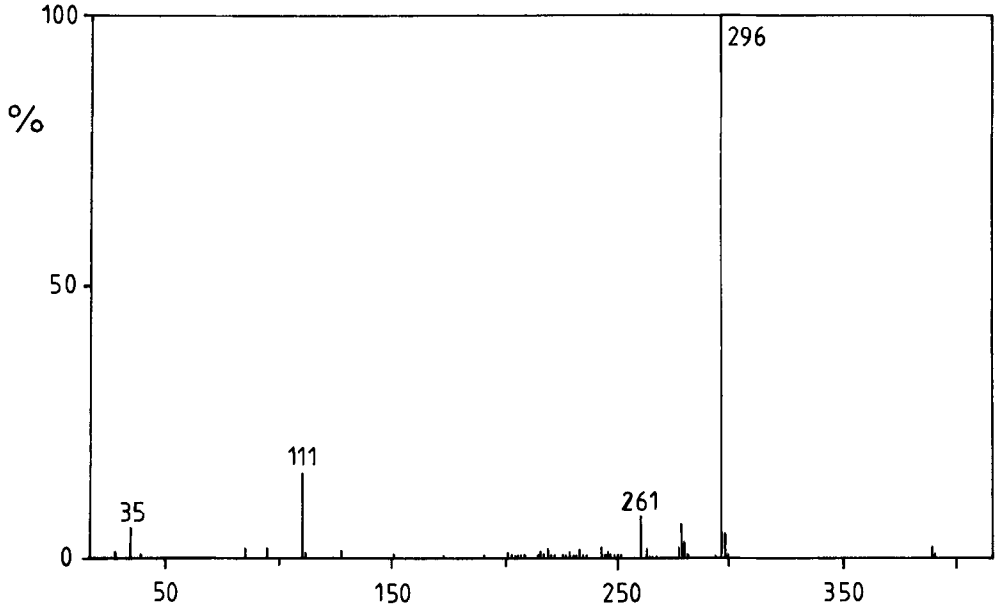
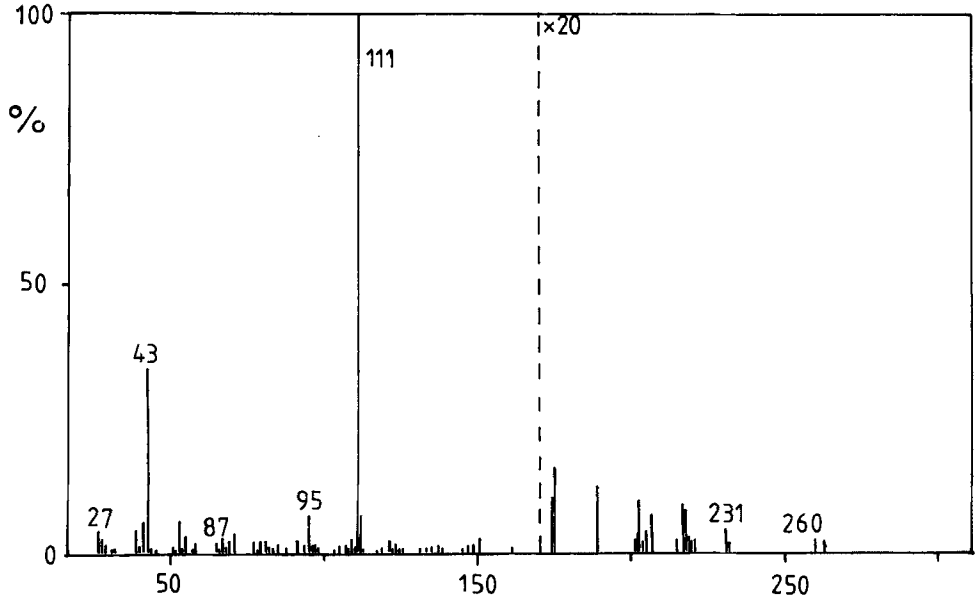
# Artecanin

$C_{15}H_{18}O_5$

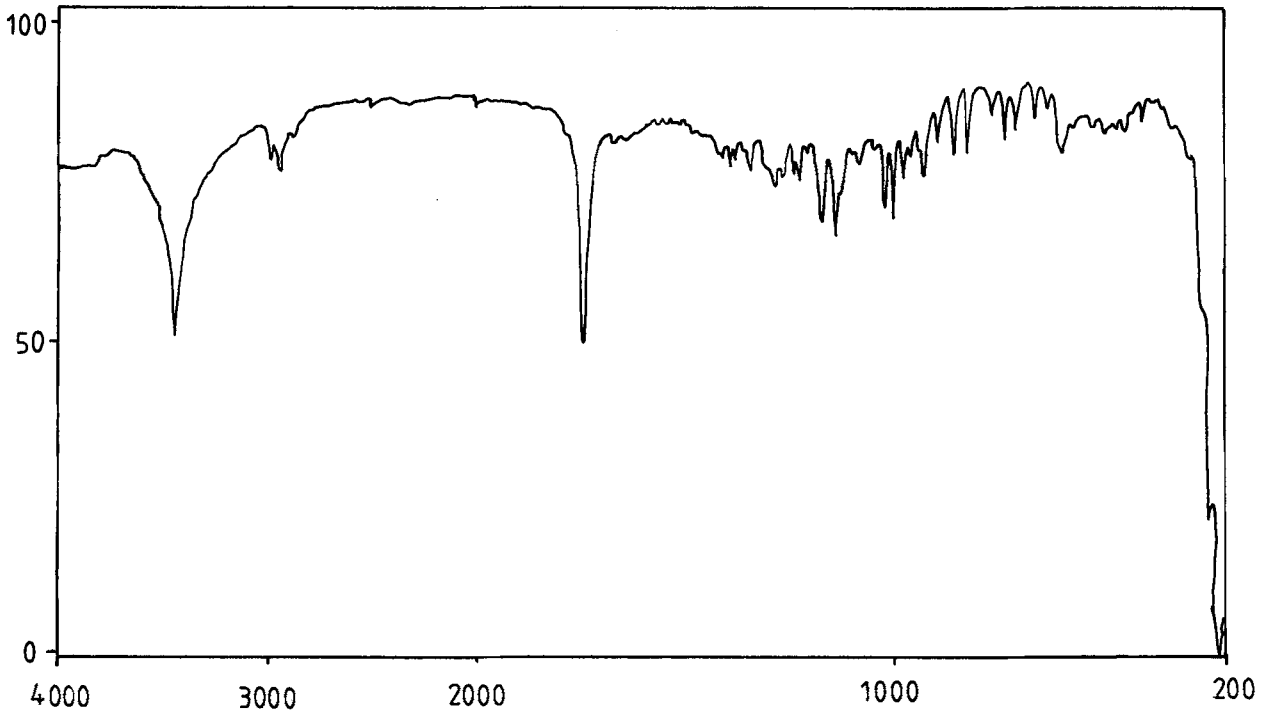



**Mass spectrum** [70 eV]

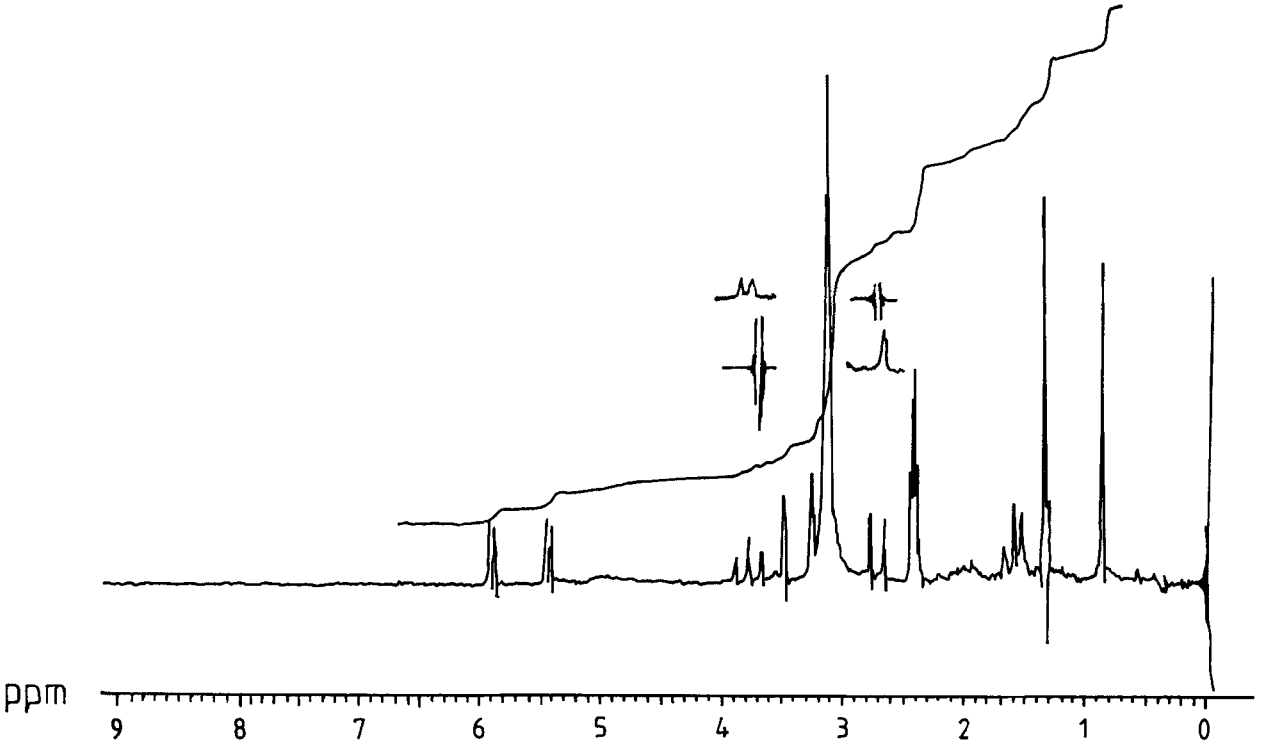


**IR spectrum** [KBr]

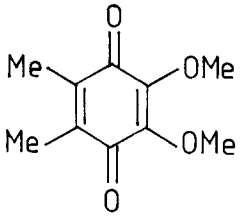



**$^1$H-NMR** [90 MHz, DMSO-$d_6$]

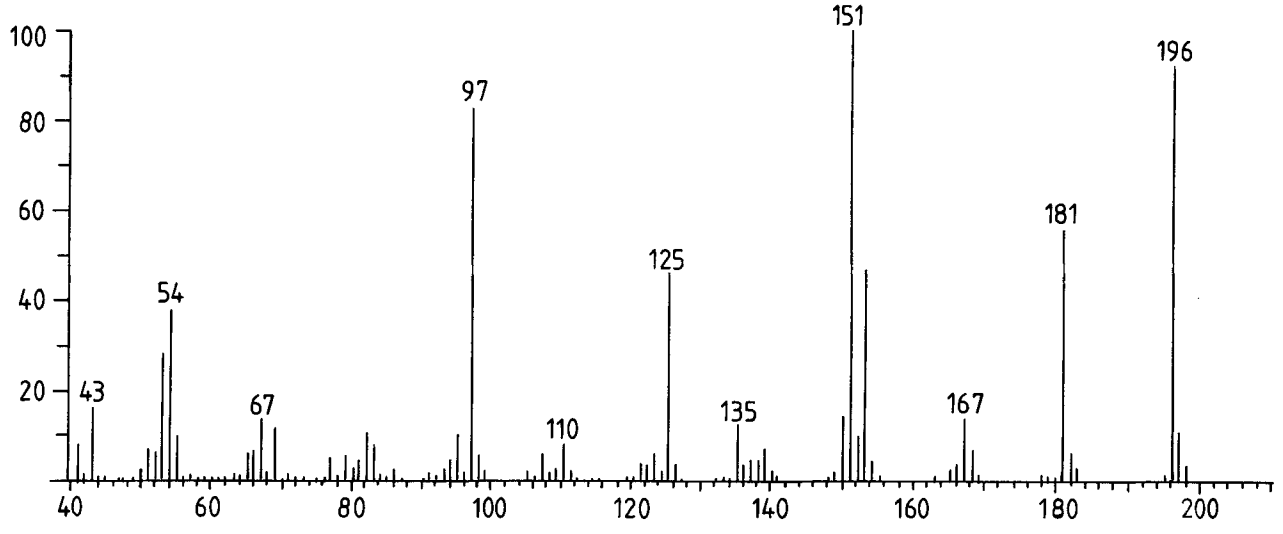


# Aurantiogliocladin

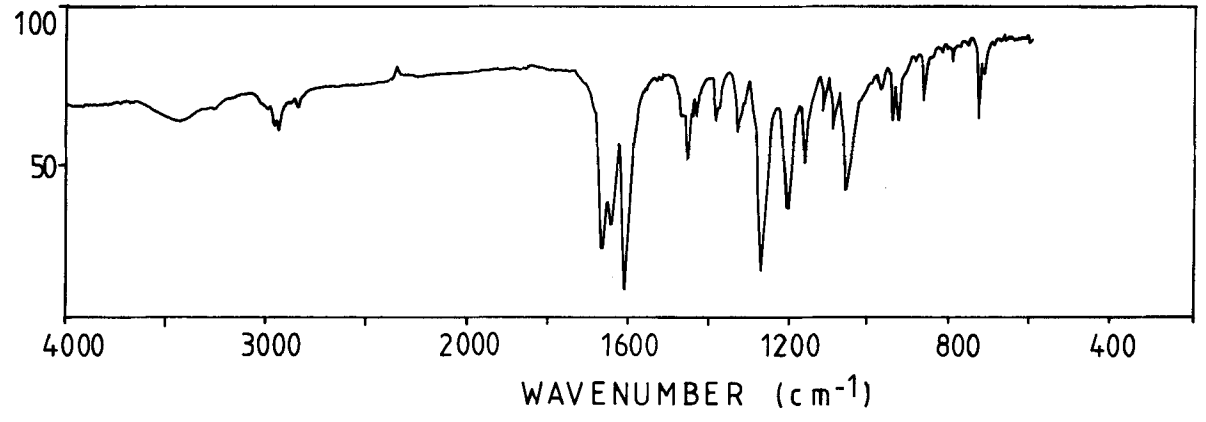
$C_{10}H_{12}O_4$



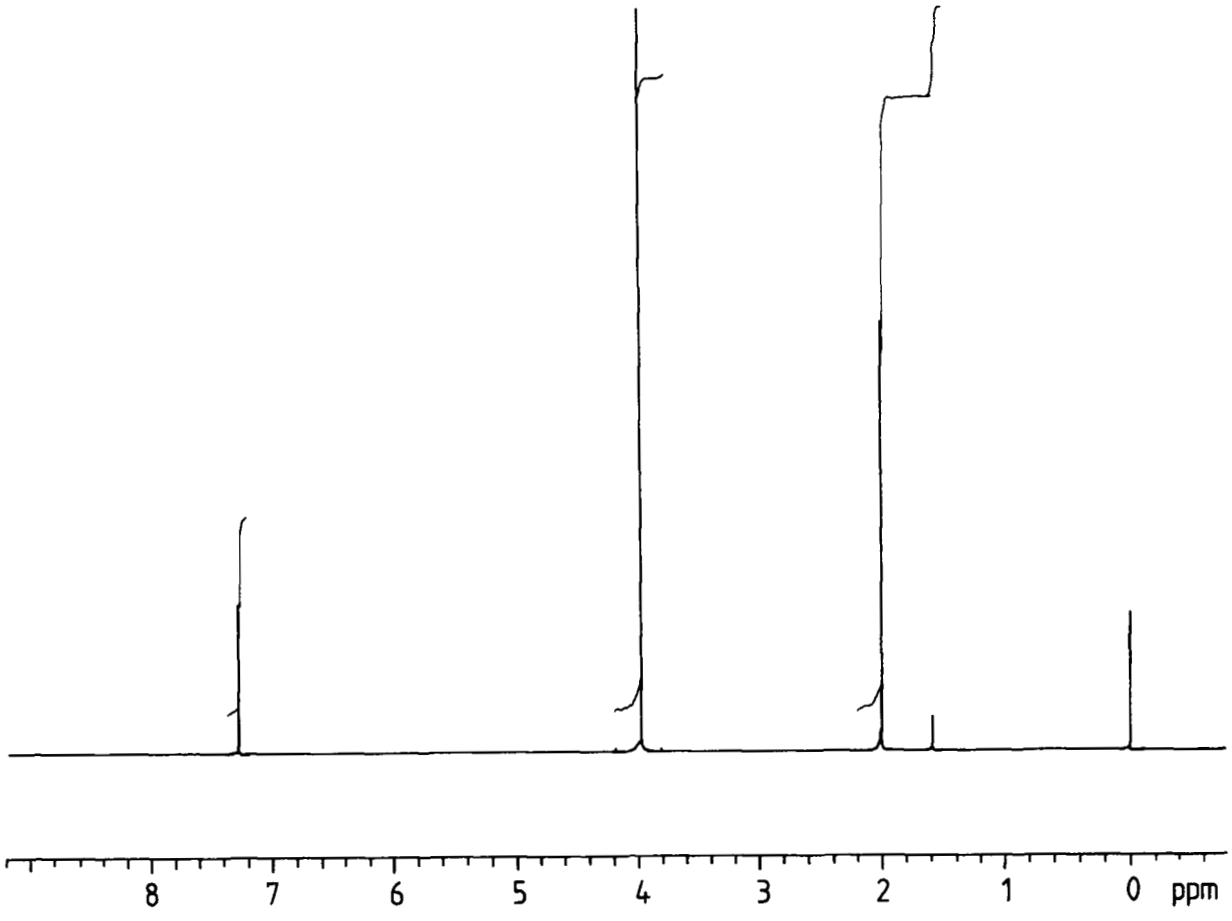

**Mass spectrum** [70 eV]



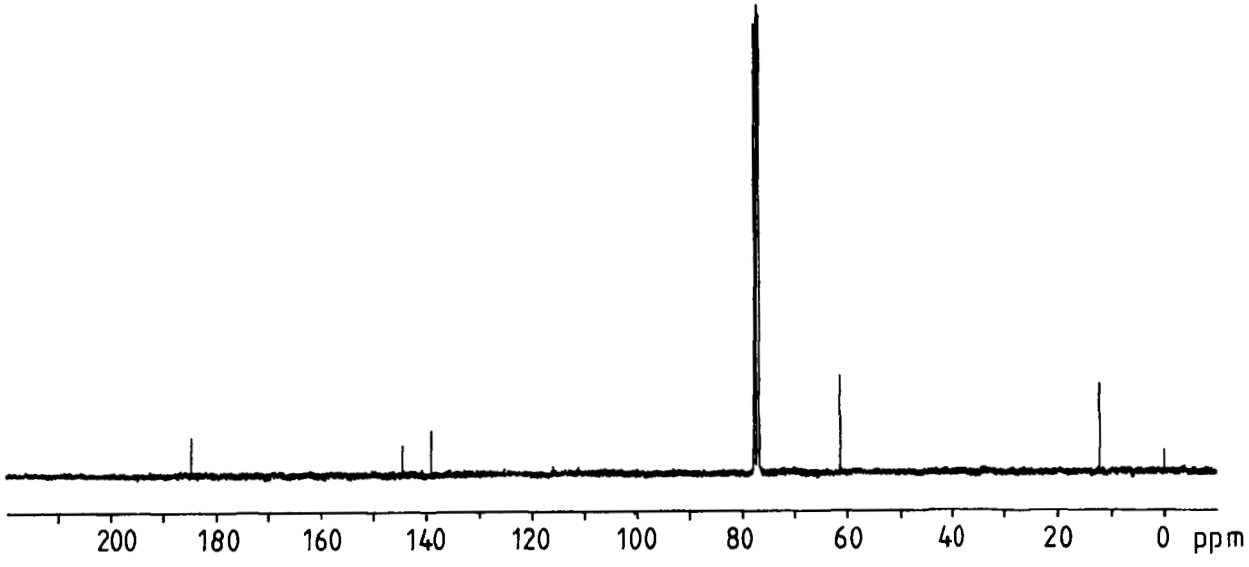

**IR spectrum** [KBr]



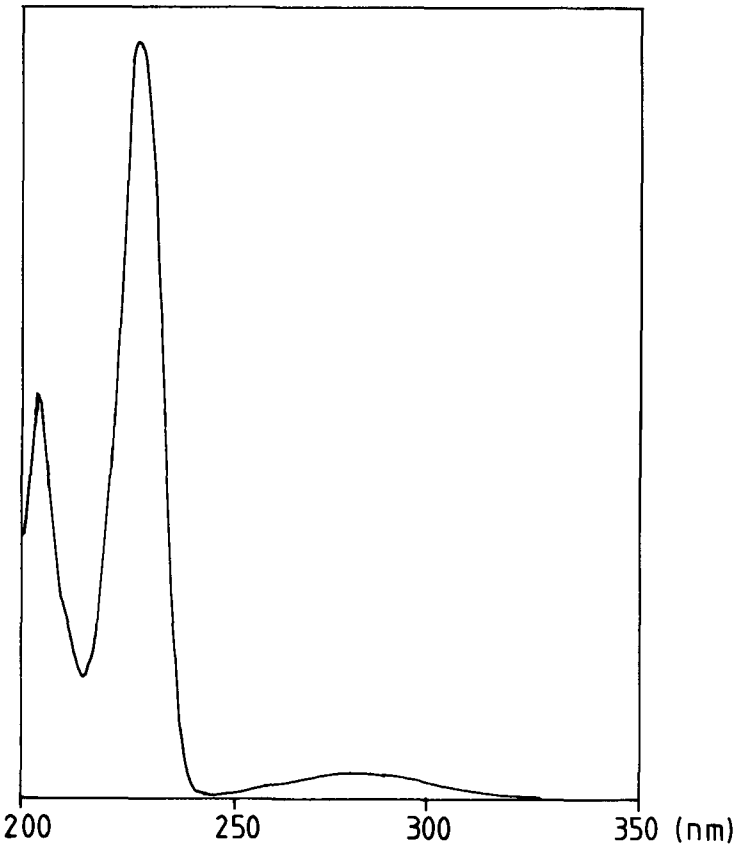
**$^1$H-NMR** [400 MHz, $CDCl_3$]




**$^{13}$C-NMR** [100 MHz, $CDCl_3$]



**UV spectrum** [methanol]



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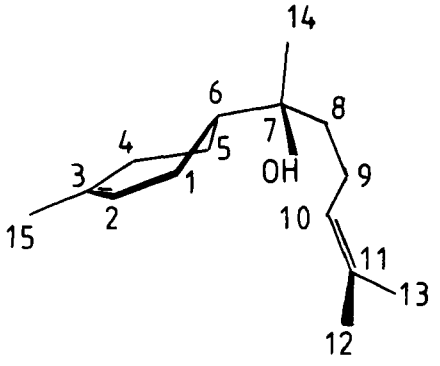
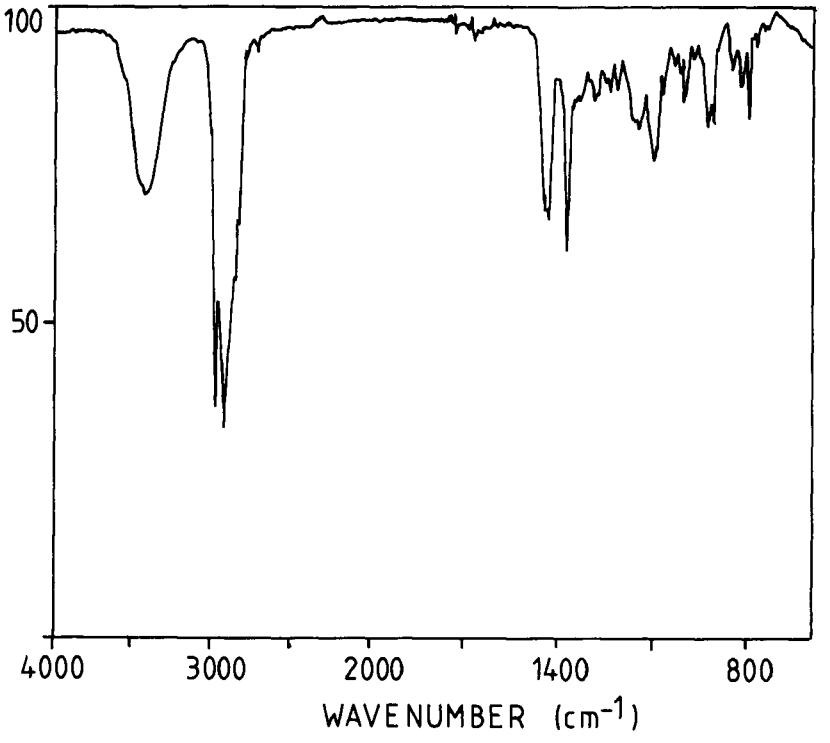
# (-)-α-Bisabolol

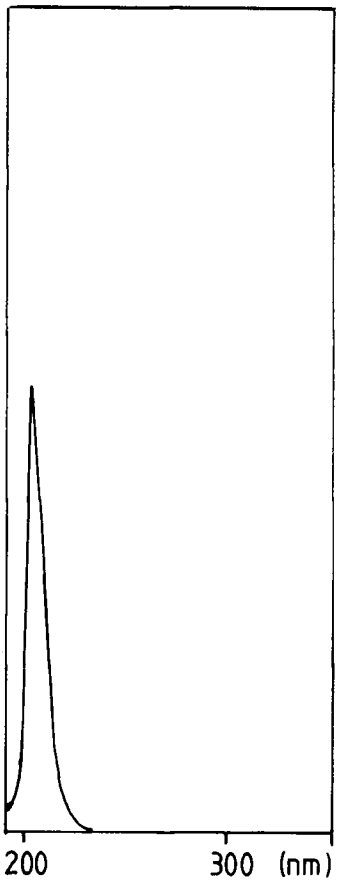
$C_{15}H_{26}O$




**IR spectrum** [capillary film]

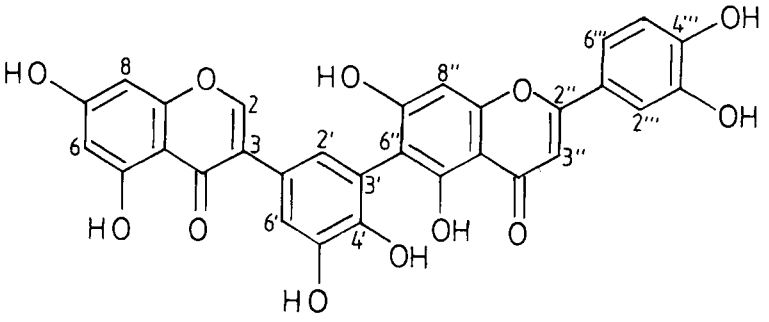


**UV spectrum** [methanol]

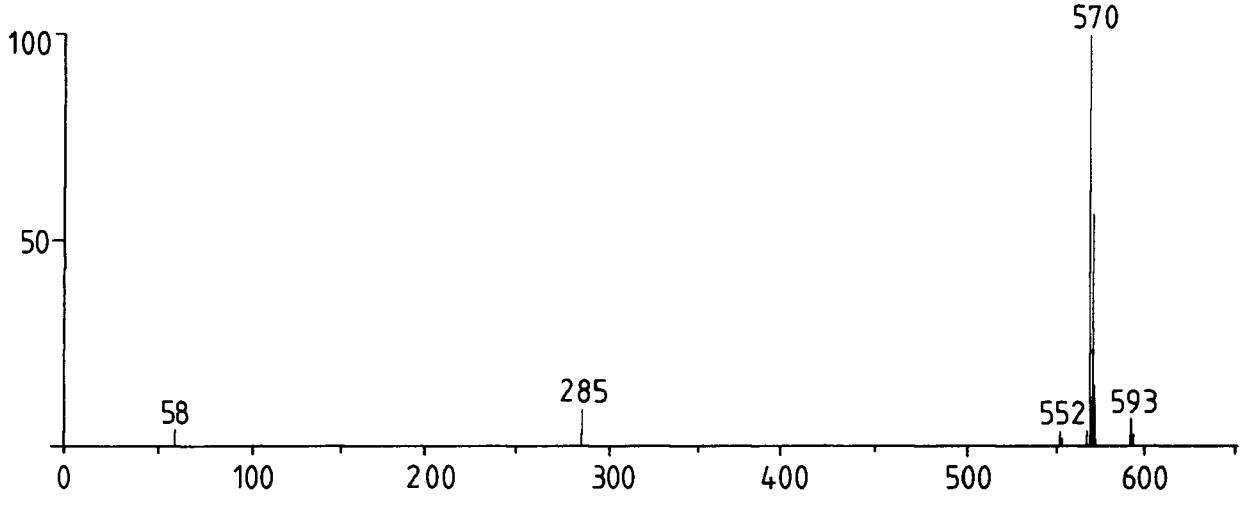


# Bryoflavone

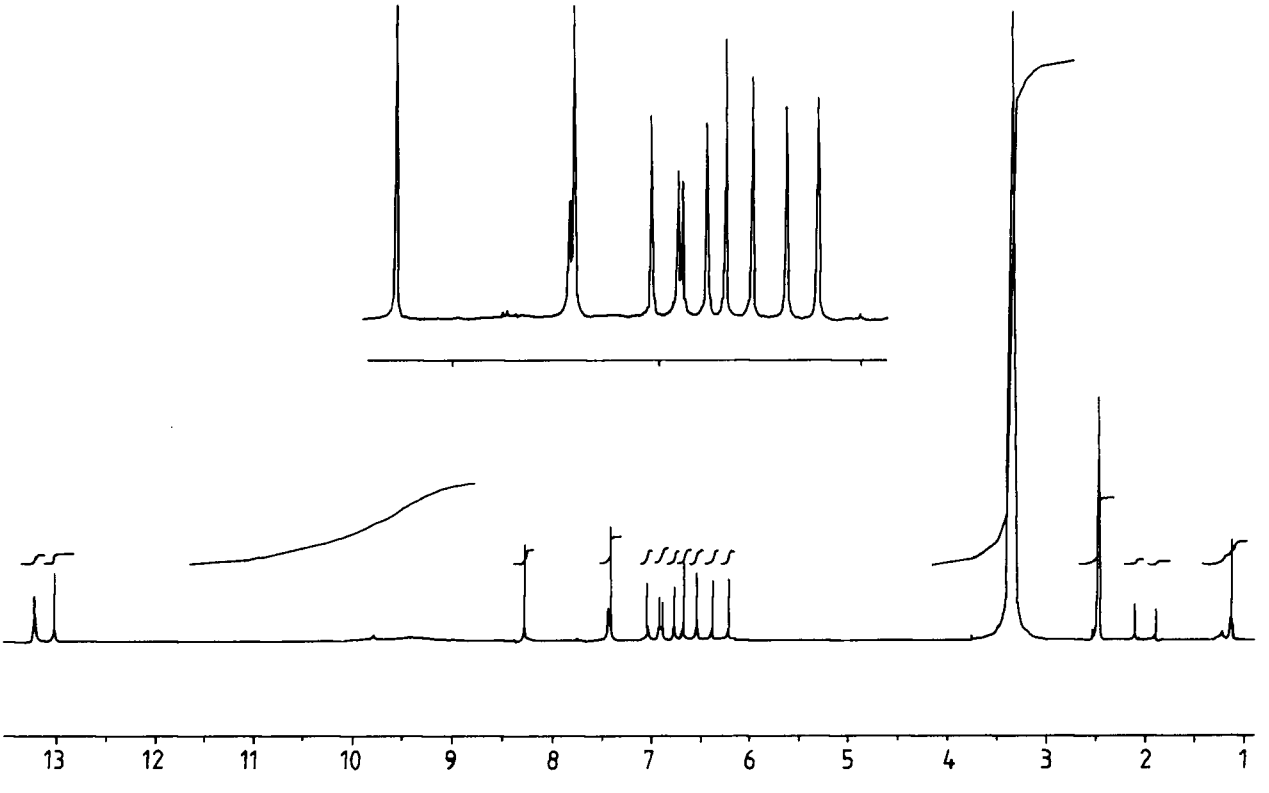
$C_{30}H_{18}O_{12}$



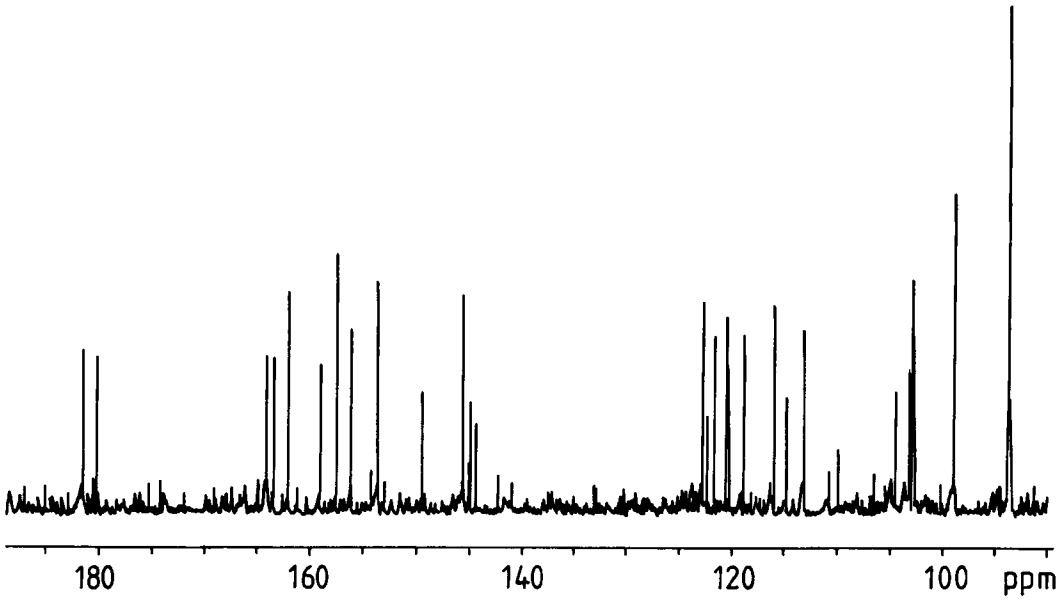

**Mass spectrum**



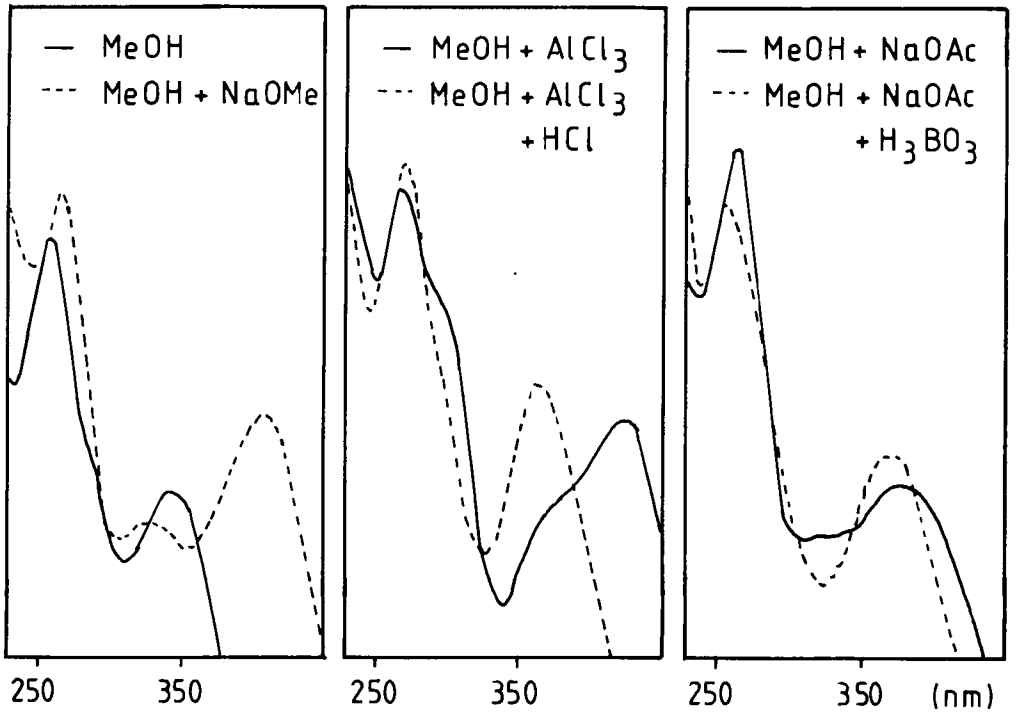

**$^1$H-NMR** [400 MHz, DMSO-$d_6$]



**$^{13}$C-NMR** [100 MHz, DMSO-$d_6$]

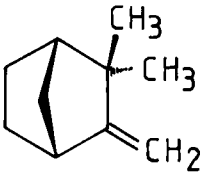



**UV spectra**

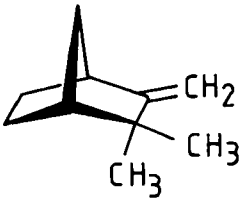


# Camphene

$C_{10}H_{16}$

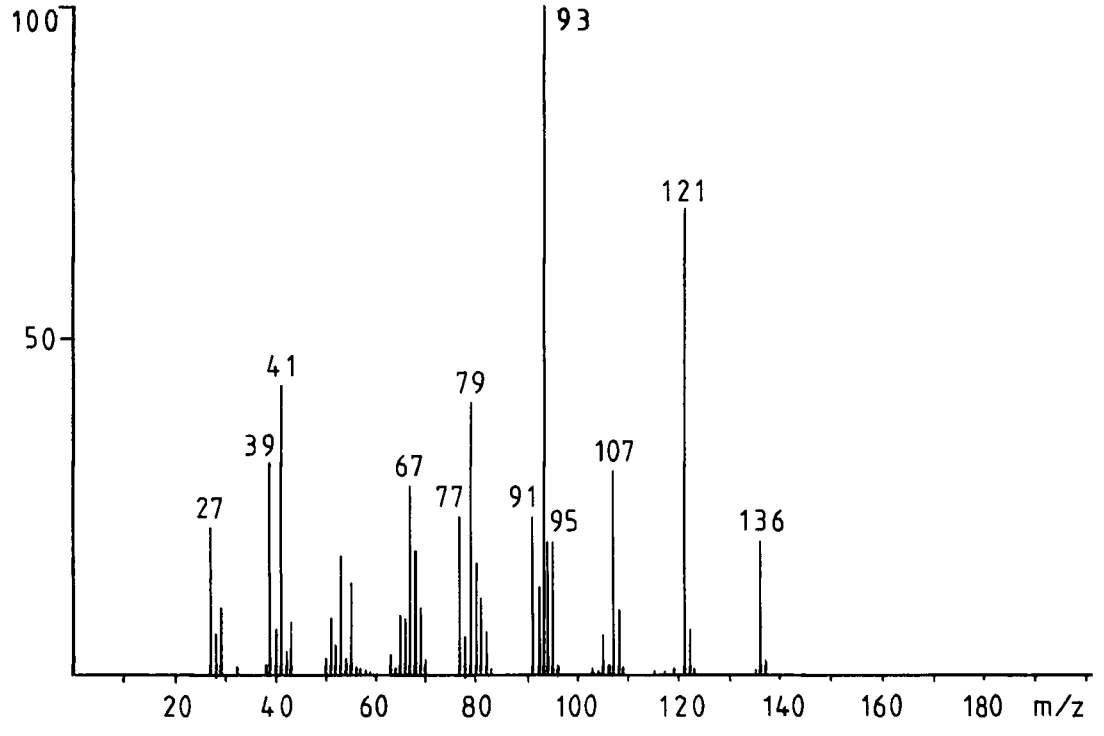



(1S,4R)-(-)-camphene

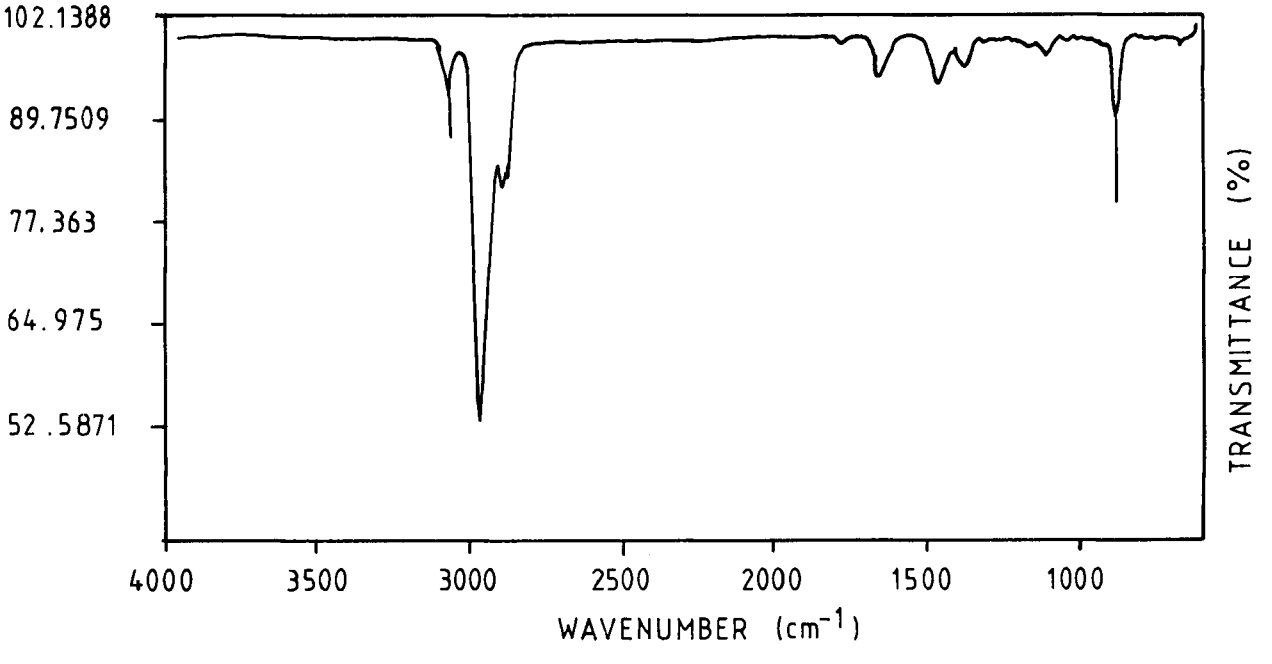



(1R,4S)-(+)-camphene

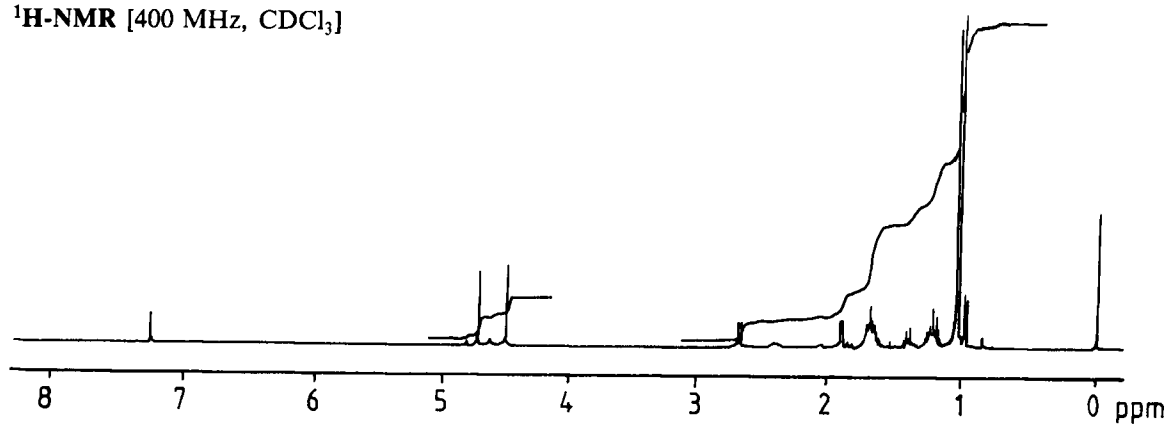
**Mass spectrum** [70 eV]



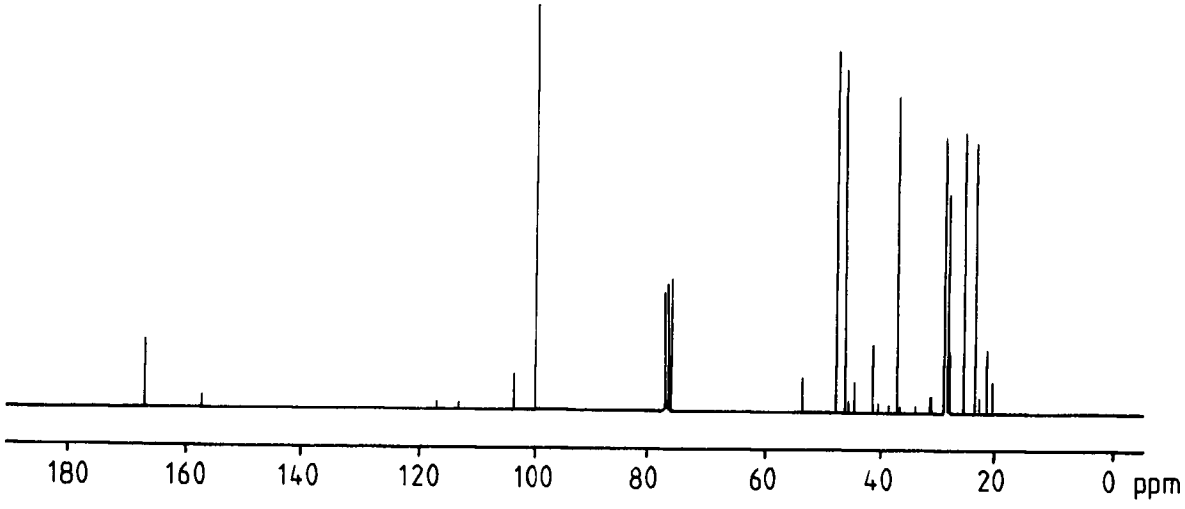

**IR spectrum** [GC-FTIR gas phase]



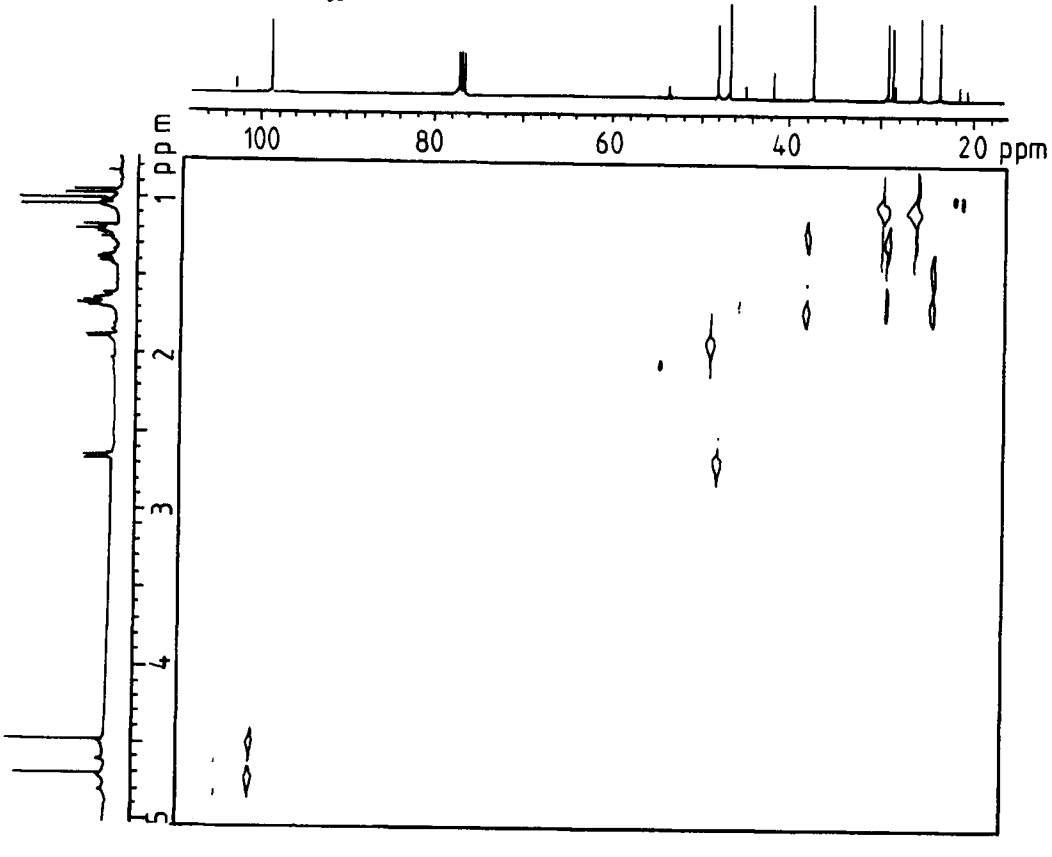
**$^{1}$H-NMR** [400 MHz, $CDCl_3$]




**$^{13}$C-NMR** [100 MHz, $CDCl_3$]



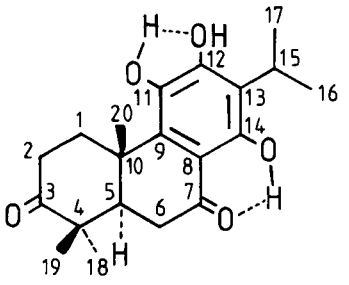

**2D-NMR** [400 MHz, $CDCl_3$]

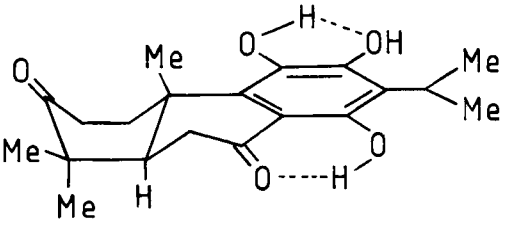



Camphene

# Candelabrone

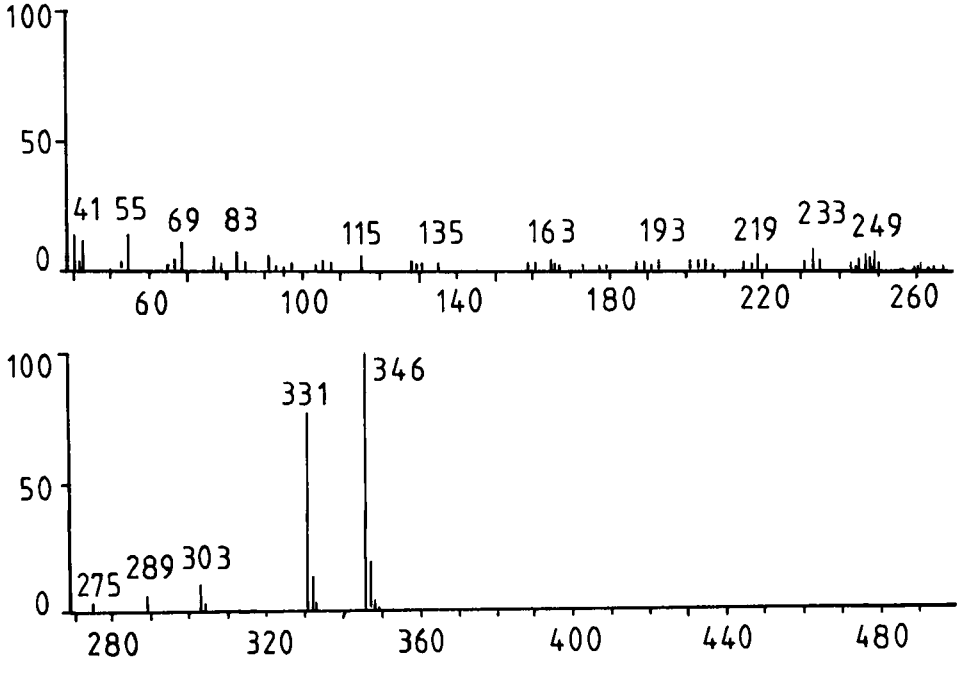
$C_{20}H_{26}O_5$



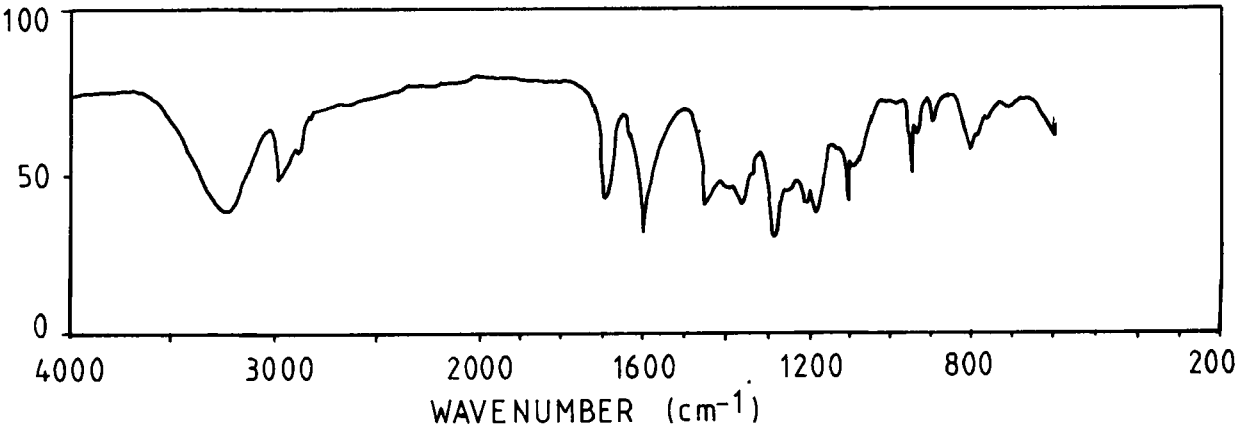


The proposed conformation

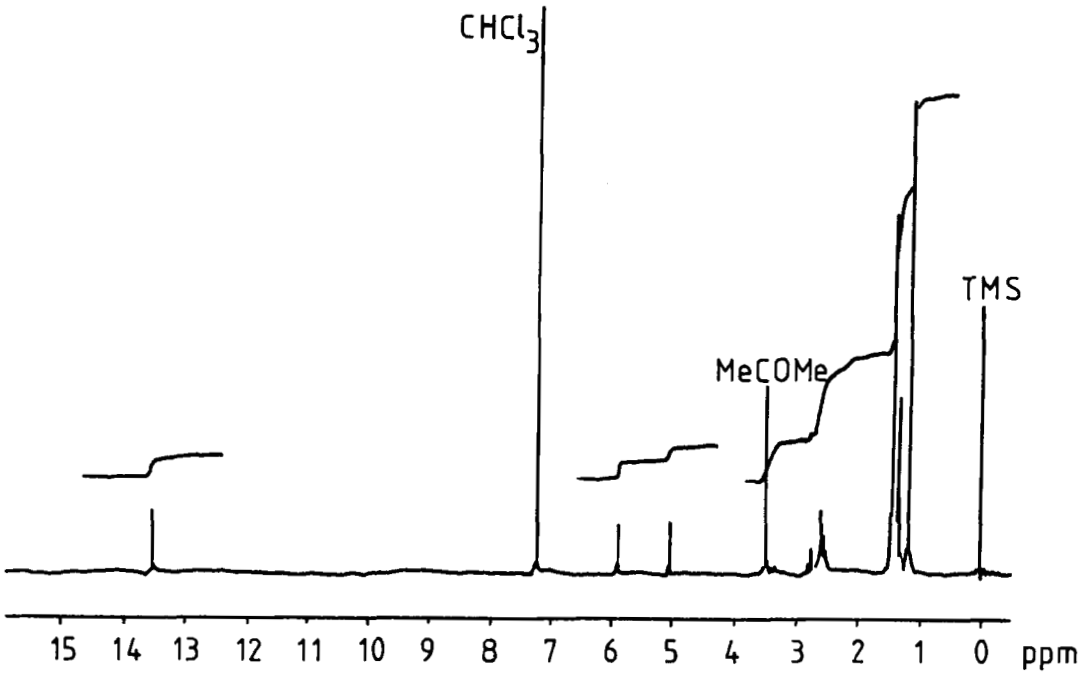
**Mass spectrum**



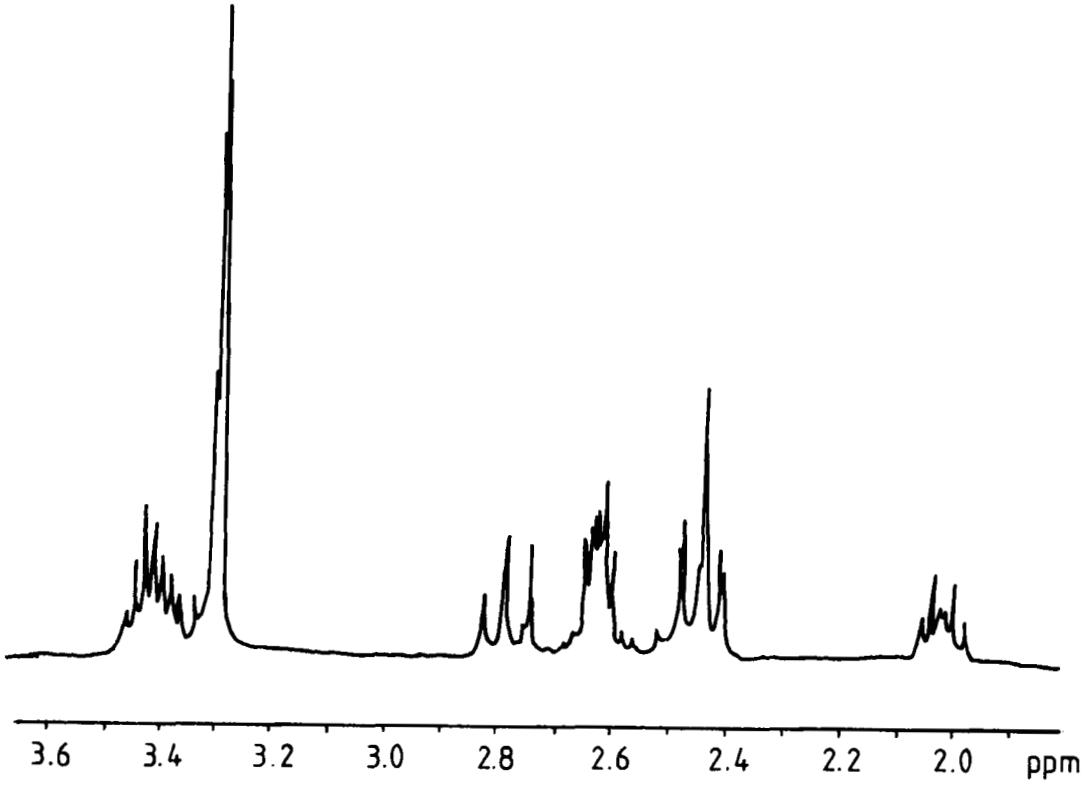

**IR spectrum** [KBr]



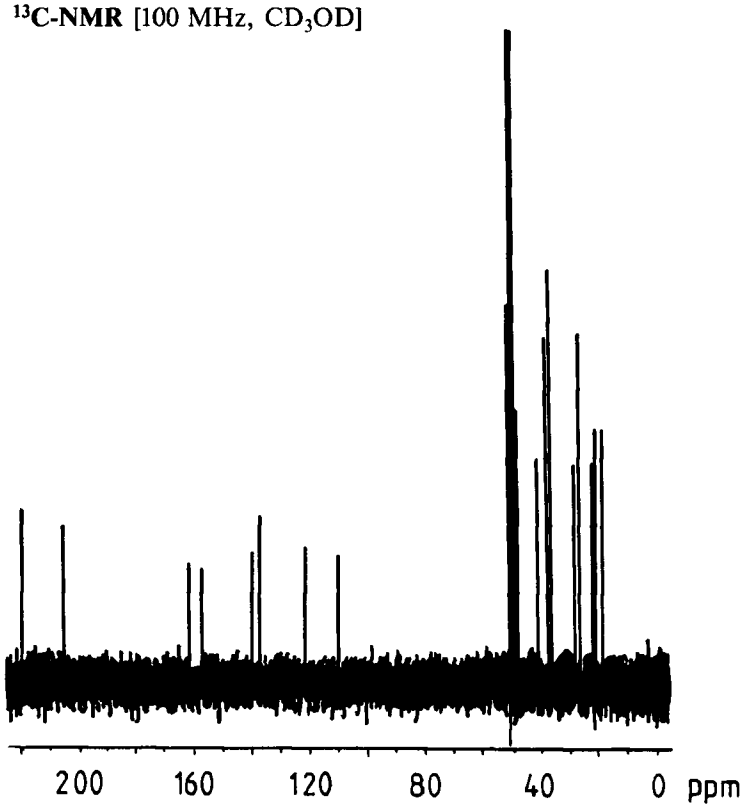
**$^1$H-NMR** [80 MHz, $CDCl_3$]

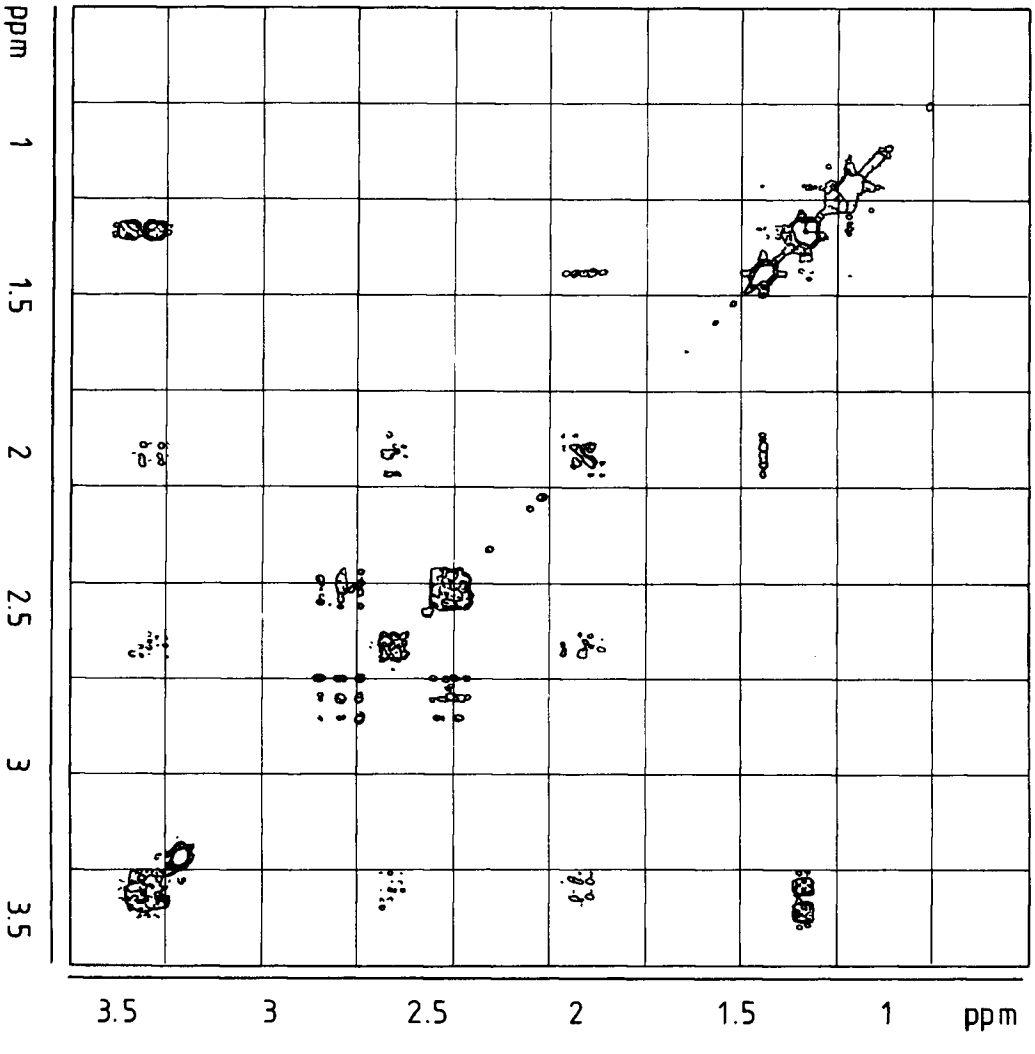



**$^1$H-NMR** [400 MHz, $CD_3OD$]



**$^{13}$C-NMR** [100 MHz, $CD_3OD$]



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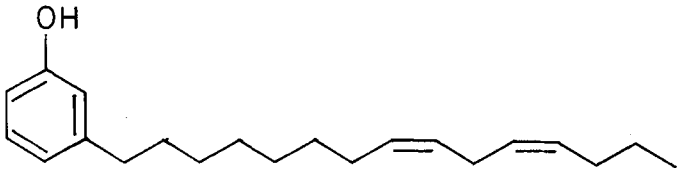
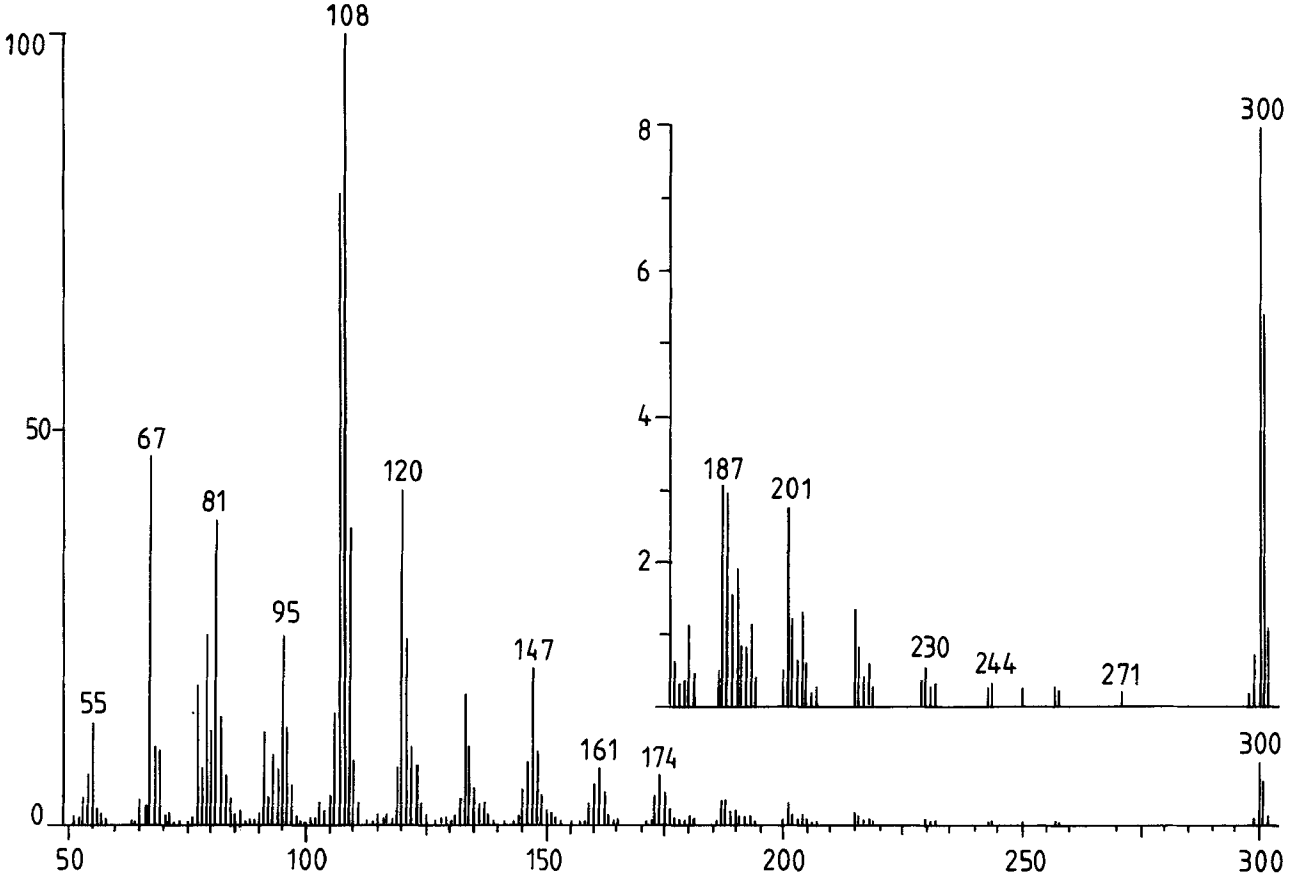
# Cardanol, Diene

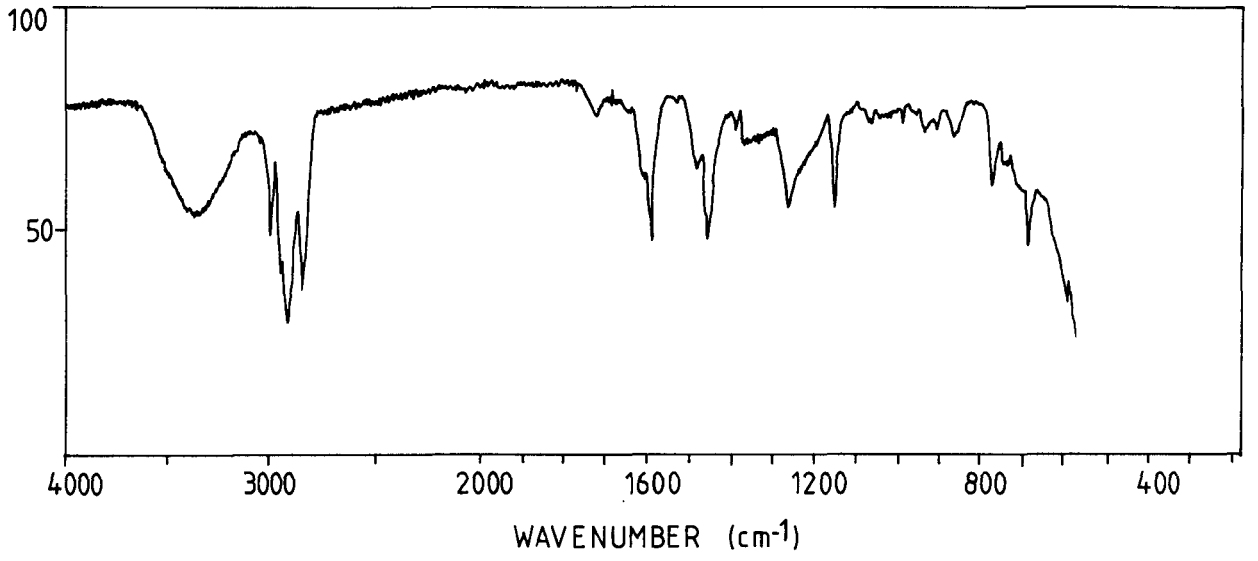
$C_{31}H_{32}O$



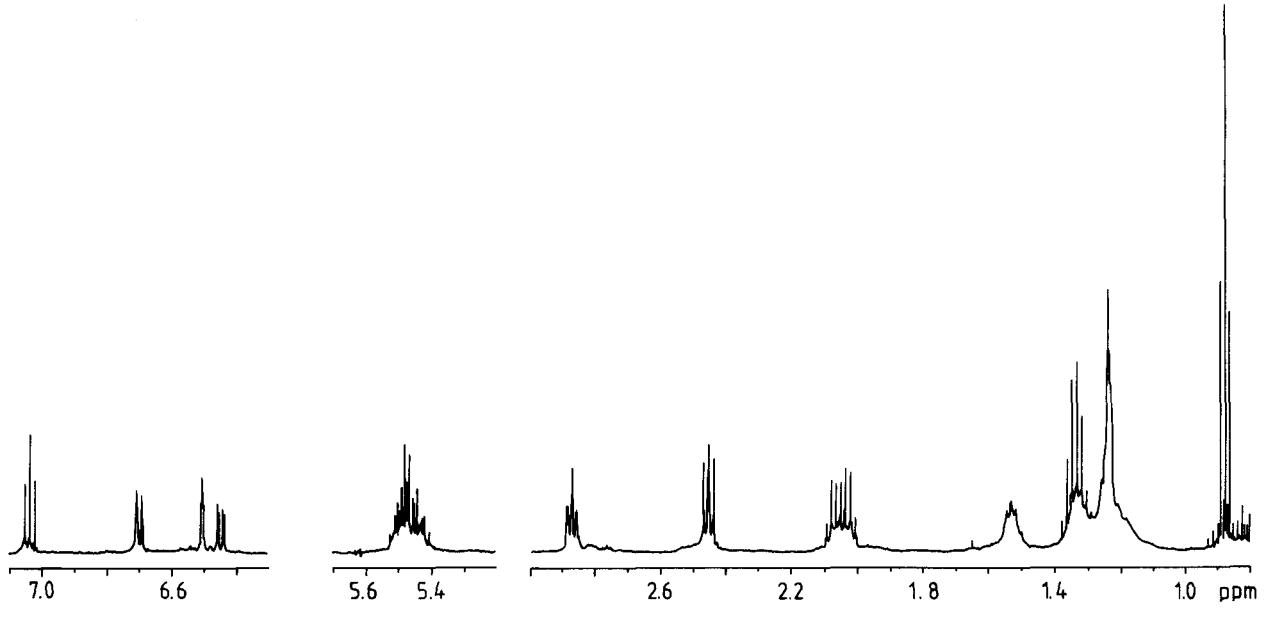

**Mass spectrum** [100 eV]



**IR spectrum** [film]

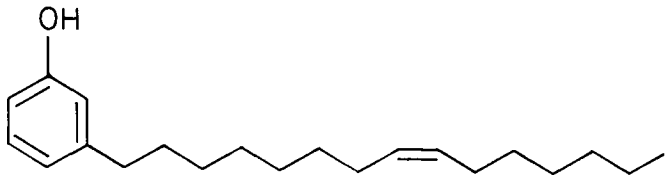



**$^1$H-NMR** [500 MHz, $C_6D_6$]

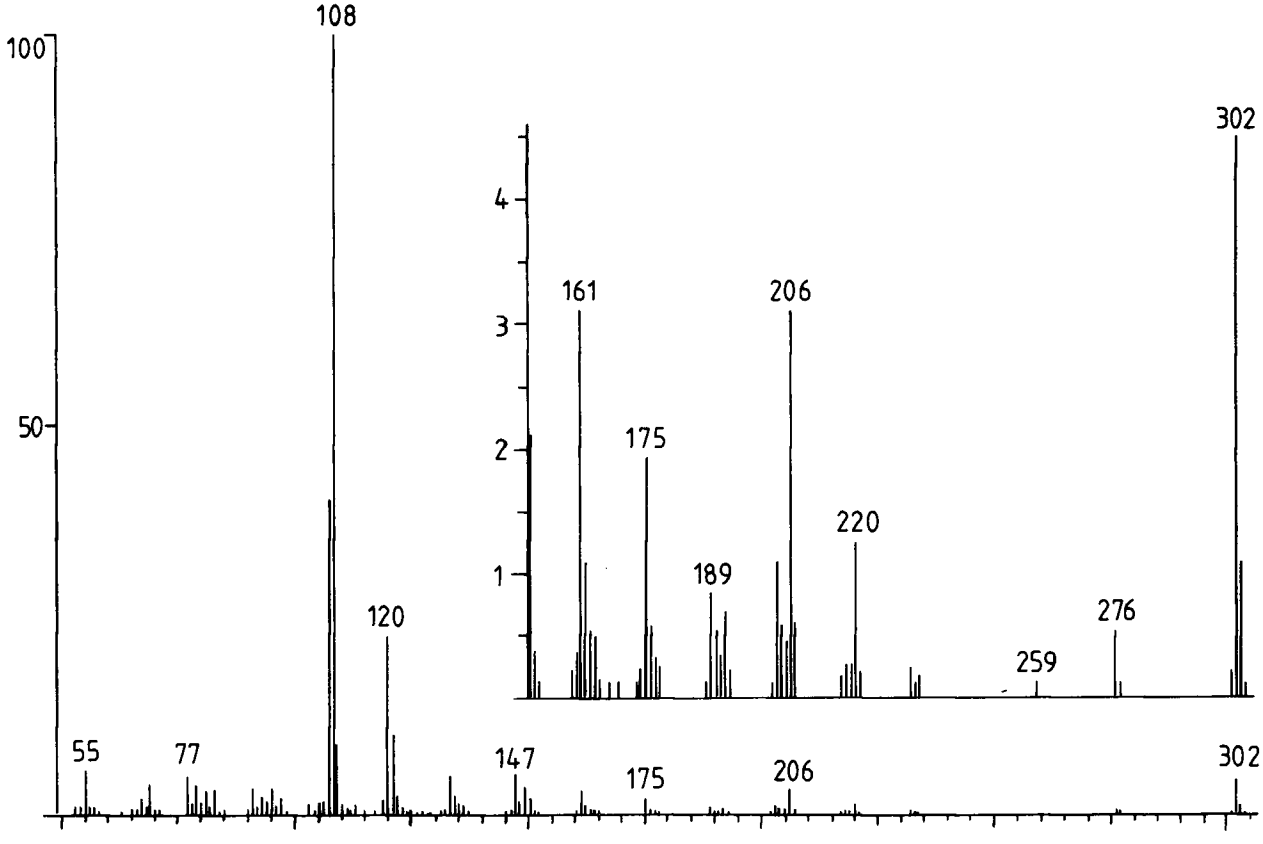


# Cardanol, Monoene

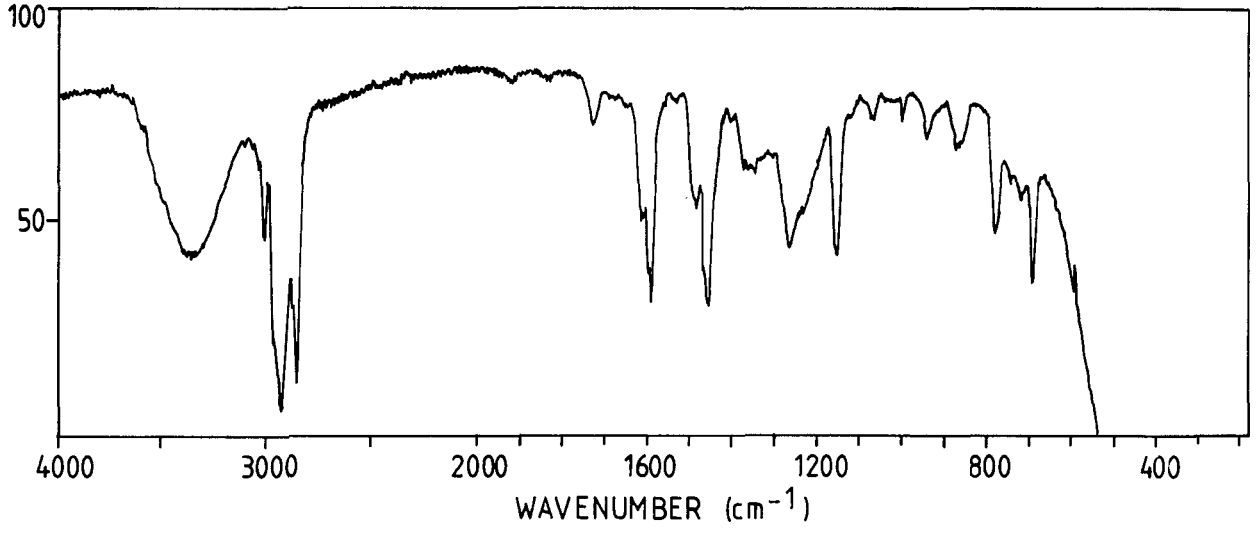
$C_{21}H_{34}O$



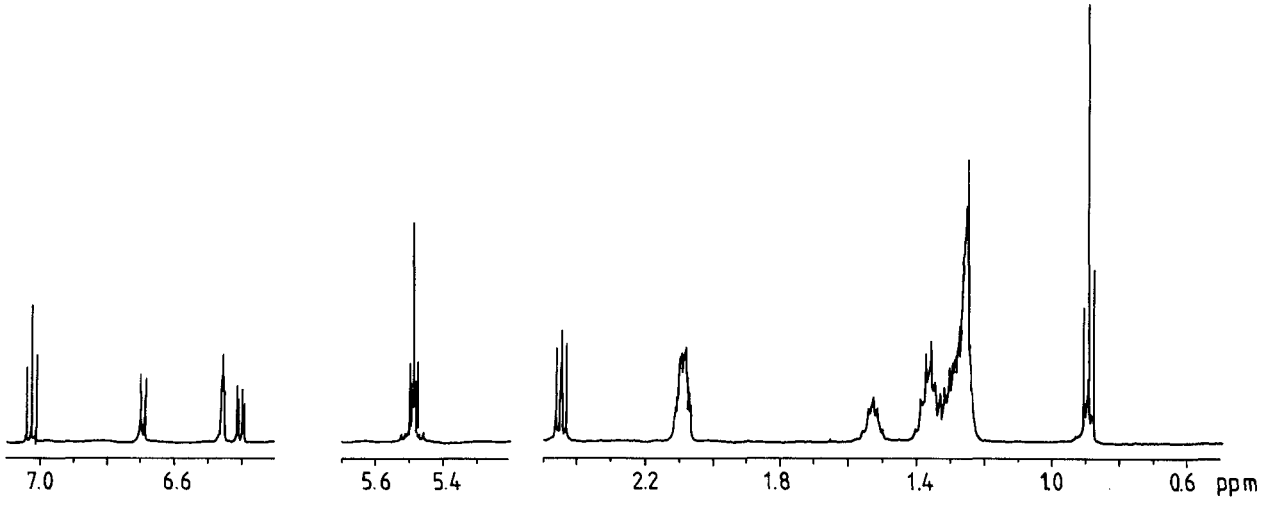

**Mass spectrum** [100 eV]



**IR spectrum** [film]

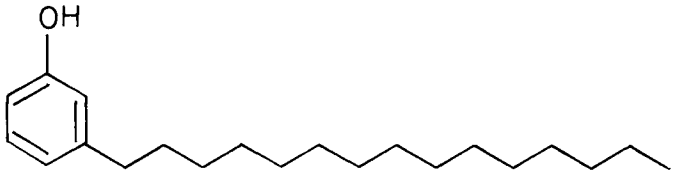



**$^1$H-NMR** [500 MHz, $C_6D_6$]

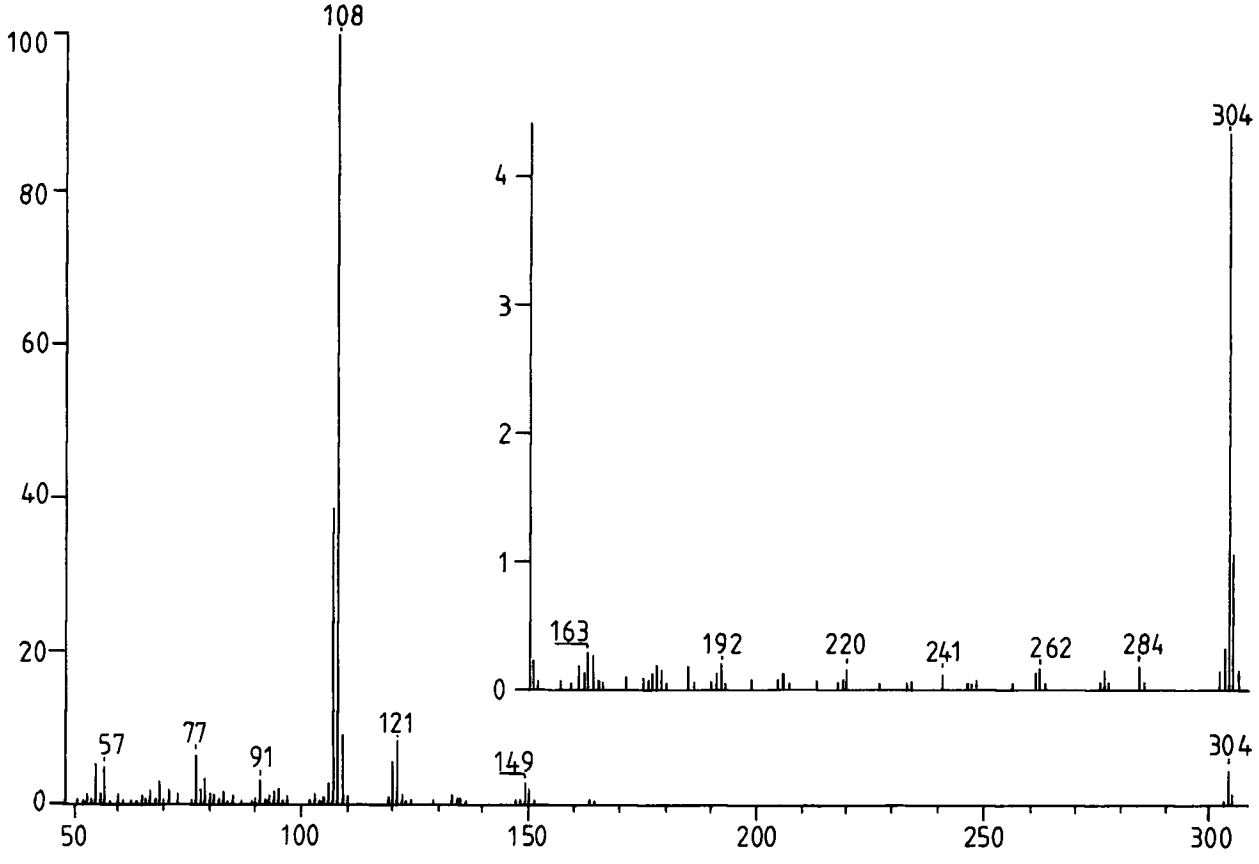


# Cardanol, saturated

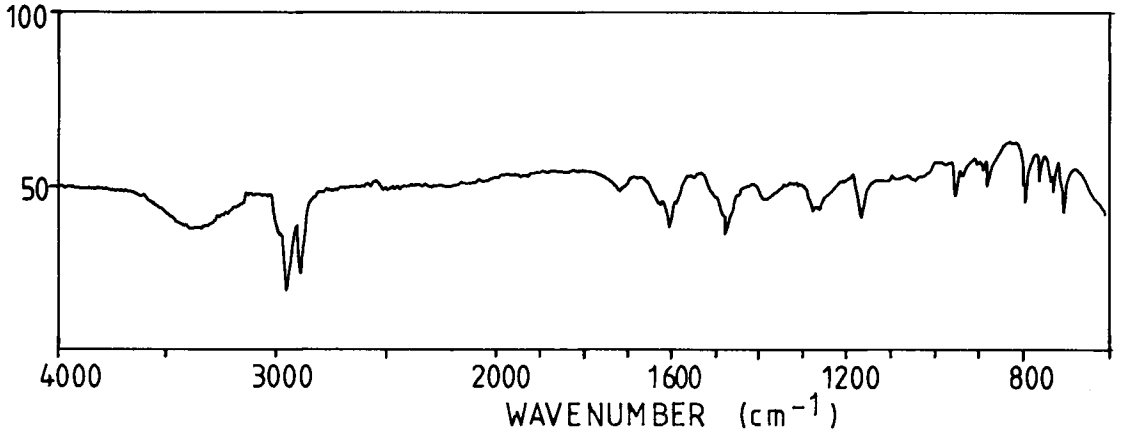
$C_{21}H_{36}O$



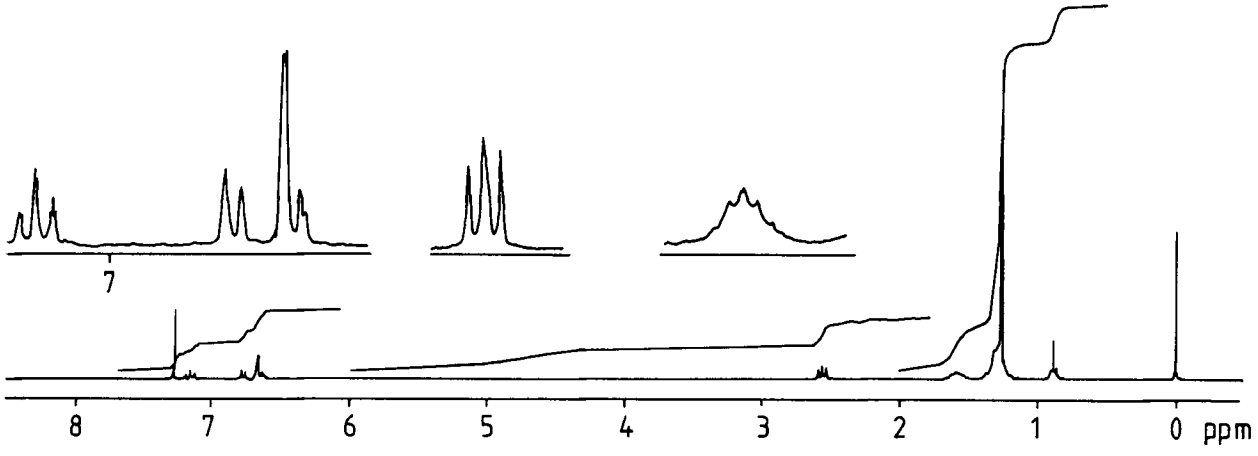

**Mass spectrum** [100 eV]



**IR spectrum** [KBr]

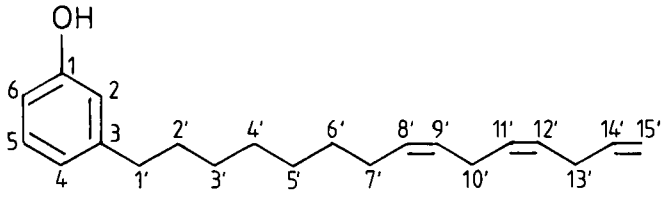



**$^1$H-NMR** [250 MHz, $CDCl_3$]

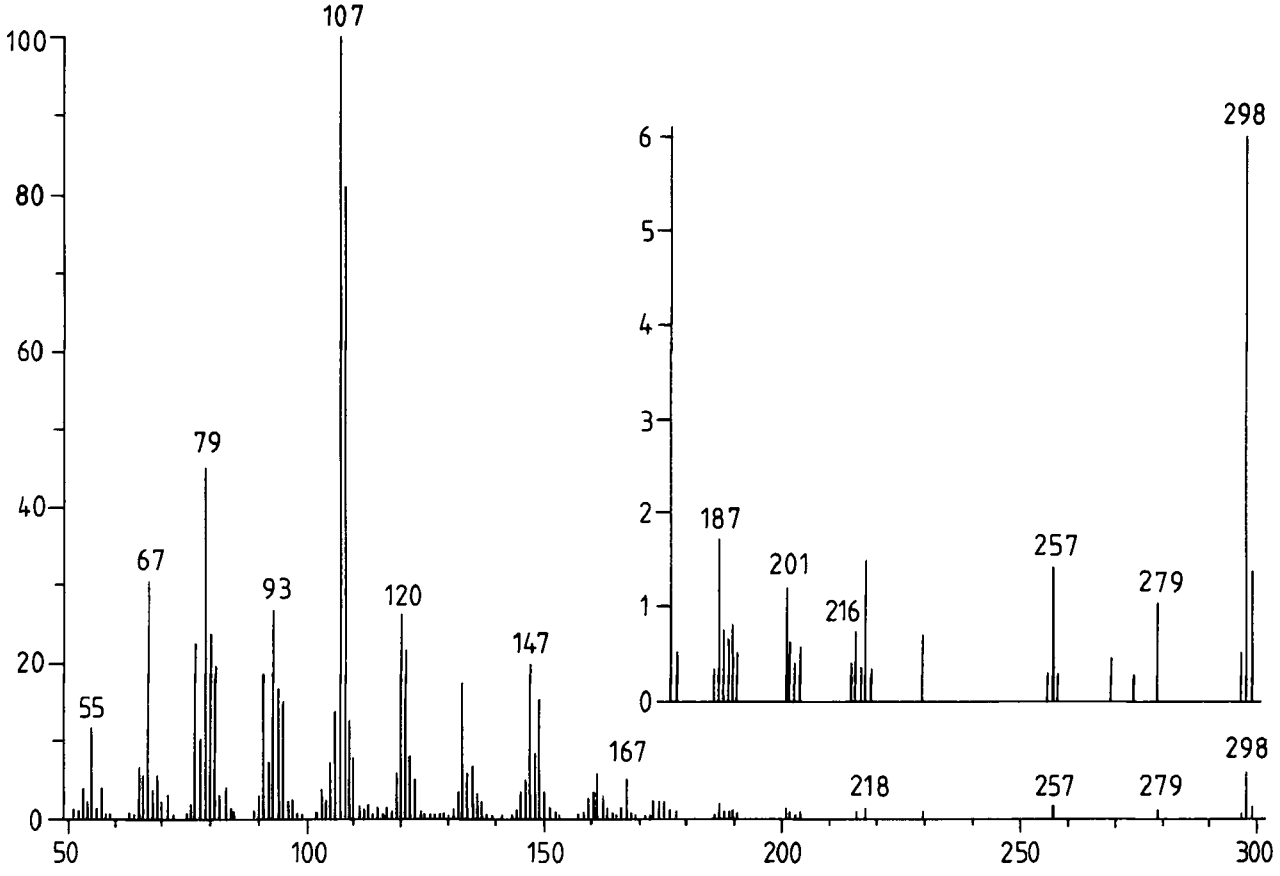


# Cardanol, Triene

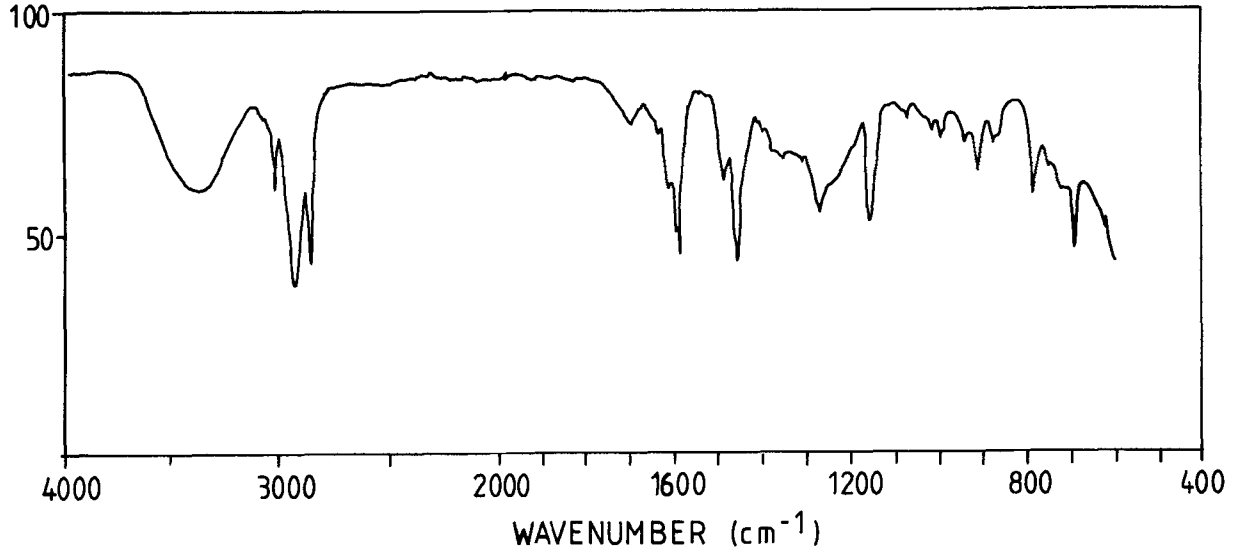
$C_{21}H_{30}O$



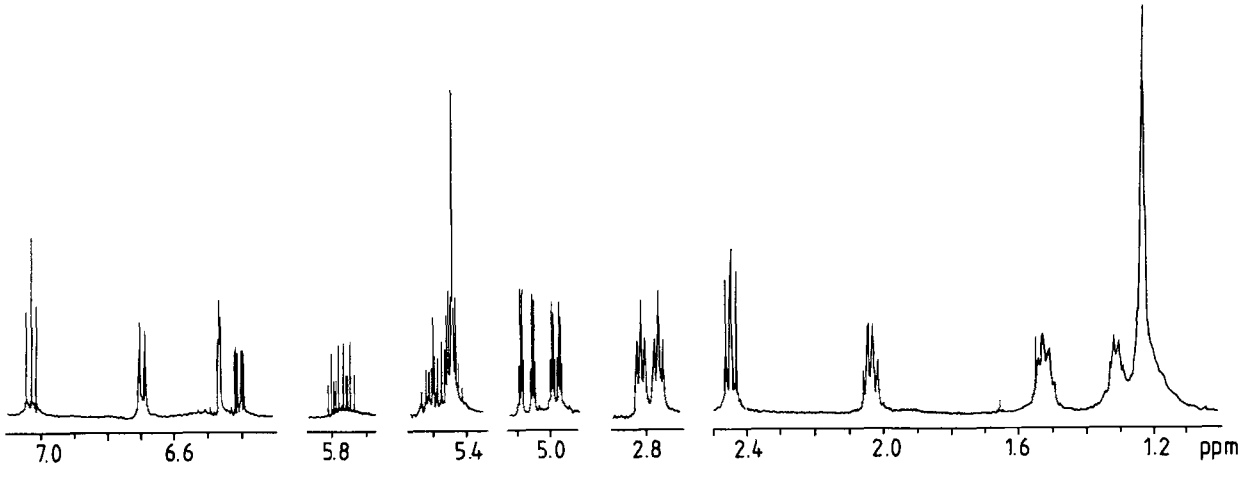

**Mass spectrum** [100 eV]



**IR spectrum** [film]

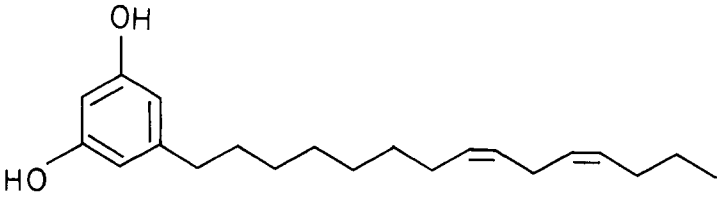



**$^1$H-NMR** [500 MHz, $C_6D_6$]

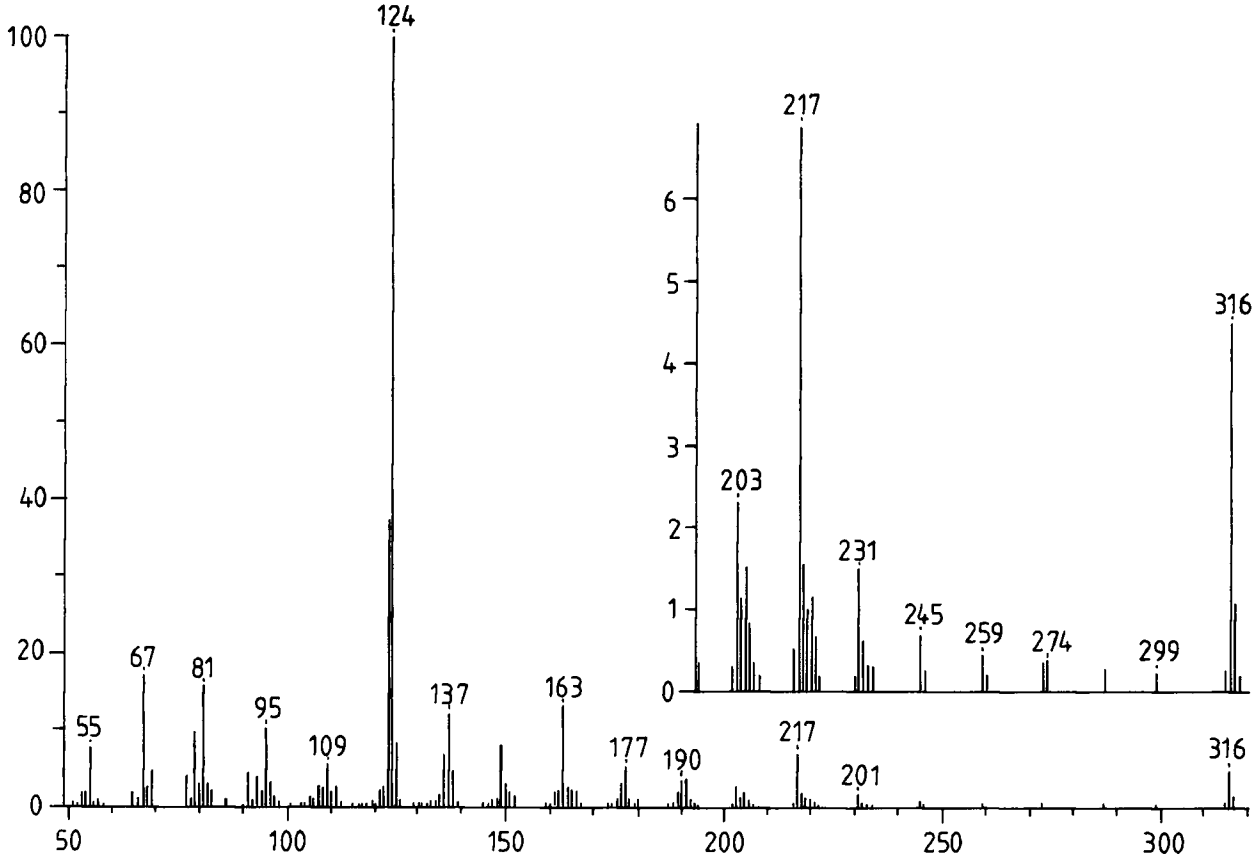


# Cardol, Diene

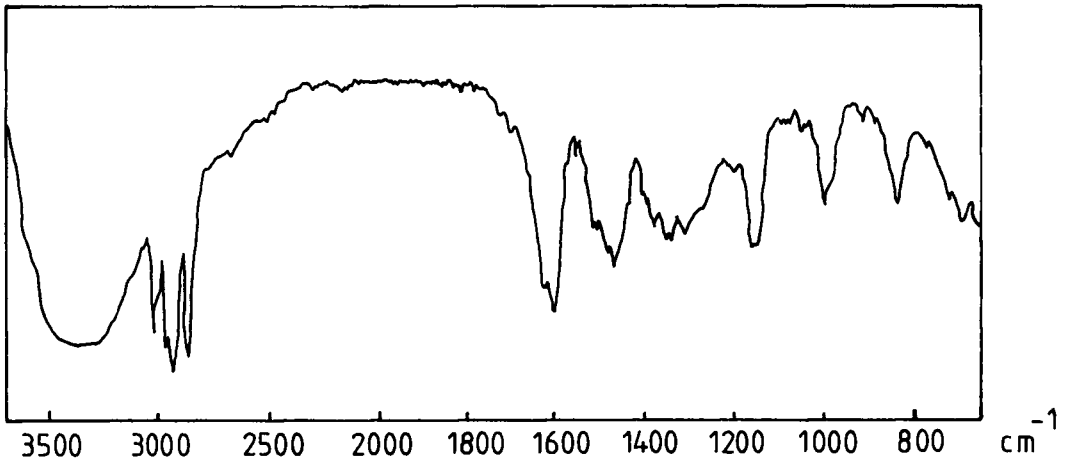
$C_{21}H_{32}O_2$



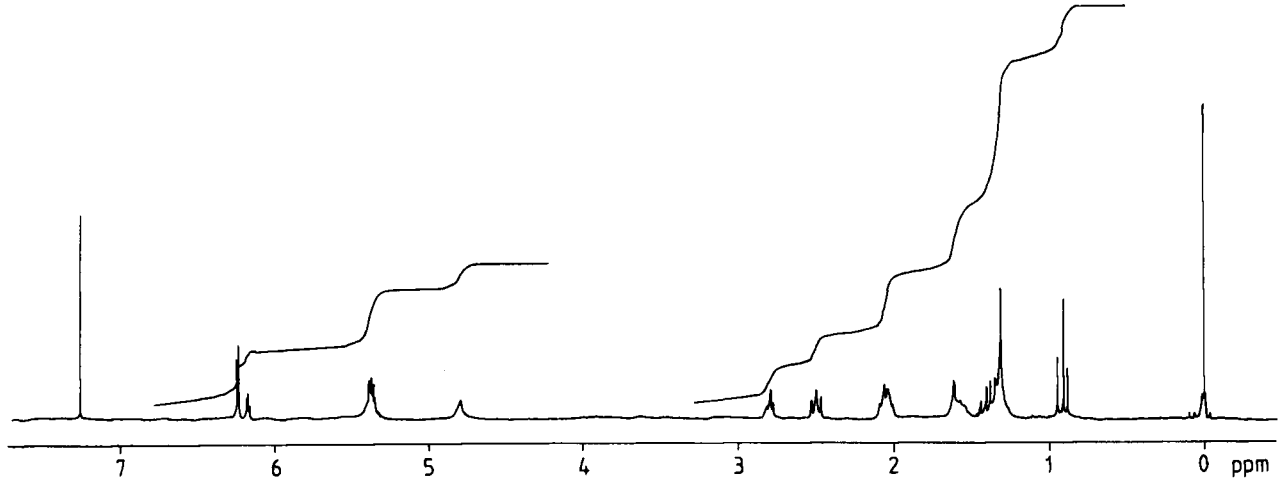

**Mass spectrum** [100 eV]



**IR spectrum** [film]

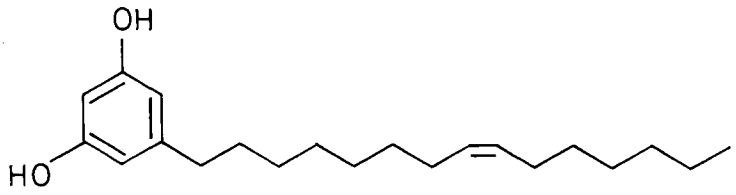



**$^1$H-NMR** [250 MHz, $CDCl_3$]

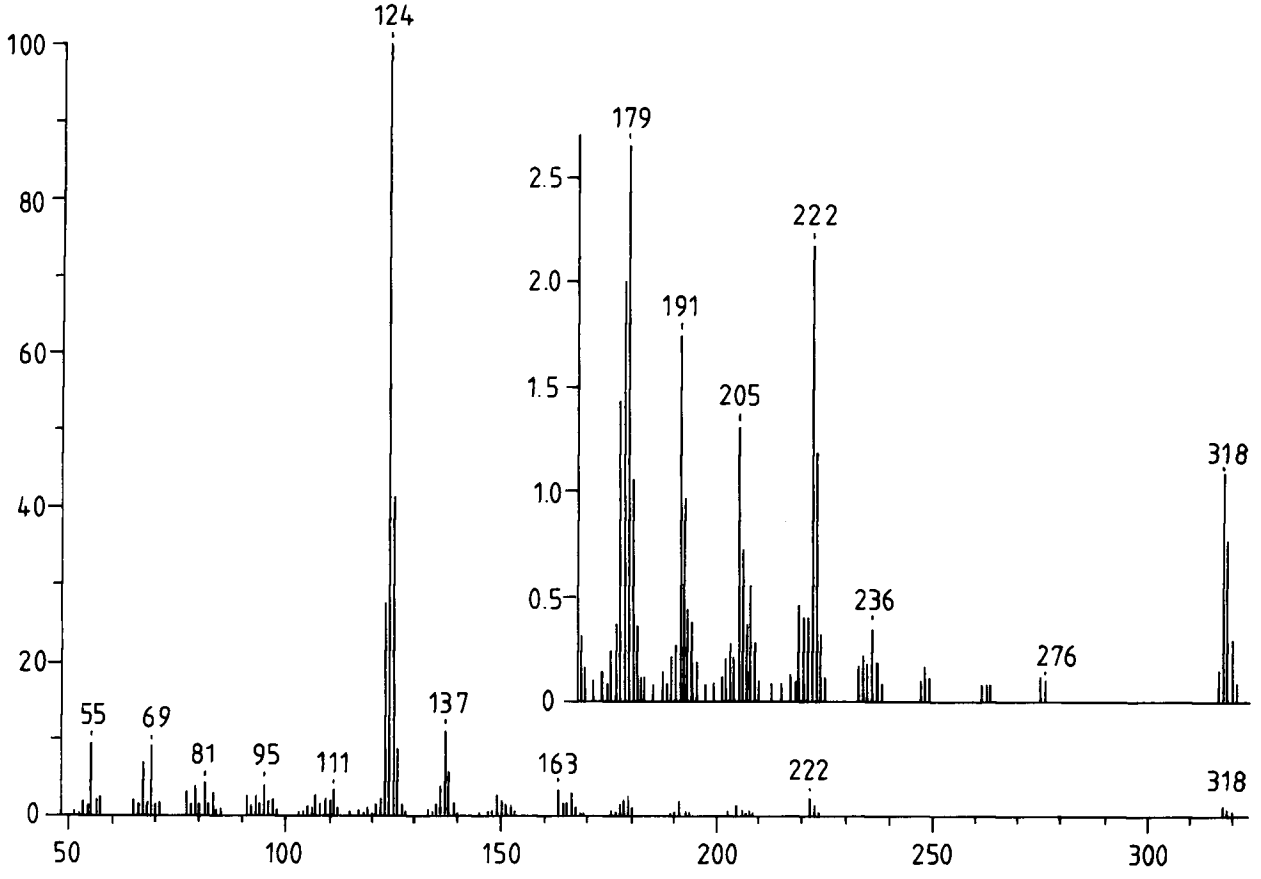


# Cardol, Monoene

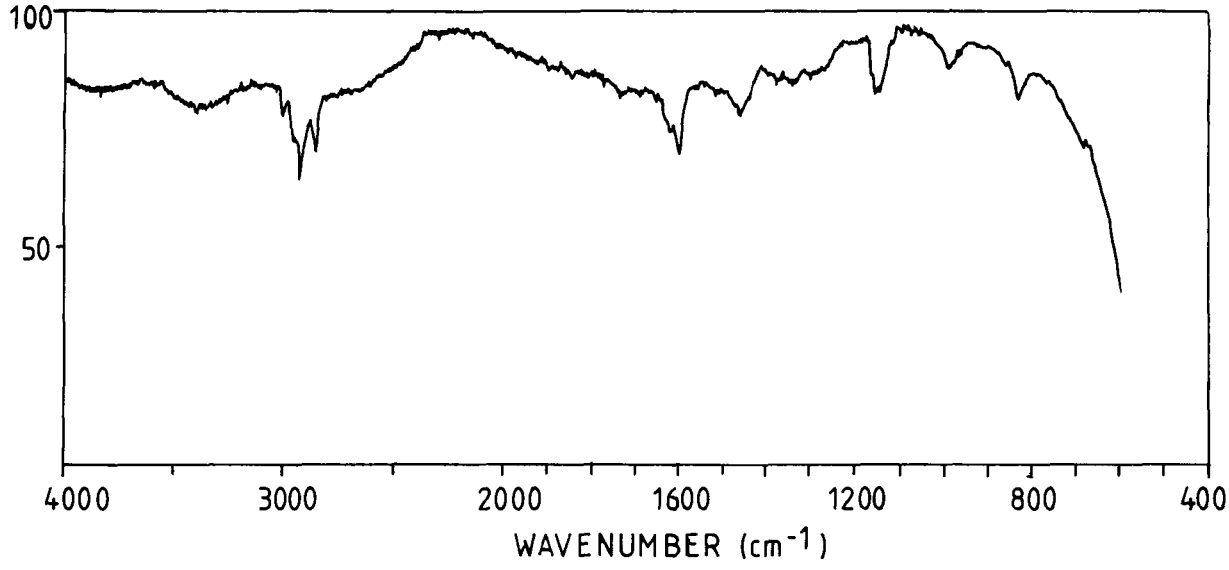
$C_{21}H_{34}O_2$



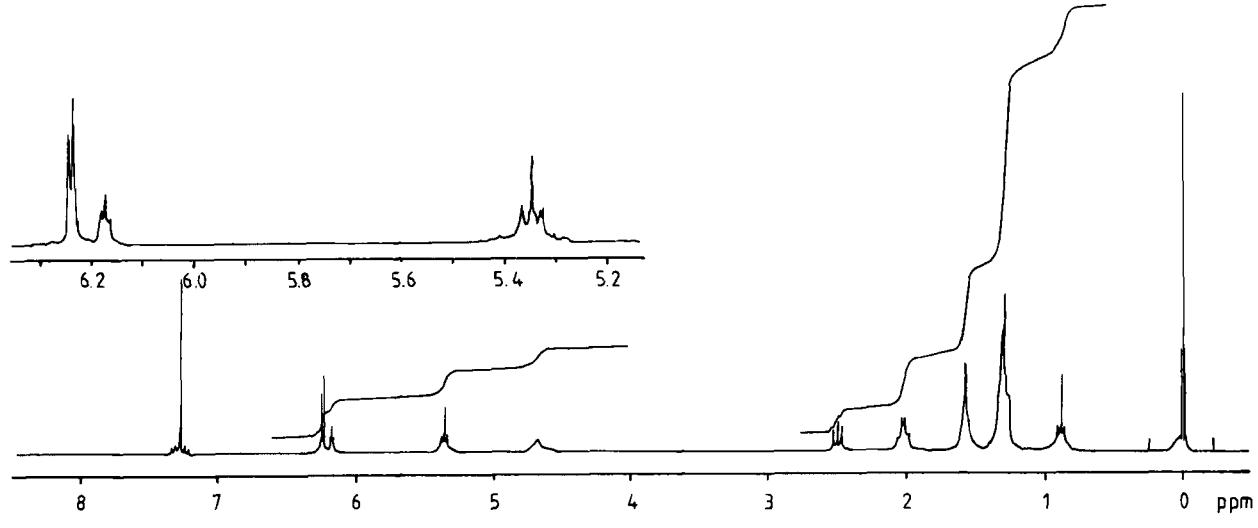

**Mass spectrum** [100 eV]



**IR spectrum** [film]

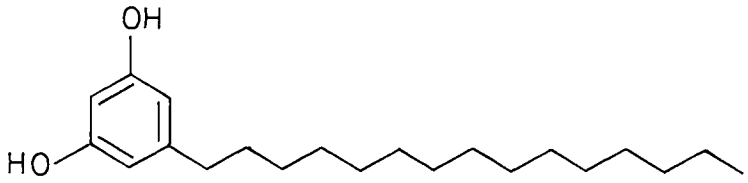



**$^1$H-NMR** [250 MHz, $CDCl_3$]

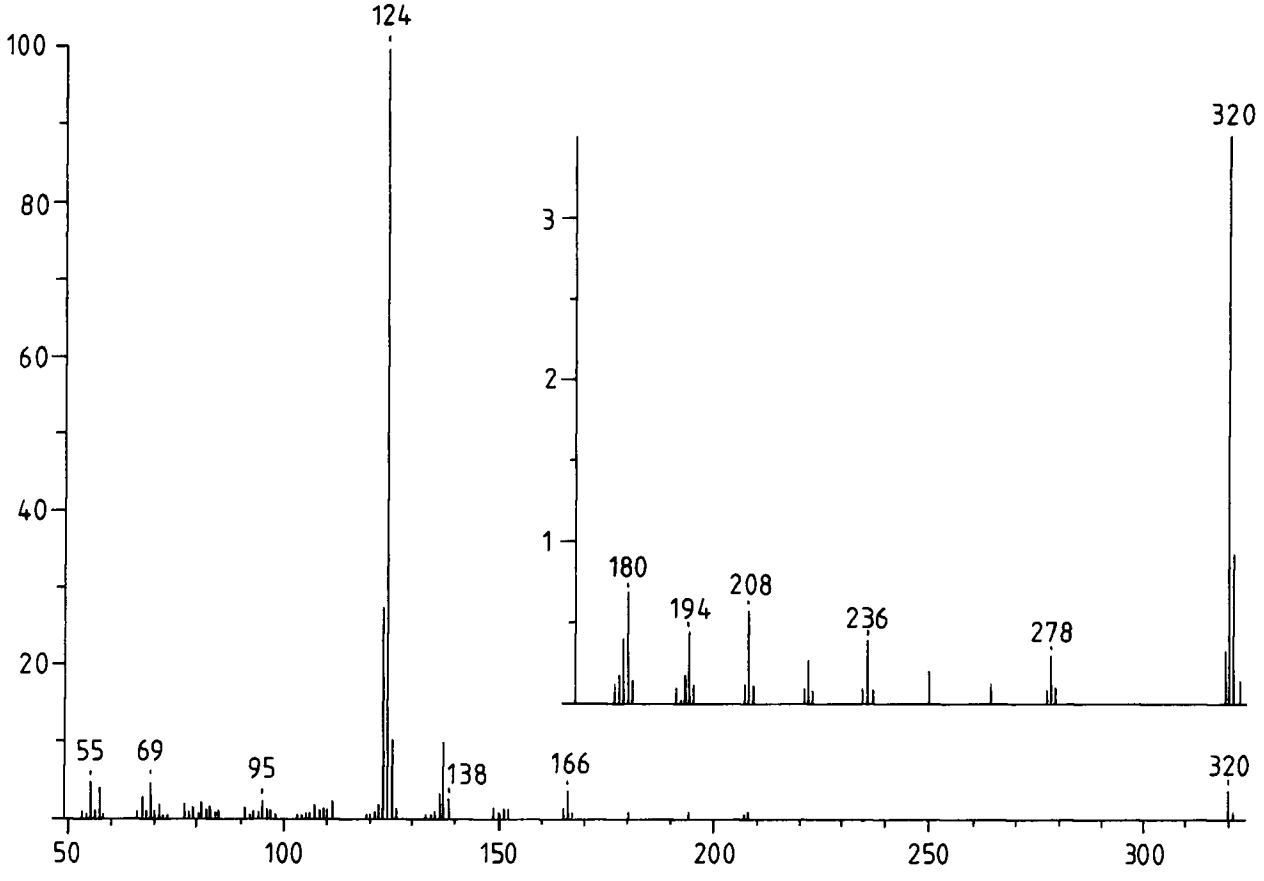


# Cardol, saturated

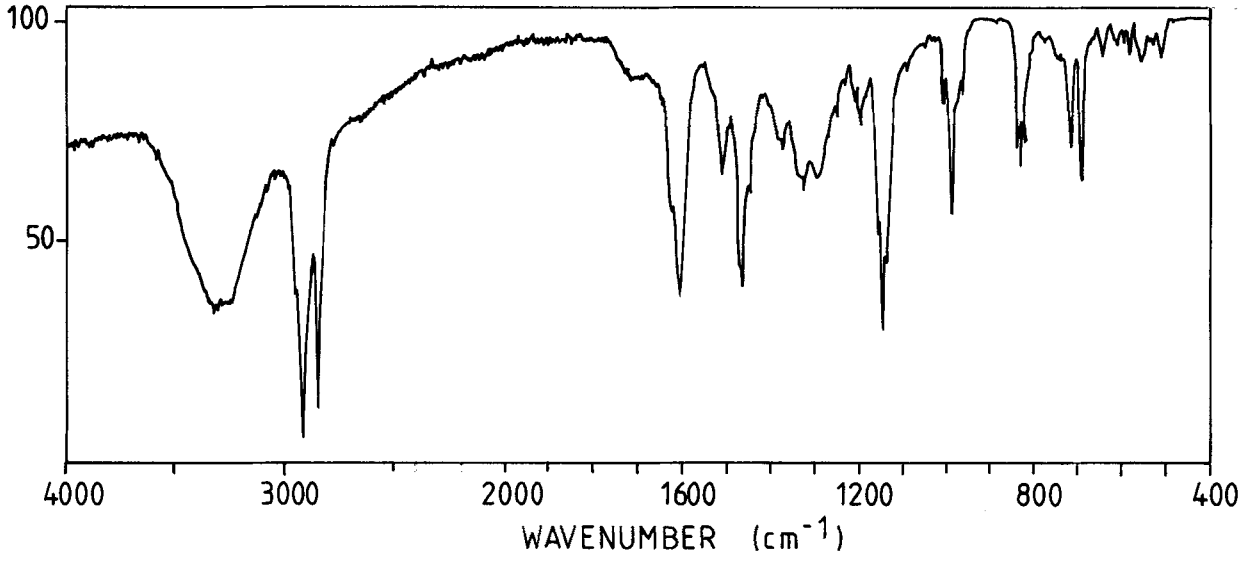
$C_{21}H_{36}O_2$



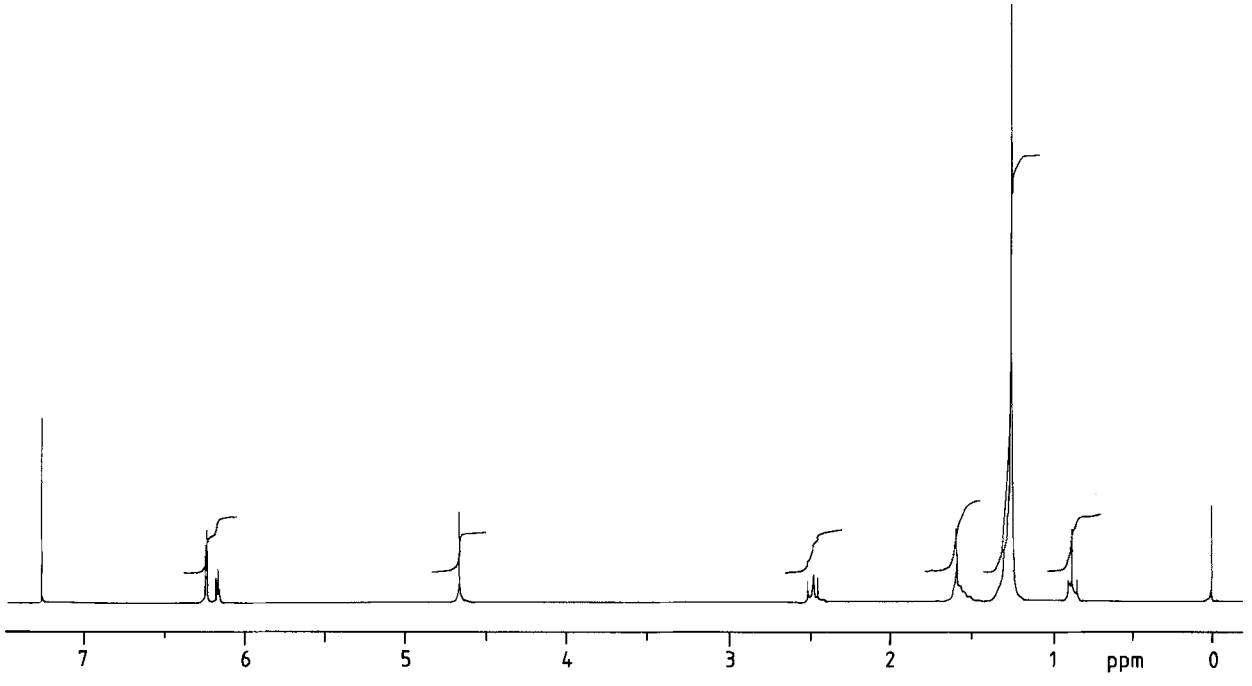

**Mass spectrum** [100 eV]



**IR spectrum** [KBr]

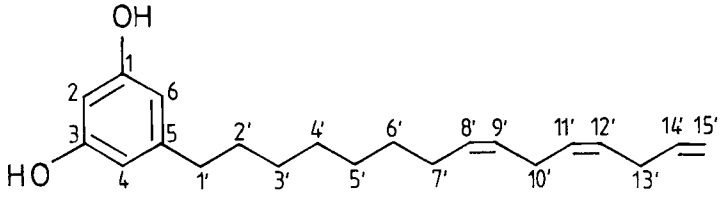



**$^1$H-NMR** [250 MHz, $CDCl_3$]

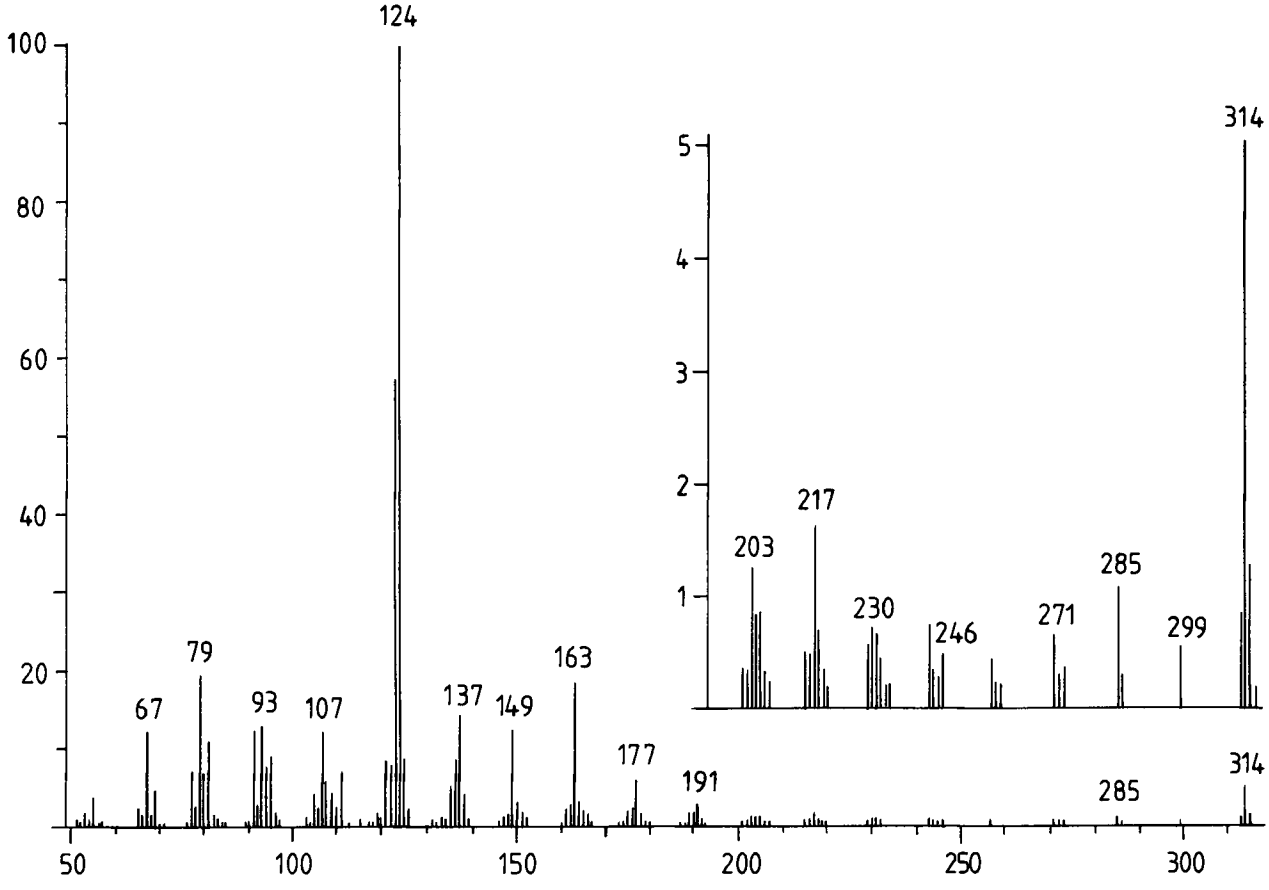


# Cardol, Triene

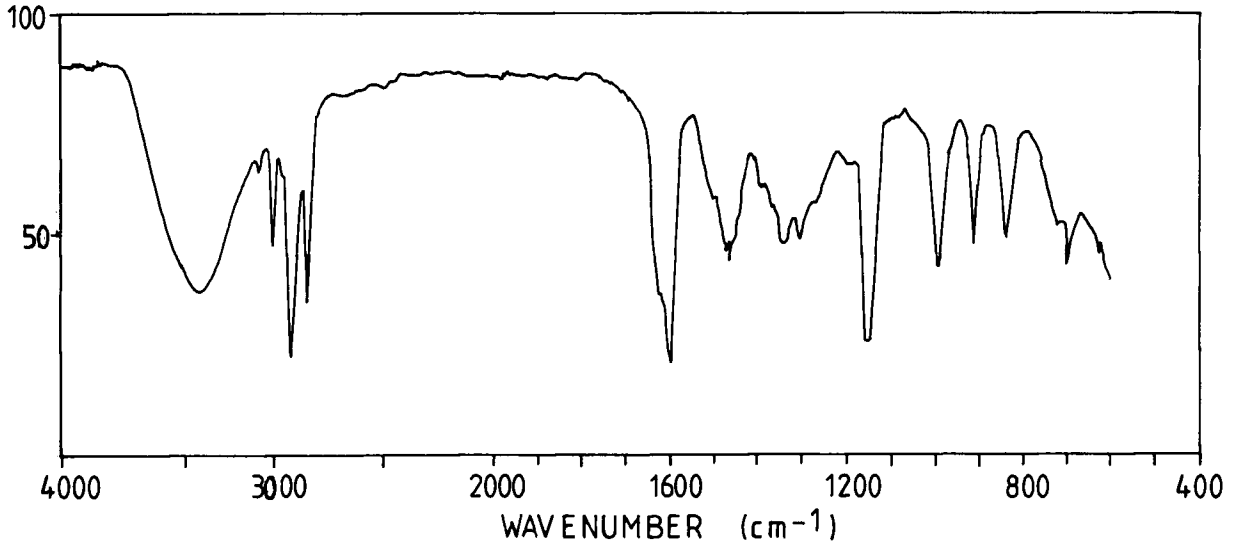
$C_{21}H_{30}O_2$



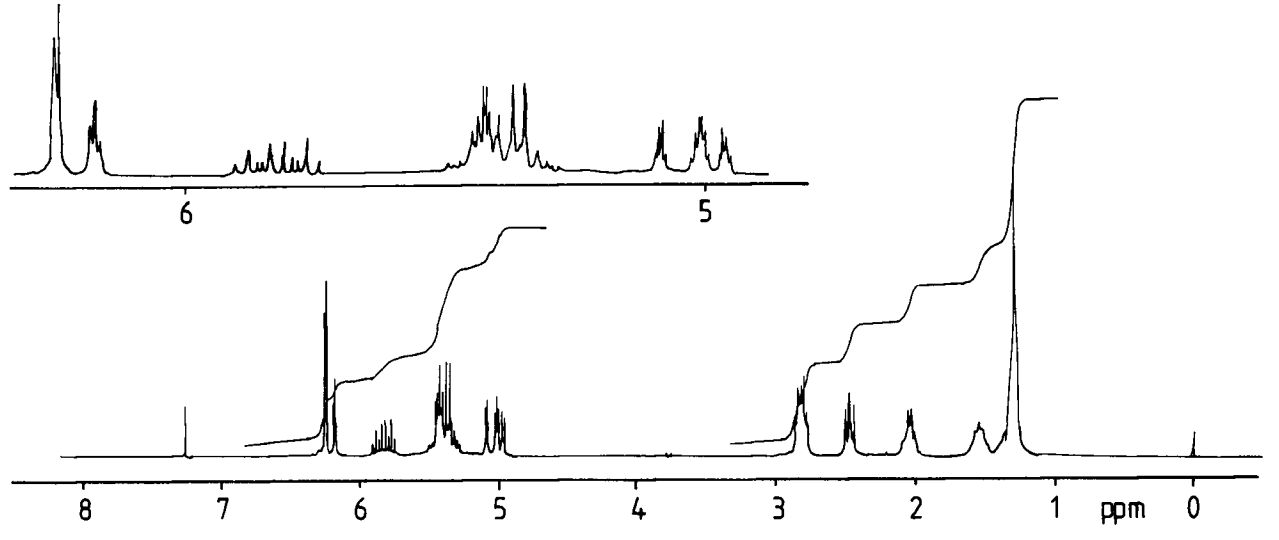

**Mass spectrum** [100 eV]



**IR spectrum** [film]

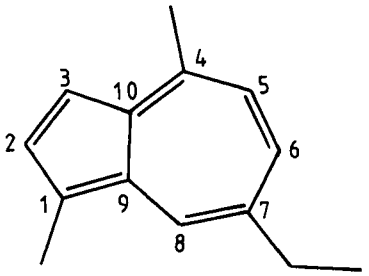



**$^1$H-NMR** [250 MHz, $CDCl_3$]

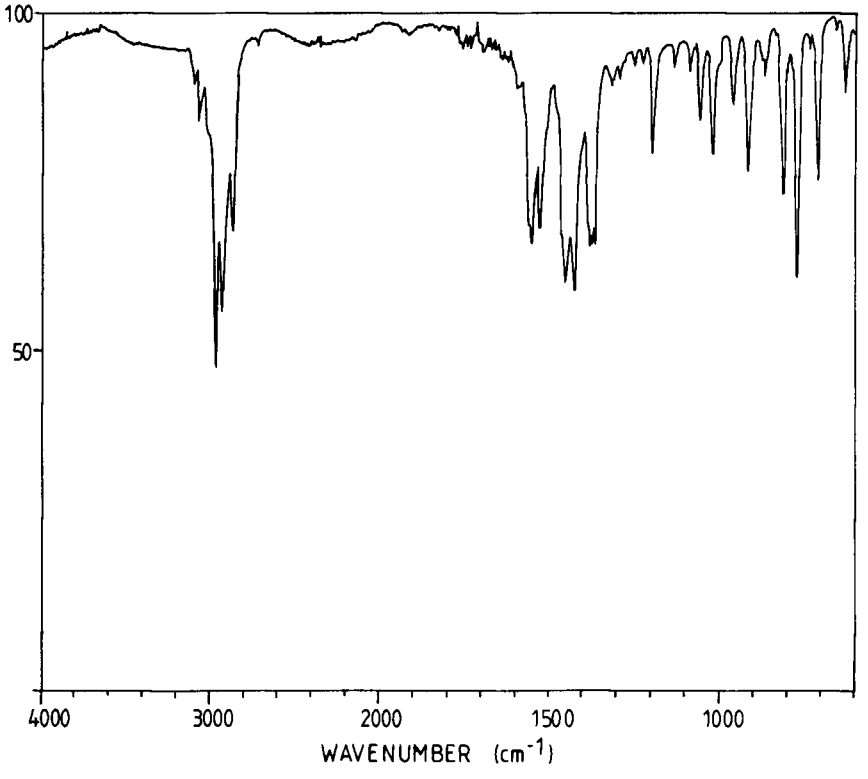


# Chamazulene

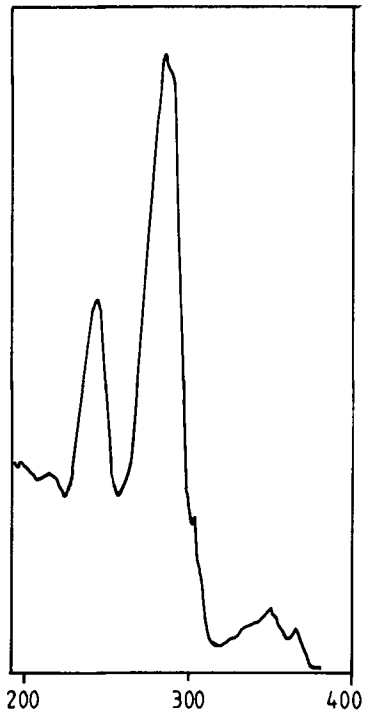
$C_{14}H_{16}$




**IR spectrum** [film between $C_5I$-plates]

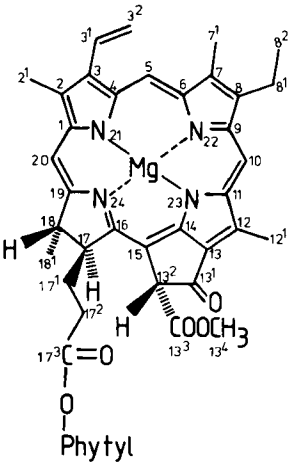


**UV spectrum** [Cyclohexane]

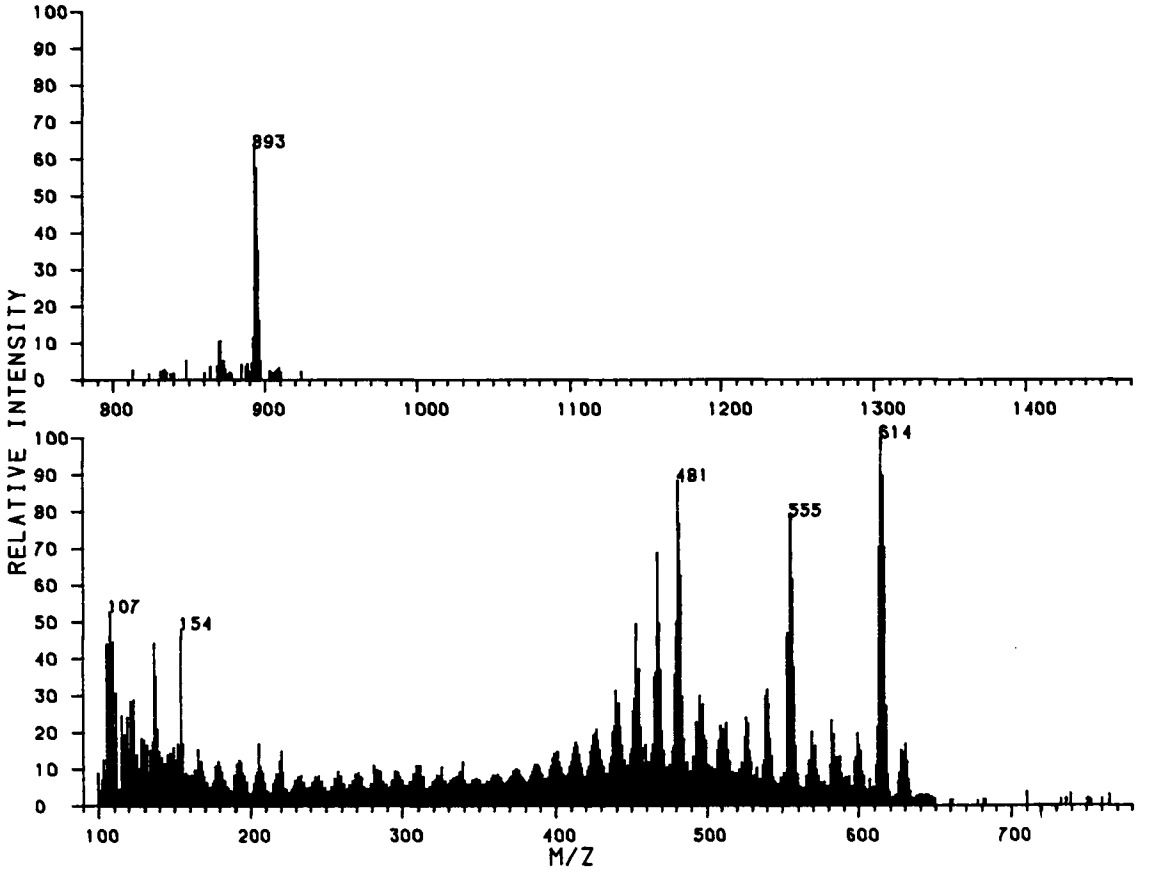


# Chlorophyll a

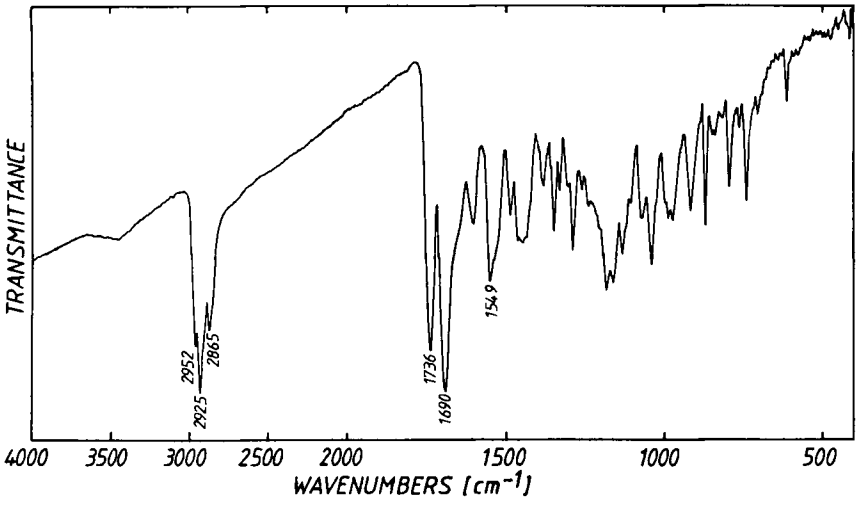
$C_{55}H_{72}MgN_4O_5$



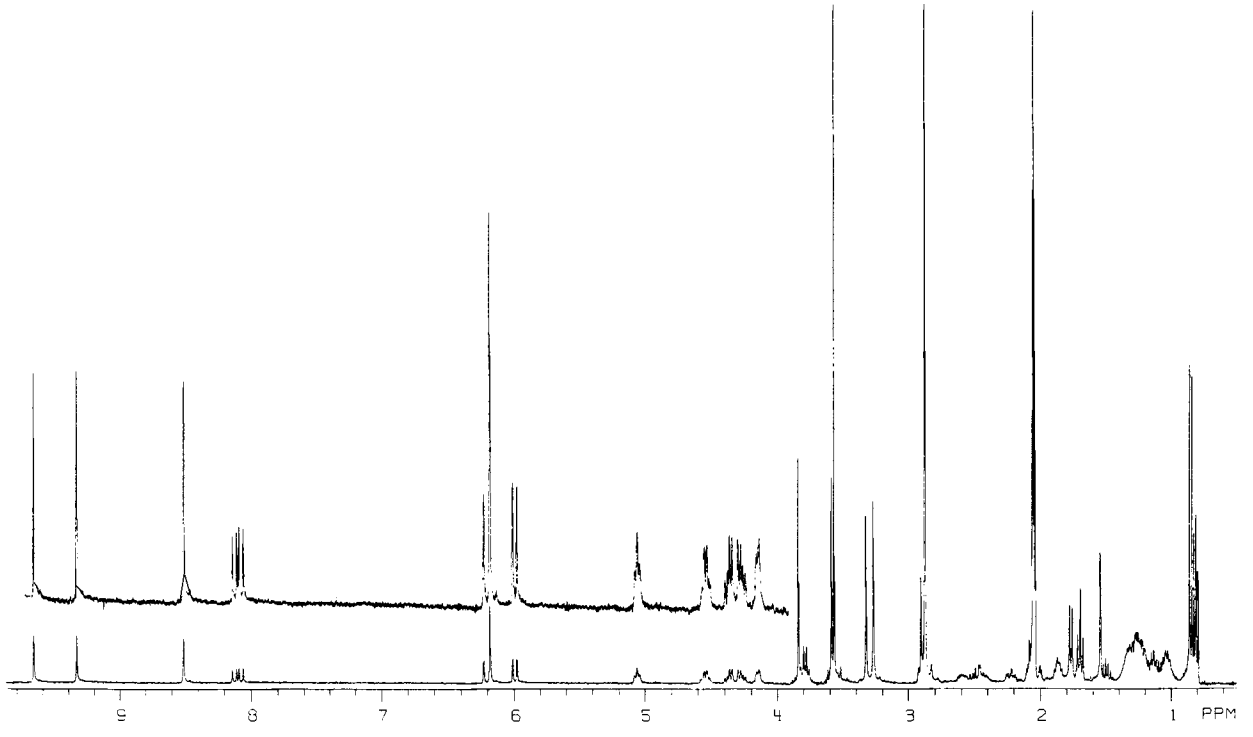

**Mass spectrum** [7–8 keV Ar-Atoms]



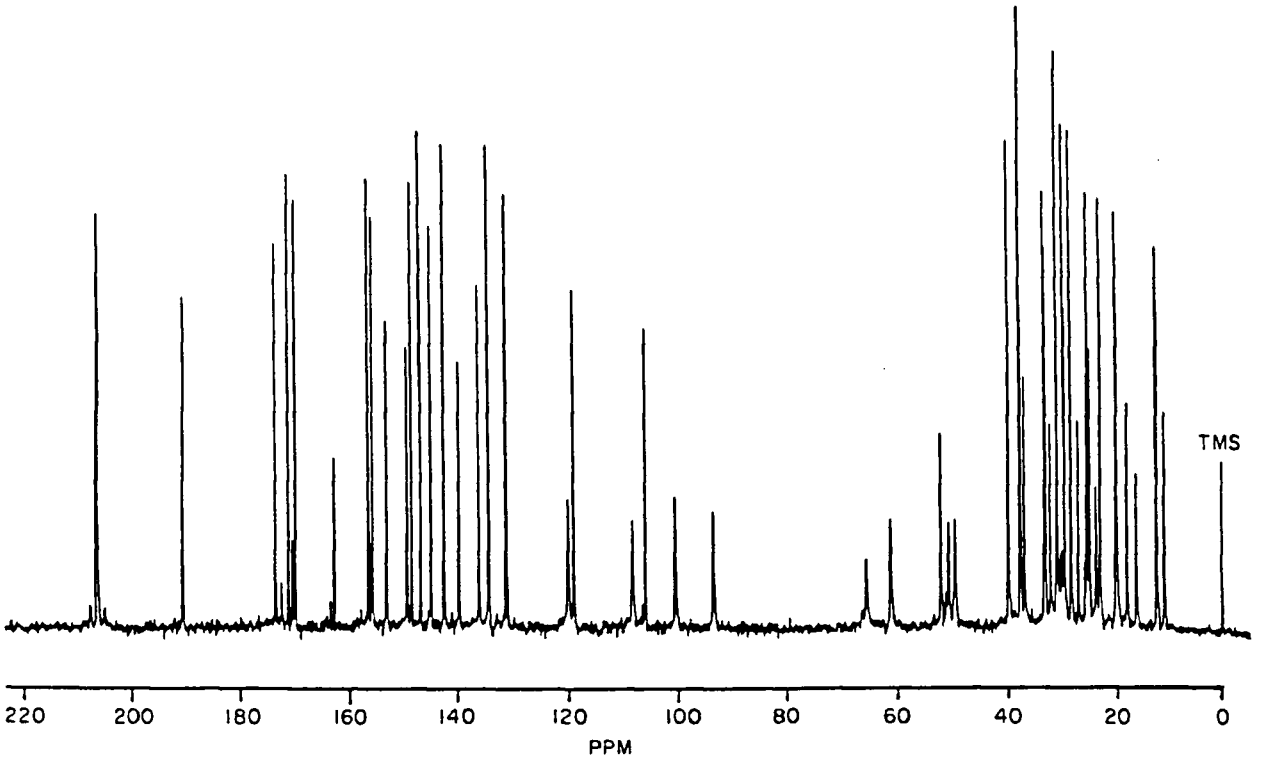
**IR spectrum** [KBr]



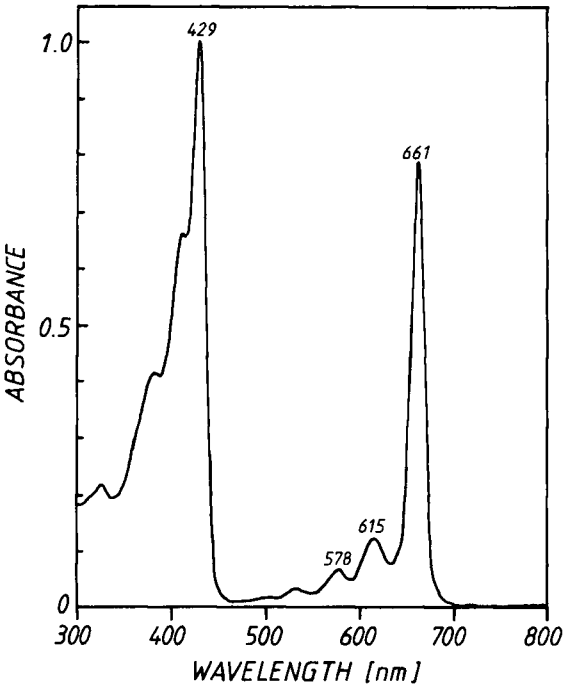

**$^1$H-NMR** [360 MHz, $d_6$-acetone]



**$^{13}$C-NMR** [15.04 MHz, $d_6$-acetone]




**UV spectrum** [diethylether]



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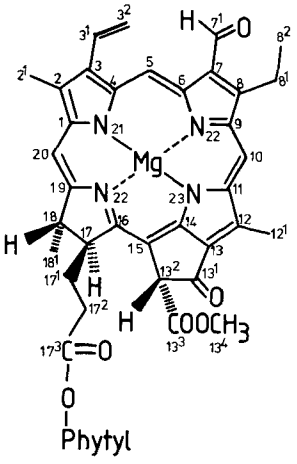
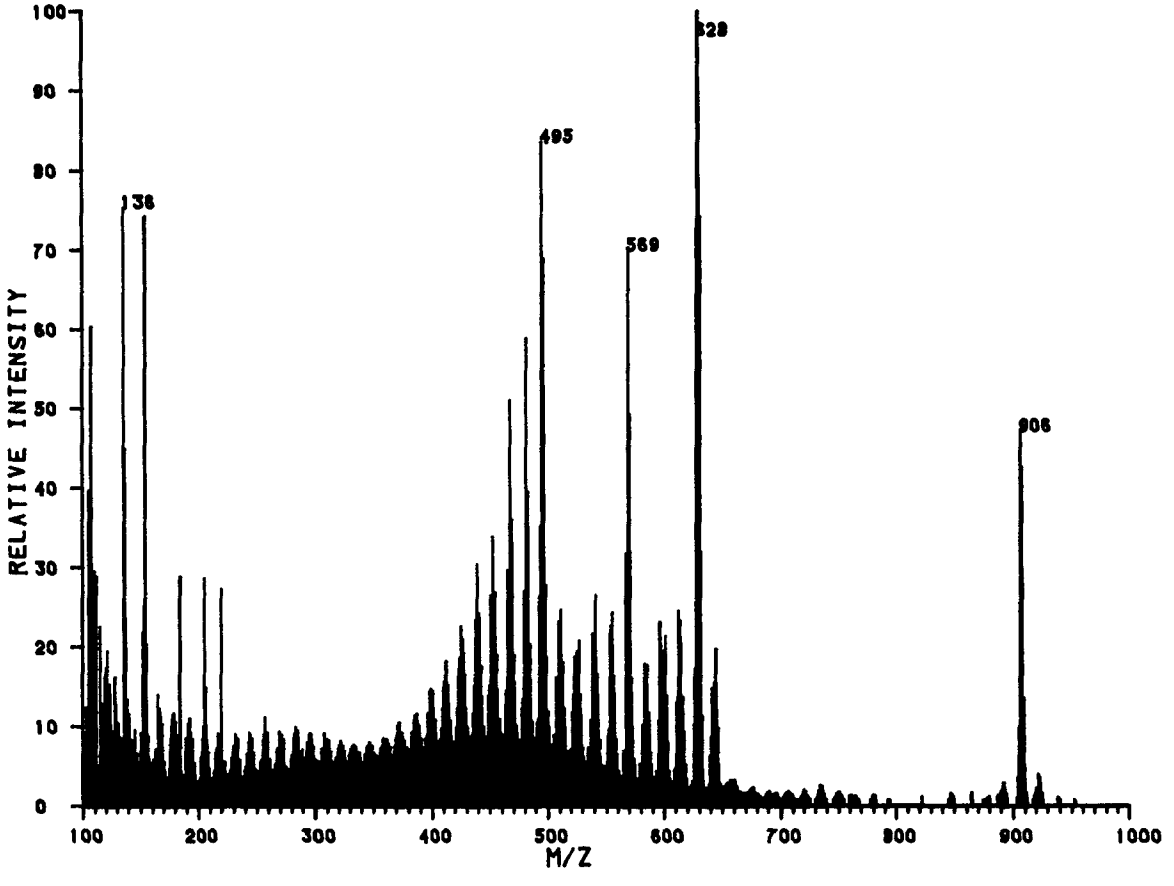
# Chlorophyll b

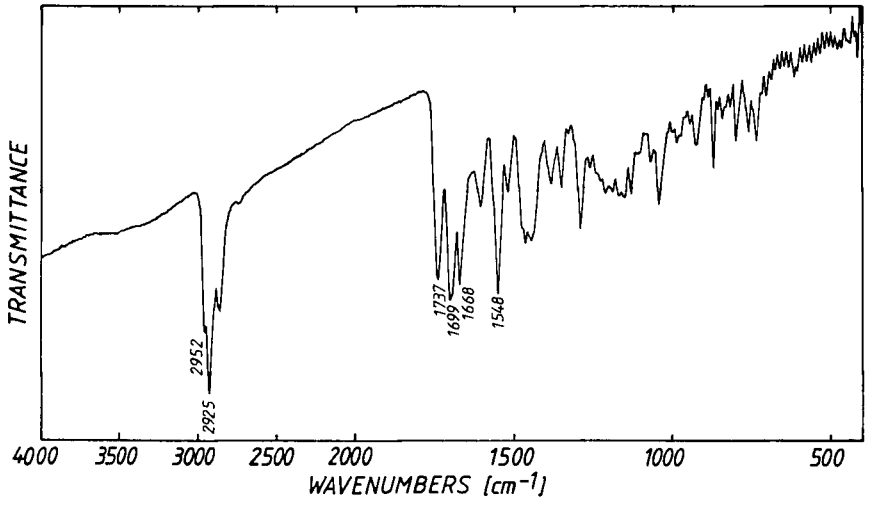
$C_{55}H_{70}MgN_4O_6$



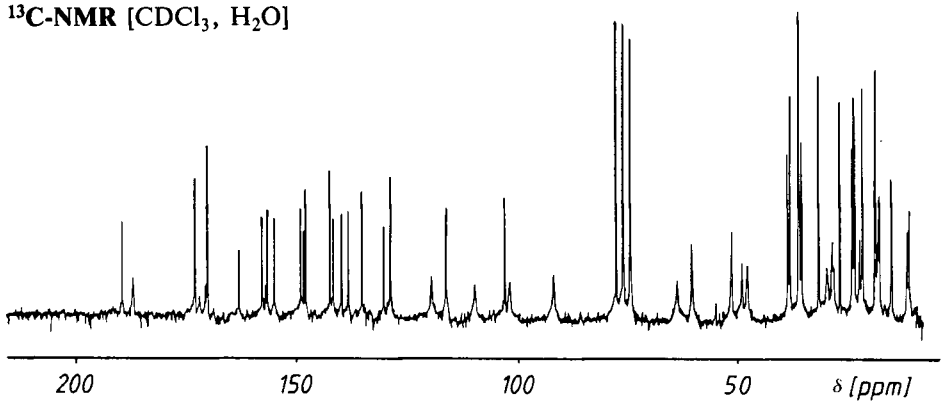

**Mass spectrum** [7–8 keV Ar-Atoms]



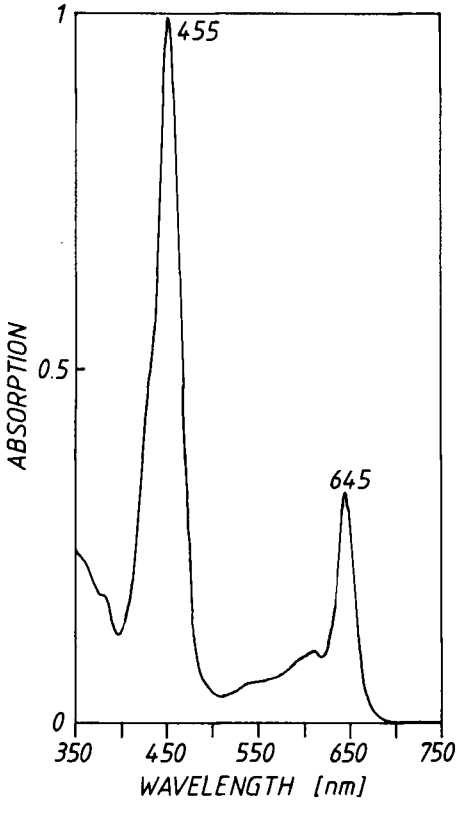
**IR spectrum** [KBr]




**$^{13}$C-NMR** [$CDCl_3$, $H_2O$]



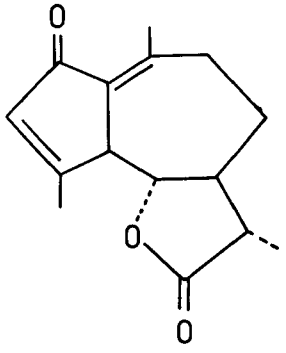

**UV spectrum** [acetone]



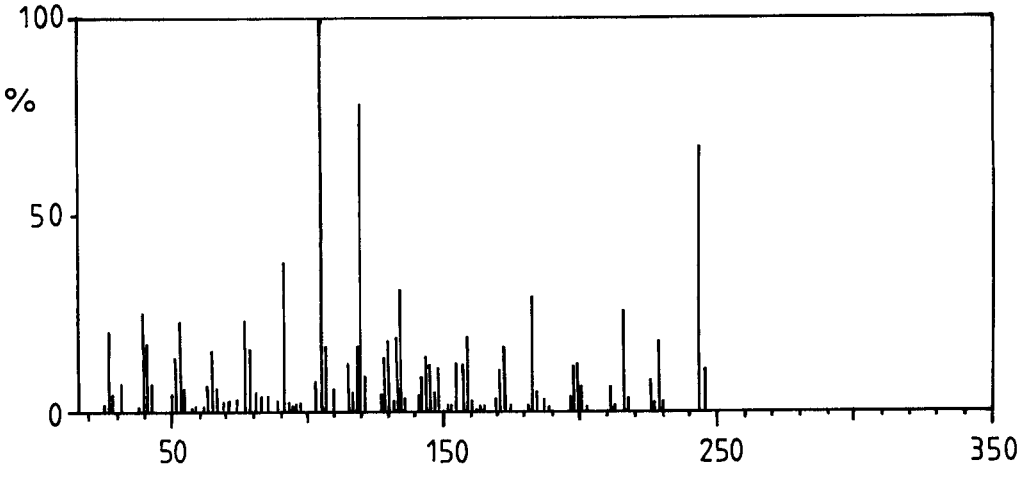

Chlorophyll b

# Deacetoxymatricarin

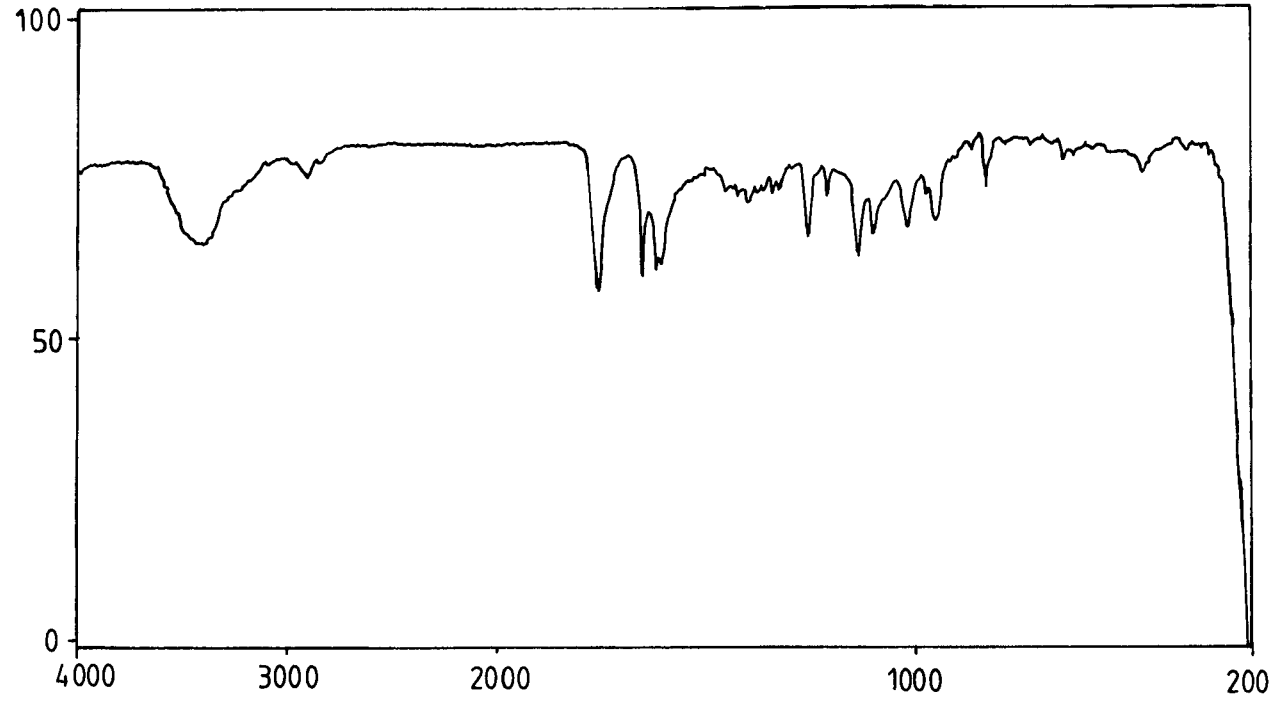
$C_{15}H_{18}O_3$



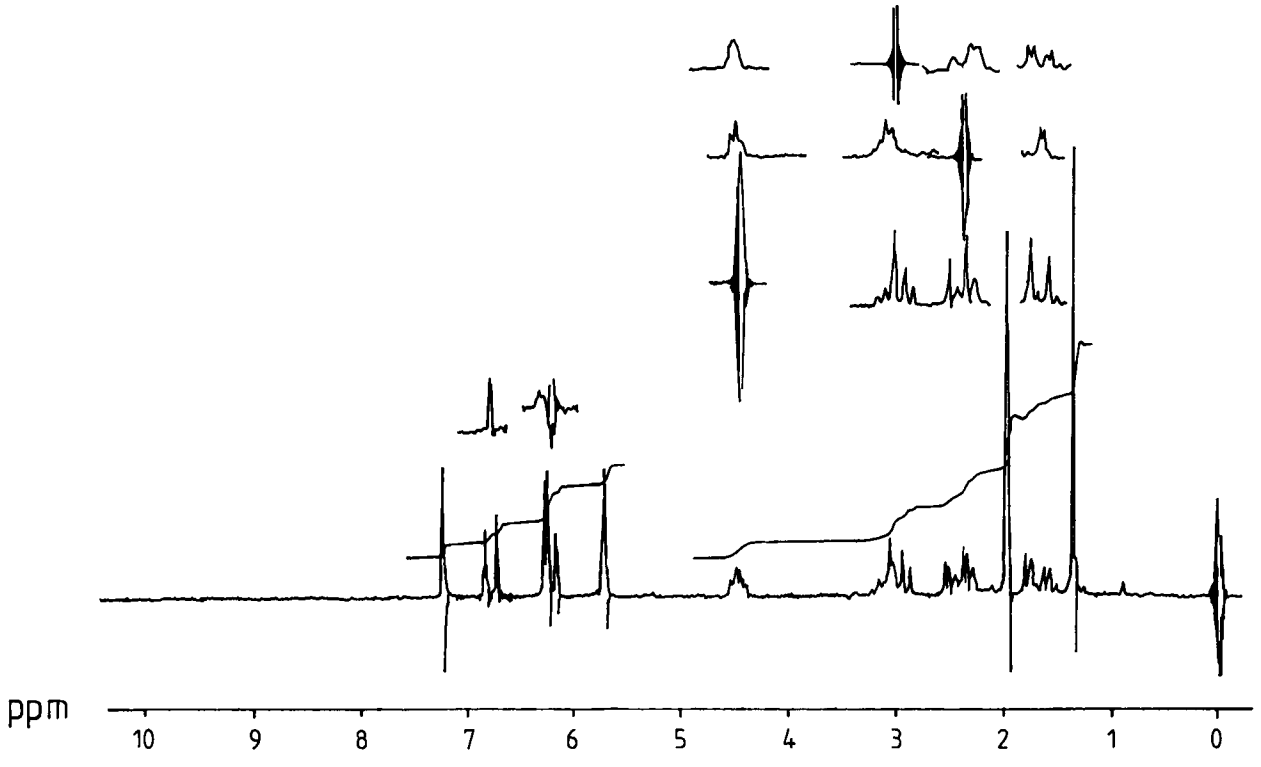

**Mass spectrum** [70 eV]




**IR spectrum** [KBr]

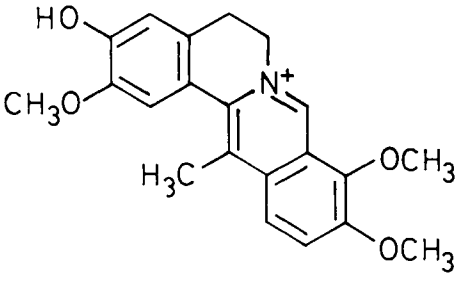


**$^1$H-NMR** [90 MHz, $CDCl_3$]

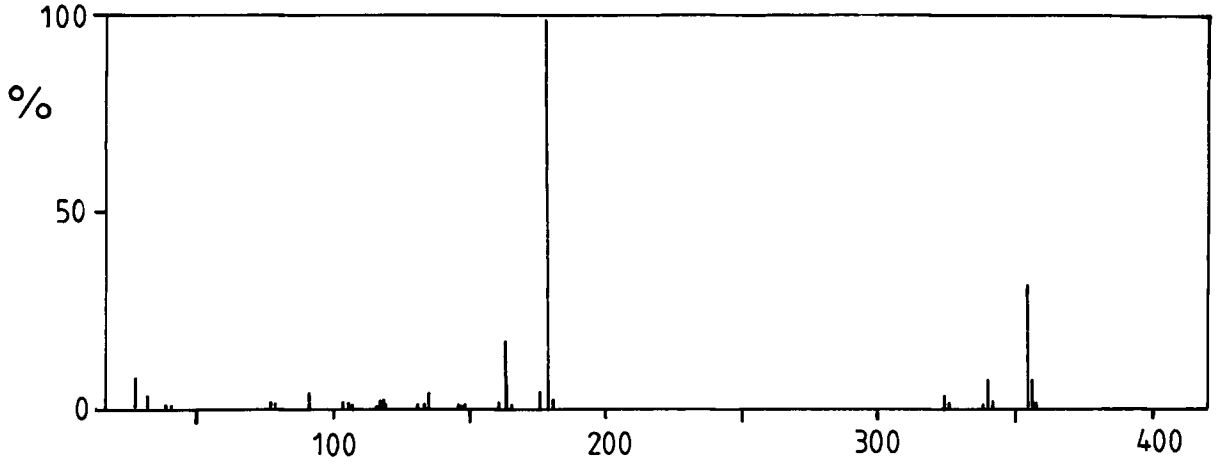


# Dehydrocorybulbine

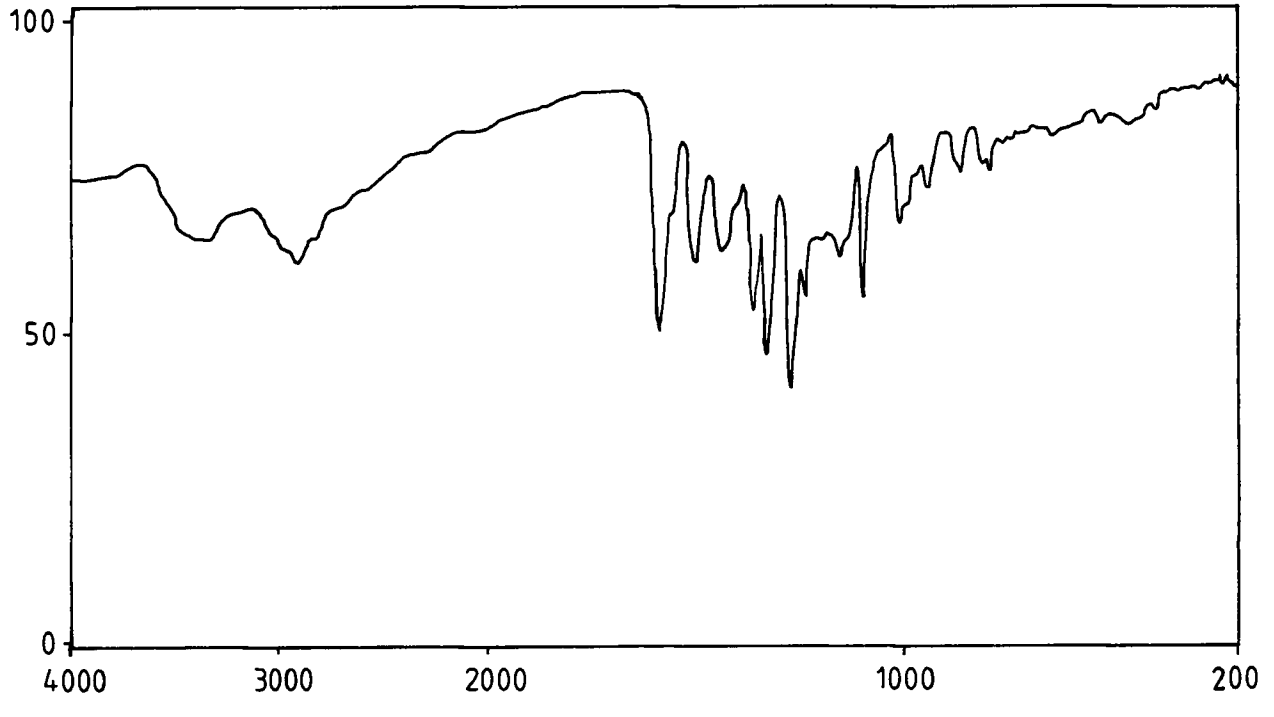
$C_{21}H_{21}O_4N^+$



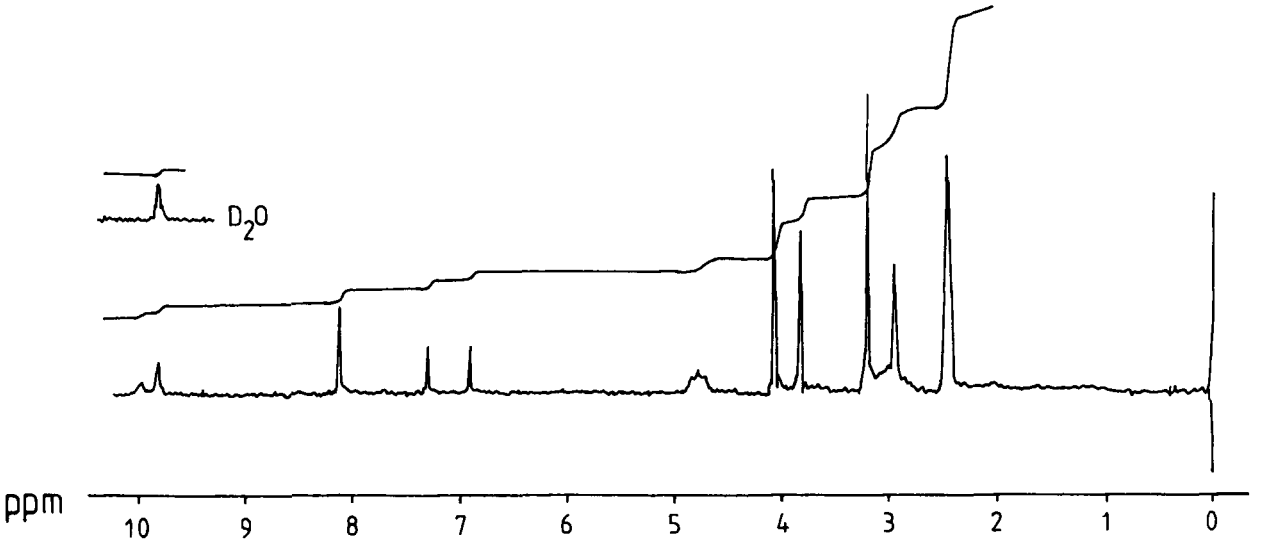

**Mass spectrum** [70 eV]




**IR spectrum** [KBr]

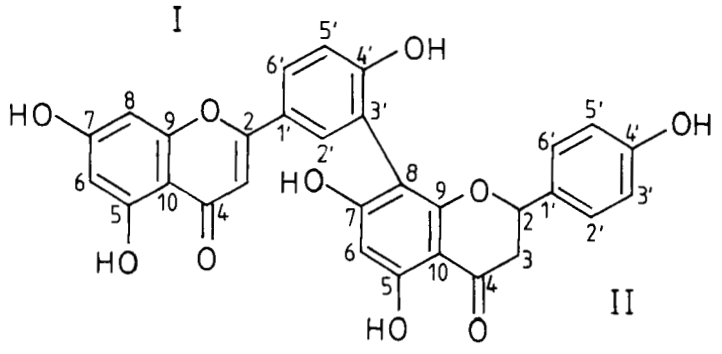


**$^1$H-NMR** [90 MHz, DMSO-$d_6$]

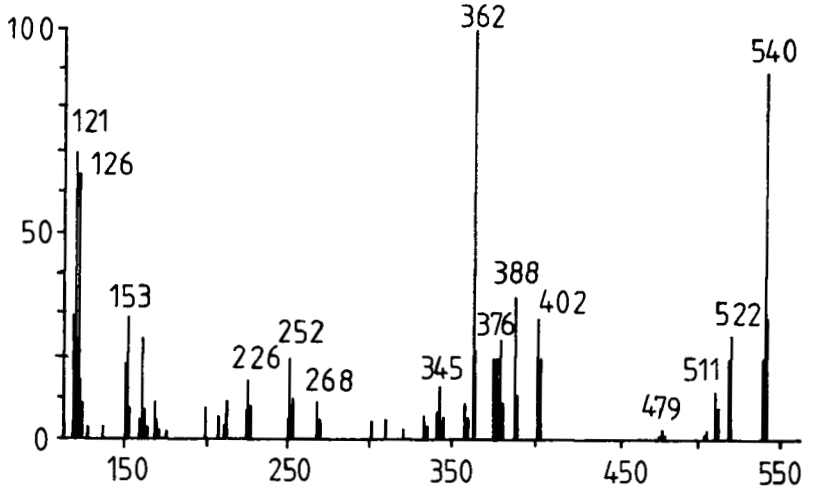


# II-2,3-Dihydroamentoflavone

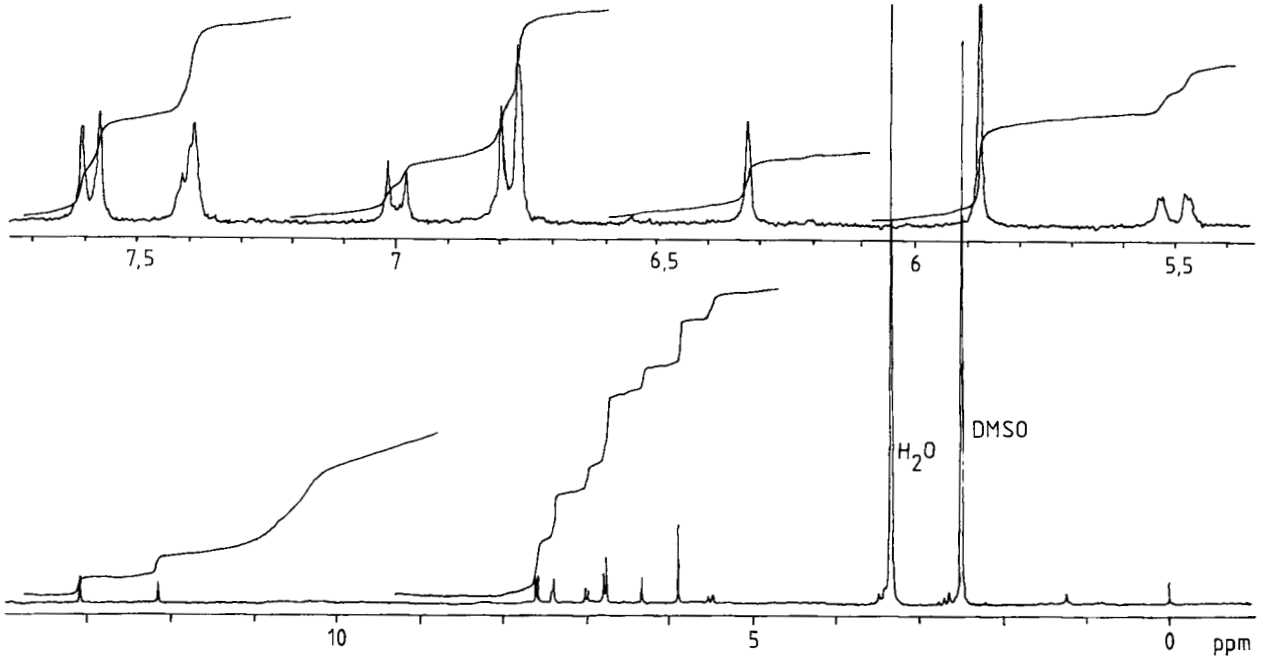
**$C_{30}H_{20}O_{10}$**



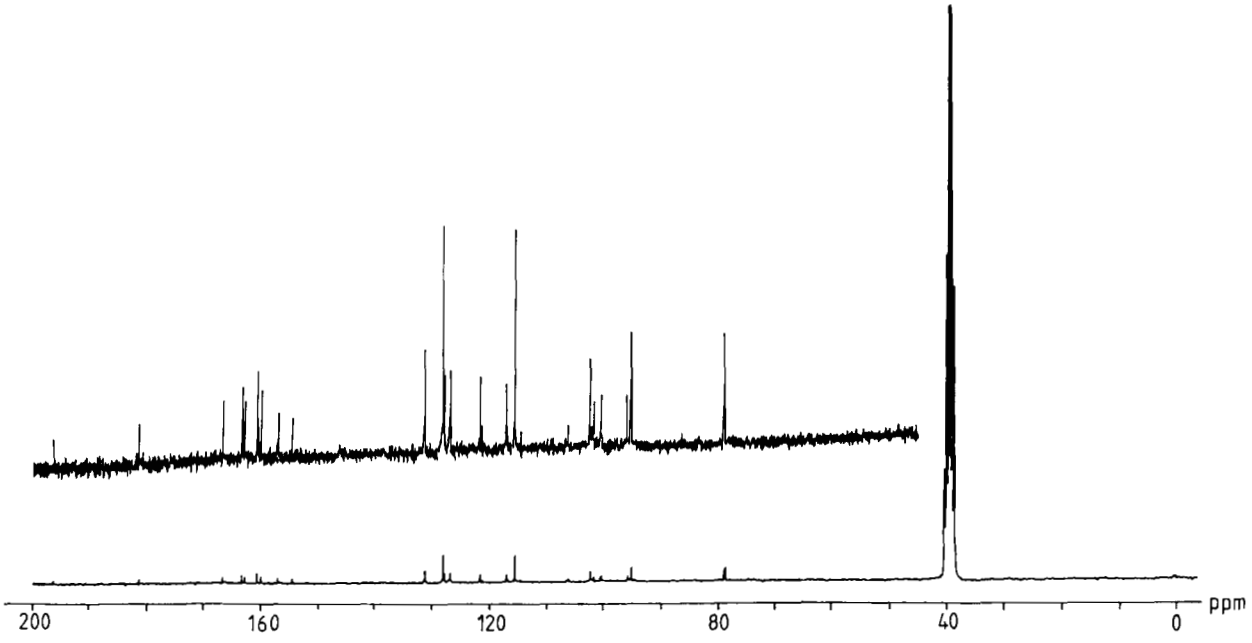

**Mass spectrum** [70 eV]



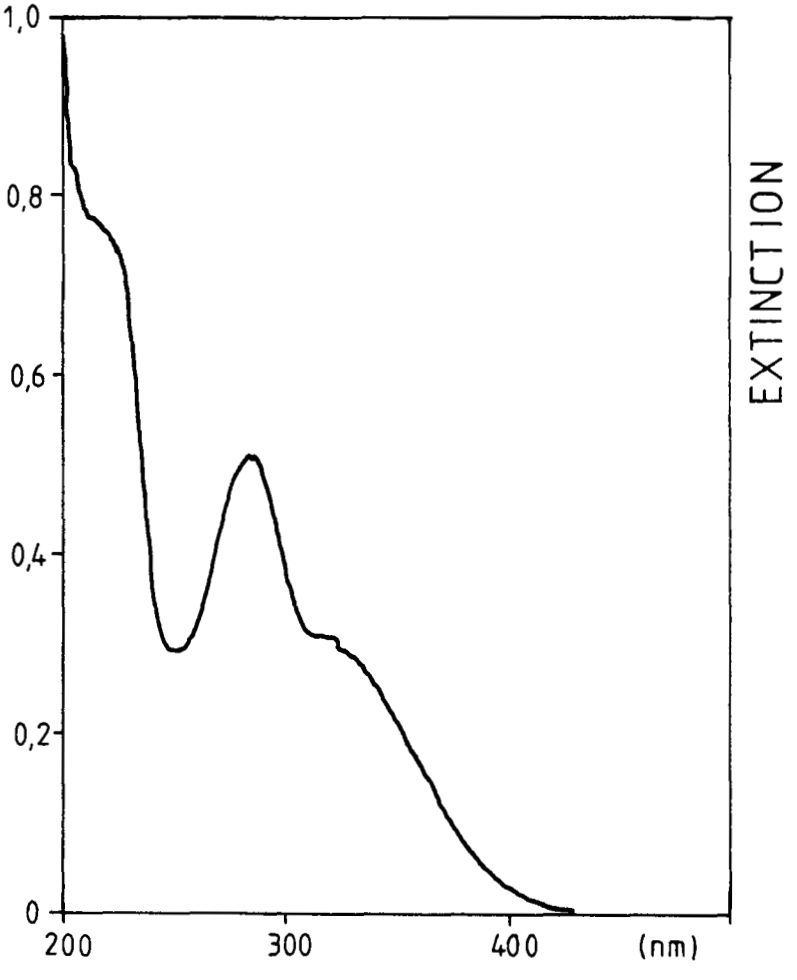

**$^1$H-NMR** [250 MHz, DMSO-$d_6$]



**$^{13}$C-NMR** [62.9 MHz, DMSO-$d_6$]

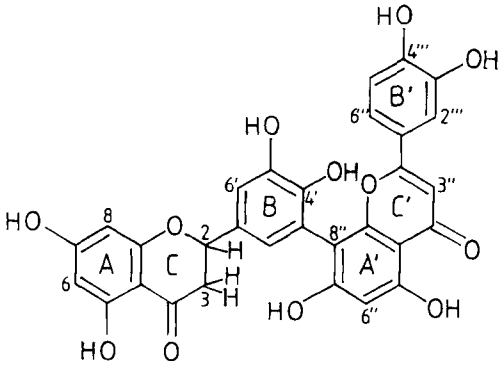



**UV spectrum** [methanol]

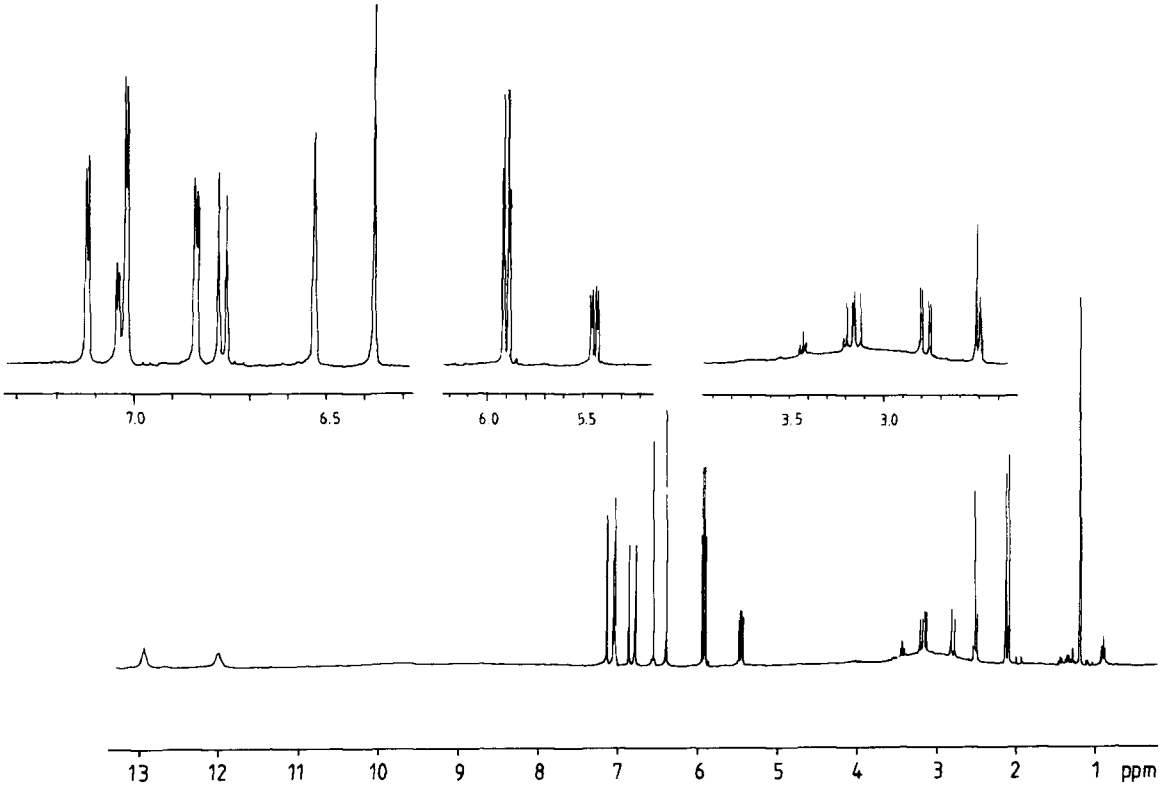


# 2,3-Dihydro-5′,3‴-dihydroxyamentoflavone

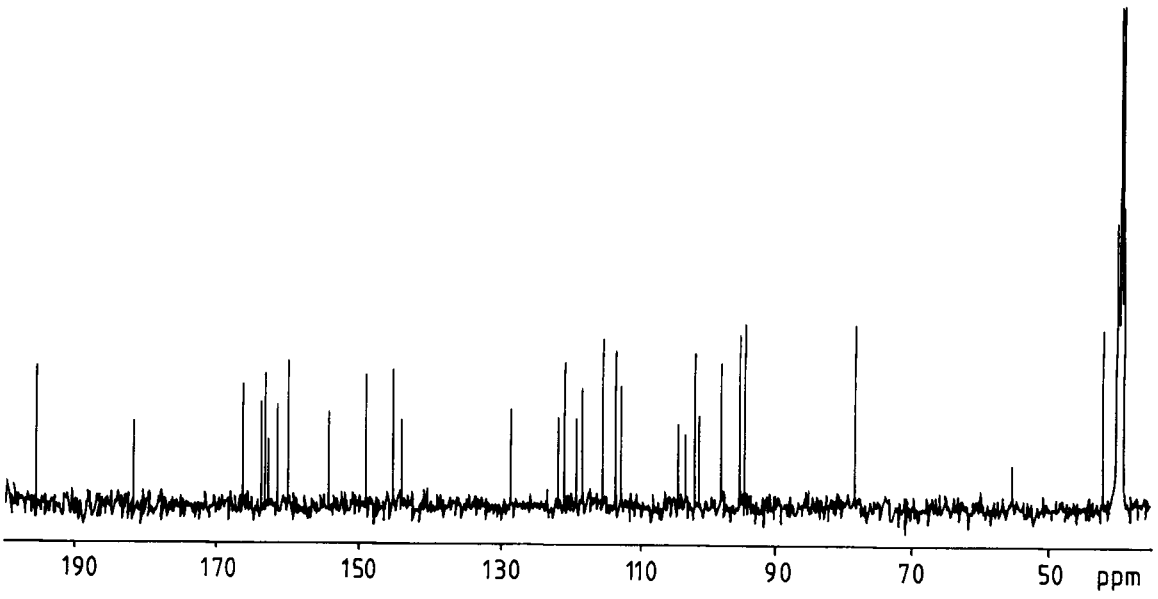
**$C_{30}H_{20}O_{12}$**



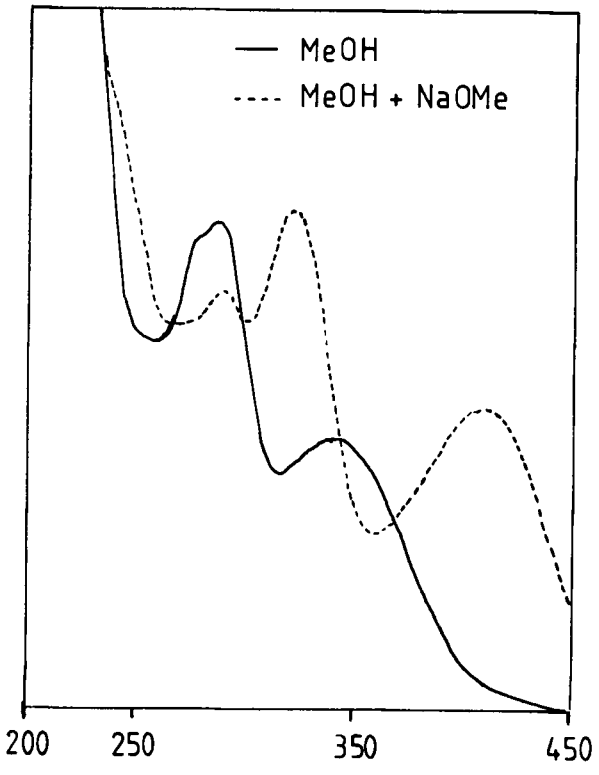

**$^1$H-NMR** [400 MHz, DMSO-$d_6$]



**$^{13}$C-NMR** [100 MHz, DMSO-$d_6$]

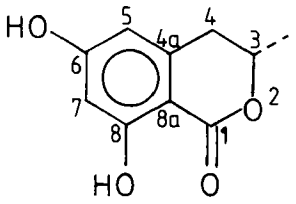



**UV spectrum**

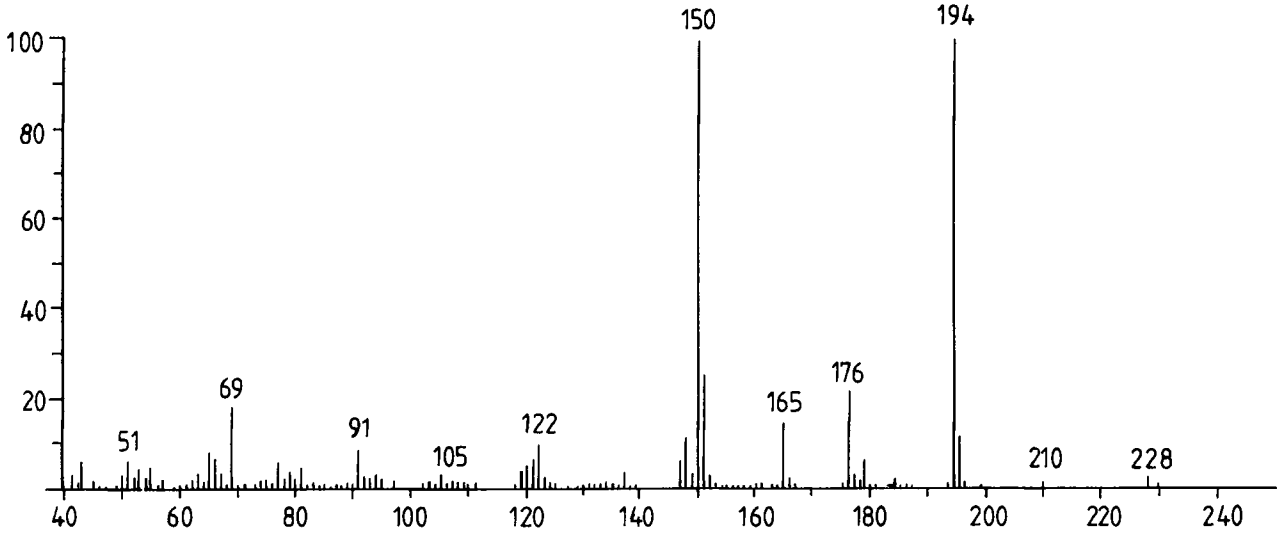


# (-)-3,4-Dihydro-6,8-dihydroxy-3-methylisocoumarin

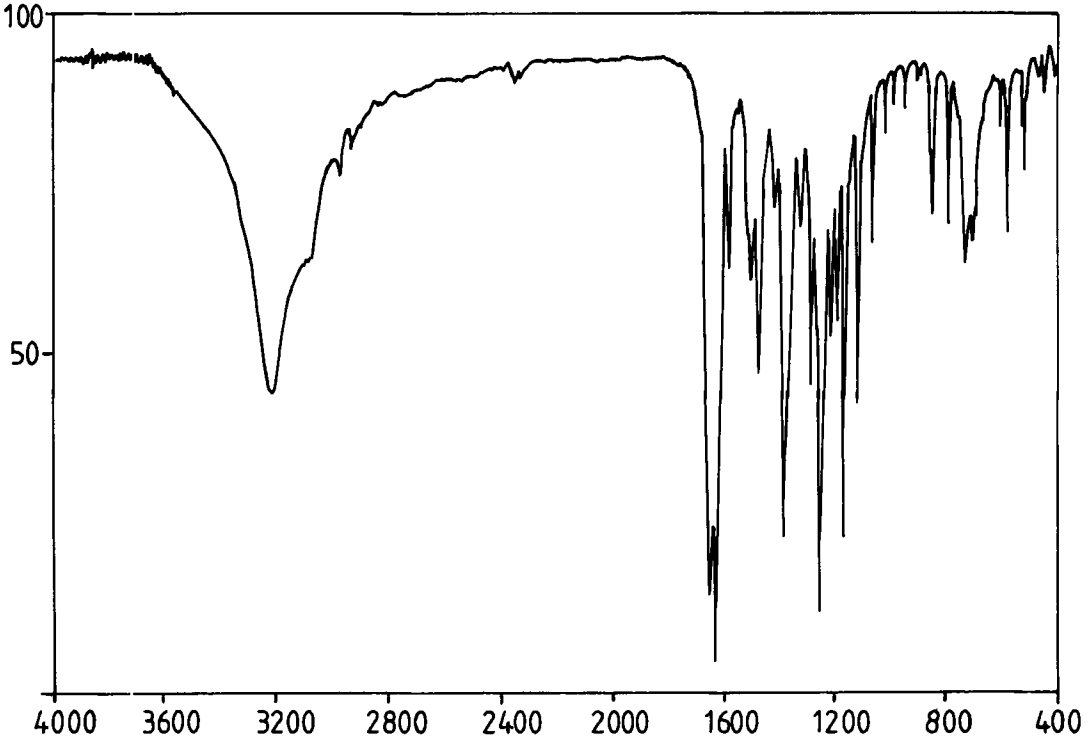
$C_{10}H_{10}O_4$



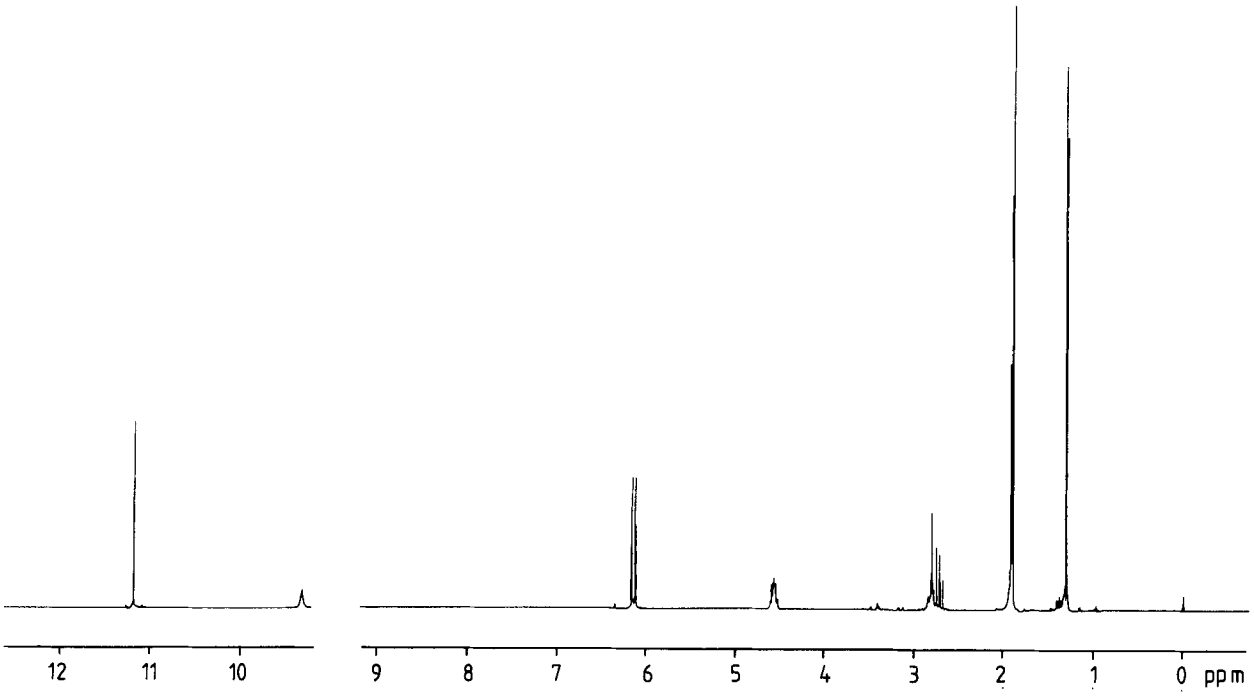

**Mass spectrum** [70 eV]



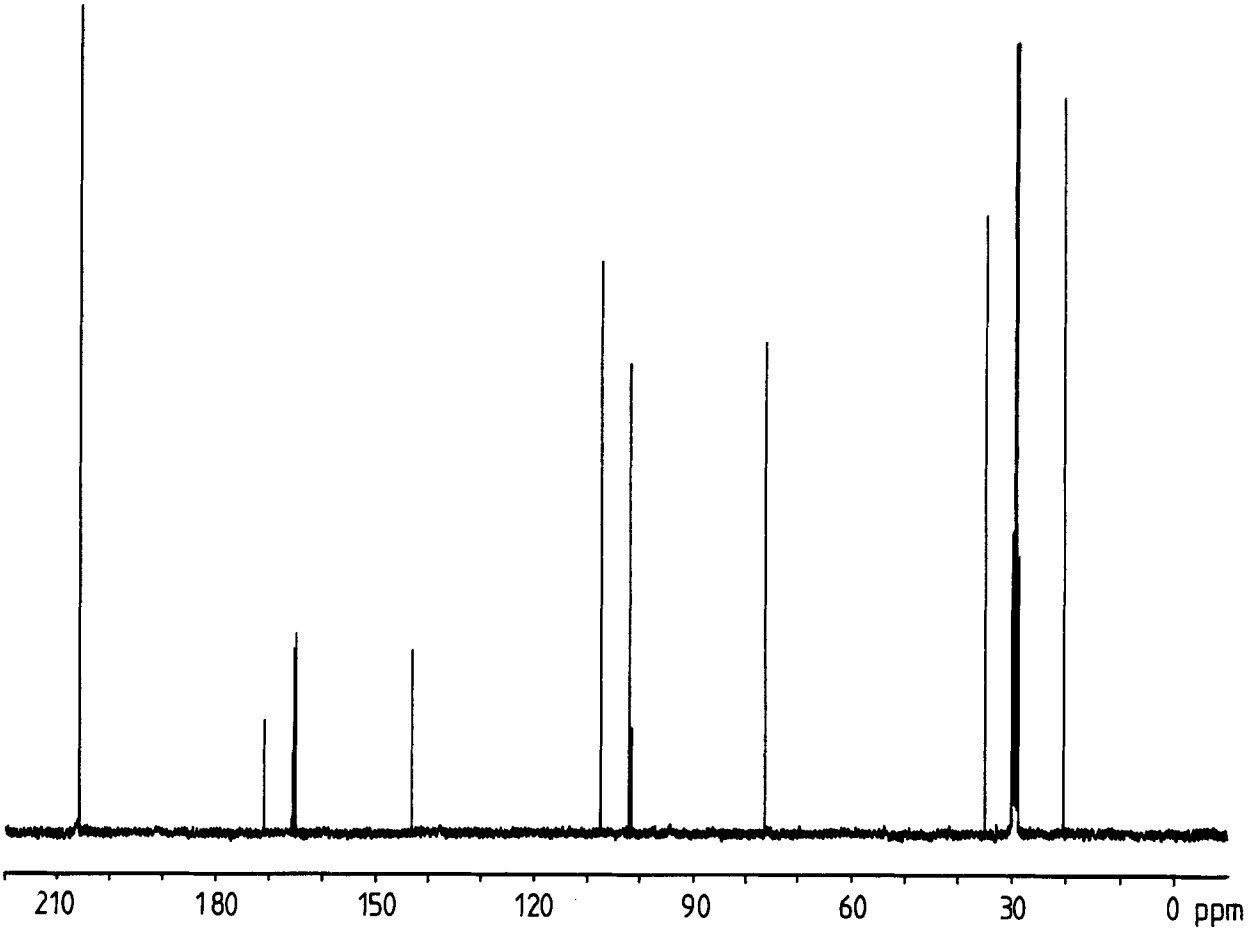

**IR spectrum** [KBr]



**$^1$H-NMR** [400 MHz, $d_6$-acetone]

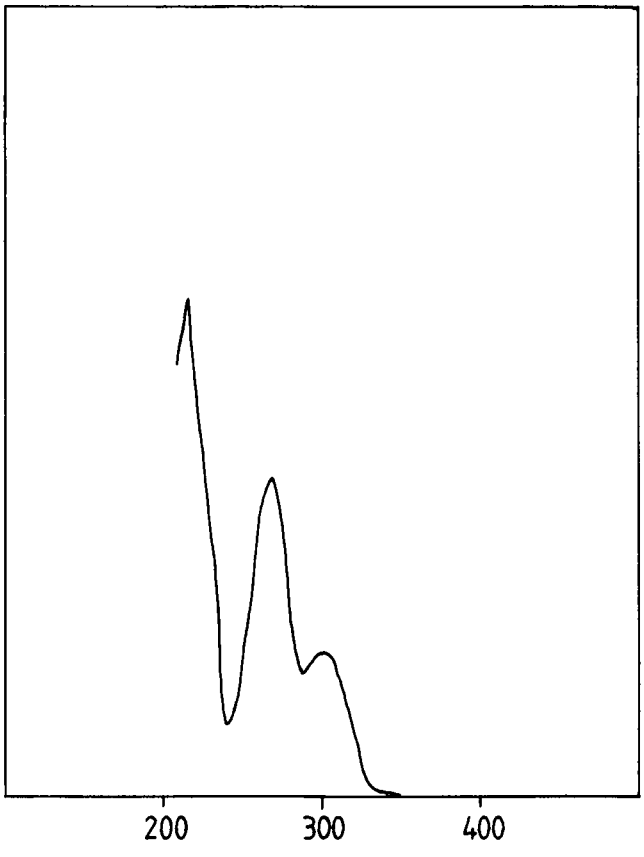



**$^{13}$C-NMR** [100 MHz, $d_6$-acetone]




(-)-3,4-Dihydro-6,8-dihydroxy-3-methylisocoumarin

**UV spectrum** [methanol]



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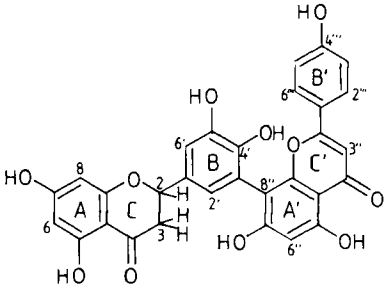
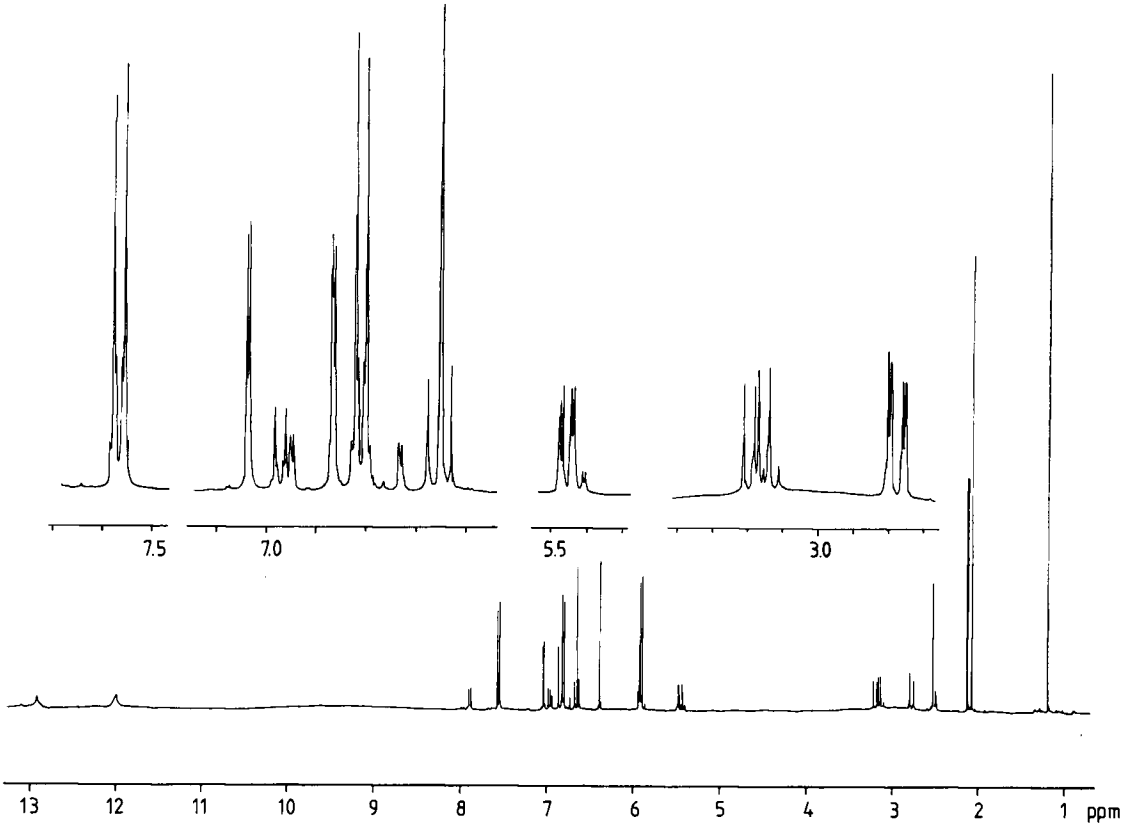
# 2,3-Dihydro-5′-hydroxyamentoflavone

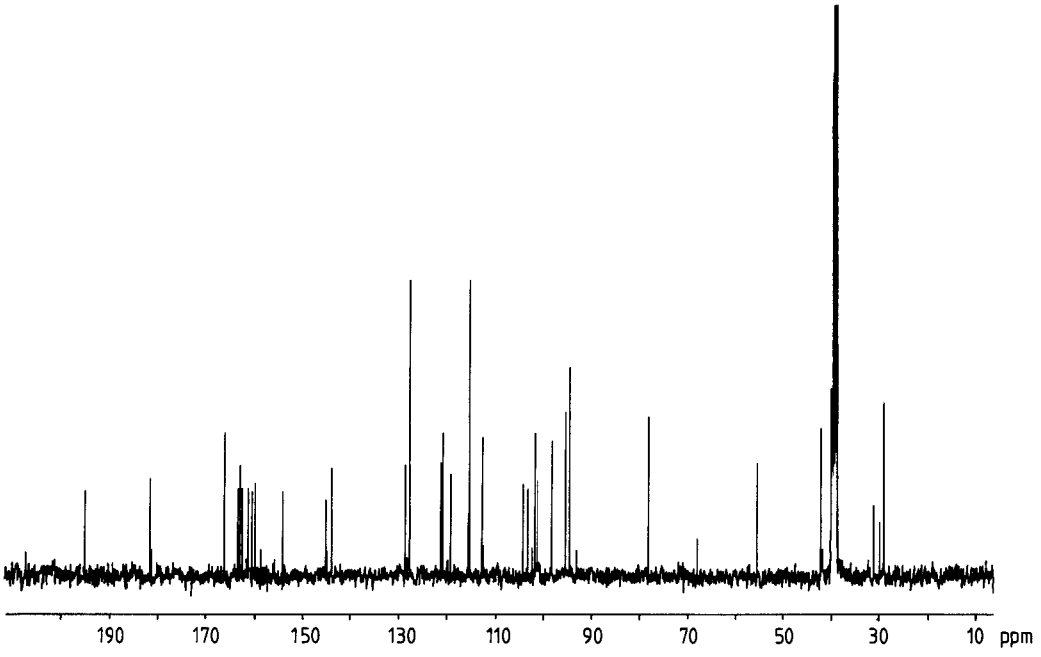
$C_{30}H_{20}O_{11}$



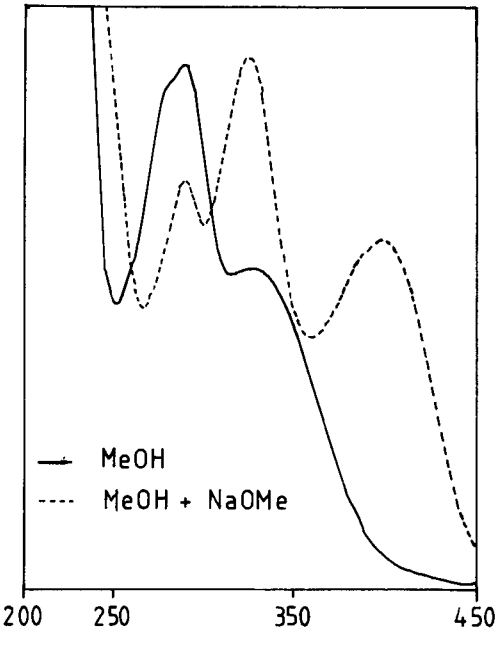
**$^1$H-NMR** [400 MHz, DMSO-$d_6$]



**$^{13}$C-NMR** [100 MHz, DMSO-$d_6$]

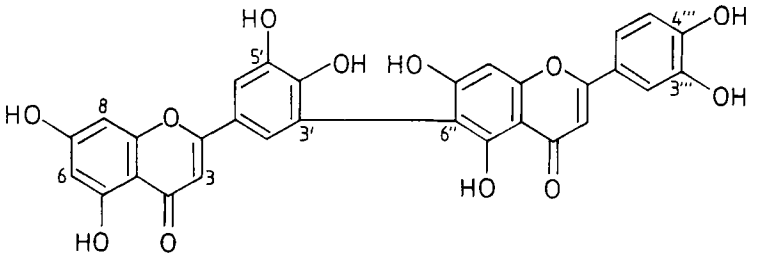



**UV spectrum** [methanol]

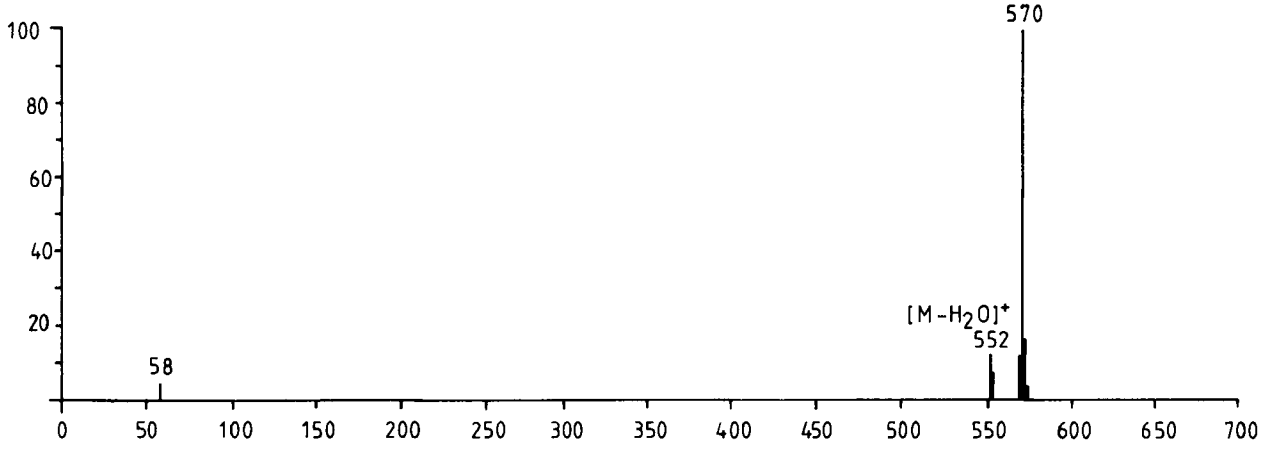


# 5′,3‴-Dihydroxyrobustaflavone

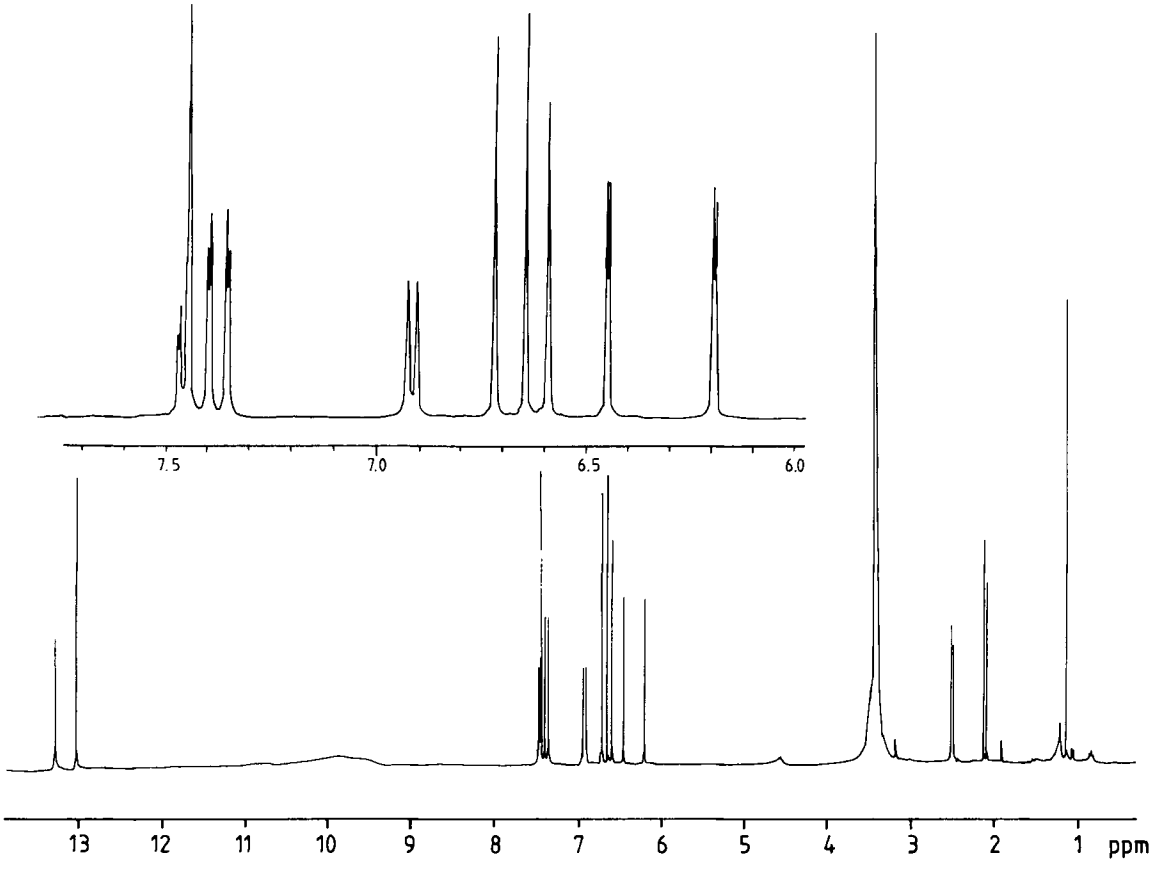
**$C_{30}H_{18}O_{12}$**



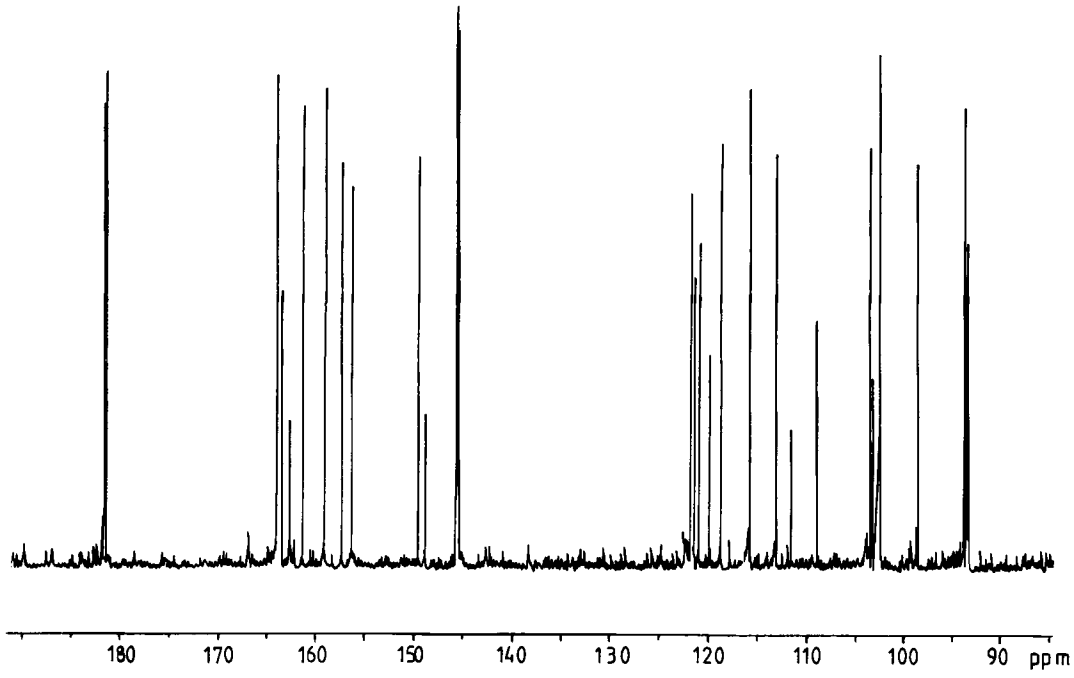

**Mass spectrum**



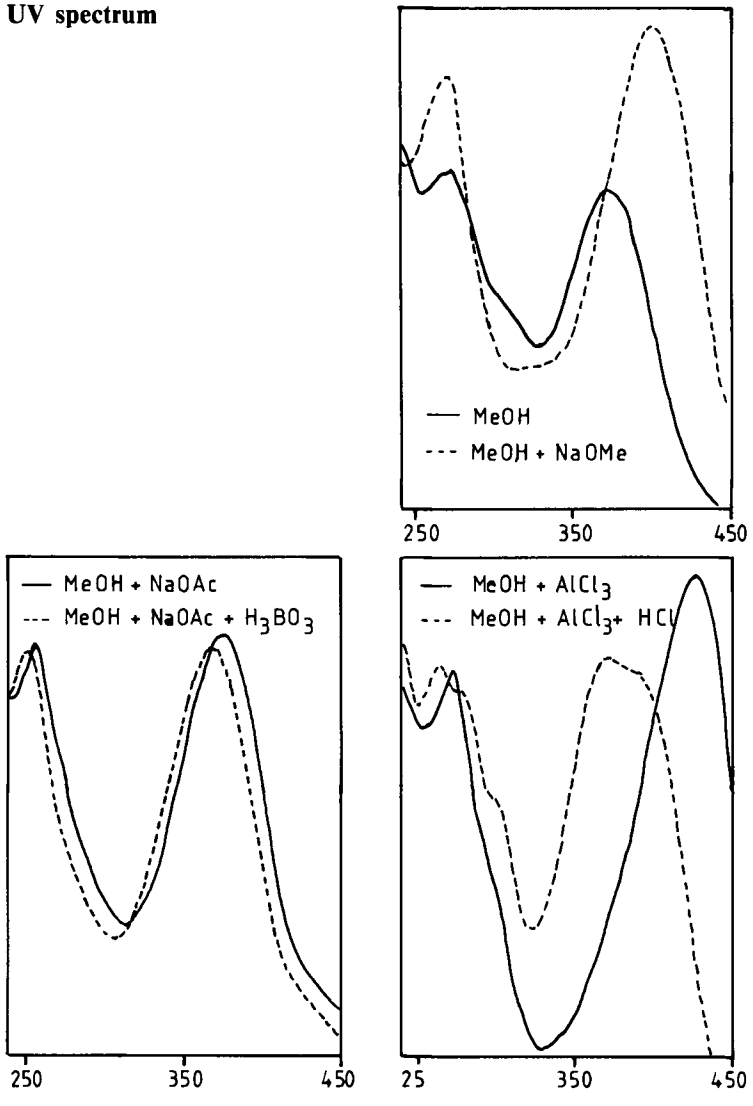

**$^1$H-NMR** [400 MHz, DMSO-$d_6$]



**$^{13}$C-NMR** [100 MHz, DMSO-$d_6$]



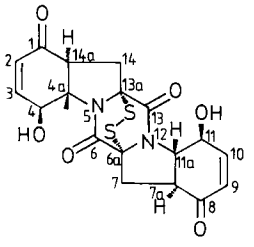

**UV spectrum**



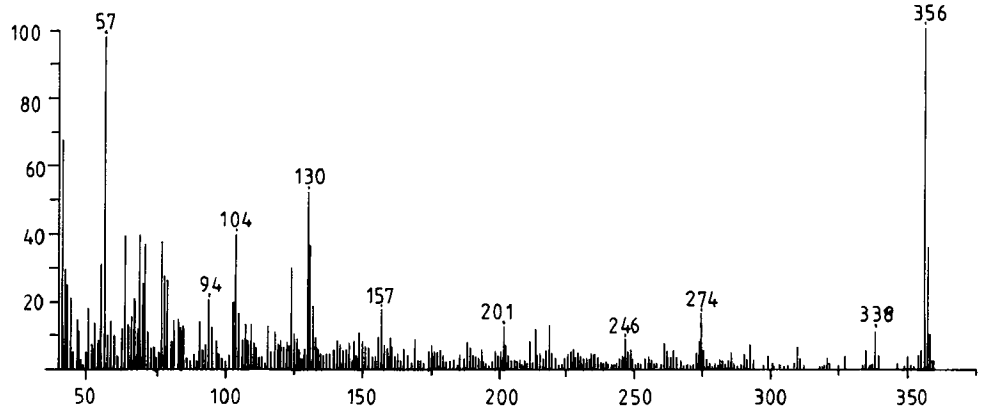

5′,3‴-Dihydroxyrobustaflavone

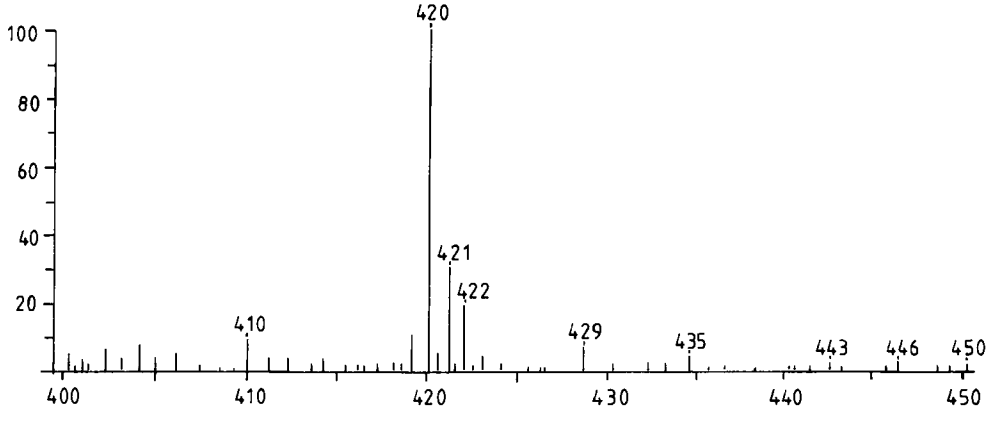
# Epicorazine A

$C_{18}H_{16}N_2O_6S_2$

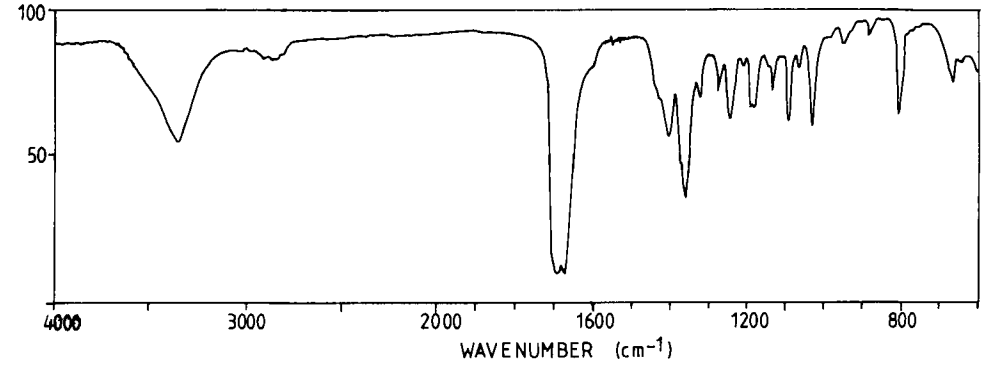


**Mass spectrum** [70 eV]

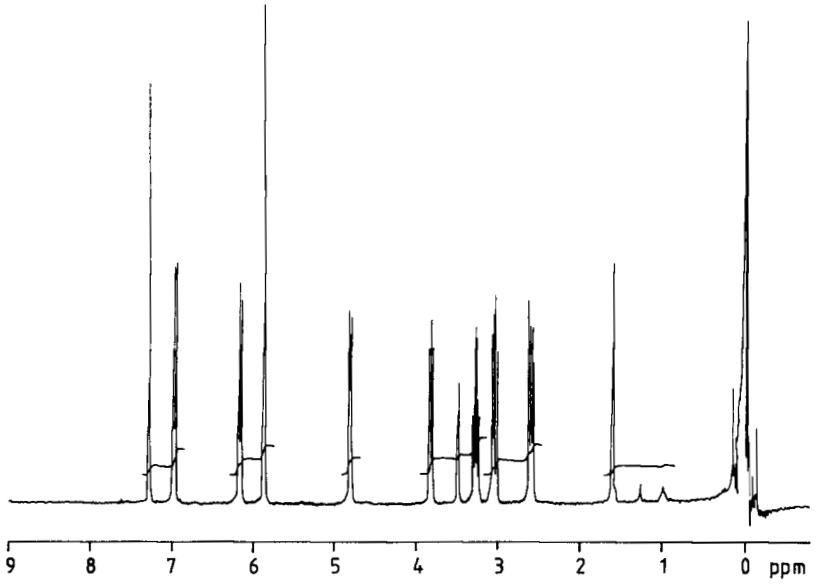


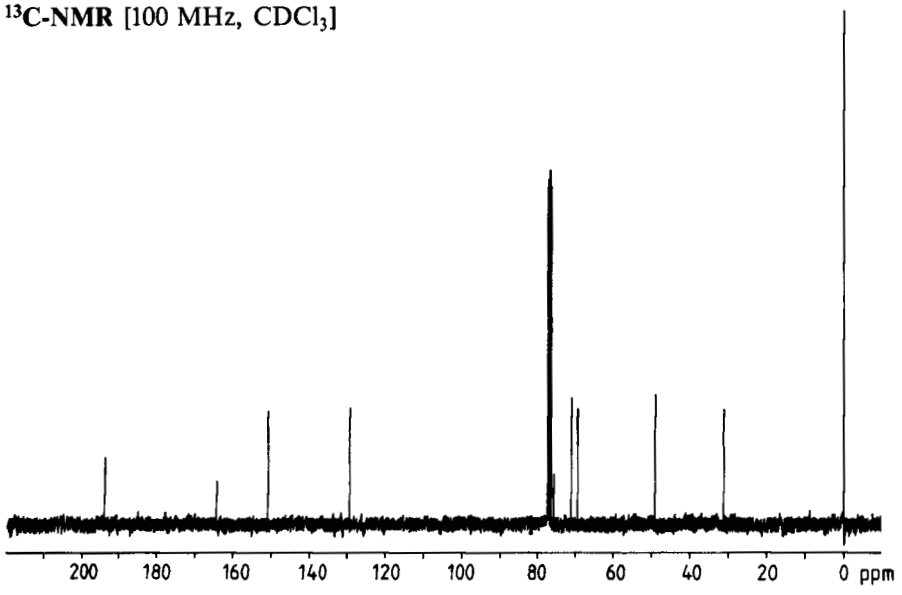

**IR spectrum** [KBr]



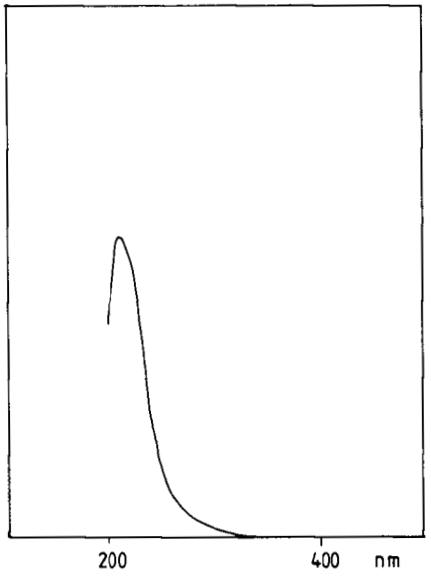
**$^1$H-NMR** [400 MHz, $CDCl_3$]




**$^{13}$C-NMR** [100 MHz, $CDCl_3$]

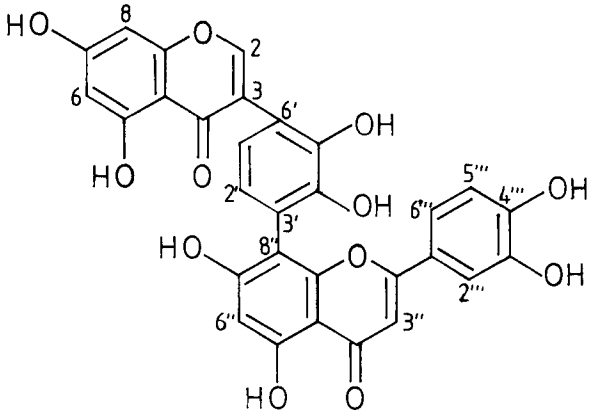



**UV spectrum**



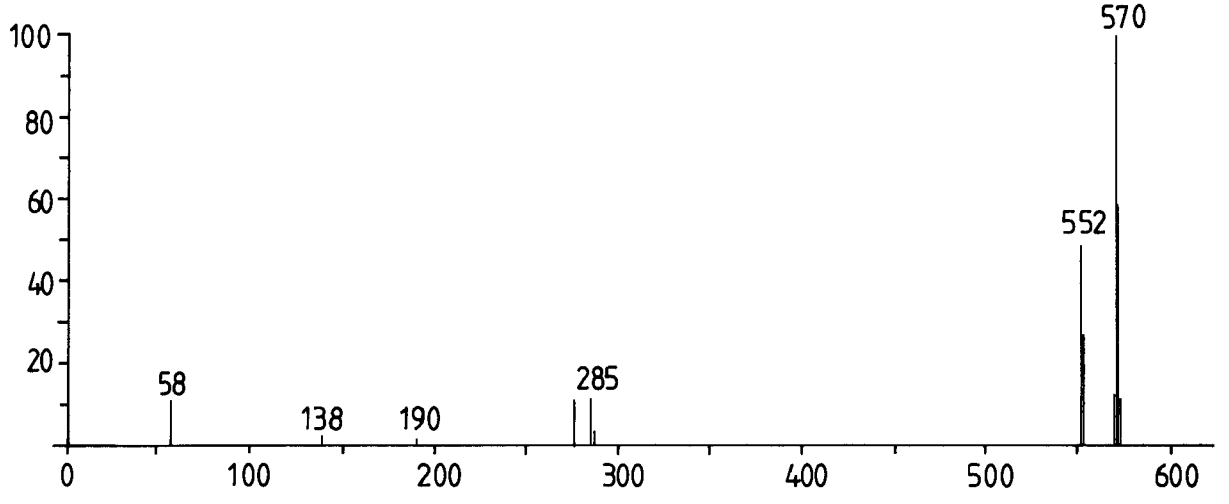

Epicorazine A

# Heterobryoflavone

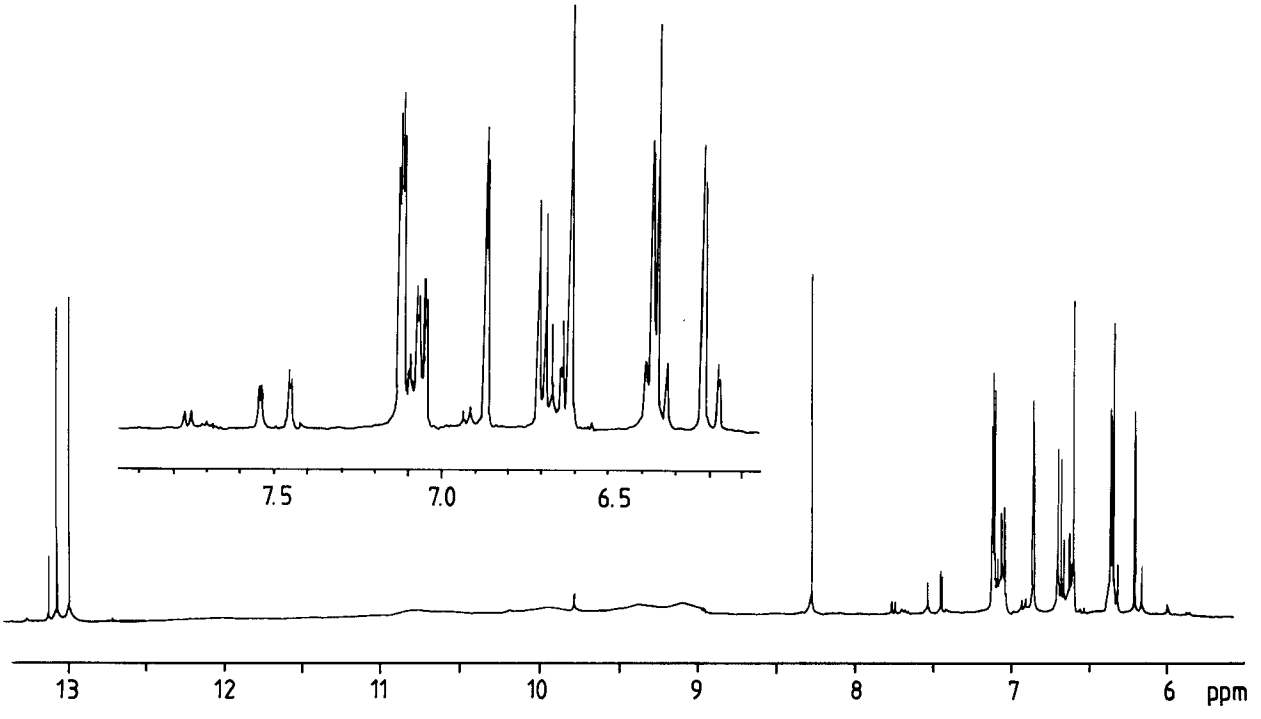



$C_{30}H_{18}O_{12}$

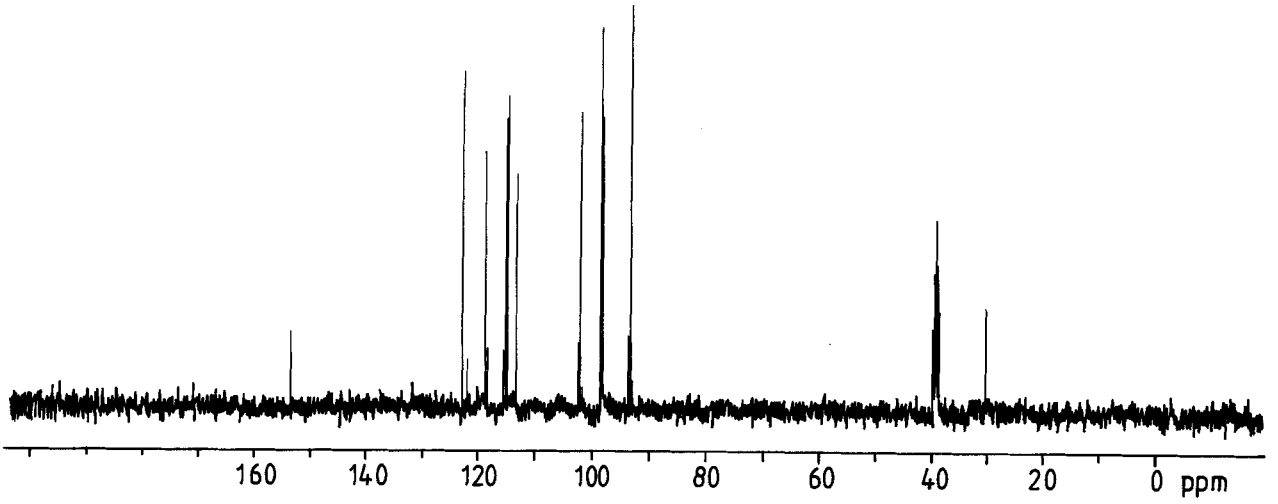
**Mass spectrum**



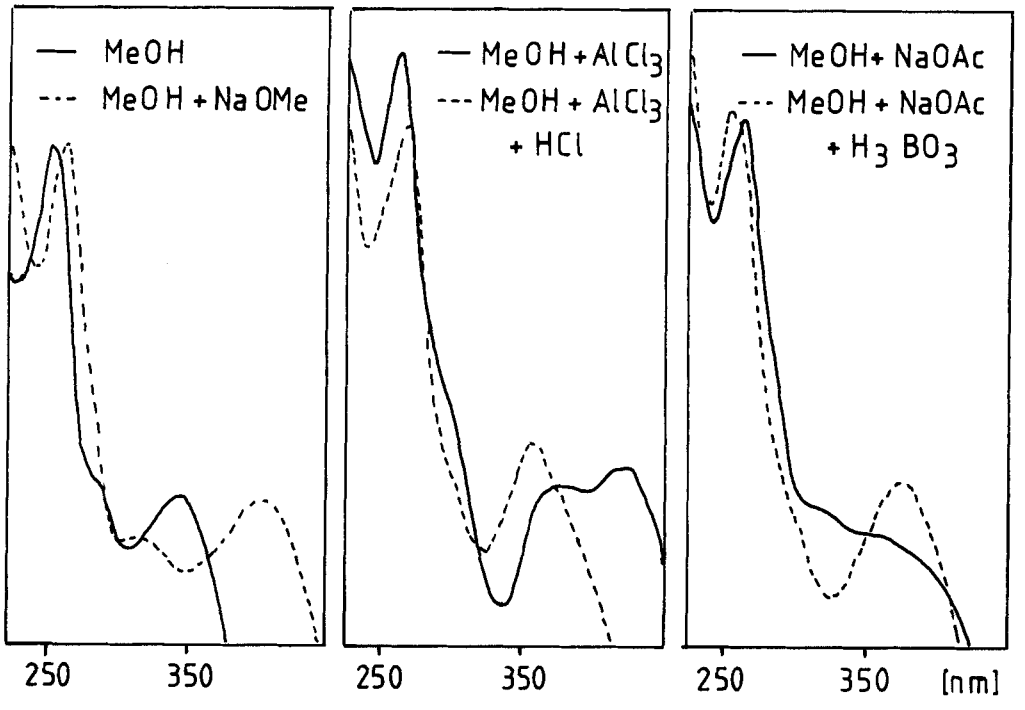

**$^1$H-NMR** [400 MHz, DMSO-$d_6$]



**$^{13}$C-NMR** [100 MHz, DMSO-$d_6$]

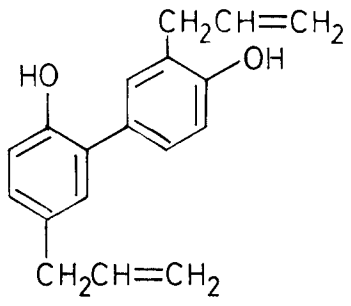



**UV spectrum**

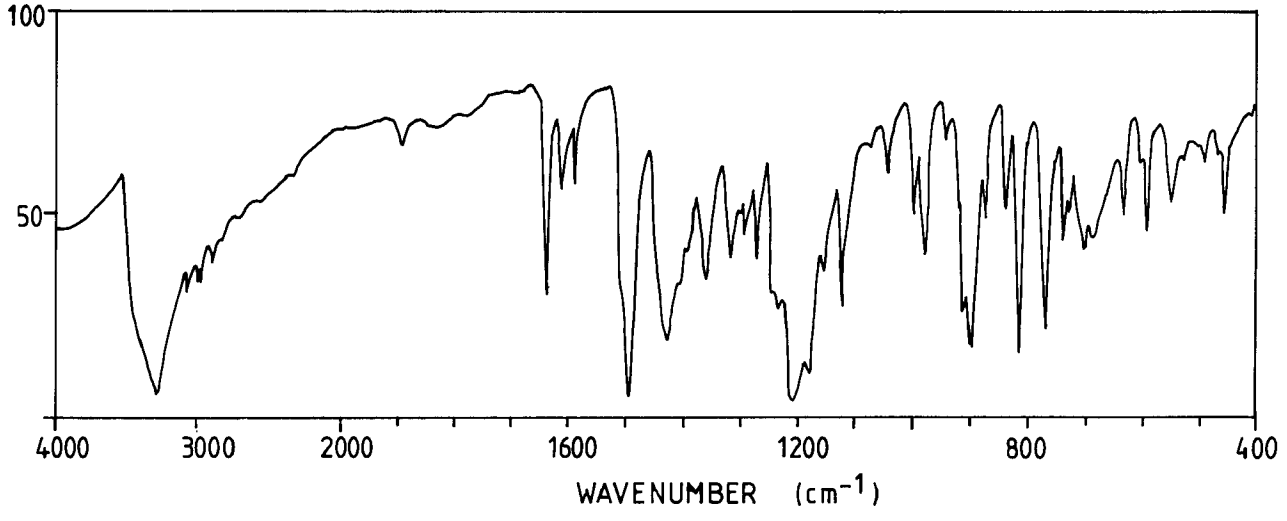


# Honokiol

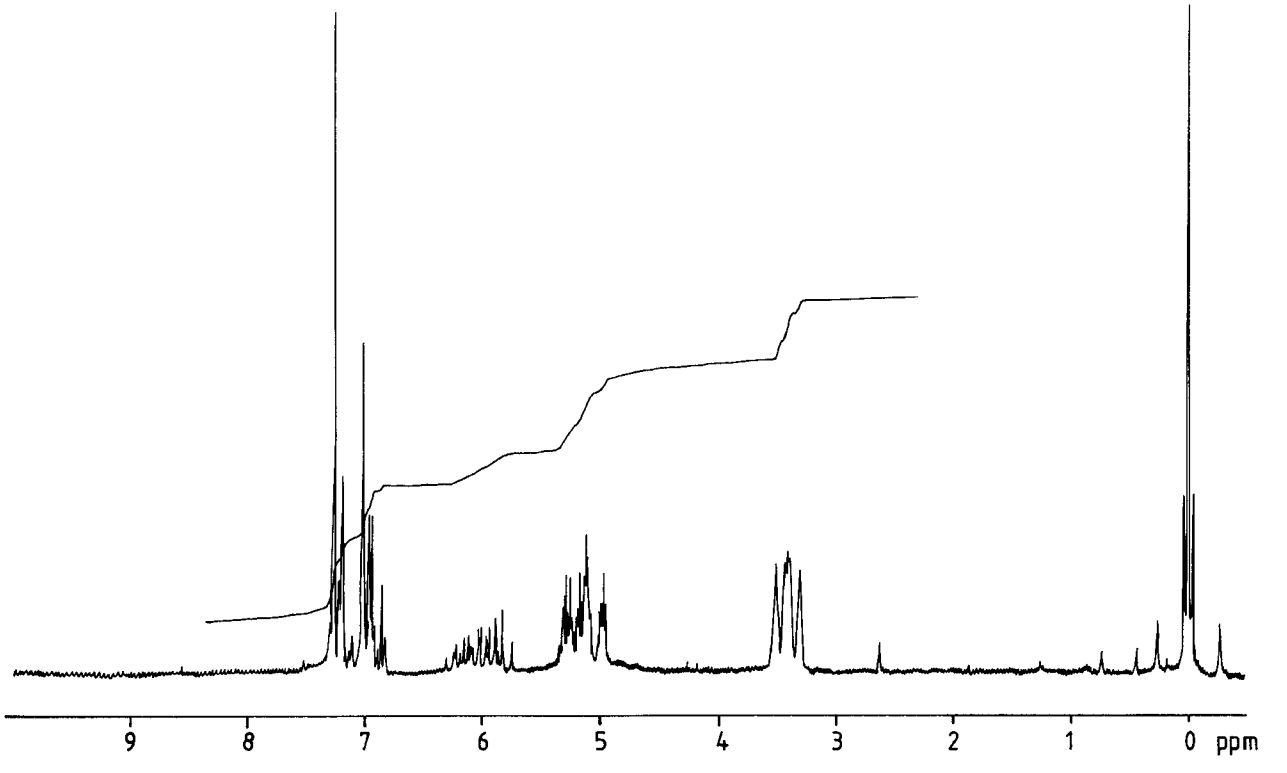
$C_{18}H_{18}O_2$




**IR spectrum** [KBr]




**$^1$H-NMR** [80 MHz, $CDCl_3$]



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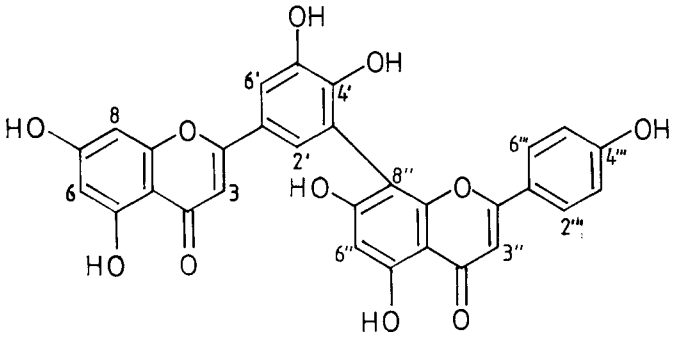
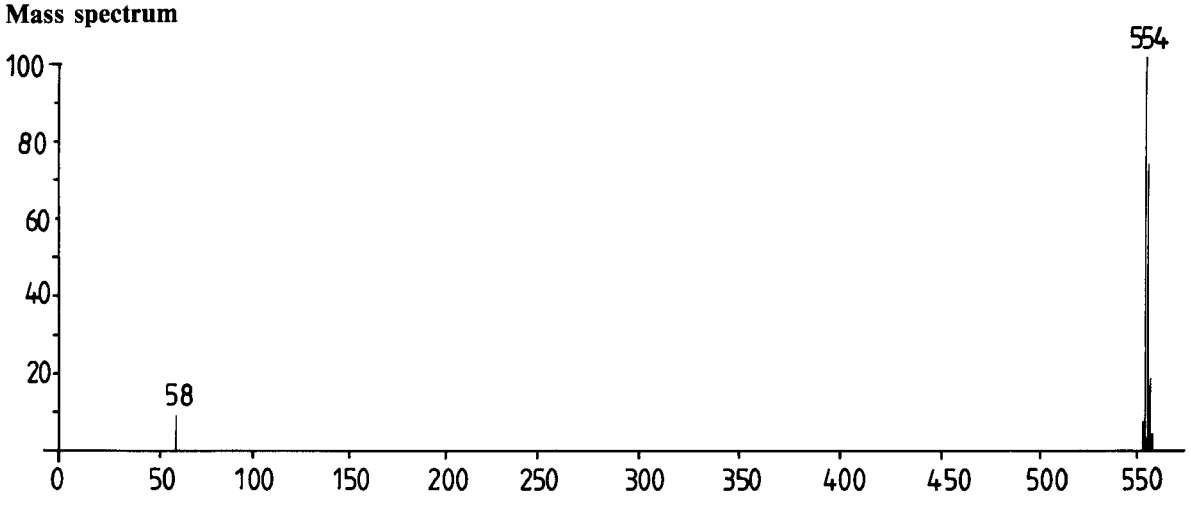
# 5′-Hydroxyamentoflavone

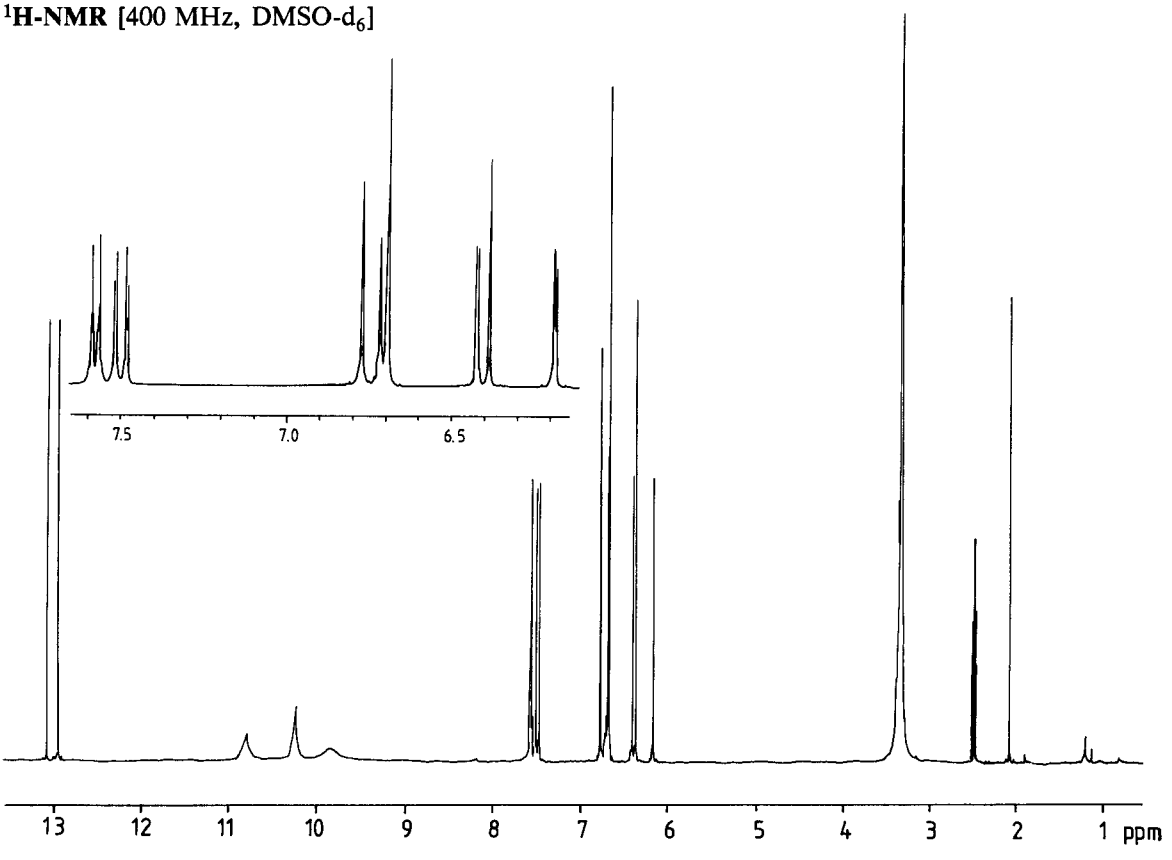
$C_{30}H_{18}O_{11}$



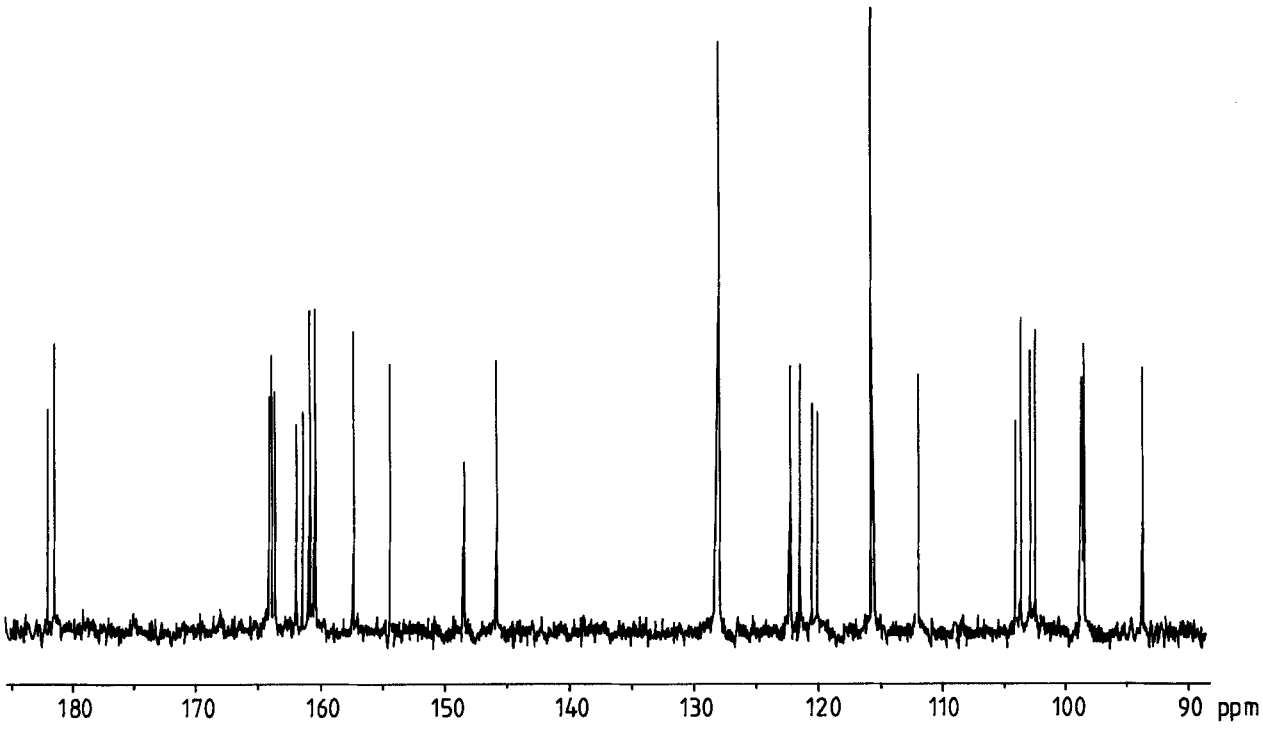

**Mass spectrum**



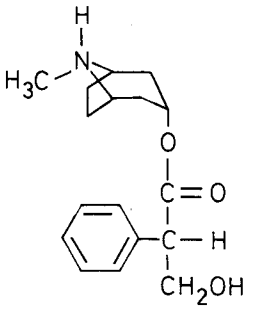

**$^1$H-NMR** [400 MHz, DMSO-$d_6$]



**$^{13}$C-NMR** [100 MHz, DMSO-$d_6$]

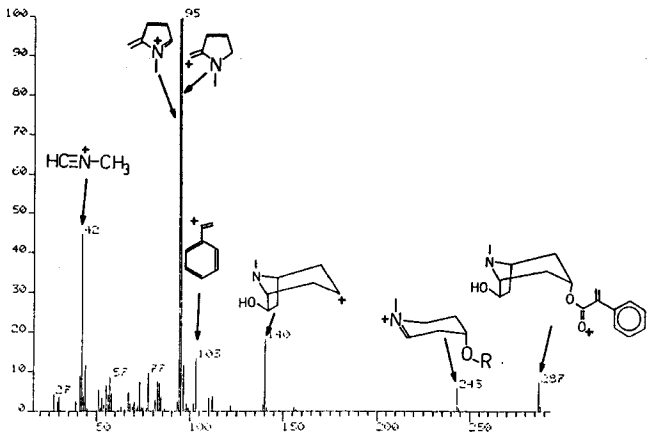


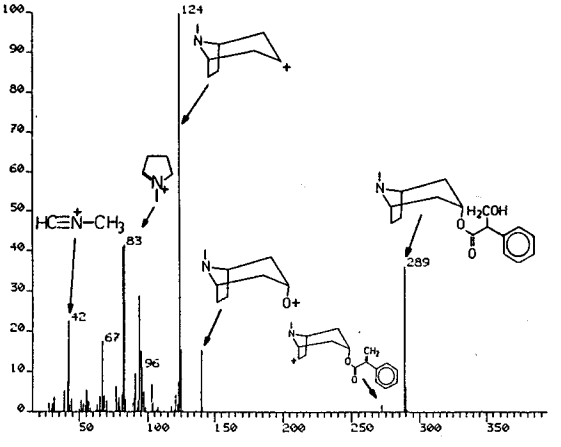
# L-Hyoscyamine

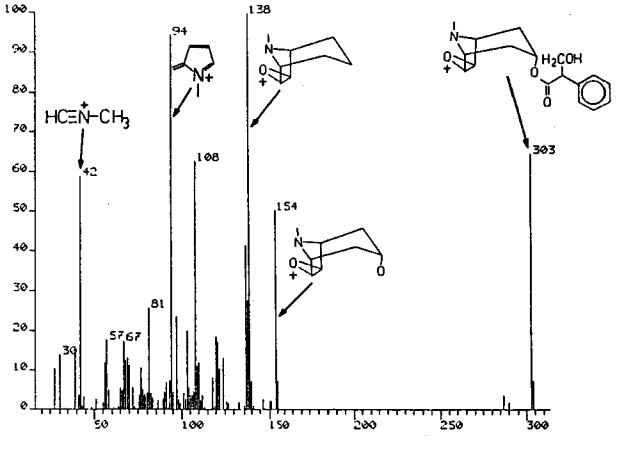


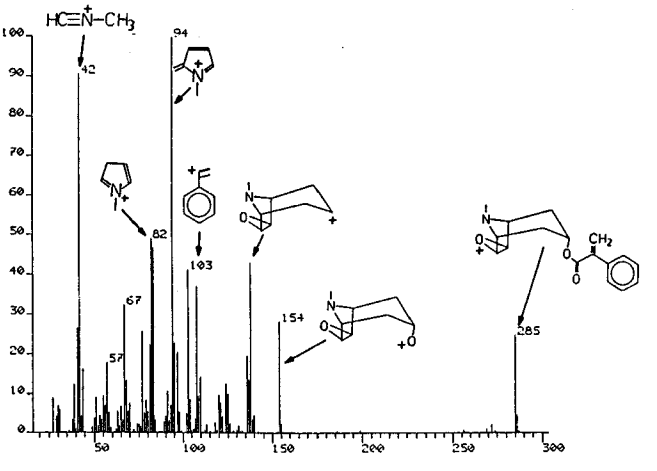
$C_{17}H_{23}NO_3$

**Mass spectra** [24 eV]









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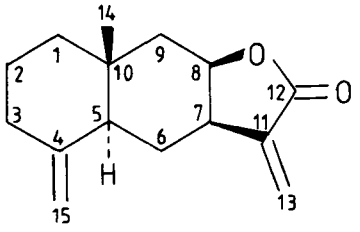
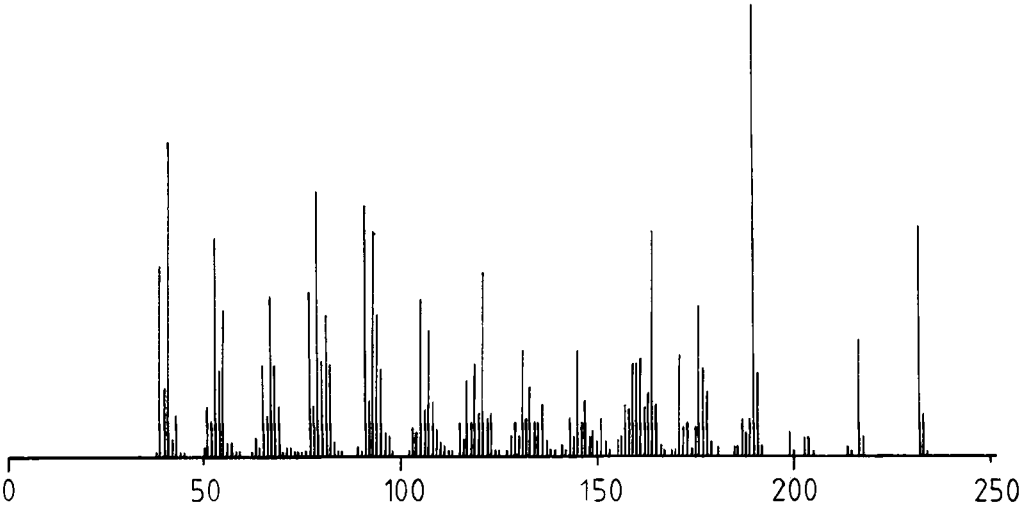
# Isoalantolactone

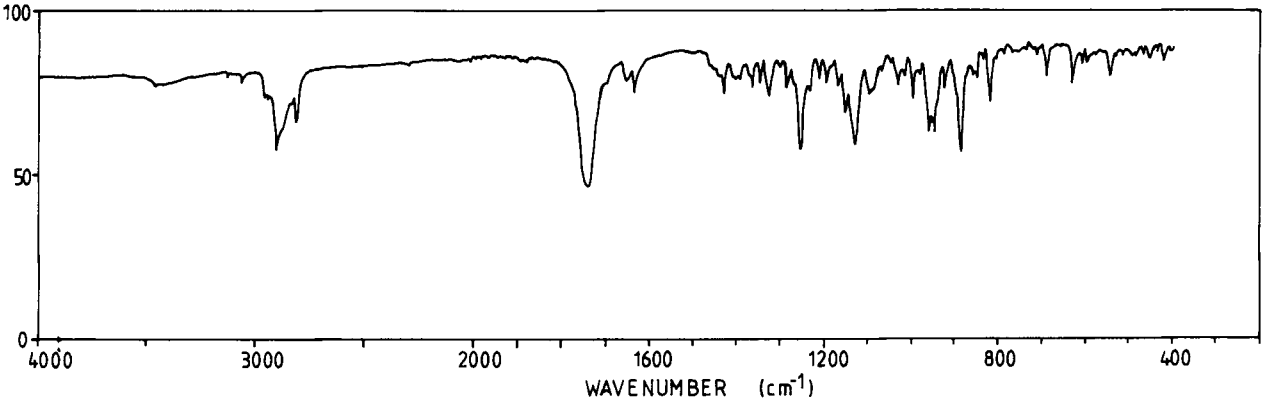
$C_{15}H_{20}O_2$



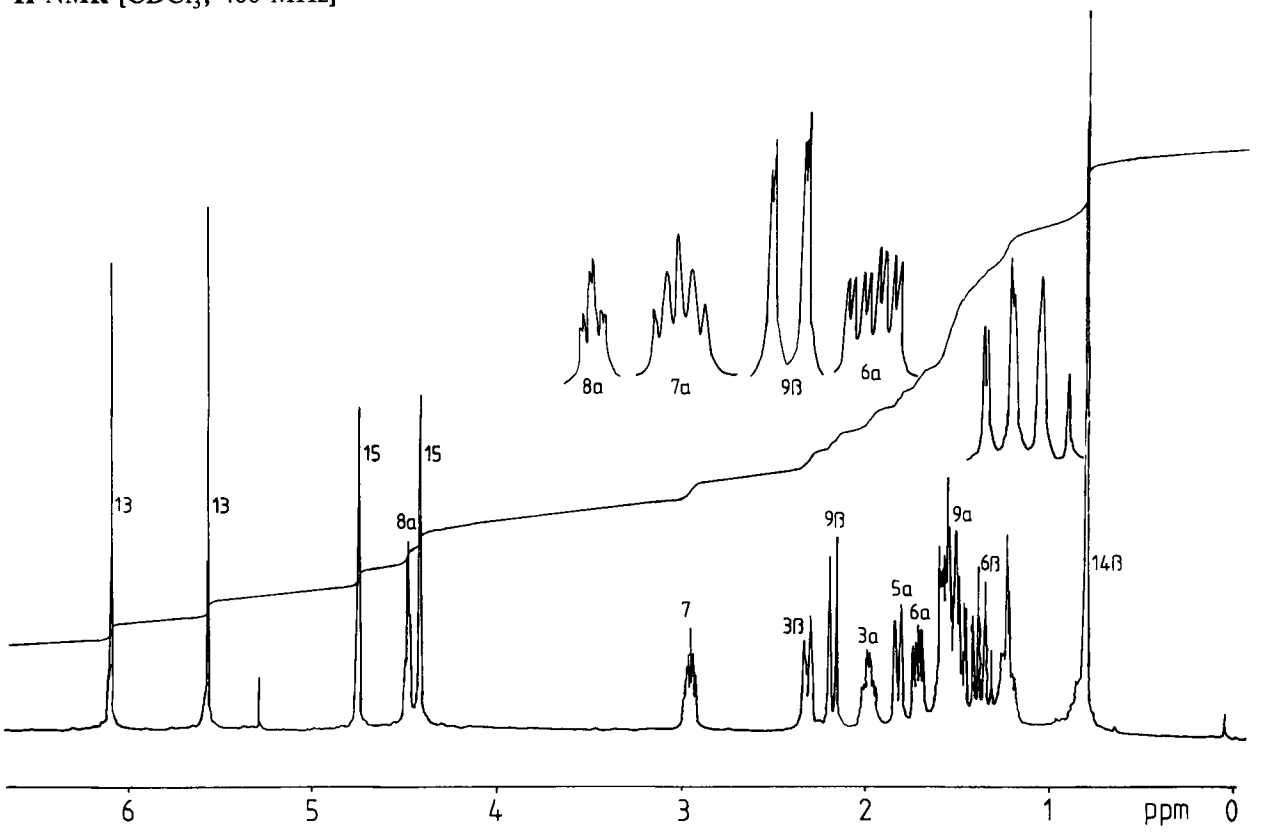

**Mass spectrum** [70 eV]




**IR spectrum** [KBr]

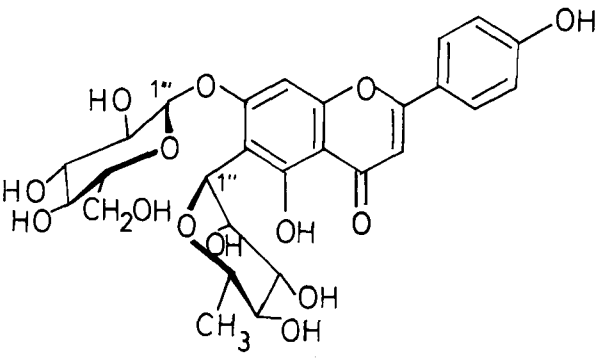


**$^1$H-NMR** [$CDCl_3$, 400 MHz]

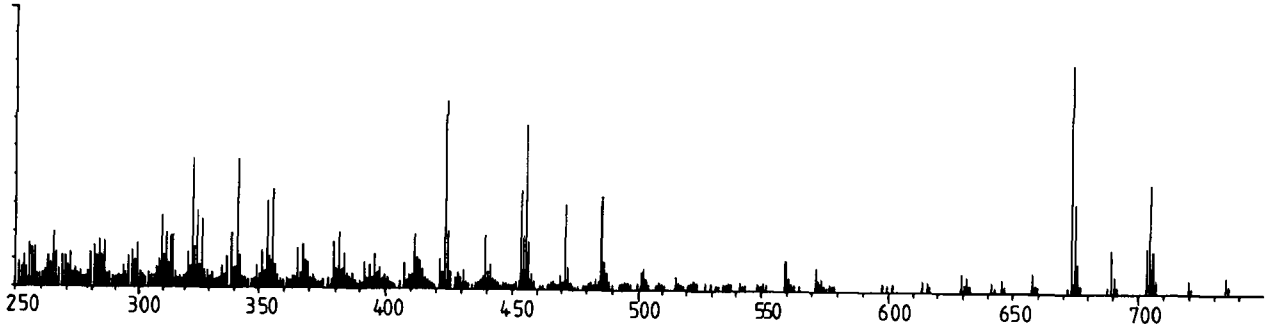


# Isofurcatain-7-O-β-D-glucoside

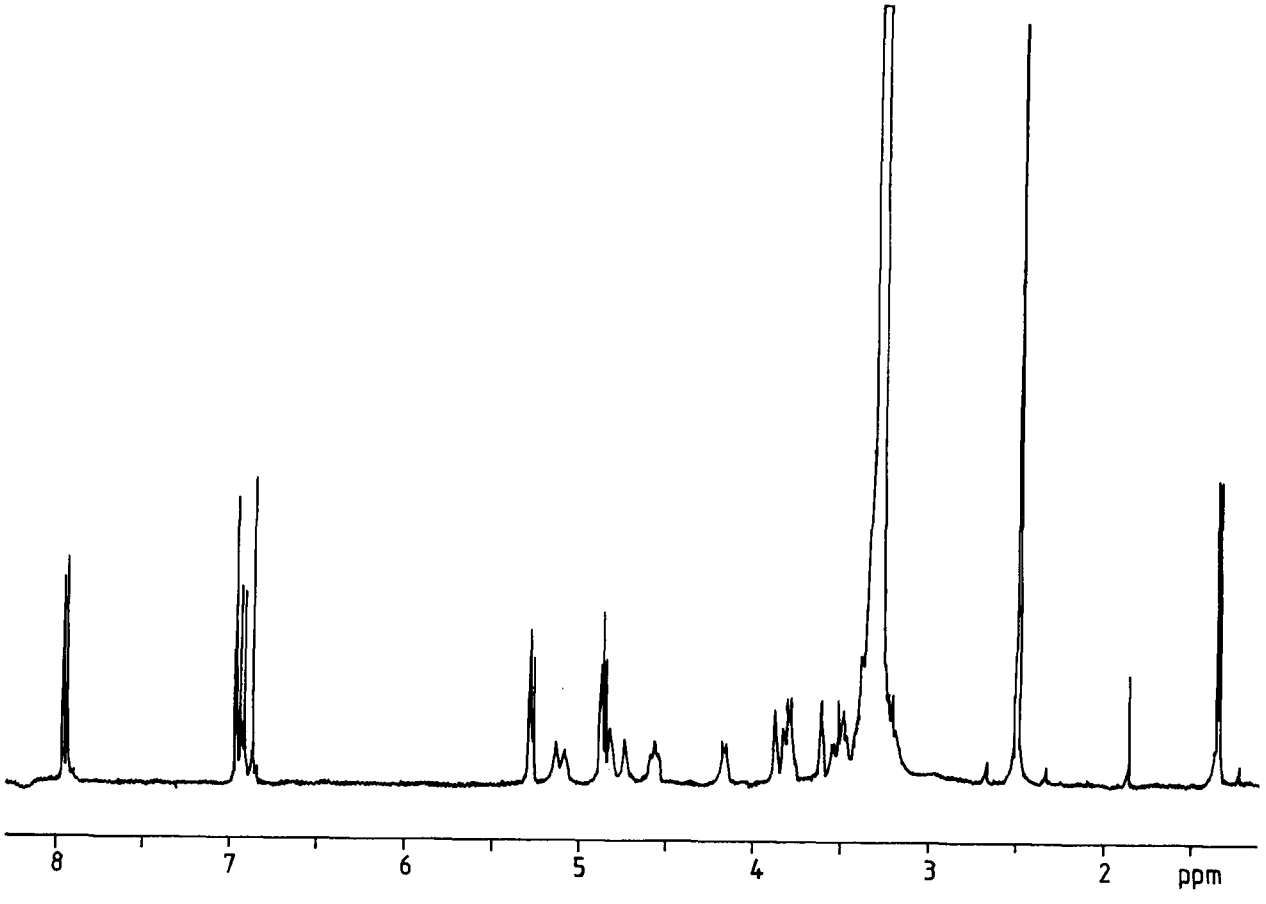
$C_{27}H_{30}O_{14}$



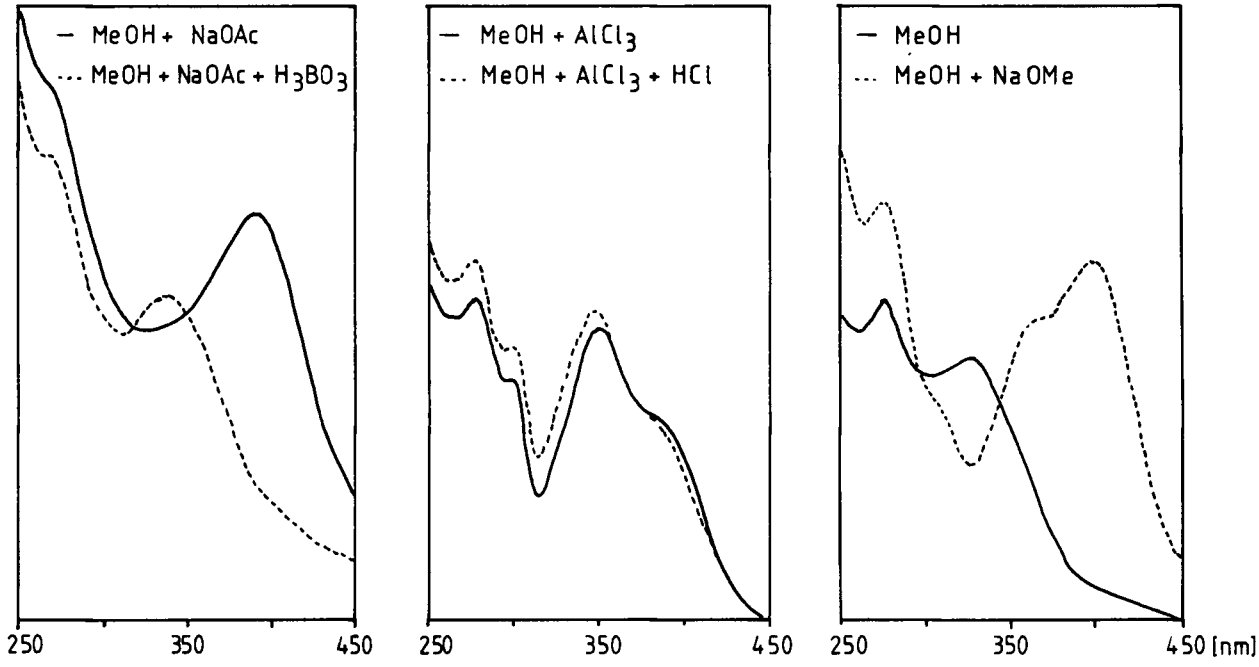

**Mass spectrum of PM-derivative** [90 eV]




**$^1$H-NMR** [400 MHz, DMSO-$d_6$]

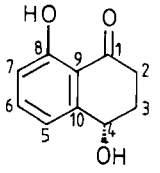


**UV spectra**

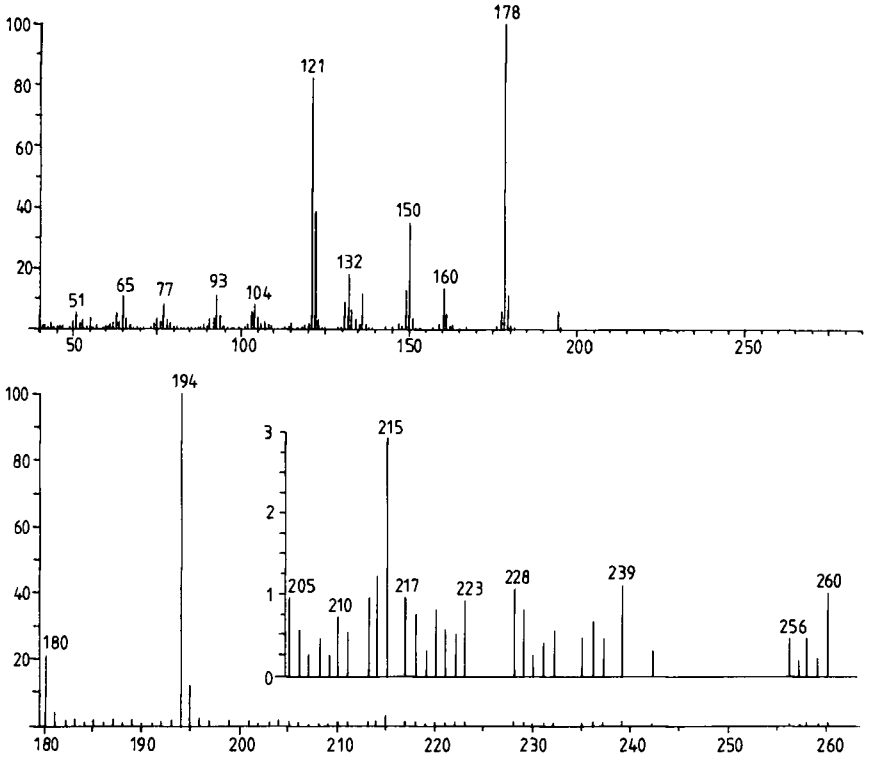

— MeOH + NaOAc
--- MeOH + NaOAc + $H_3BO_3$
— MeOH + $AlCl_3$
--- MeOH + $AlCl_3$ + HCl
— MeOH
--- MeOH + NaOMe
250
350
450
250
350
450
250
350
450 [nm]

# Isosclerone

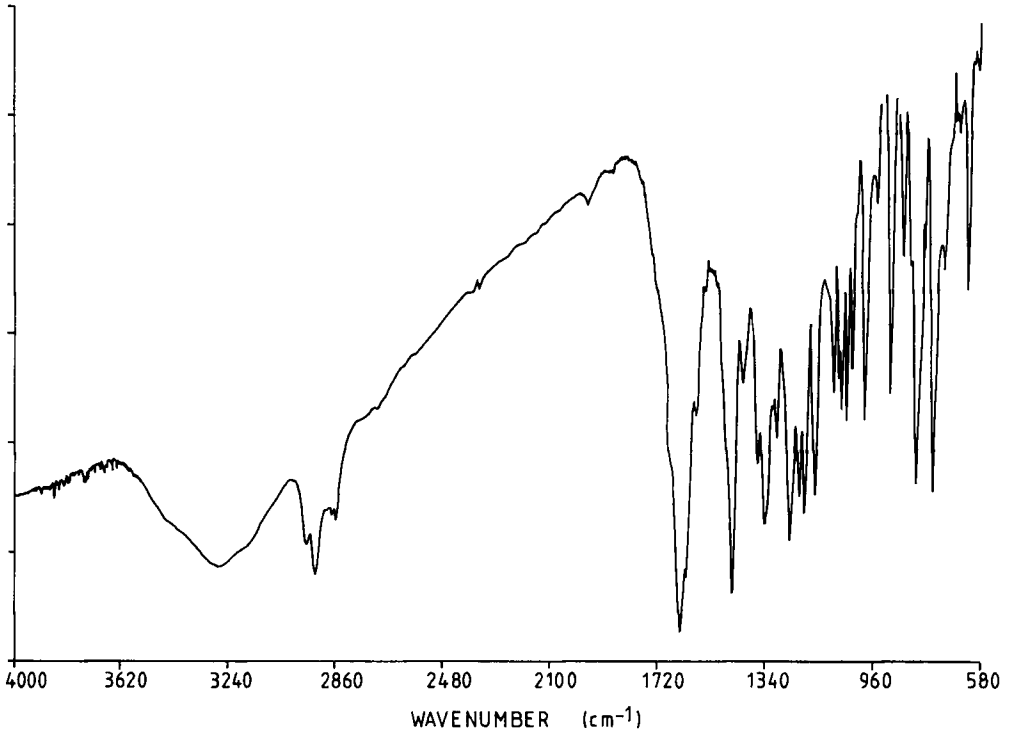
$C_{10}H_{10}O_3$



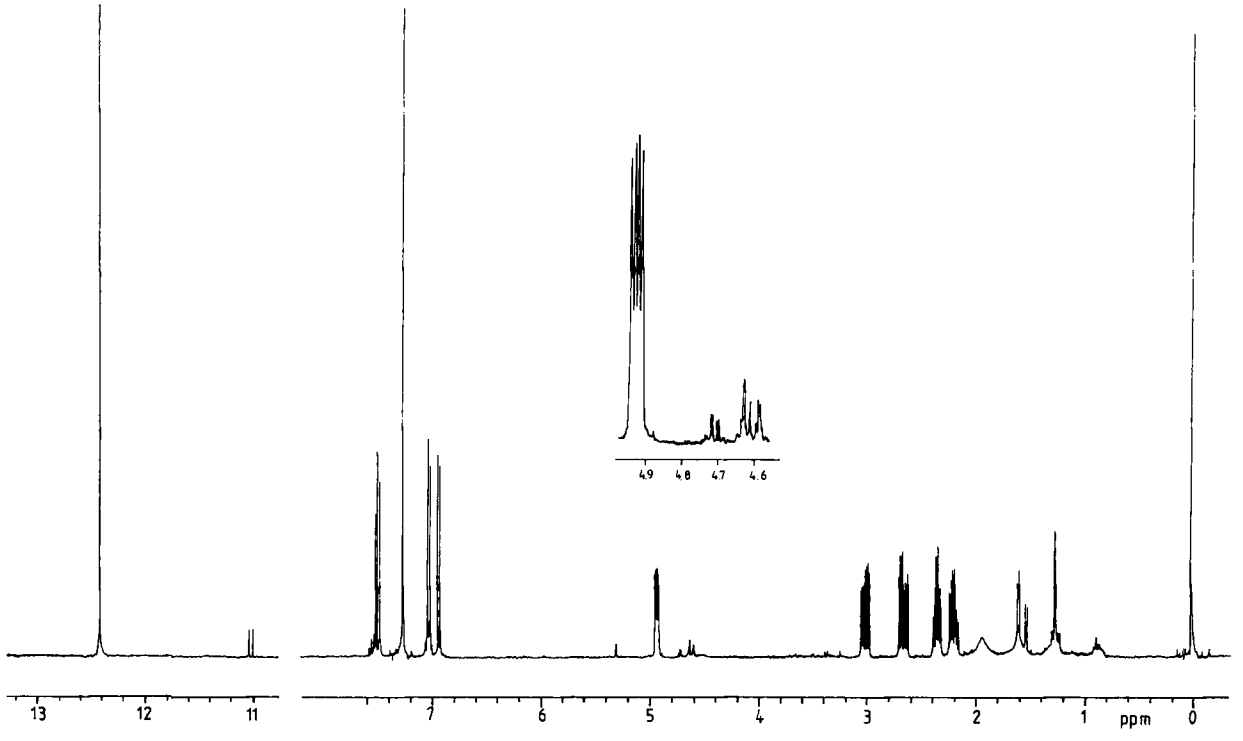

**Mass spectrum** [70 eV]



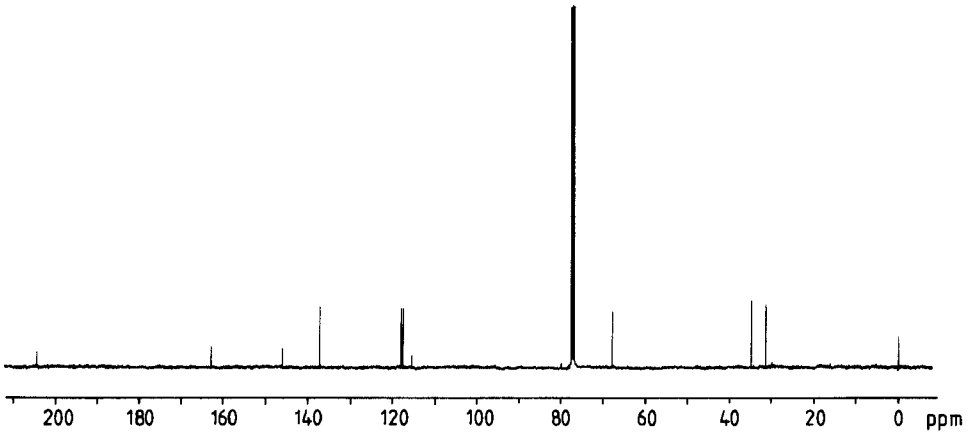
**IR spectrum** [KBr]




**$^1$H-NMR** [$CDCl_3$, 400 MHz]



**$^{13}$C-NMR** [$CDCl_3$, 75.47 MHz]



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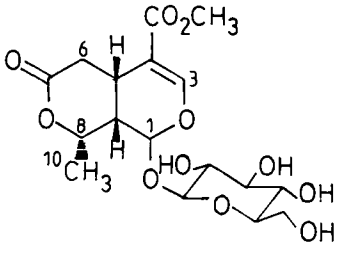
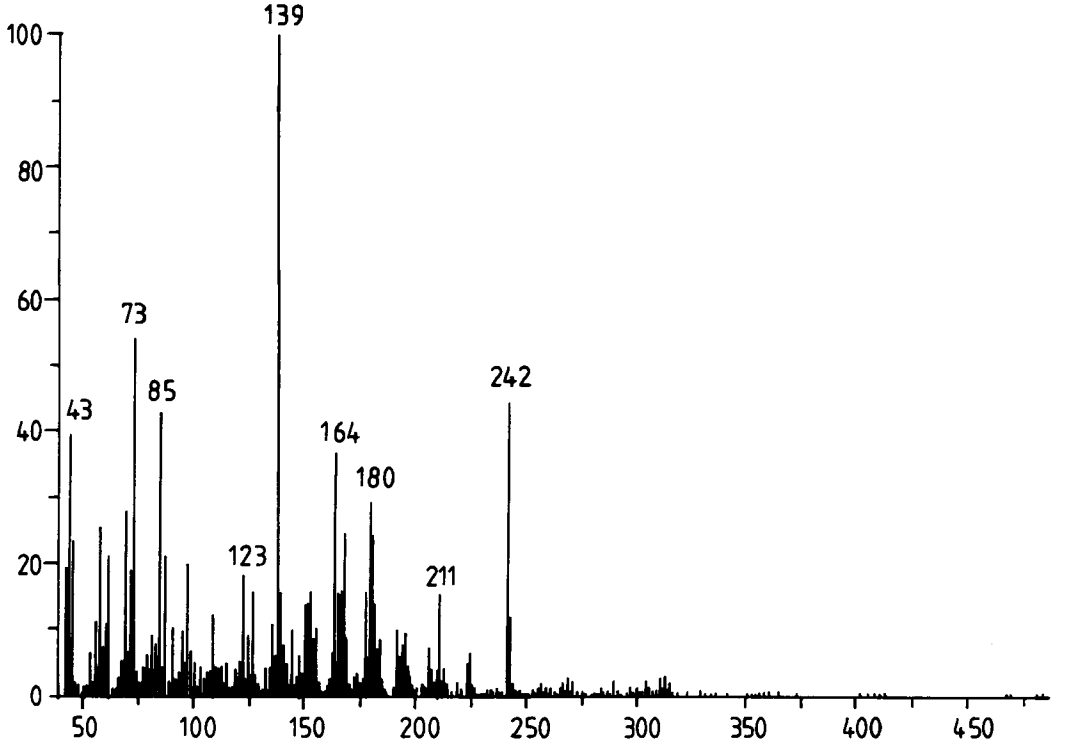
# Kingiside

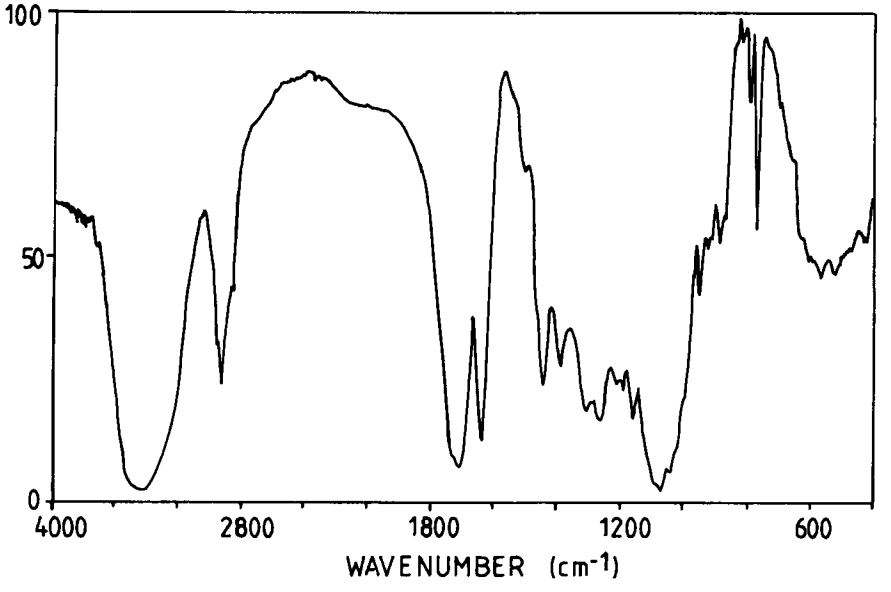
$C_{17}H_{24}O_{11}$



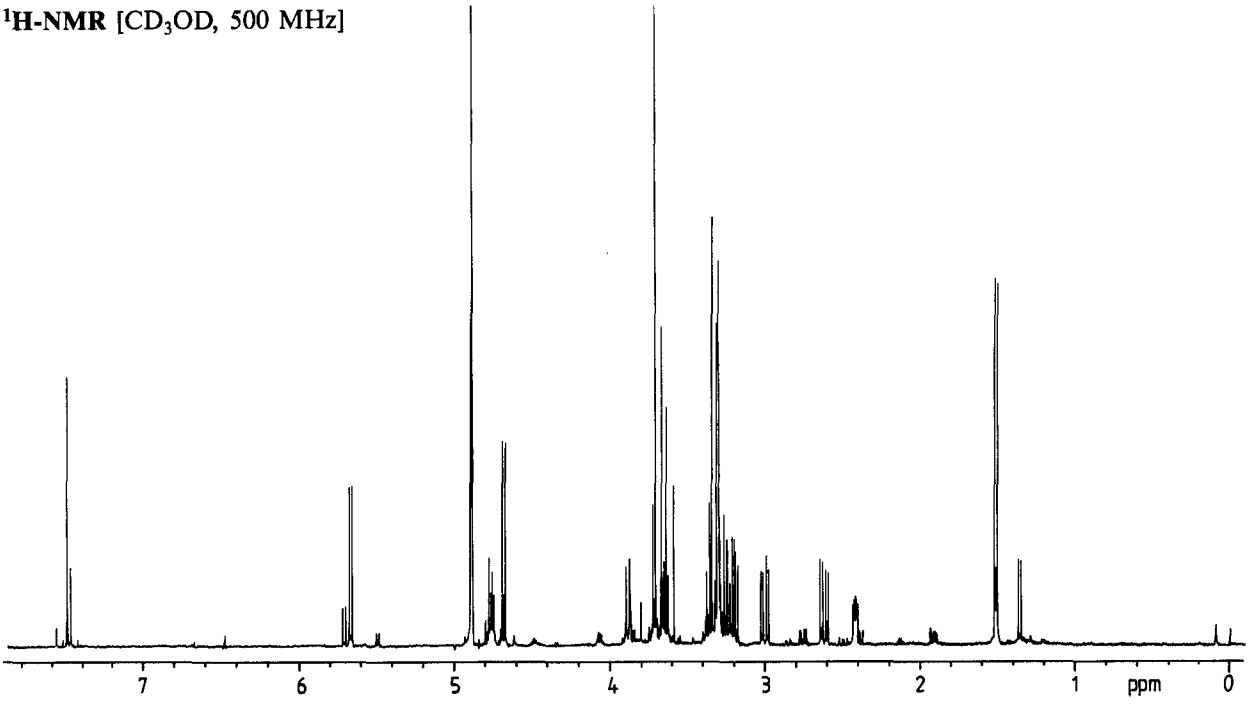

**Mass spectrum** [70 eV]



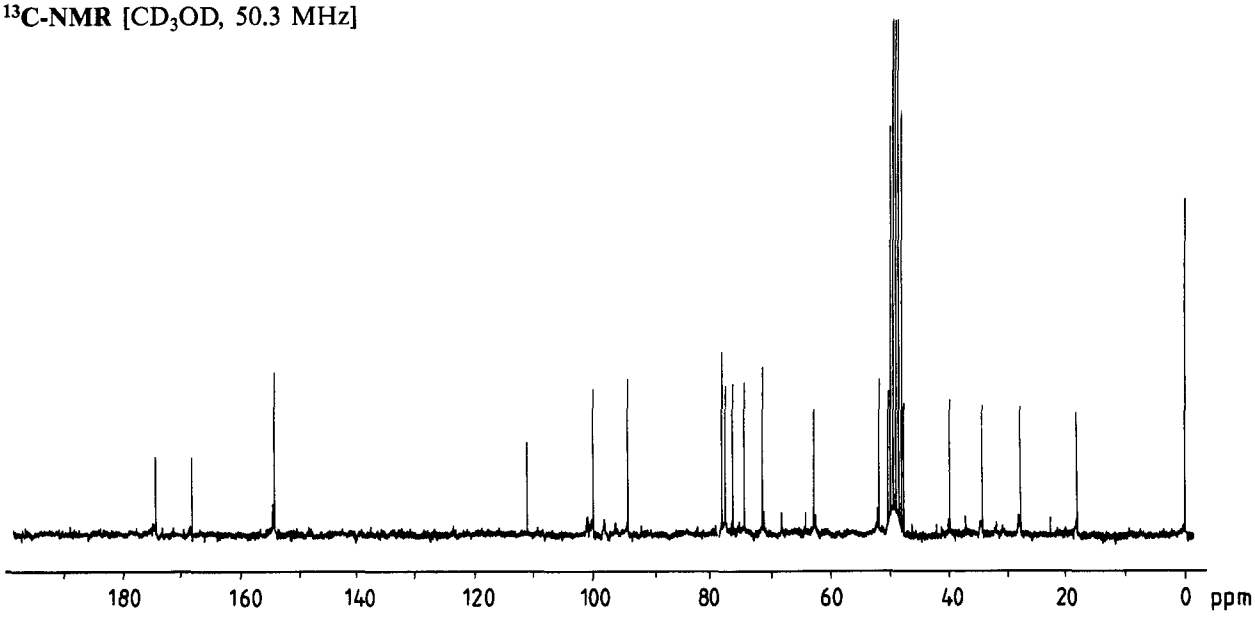

**IR spectrum** [KBr]



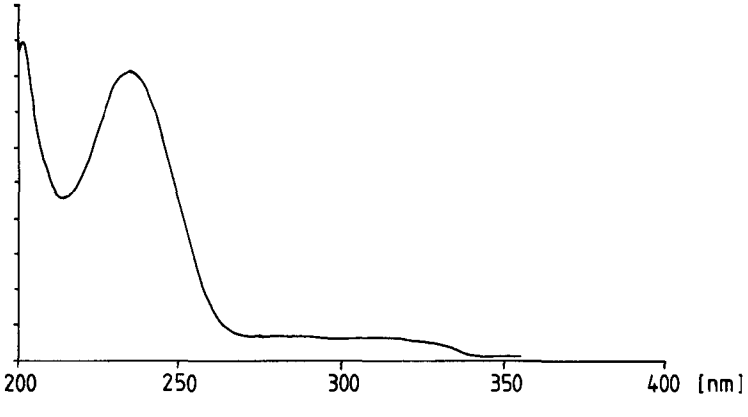
**$^1$H-NMR** [$CD_3OD$, 500 MHz]




**$^{13}$C-NMR** [$CD_3OD$, 50.3 MHz]

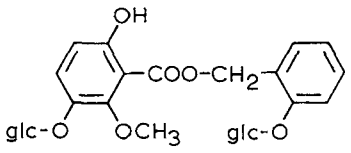



**UV spectrum** [methanol]

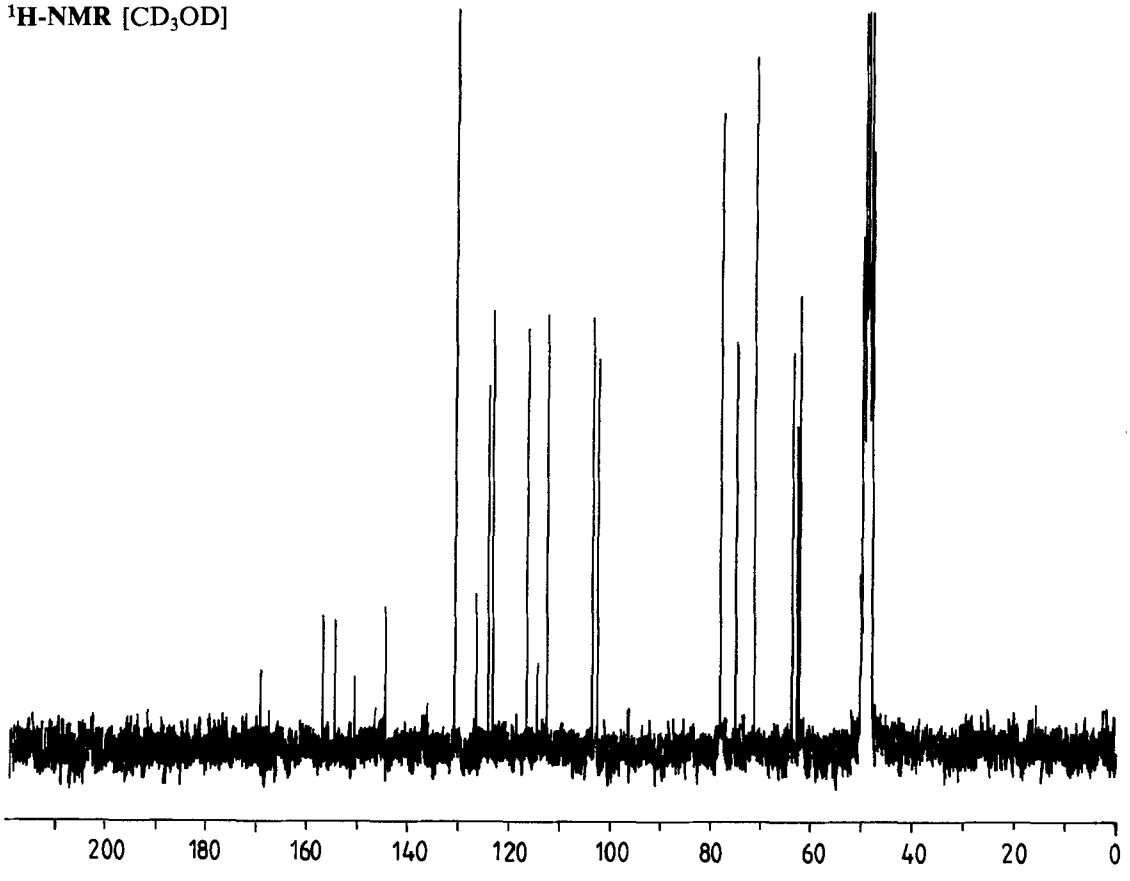


# Leiocarposide

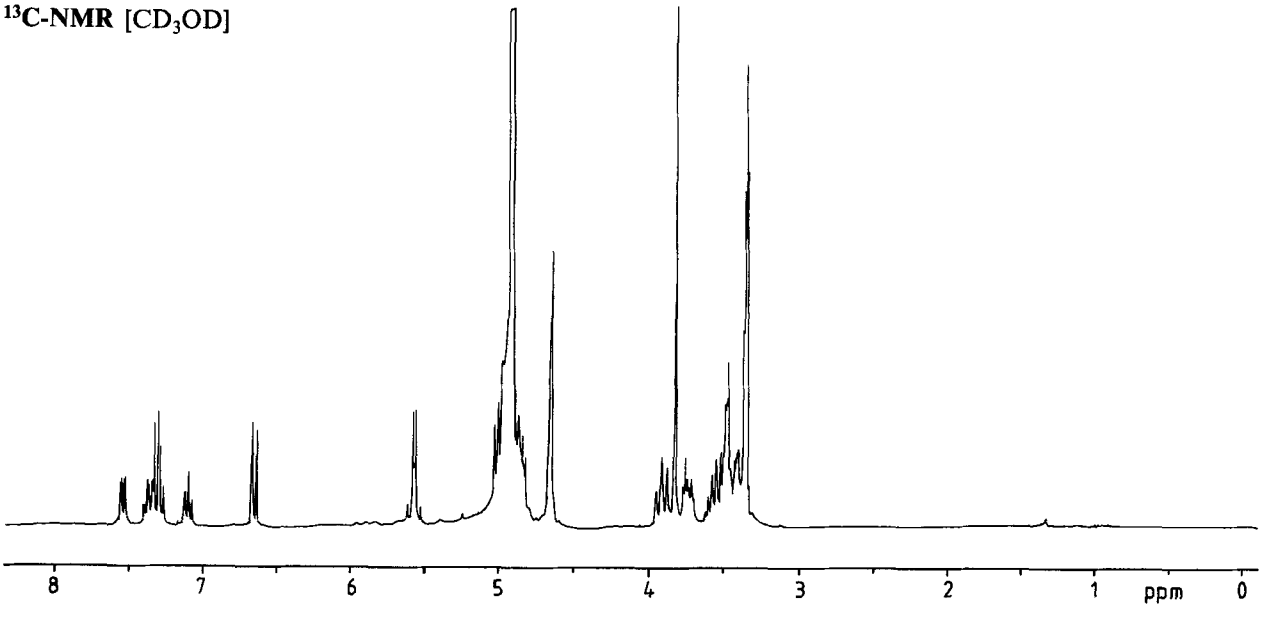
$C_{27}H_{34}O_{16}$




**$^1$H-NMR** [$CD_3OD$]




**$^{13}$C-NMR** [$CD_3OD$]



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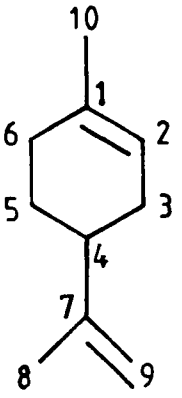
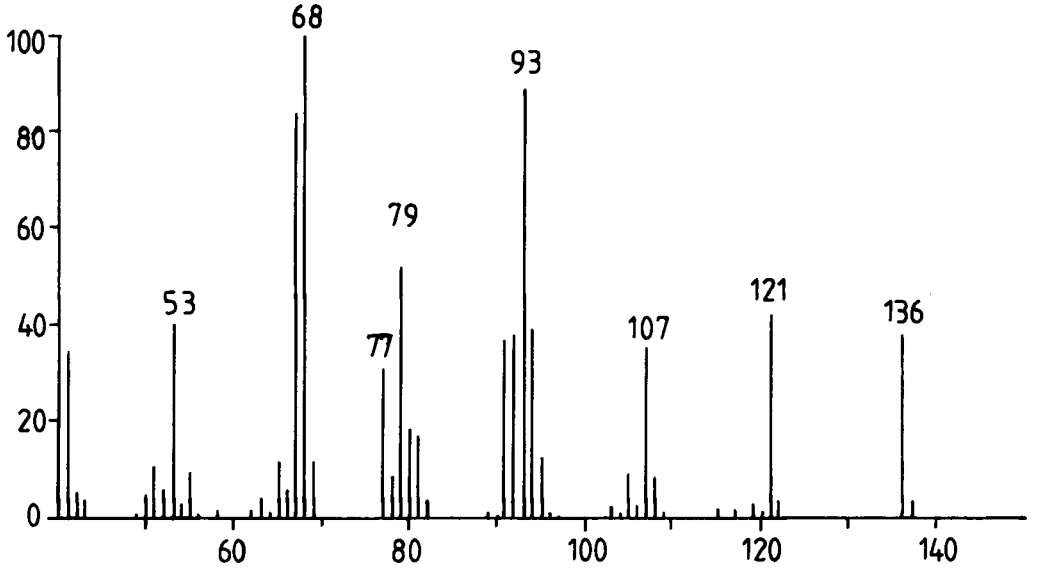
# Limonene

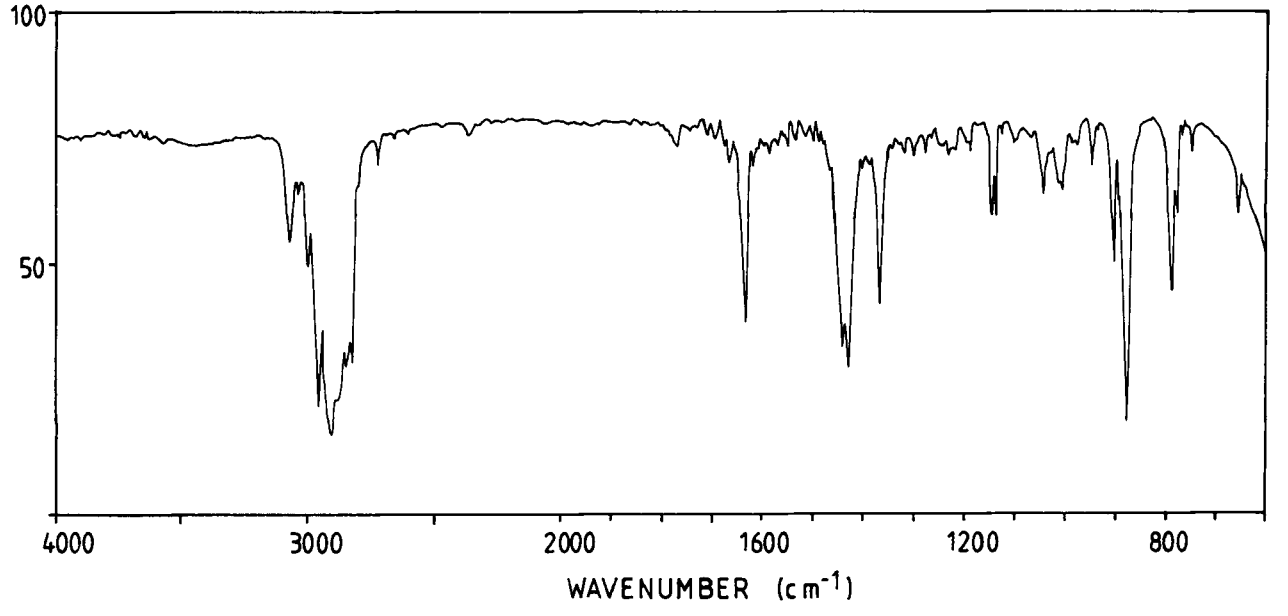
$C_{10}H_{16}$



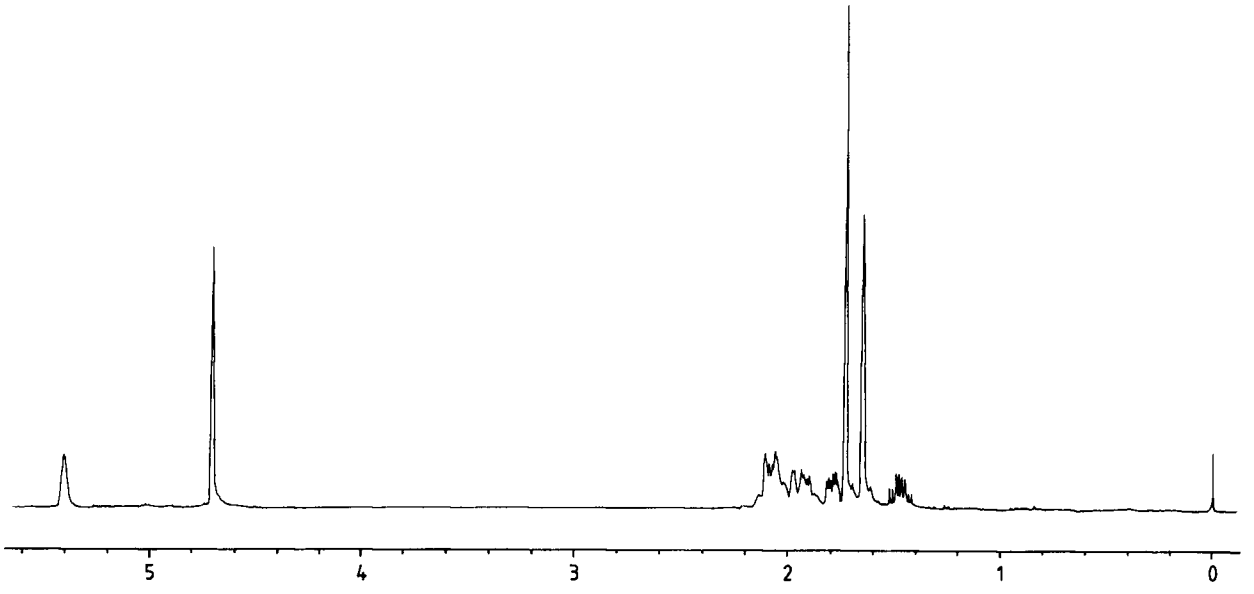

**Mass spectrum** [70 eV]



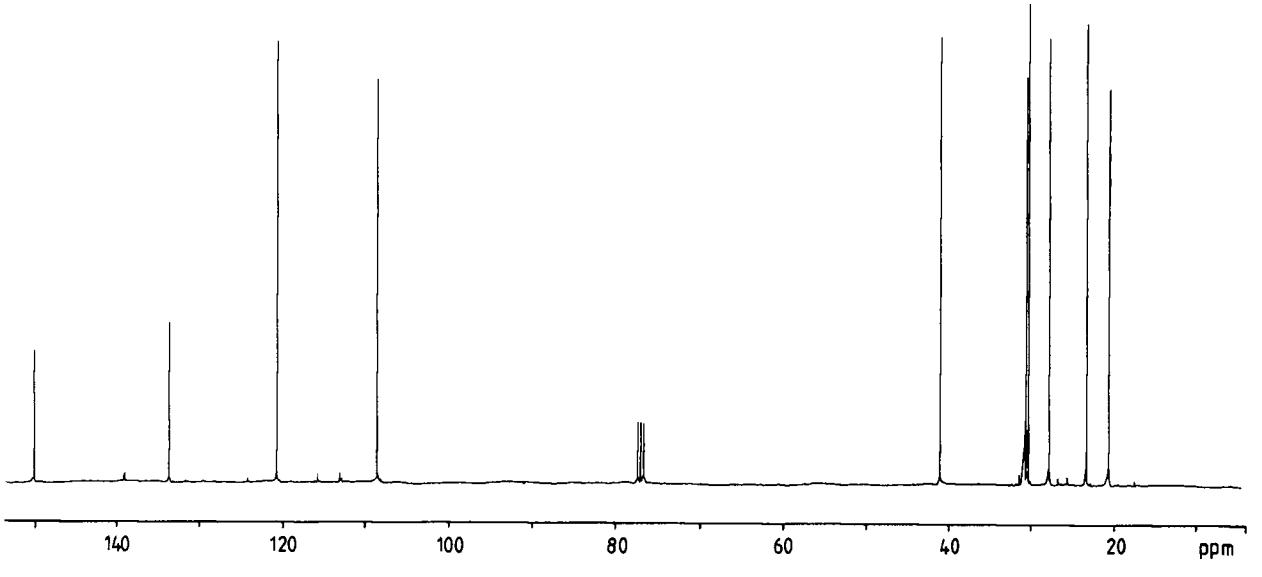

**IR spectrum** [liquid film, 100%]



**$^1$H-NMR** [$CDCl_3$, 400 MHz]



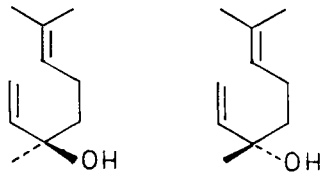

**$^{13}$C-NMR** [$CDCl_3$, 100 MHz]



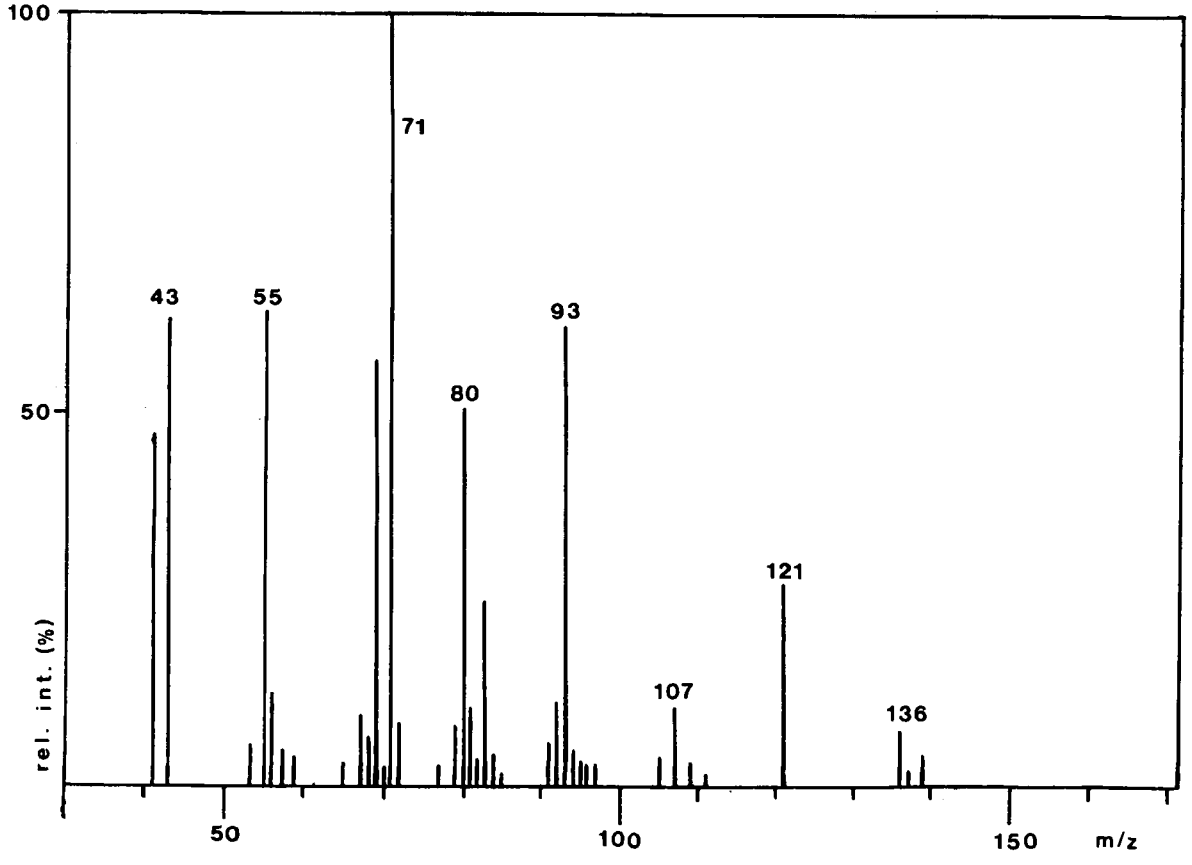
# Linalool

(-) (+)

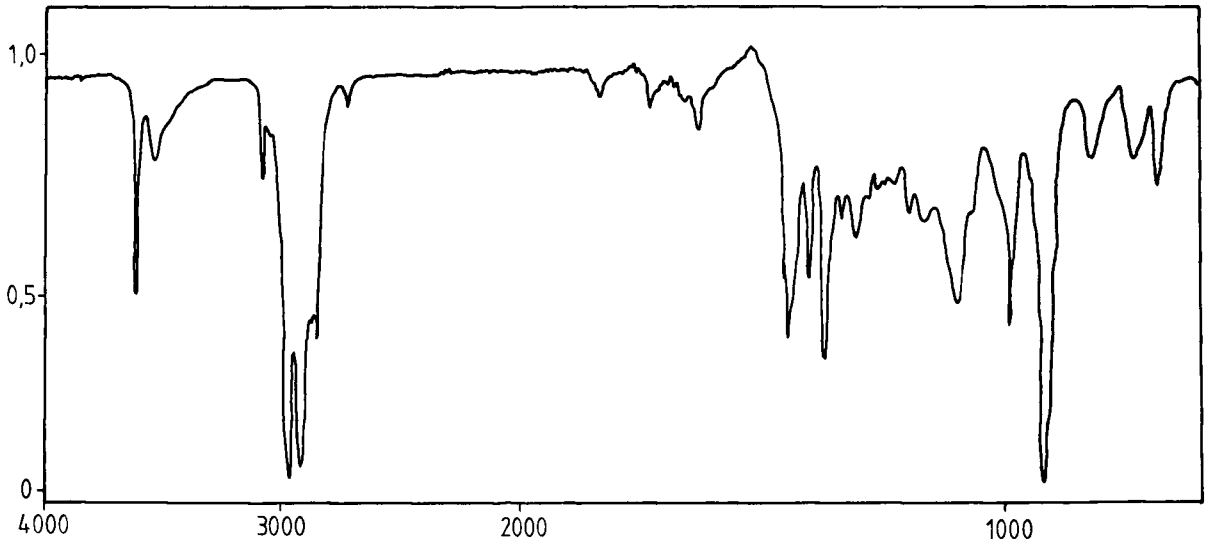
**$C_{10}H_{18}O$**



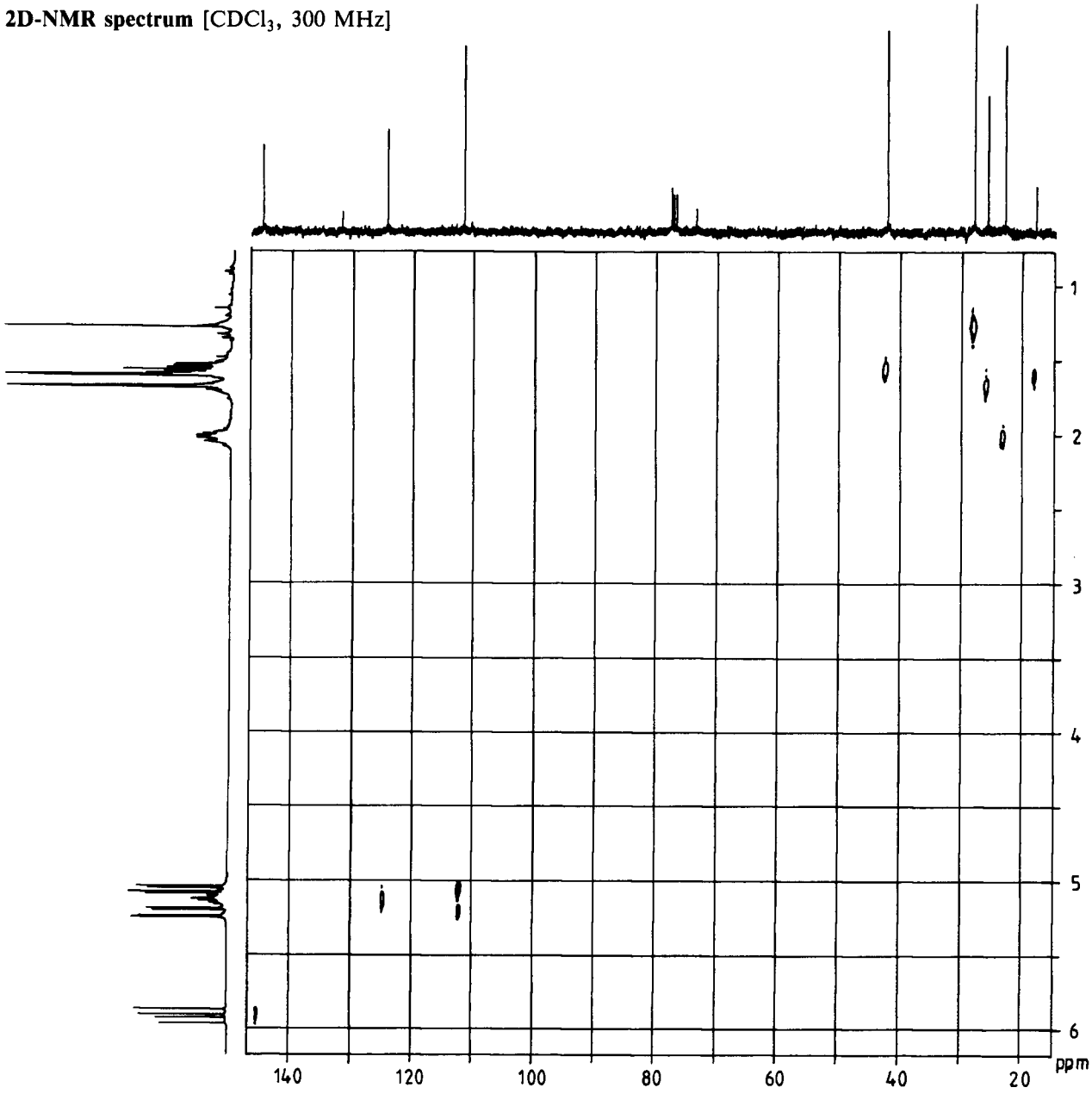

**Mass spectrum** [70 eV]



**IR spectrum** [2% in $CCl_4$]

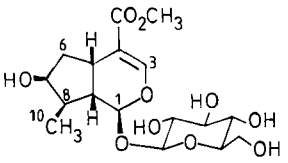



**2D-NMR spectrum** [$CDCl_3$, 300 MHz]

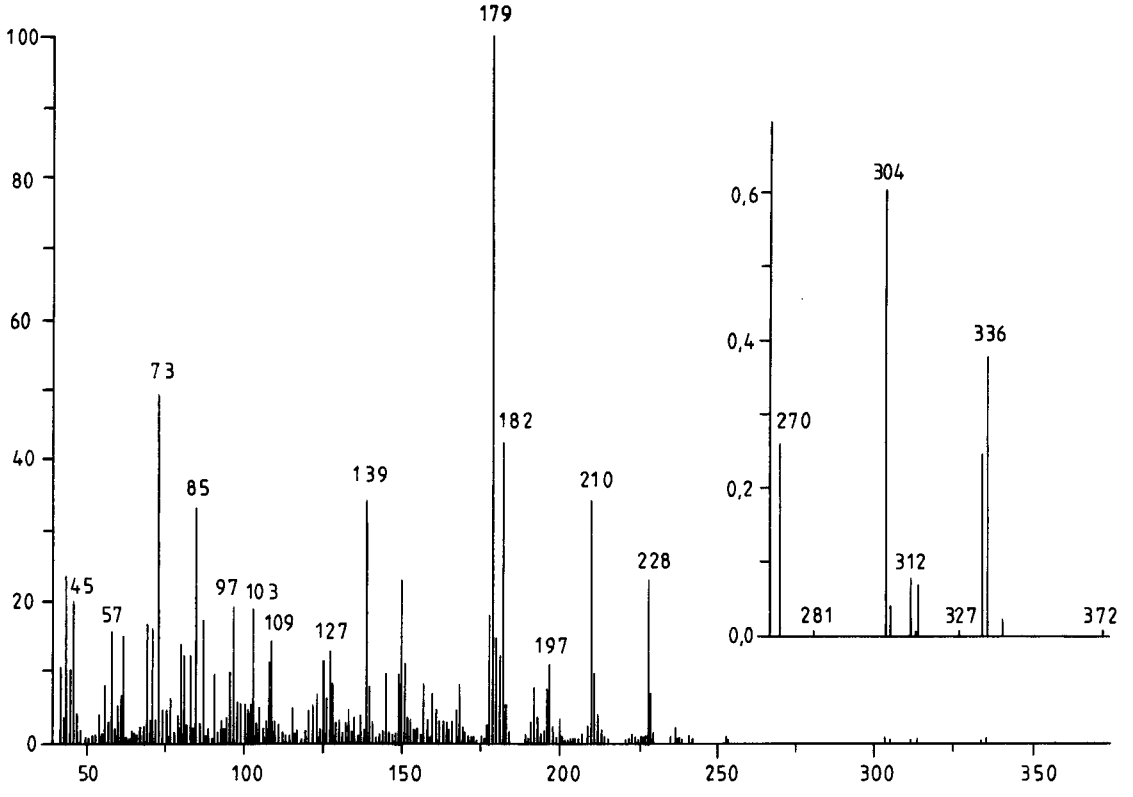


# Loganin

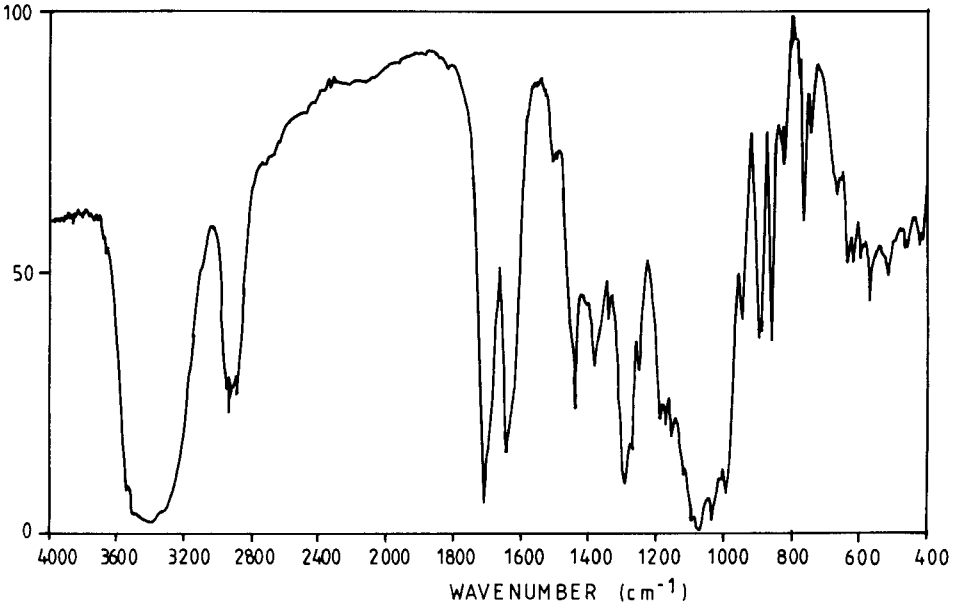
$C_{17}H_{26}O_{10}$



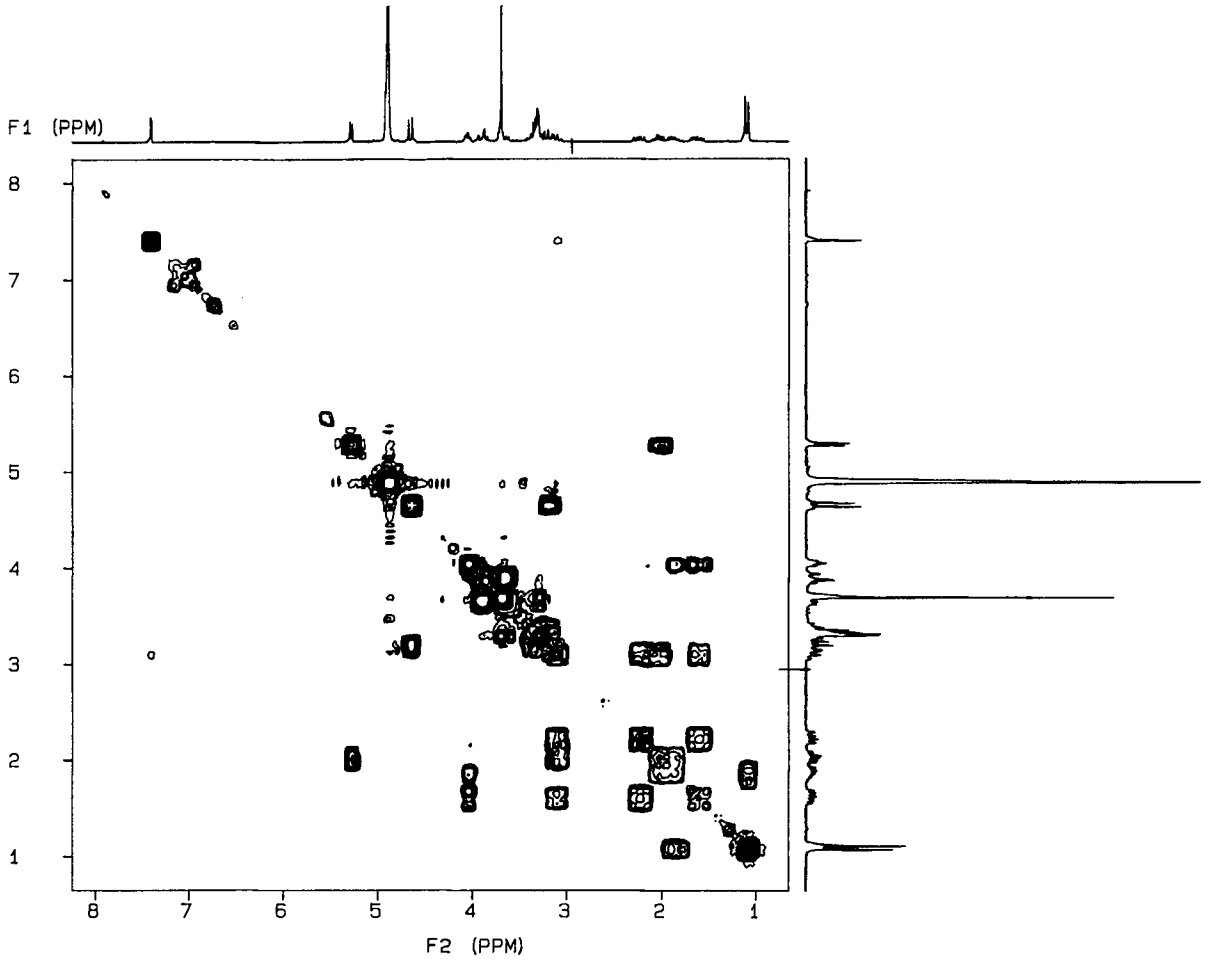

**Mass spectrum** [70 eV]



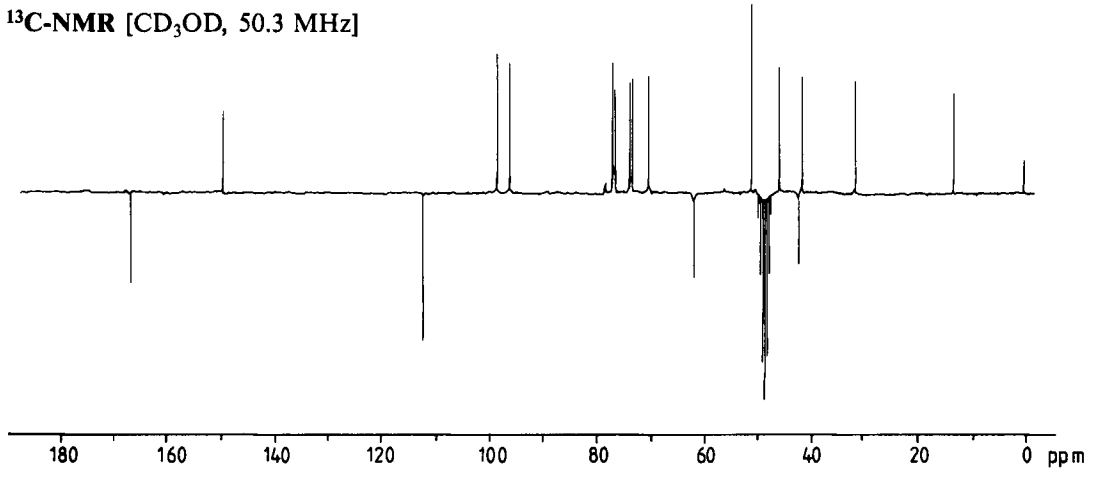

**IR spectrum** [KBr]



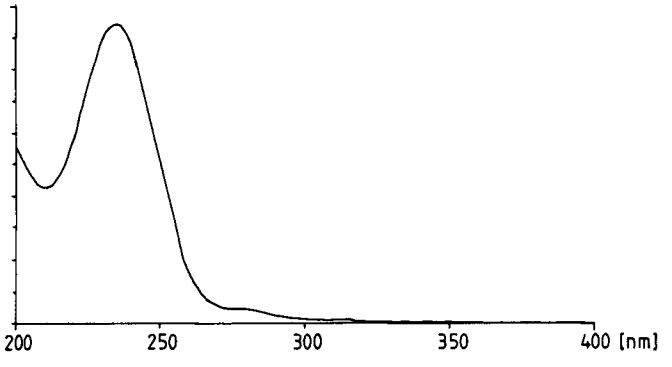
**$^1$H-NMR 2D-spectrum** [$CD_3OD$, 400 MHz]




**$^{13}$C-NMR** [$CD_3OD$, 50.3 MHz]

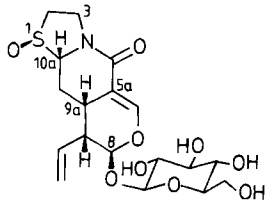



**UV spectrum** [methanol]



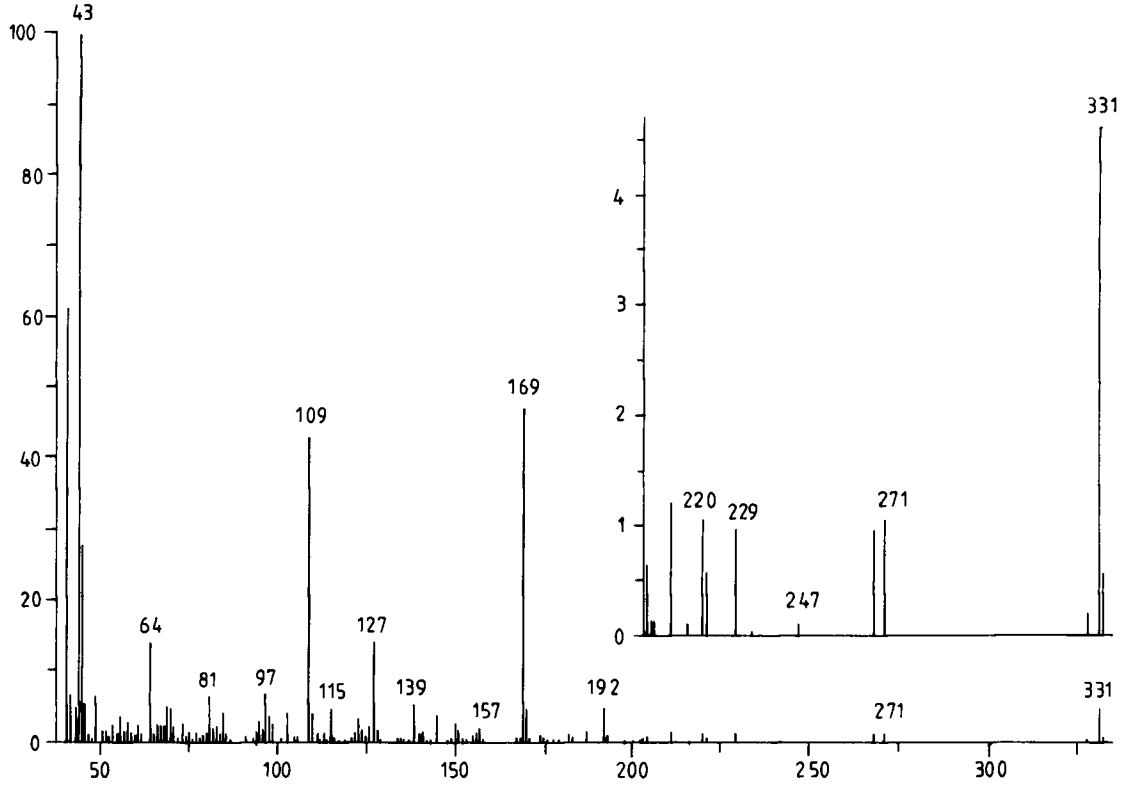

Loganin

# Loxylostosidine A

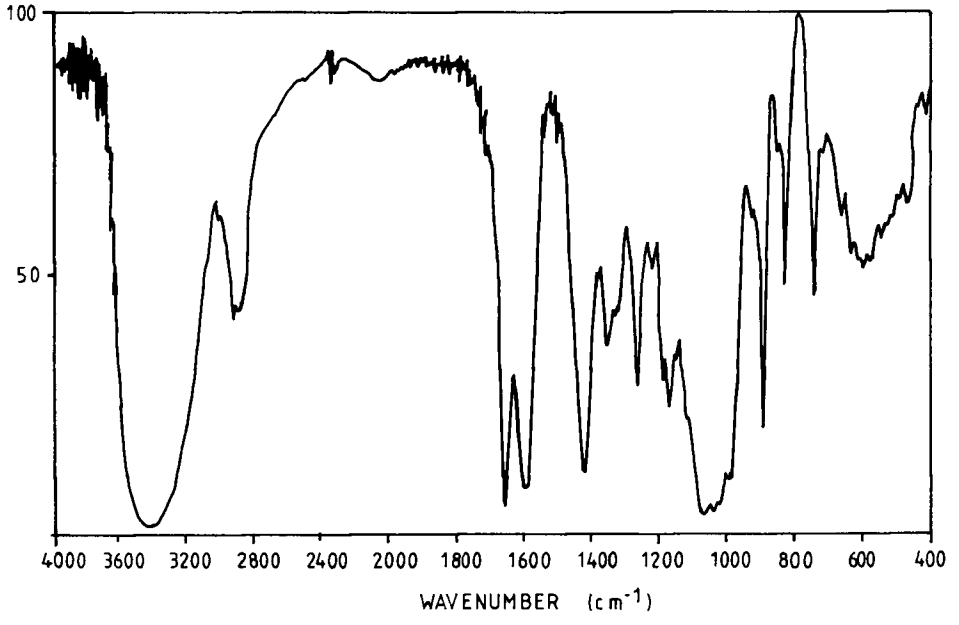


$C_{18}H_{25}NO_9S$

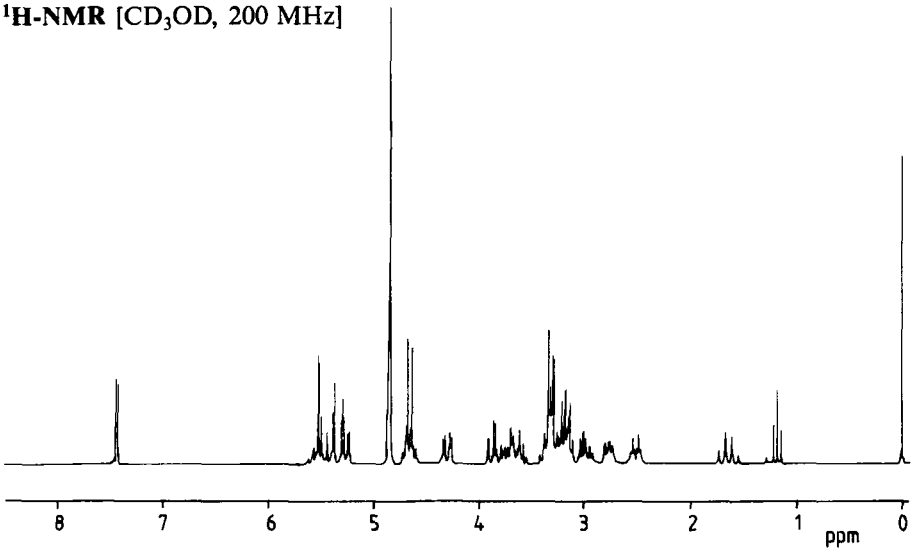
**Mass spectrum of Loxylostosidin-tetraacetate** [70 eV]



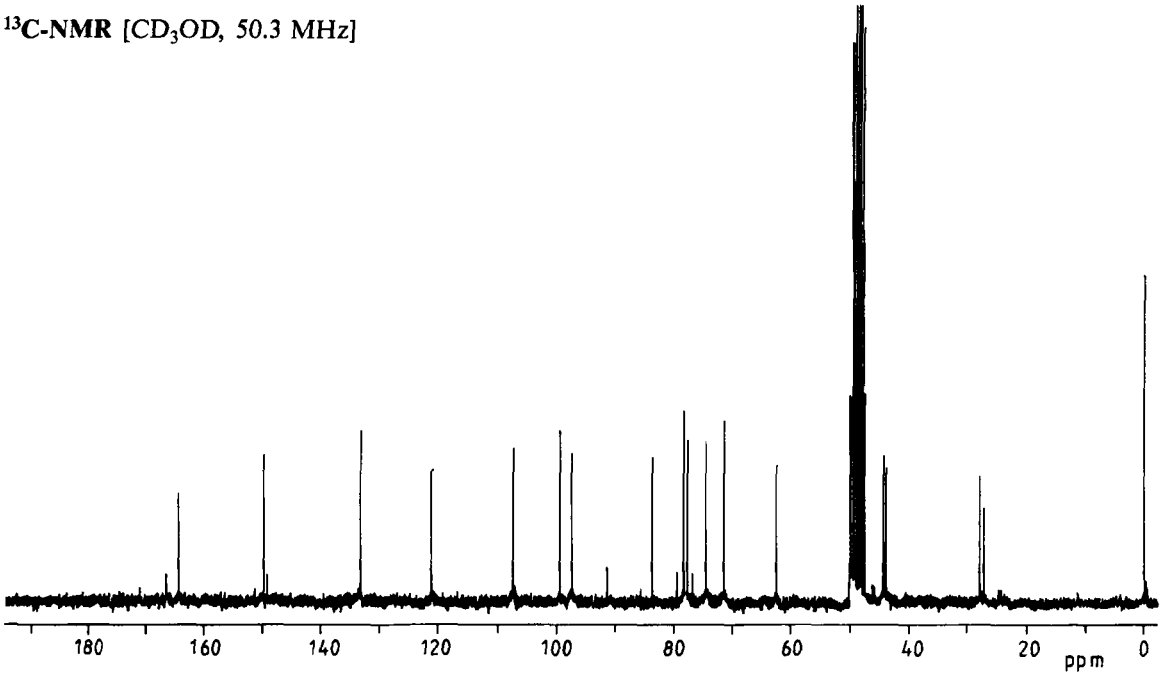

**IR spectrum** [KBr]



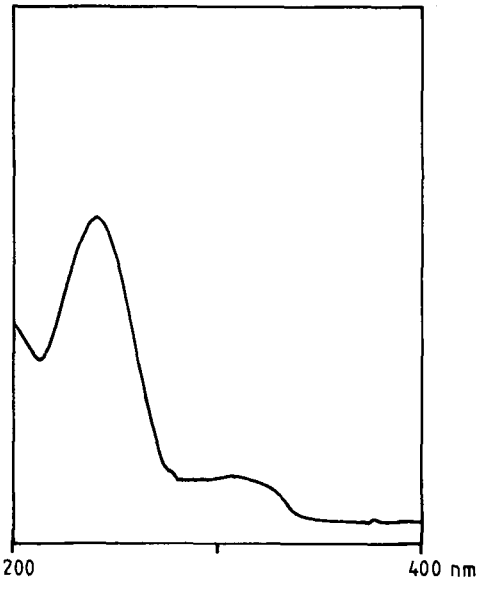
**$^1$H-NMR** [$CD_3OD$, 200 MHz]




**$^{13}$C-NMR** [$CD_3OD$, 50.3 MHz]



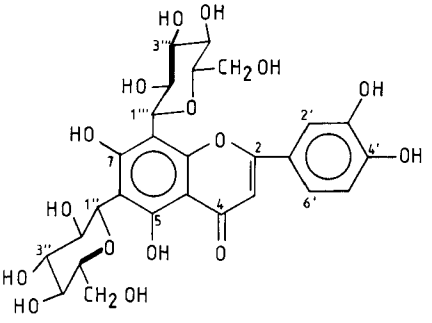

**UV spectrum** [methanol]



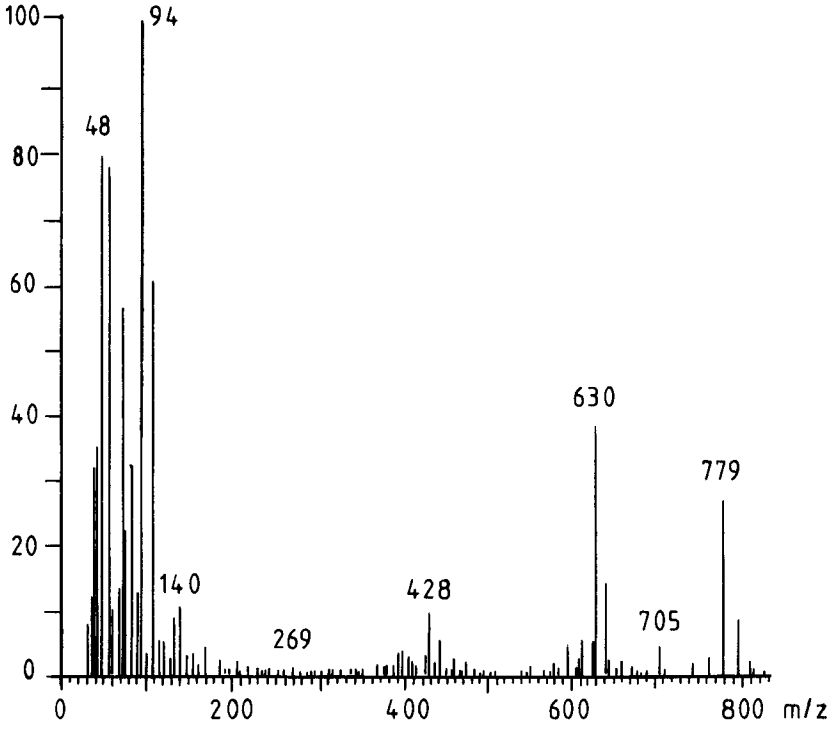

Loxylostosidine A

# Lucenin-2

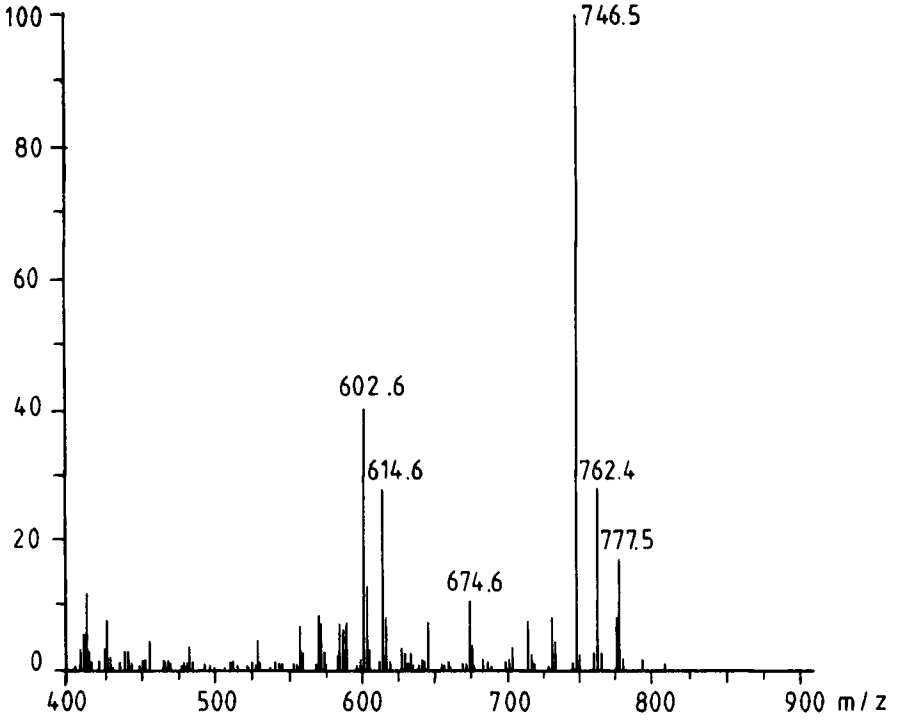
$C_{27}H_{30}O_{16}$



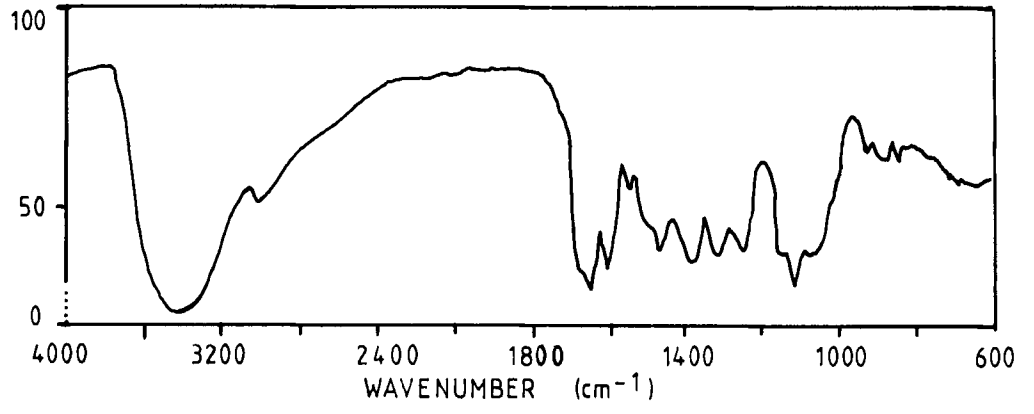
**Mass spectrum of Perdeuteromethylether-Derivative**



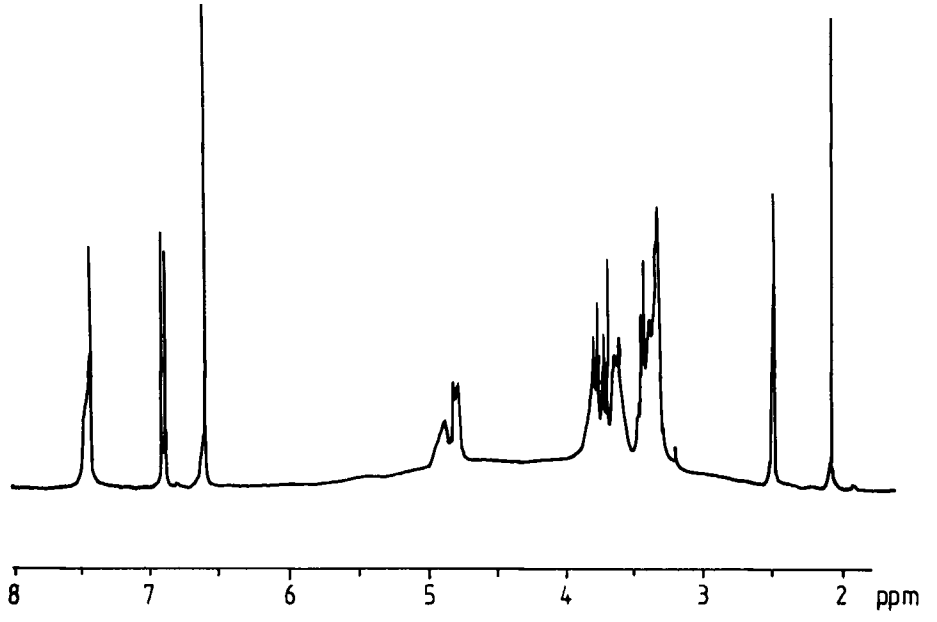
**Mass spectrum of Permethylether-Derivative**



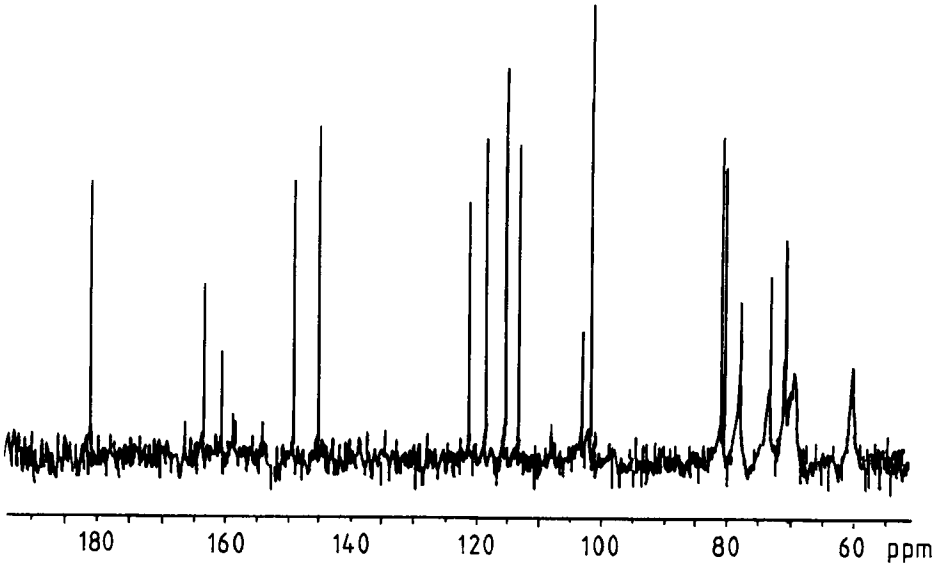

**IR spectrum**



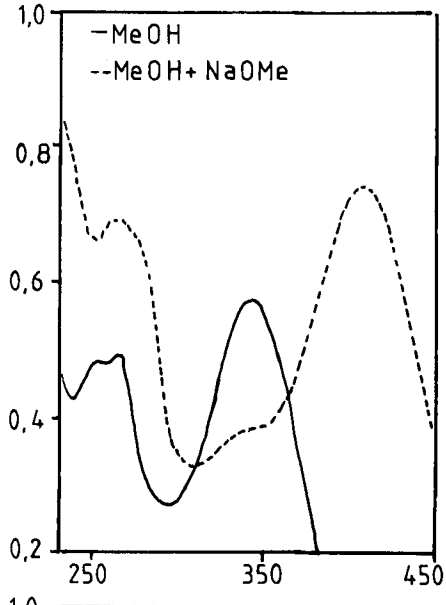

**$^1$H-NMR** [400 MHz, DMSO-$d_6$]

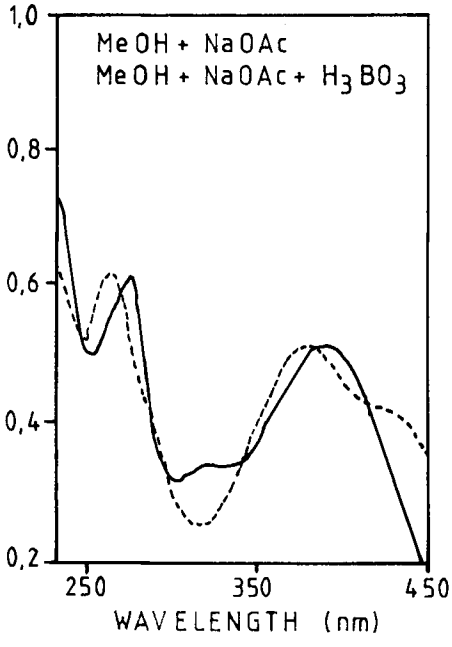


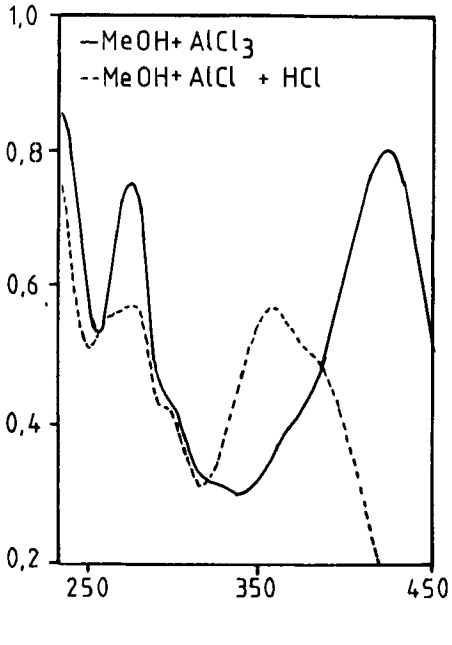
**$^{13}$C-NMR** [100 MHz, DMSO-$d_6$]




**UV spectra**



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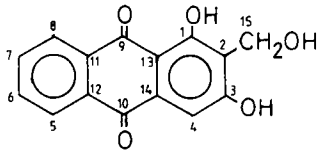
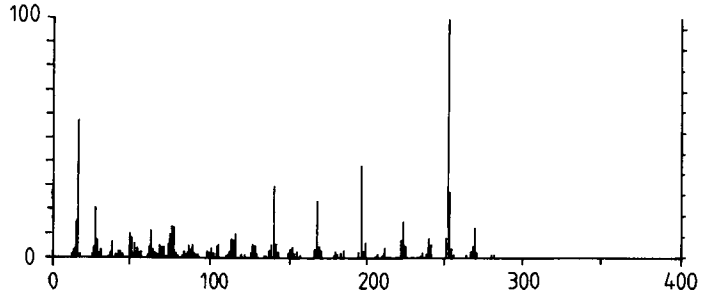
# Lucidin

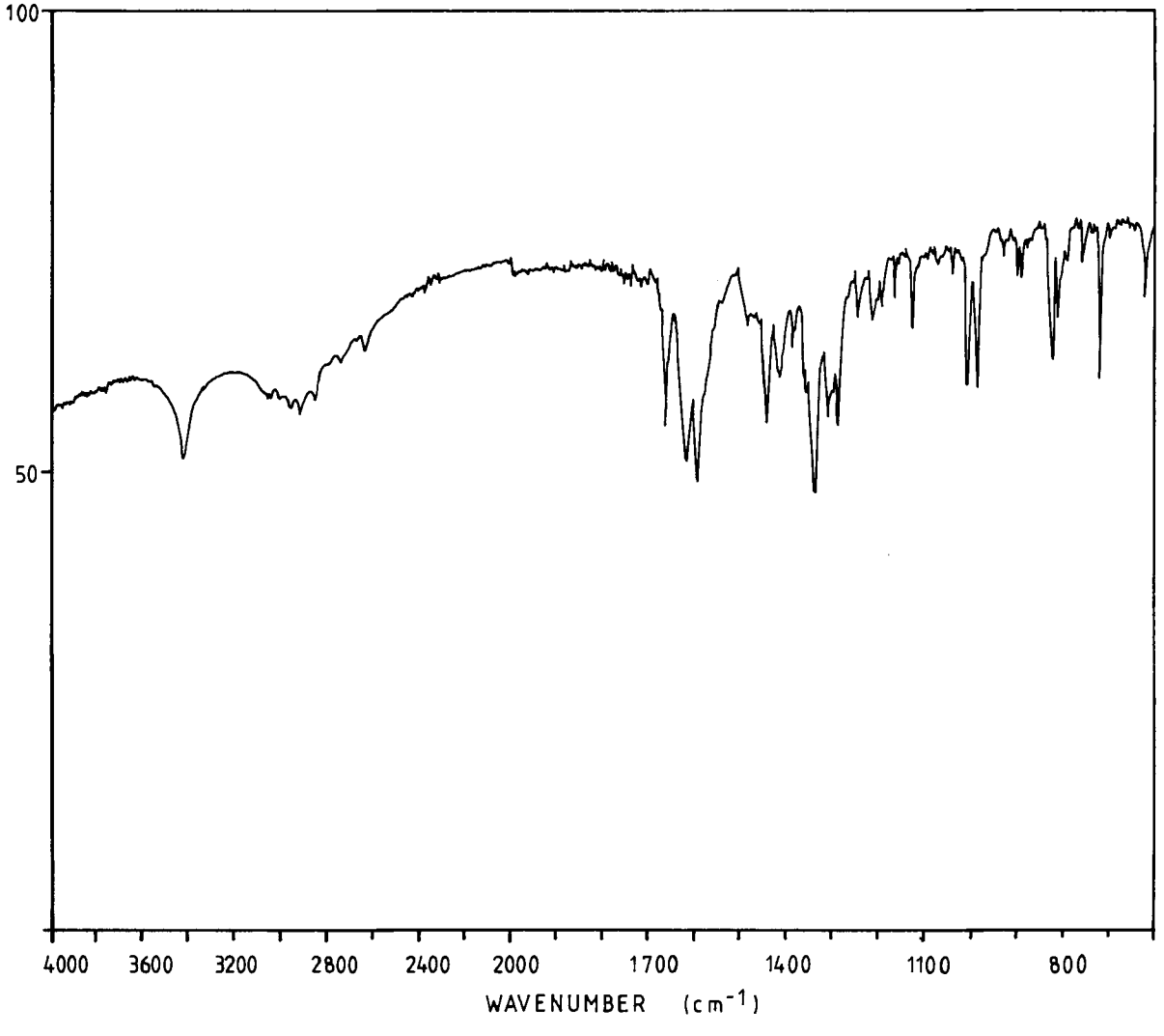
$C_{15}H_{10}O_5$



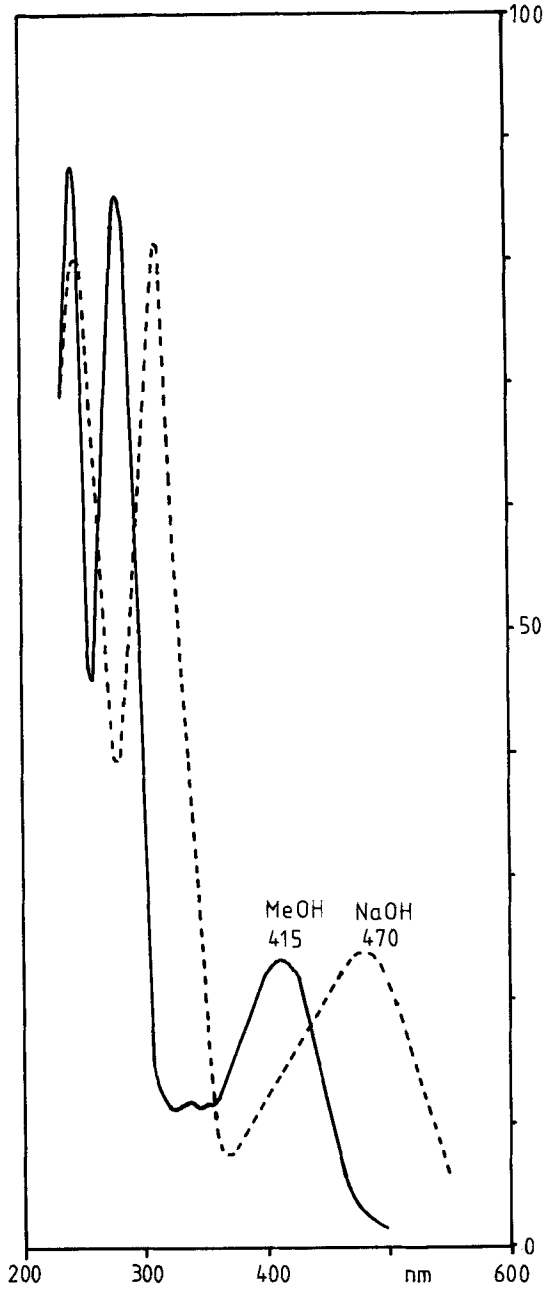

**Mass spectrum** [70 eV]




**IR spectrum** [KBr]

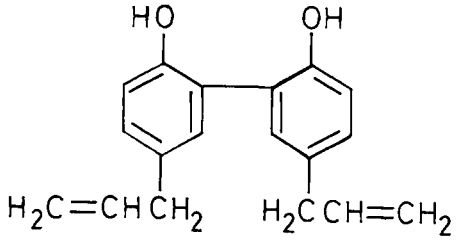


**UV spectrum**

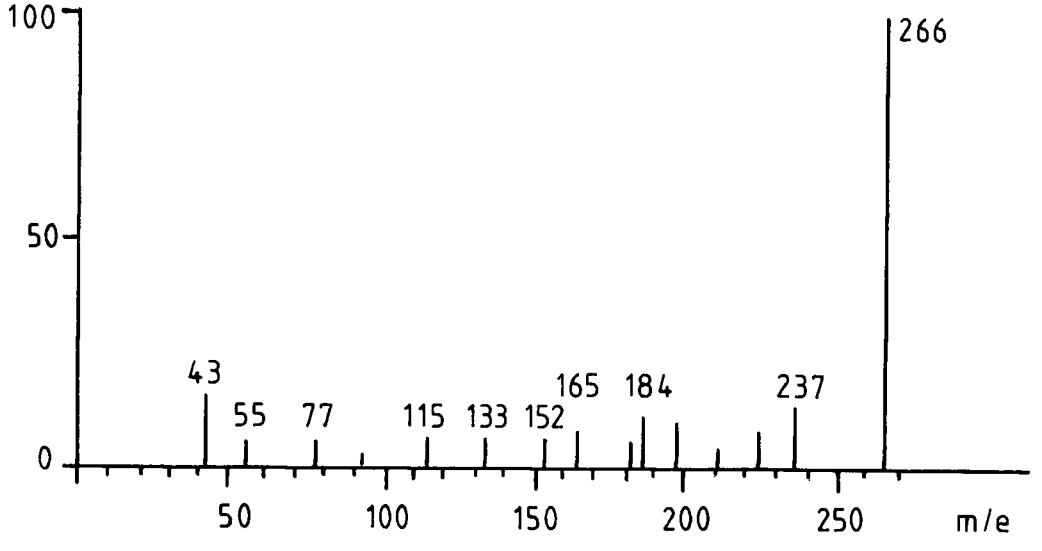


# Magnolol

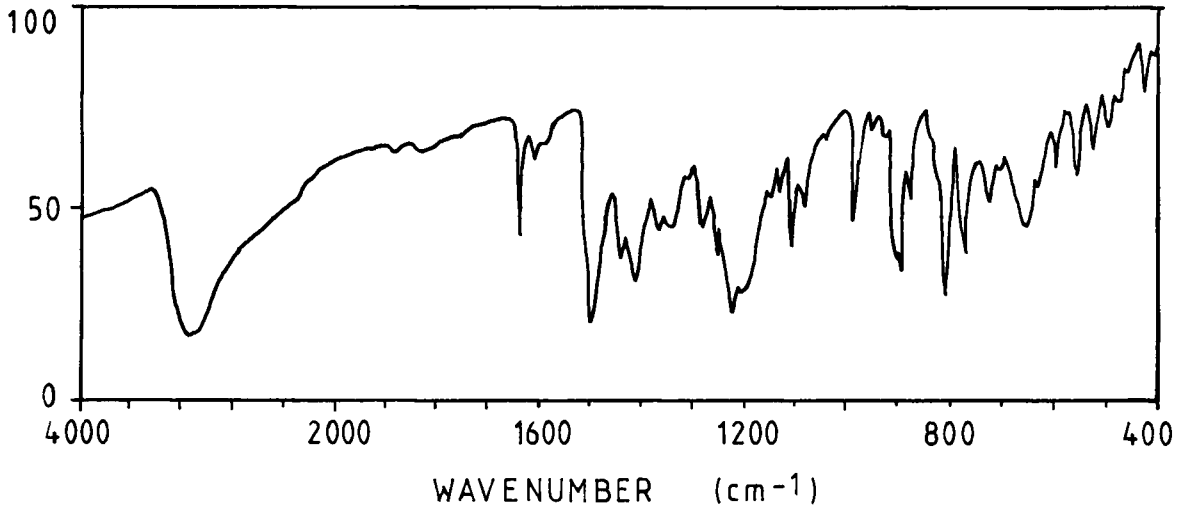
**$C_{18}H_{18}O_2$**



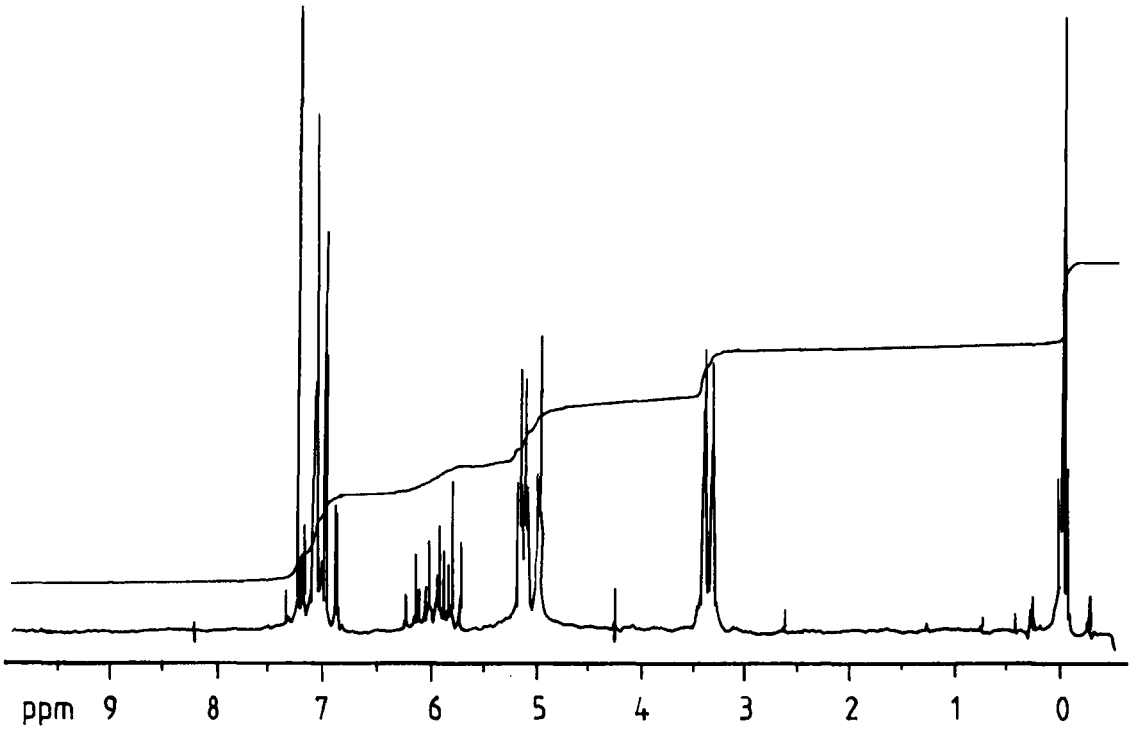

**Mass spectrum** [70 eV]




**IR spectrum** [KBr]

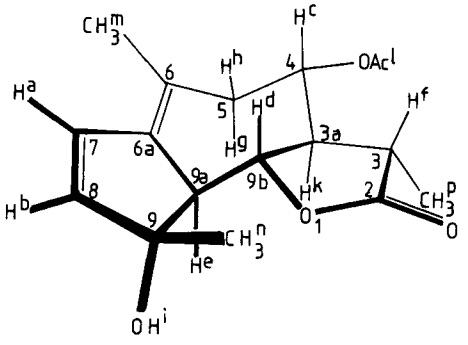


**$^1$H-NMR** [$CDCl_3$, 80 MHz]

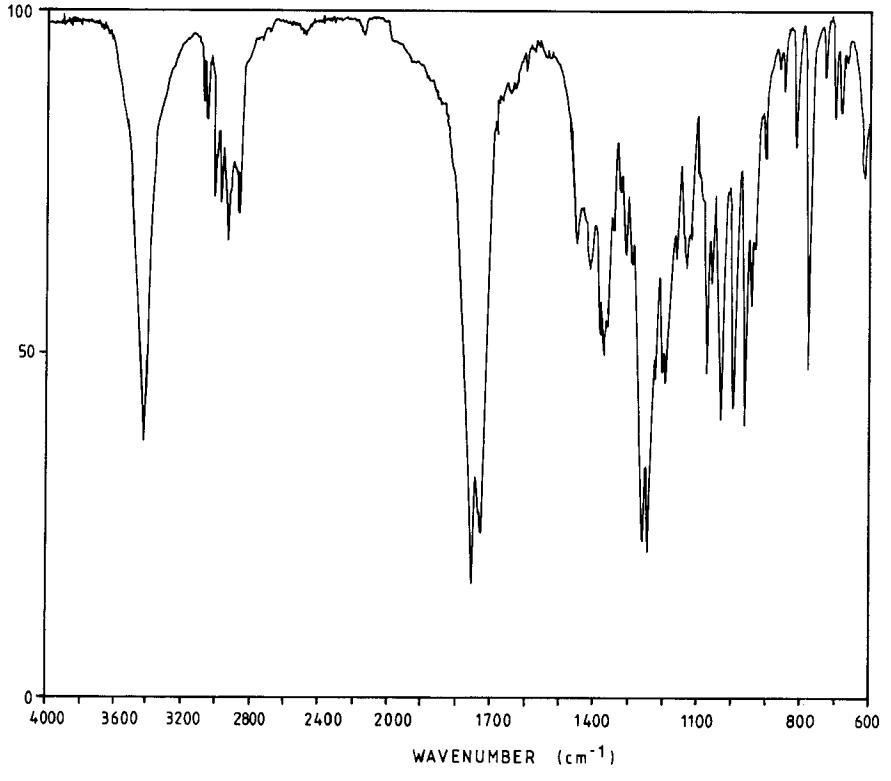


# Matricine

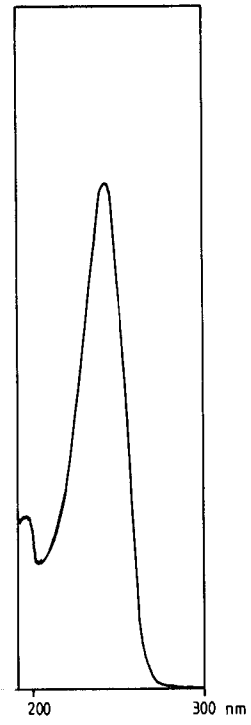
$C_{17}H_{22}O_5$




**IR spectrum** [KBr tablet]

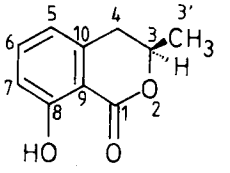


**UV spectrum** [methanol]



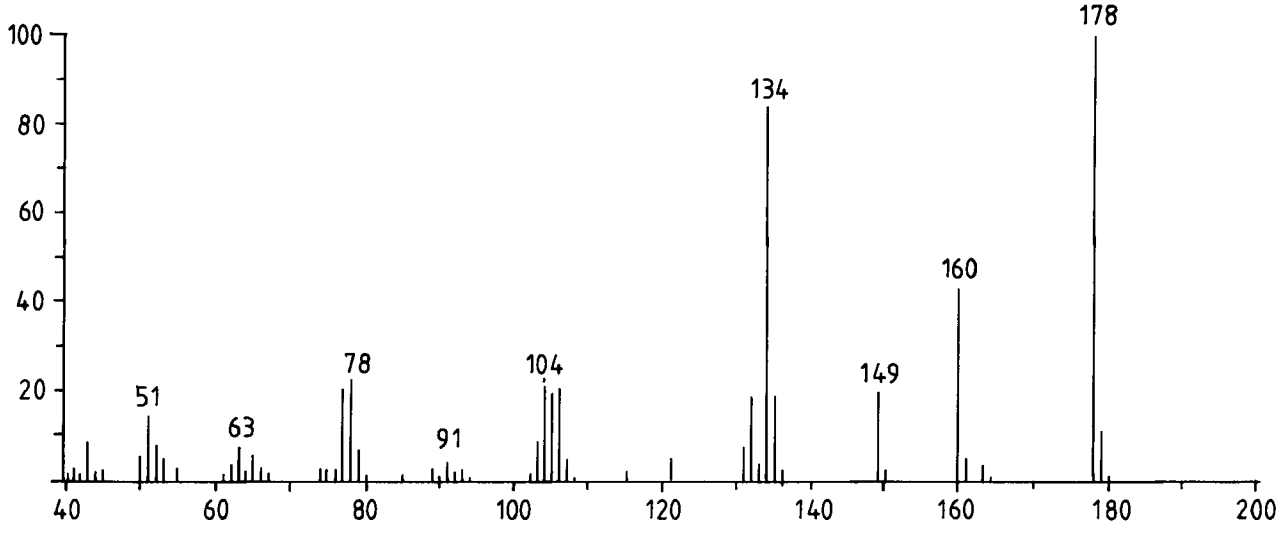
# Mellein

**$C_{10}H_{10}O_3$**

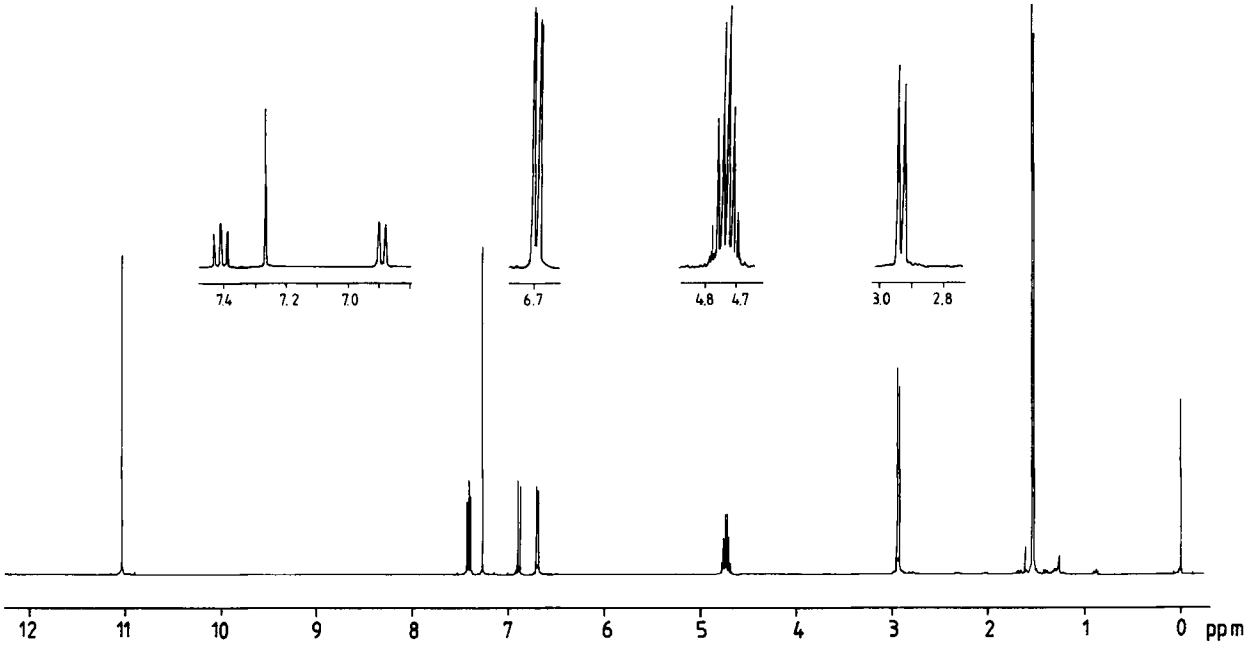



(R) - form

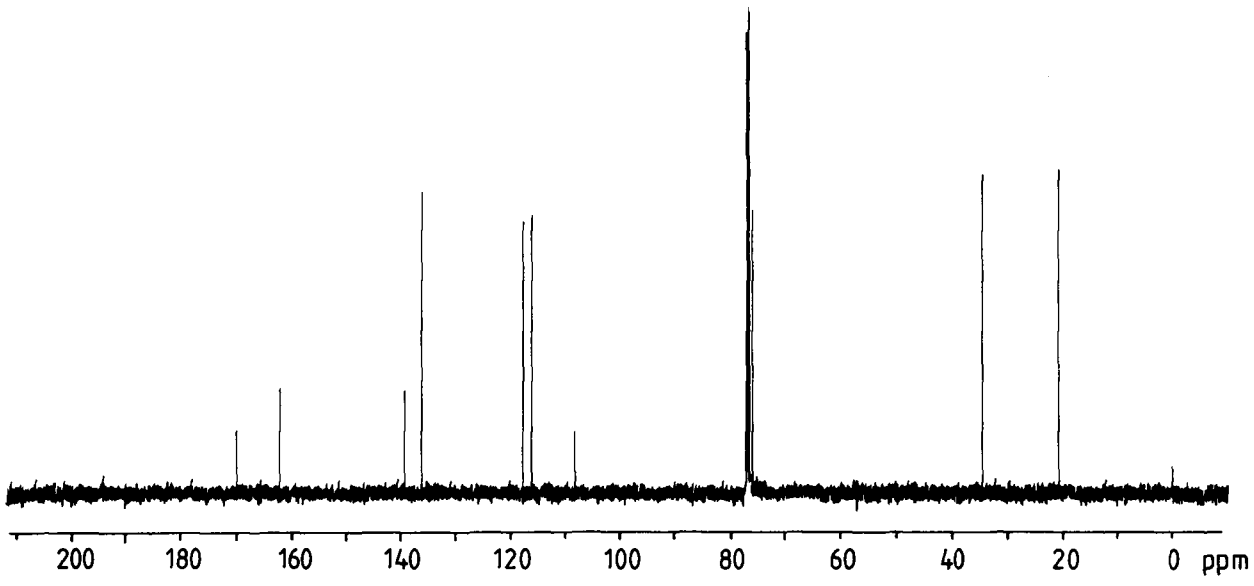
**Mass spectrum** [70 eV]




**$^1$H-NMR** [$CDCl_3$, 400 MHz]

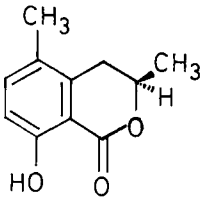


**$^{13}$C-NMR** [$CDCl_3$, 25 MHz]

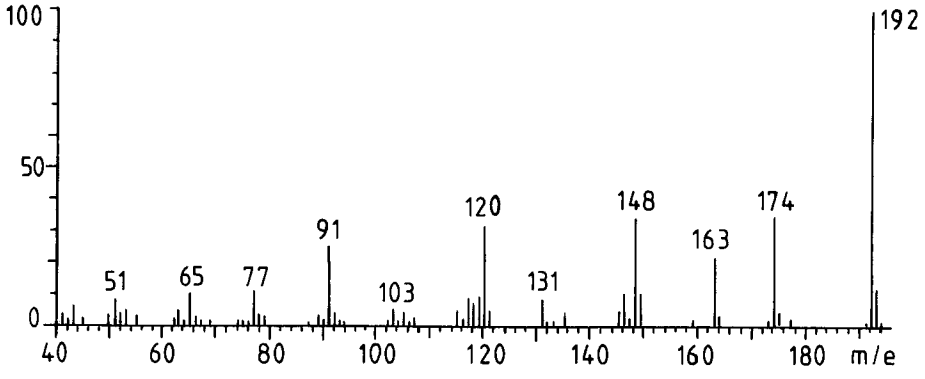


# Methylmellein

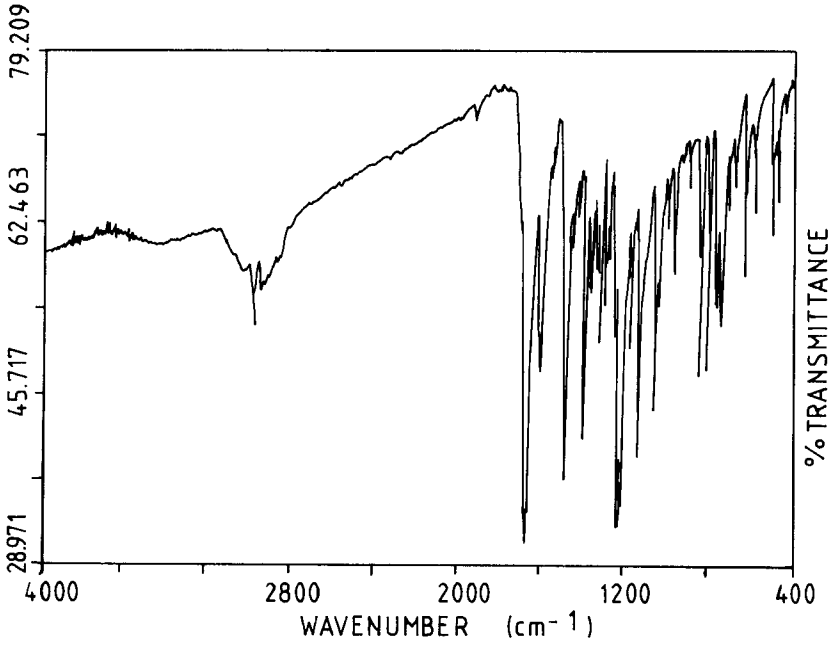
**$C_{11}H_{12}O_3$**



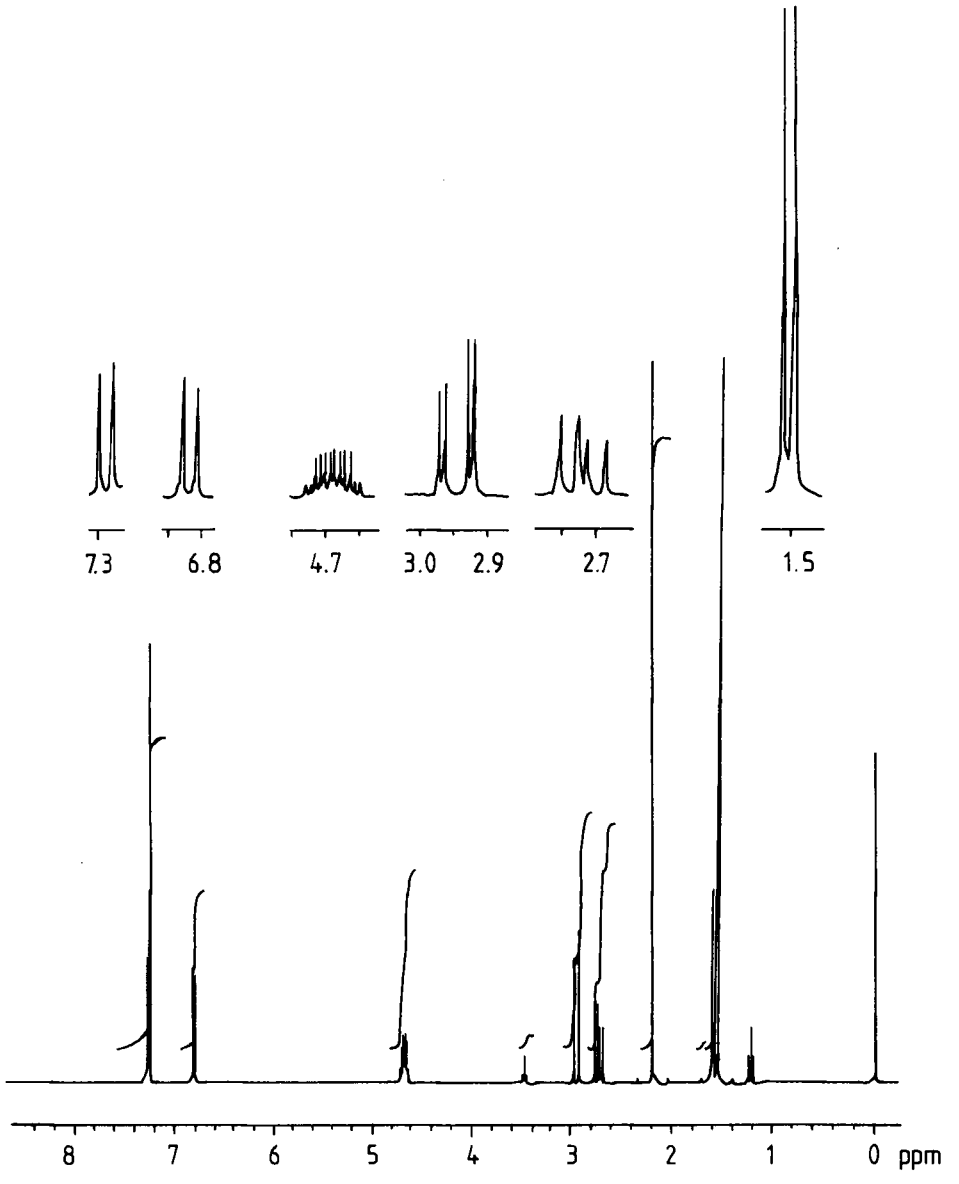

**Mass spectrum** [70 eV]



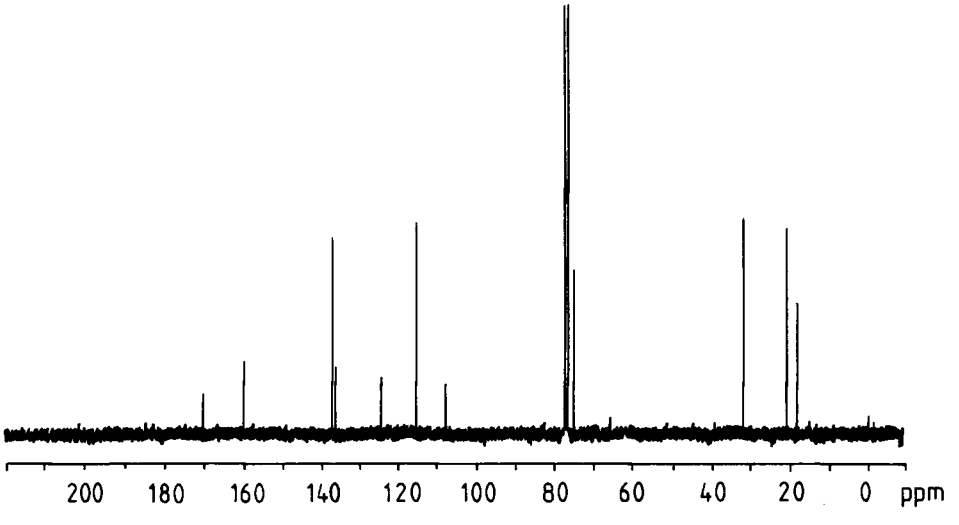

**IR spectrum** [KBr]



**$^1$H-NMR** [$CDCl_3$, 400 MHz]

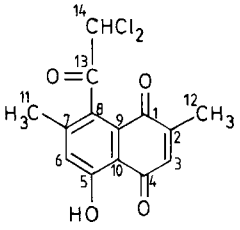



**$^{13}$C-NMR** [$CDCl_3$, 100 MHz]

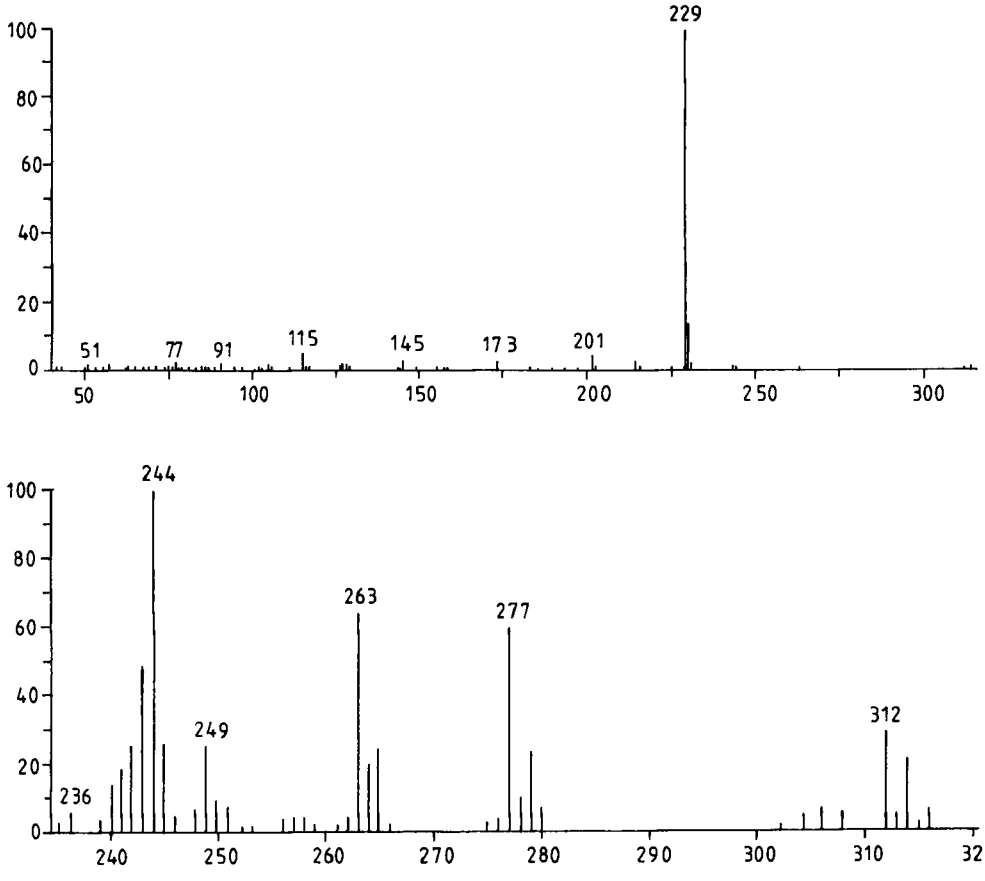


# Mollisin

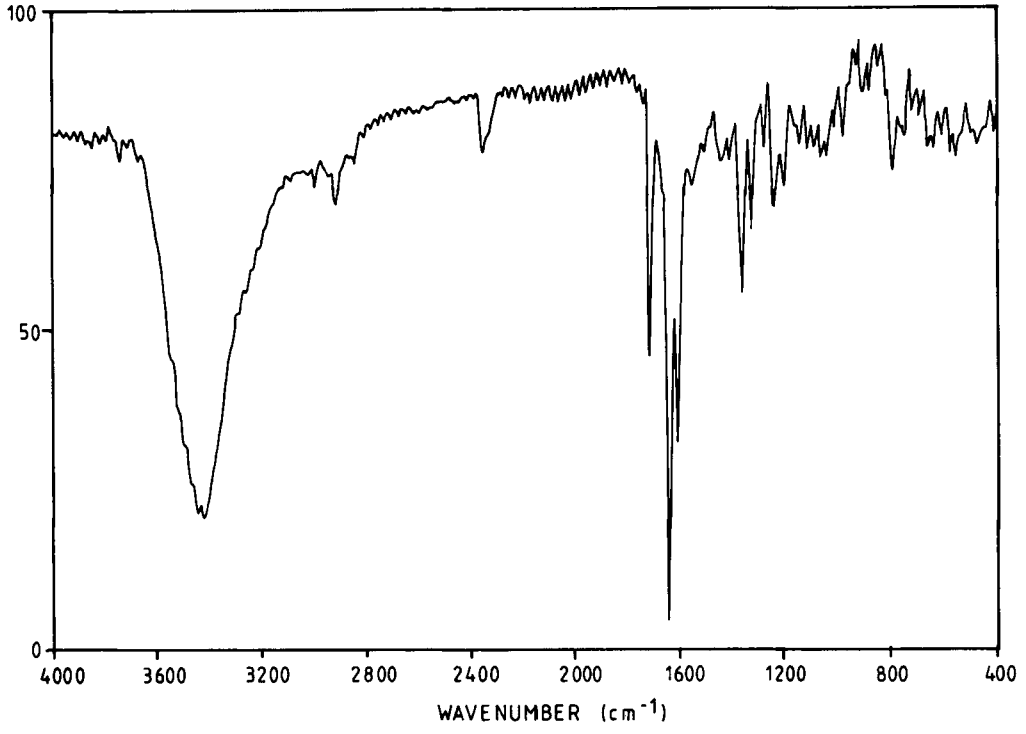
$C_{14}H_{10}O_4Cl_2$



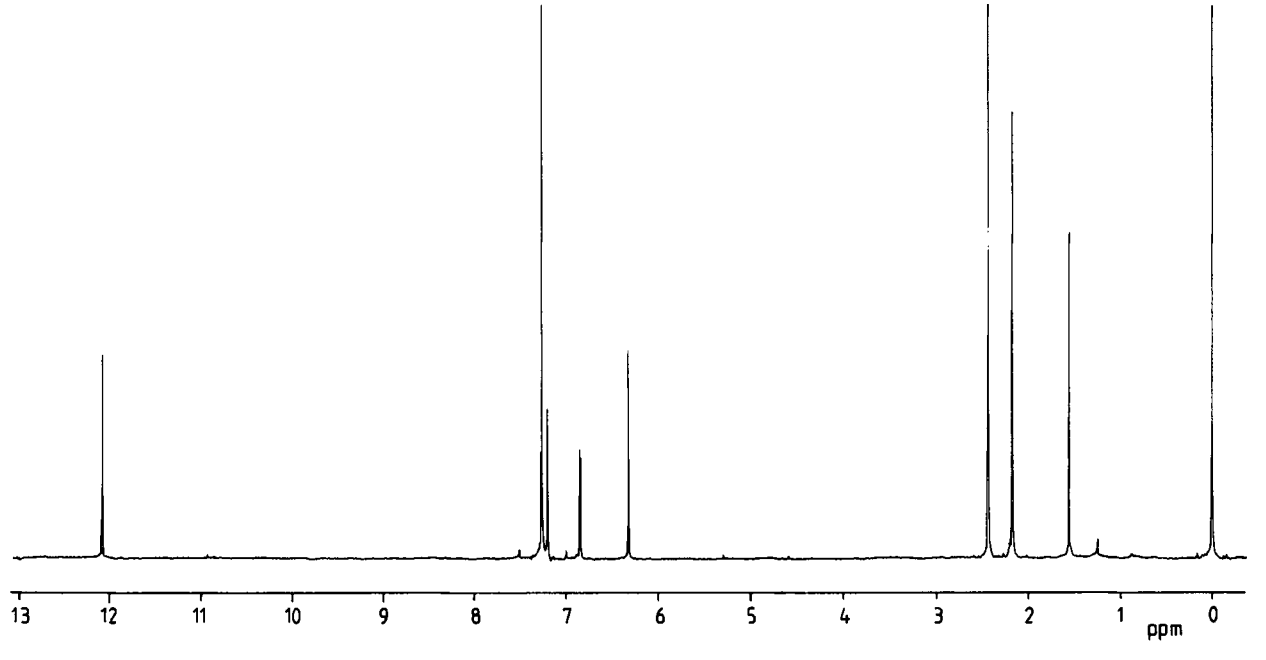

**Mass spectrum** [70 eV]



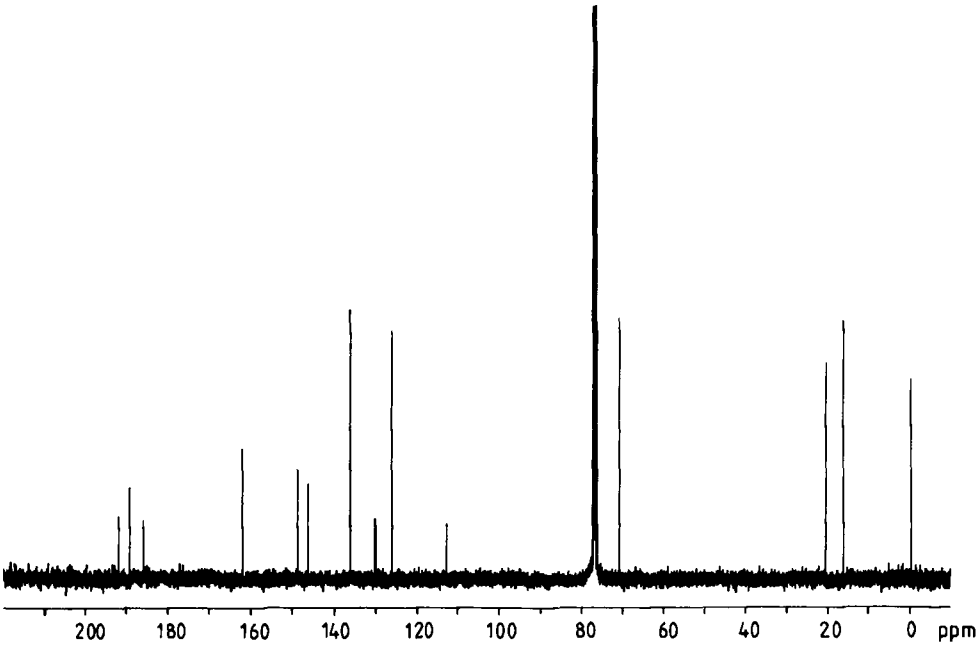
**IR spectrum** [KBr]




**$^1$H-NMR** [$CDCl_3$, 400 MHz]



**$^{13}$C-NMR** [$CDCl_3$, 400 MHz]



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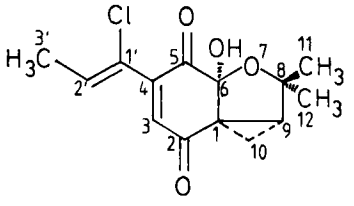
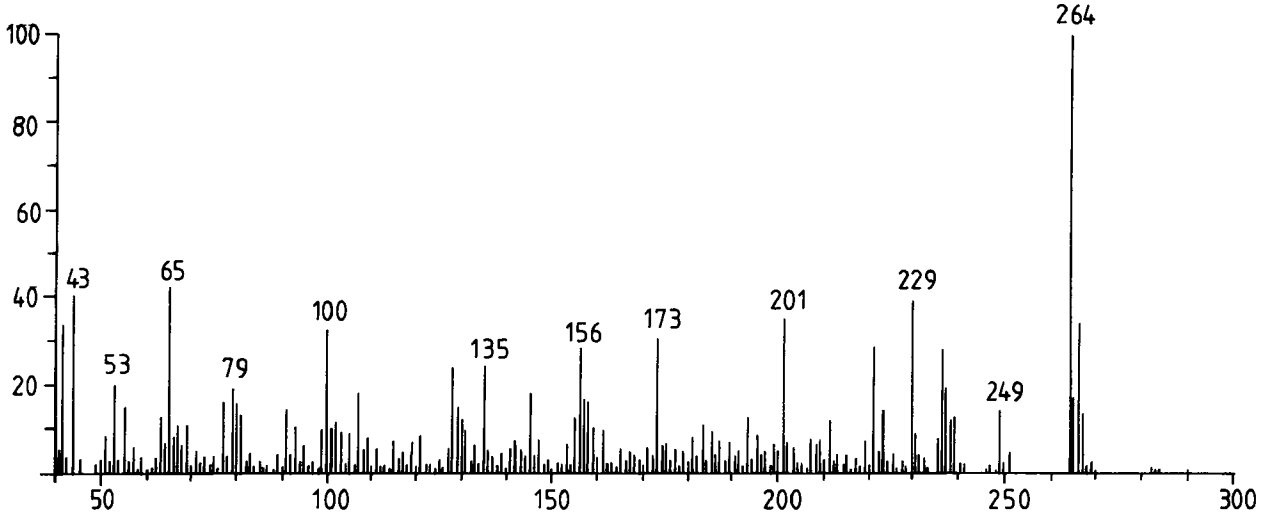
# Mycorrhizin A

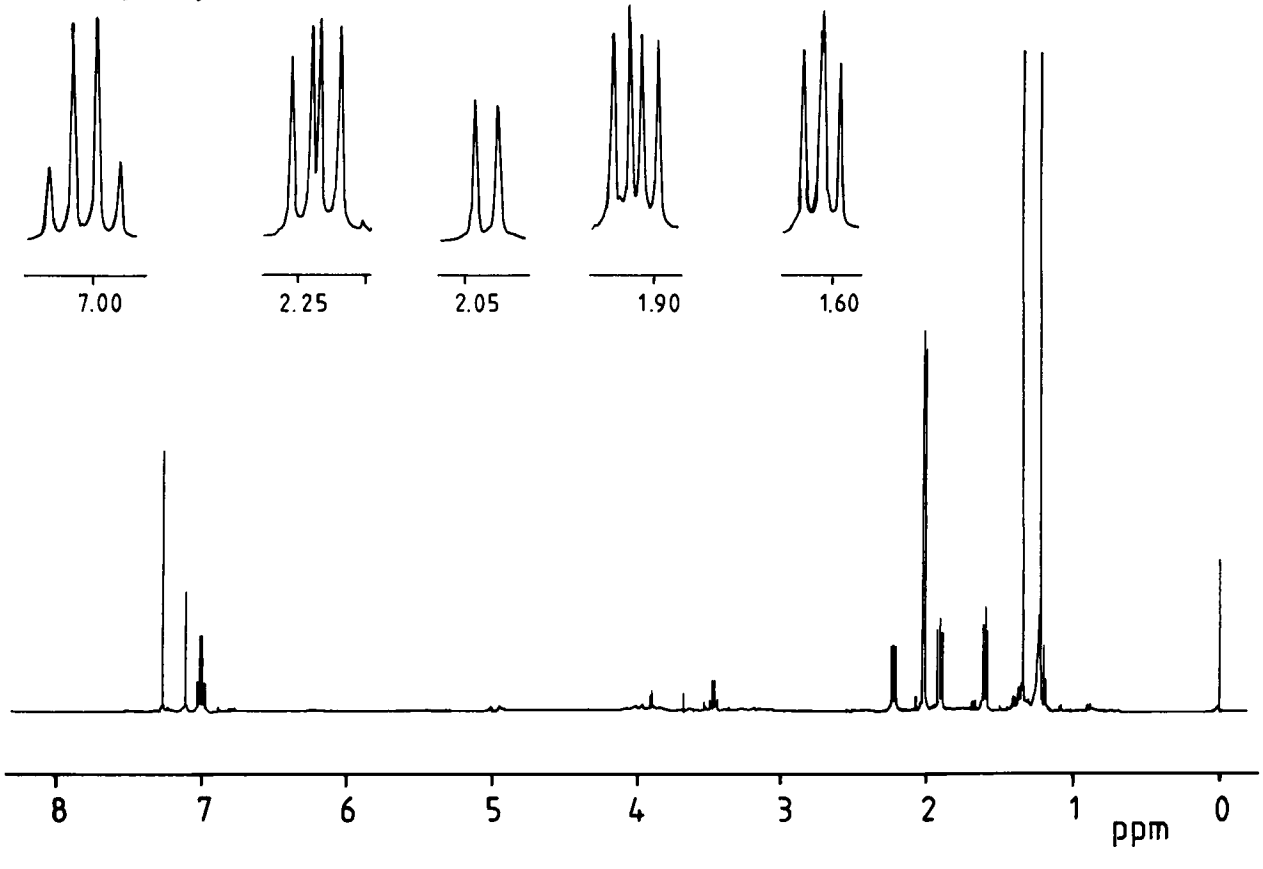
$C_{14}H_{15}O_4Cl$



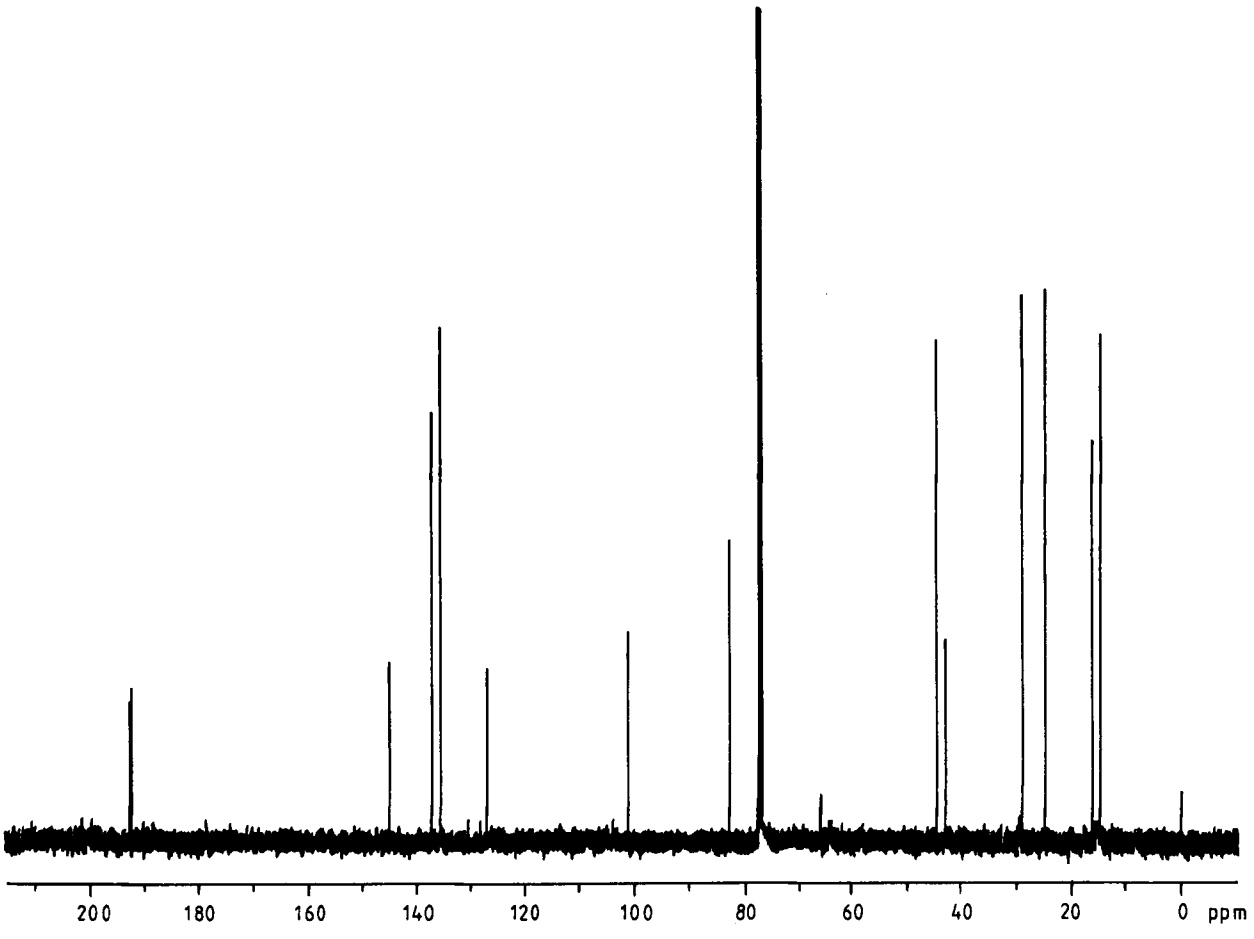

**Mass spectrum** [70 eV]




**$^1$H-NMR** [$CDCl_3$, 400 MHz]

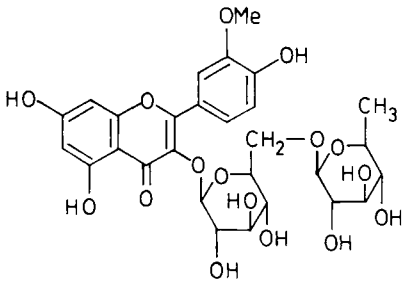


**$^{13}$C-NMR** [$CDCl_3$, 100 MHz]

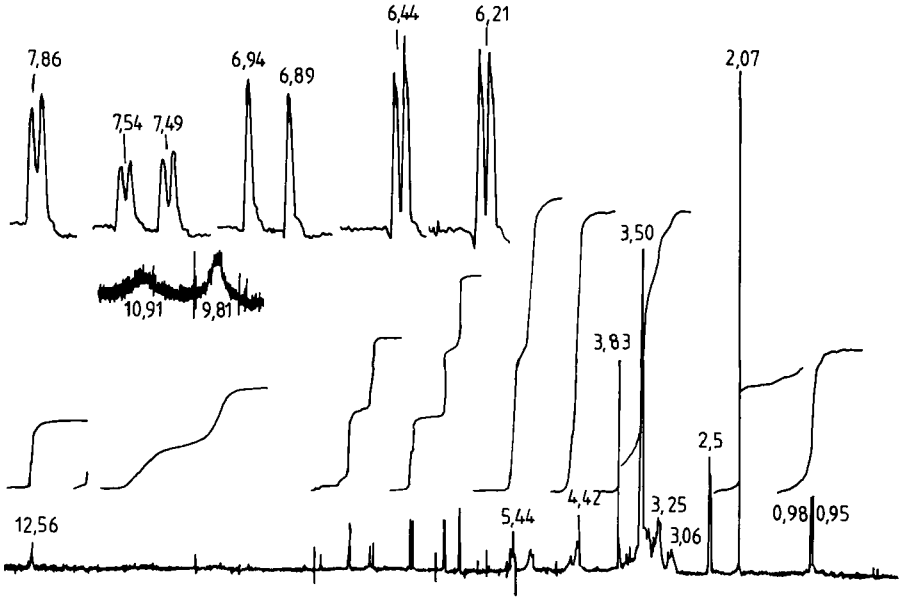


# Narcissin

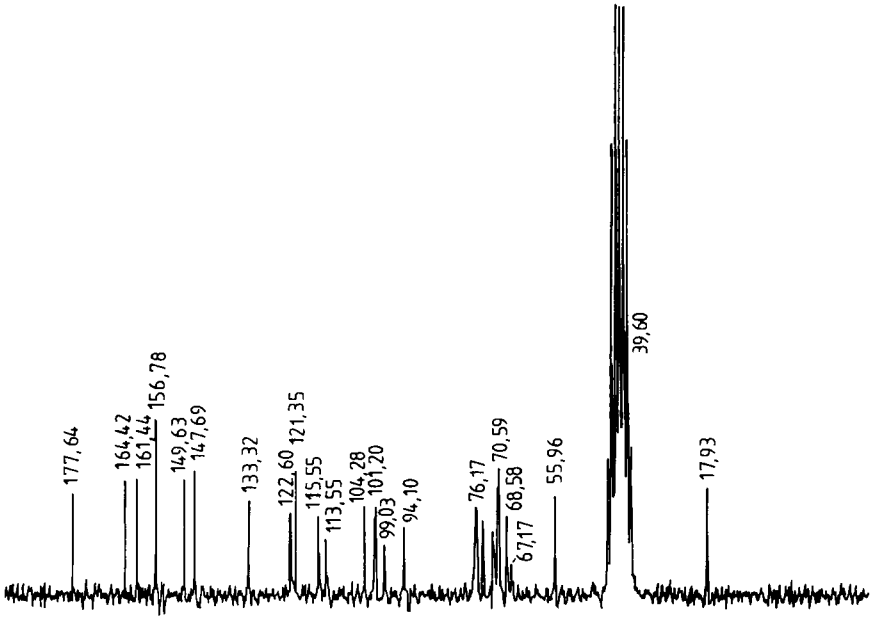
**$C_{28}H_{32}O_{16}$**




**$^1$H-NMR** [DMSO-$d_6$]




**$^{13}$C-NMR** [DMSO-$d_6$, 22.49 MHz]



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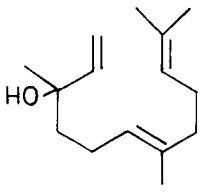
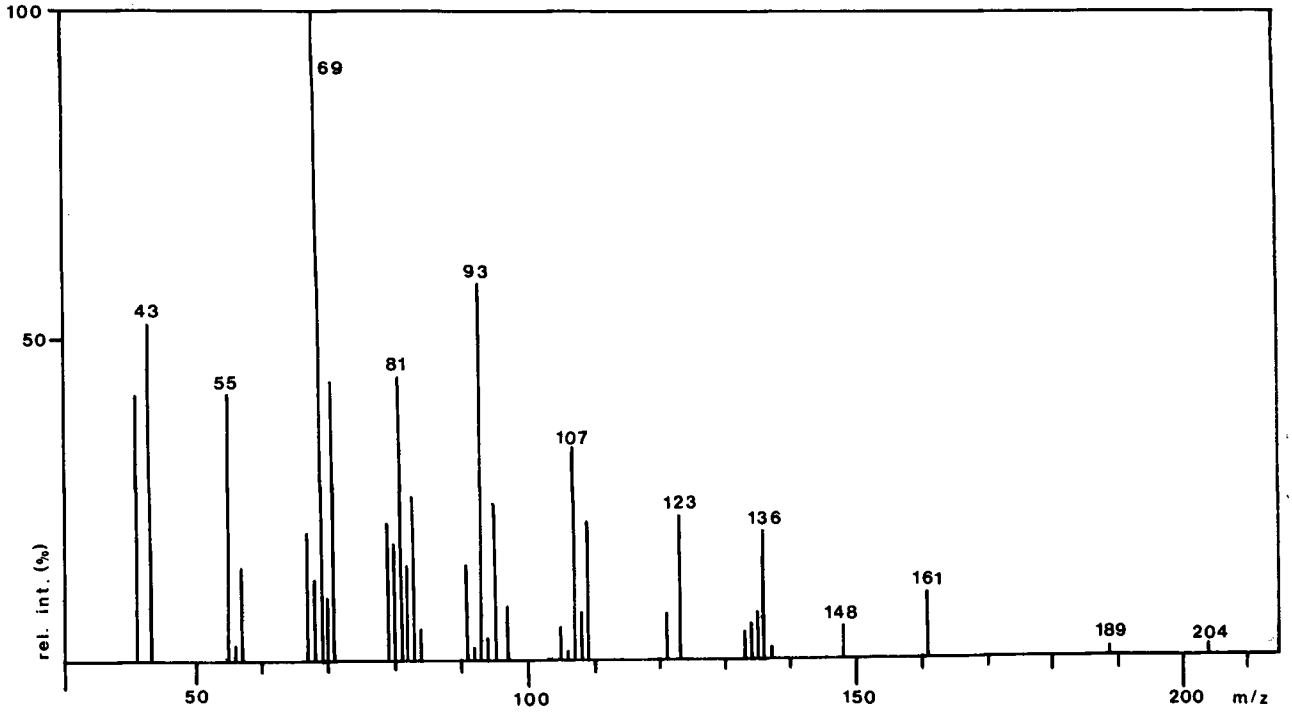
# trans-Nerolidol

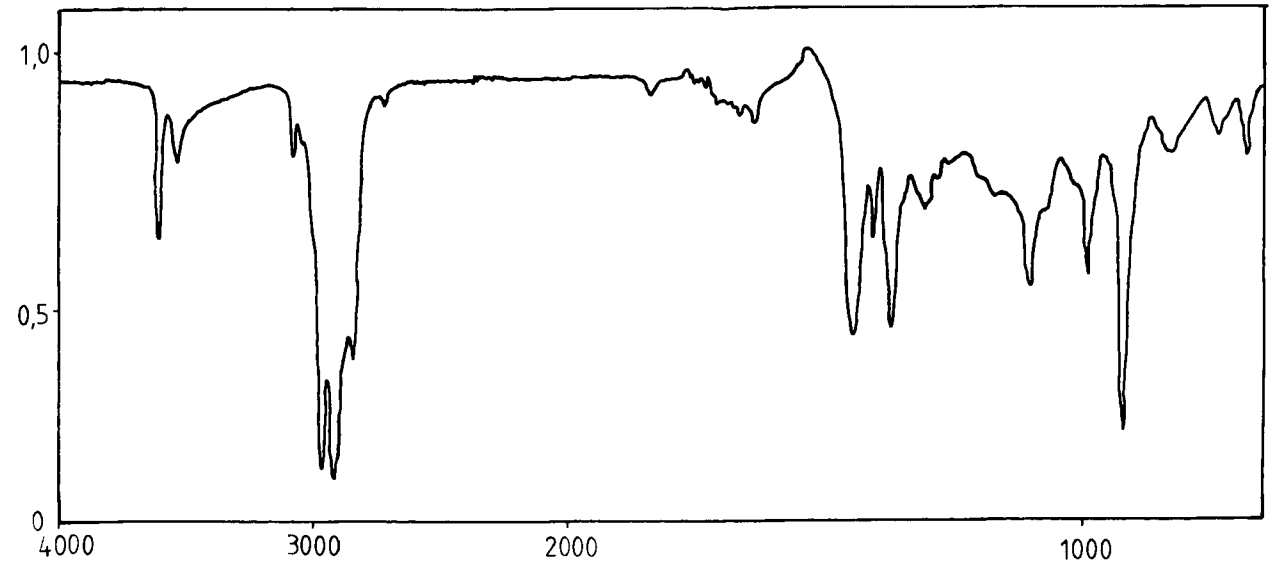
$C_{15}H_{26}O$



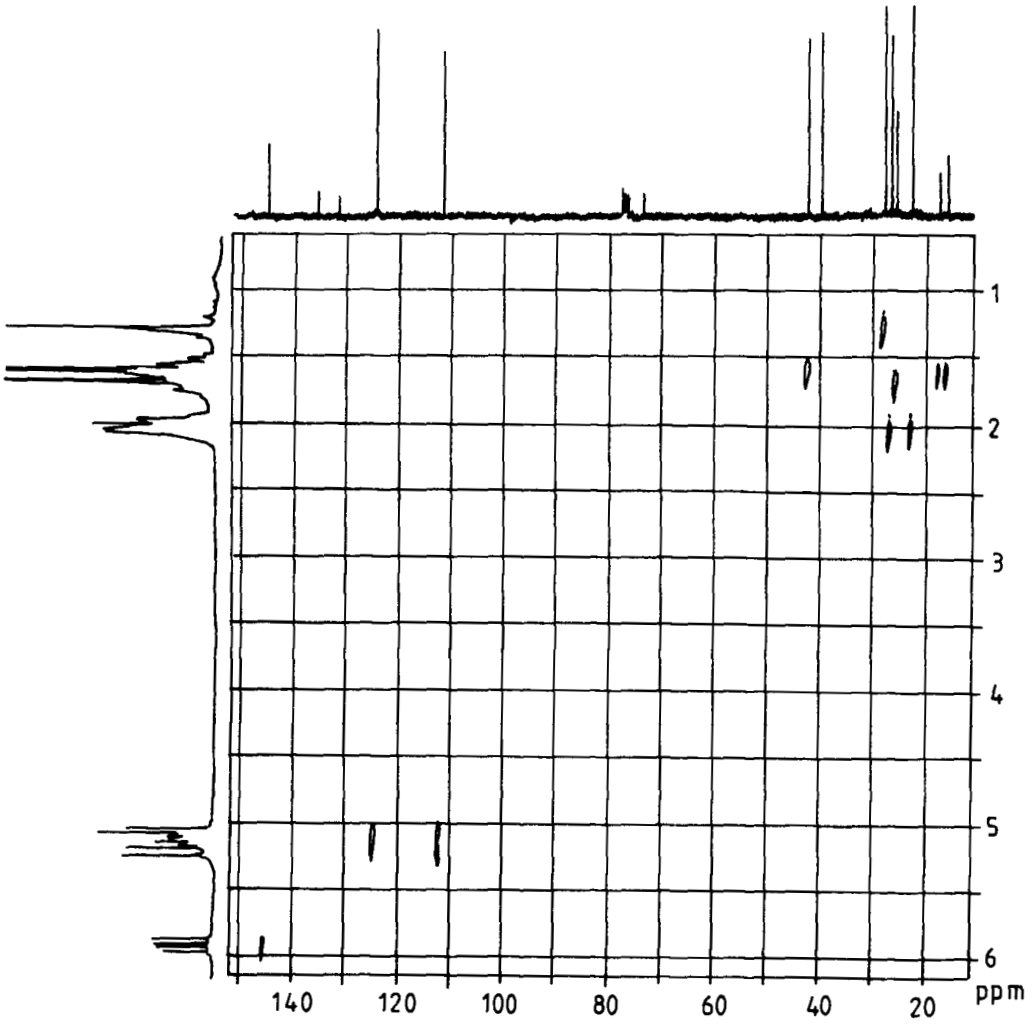

**Mass spectrum** [70 eV]




**IR spectrum** [2% in $CCl_4$]



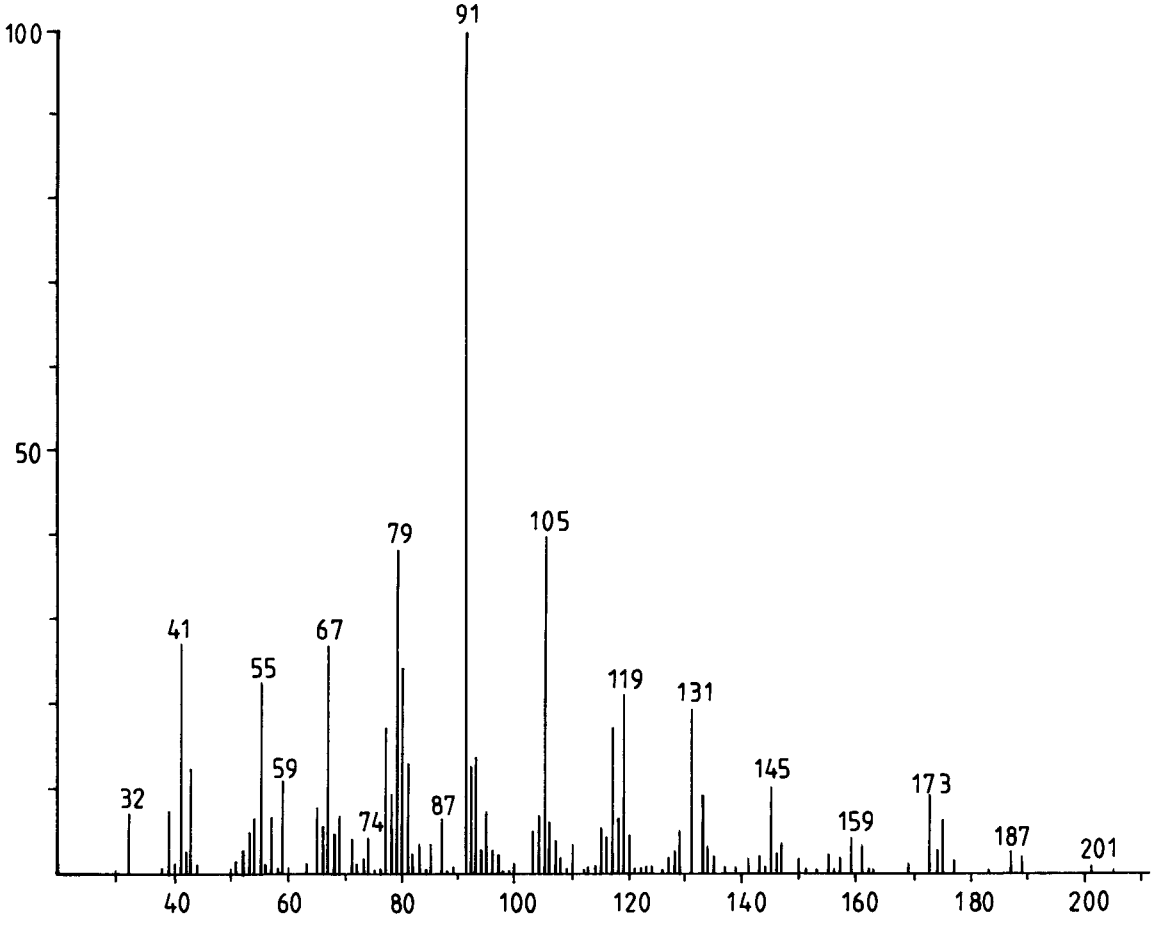
**2D NMR** [$CDCl_3$, 300 MHz]



# 9,12-Octadecadien-6-ynoic acid

**$C_{18}H_{28}O_2$** $HOOC-(CH_2)_4-C\equiv C-CH_2-CH=CH-CH_2-CH=CH-(CH_2)_4-CH_3$

**Mass spectrum as methylester** [70 eV]



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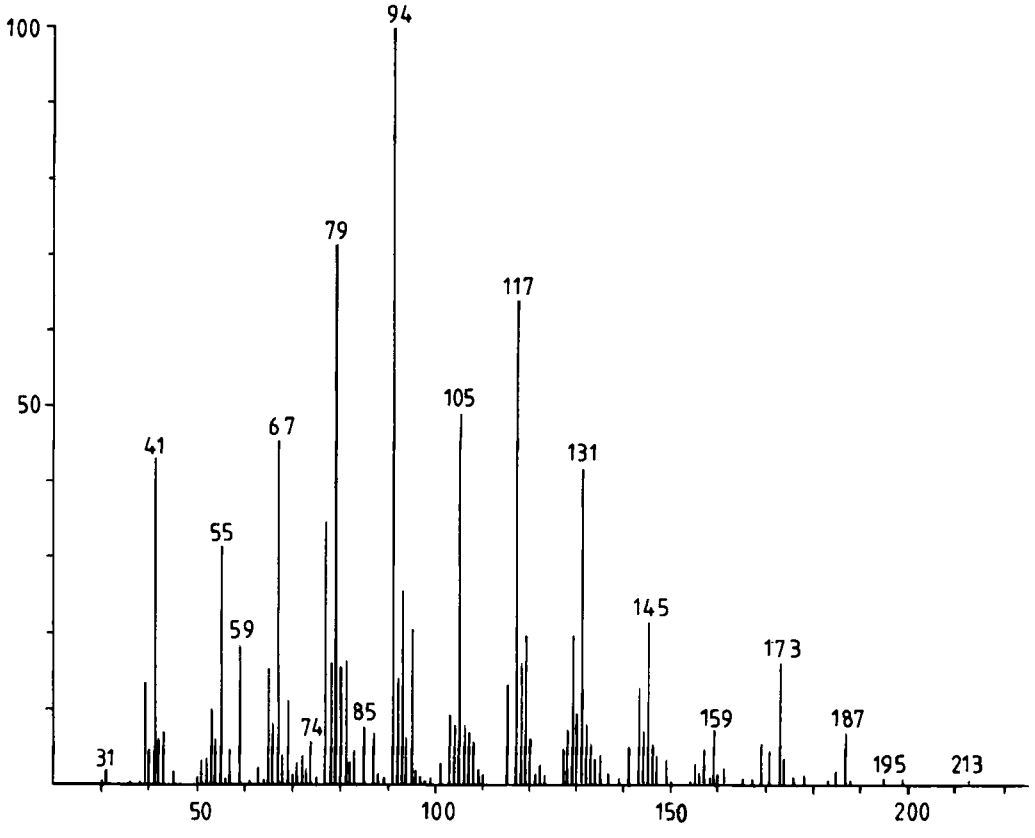
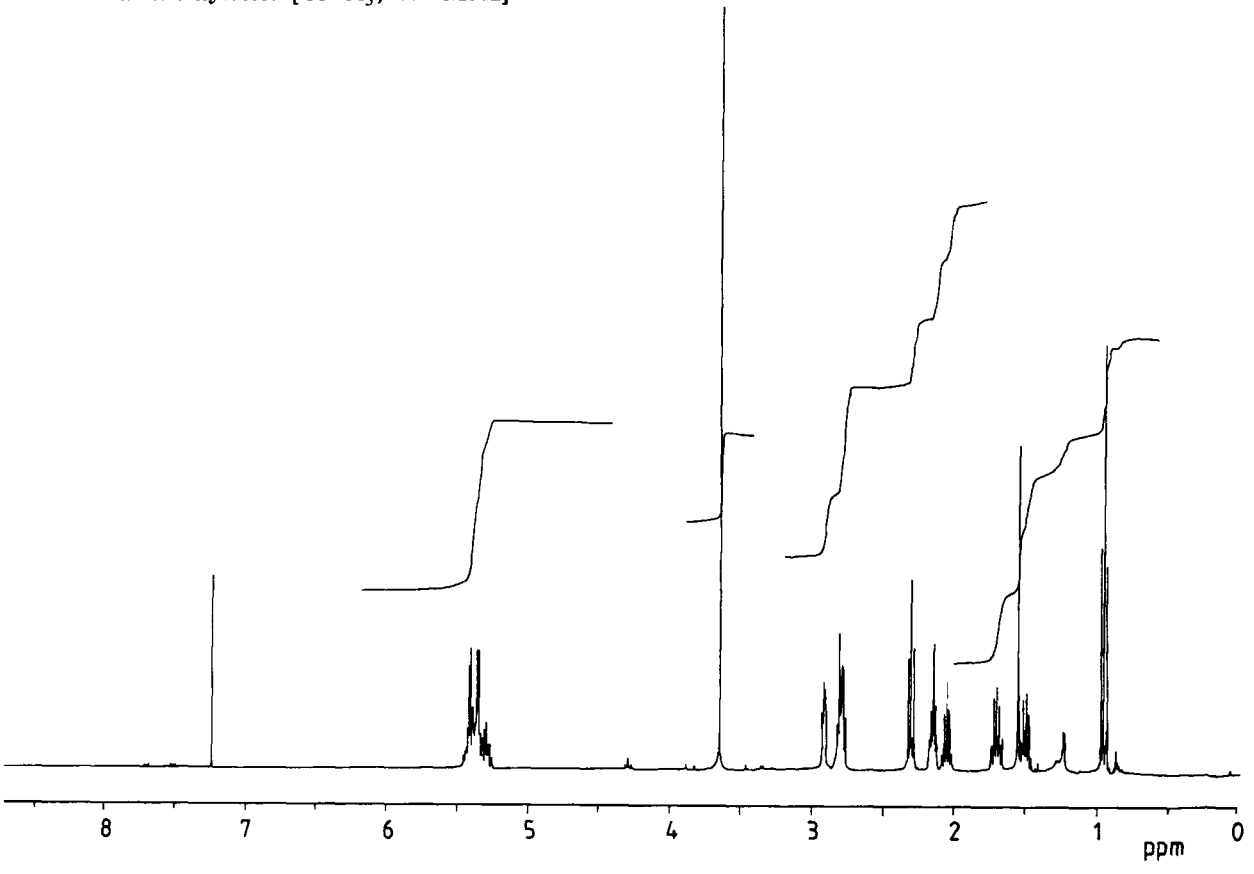
# 9,12,15-Octadecatrien-6-ynoic acid

**$C_{18}H_{26}O_2$** $HOOC-(CH_2)_4-C\equiv C-CH_2-CH=CH-CH_2-CH=CH-CH_2-CH=CH-CH_2-CH_3$

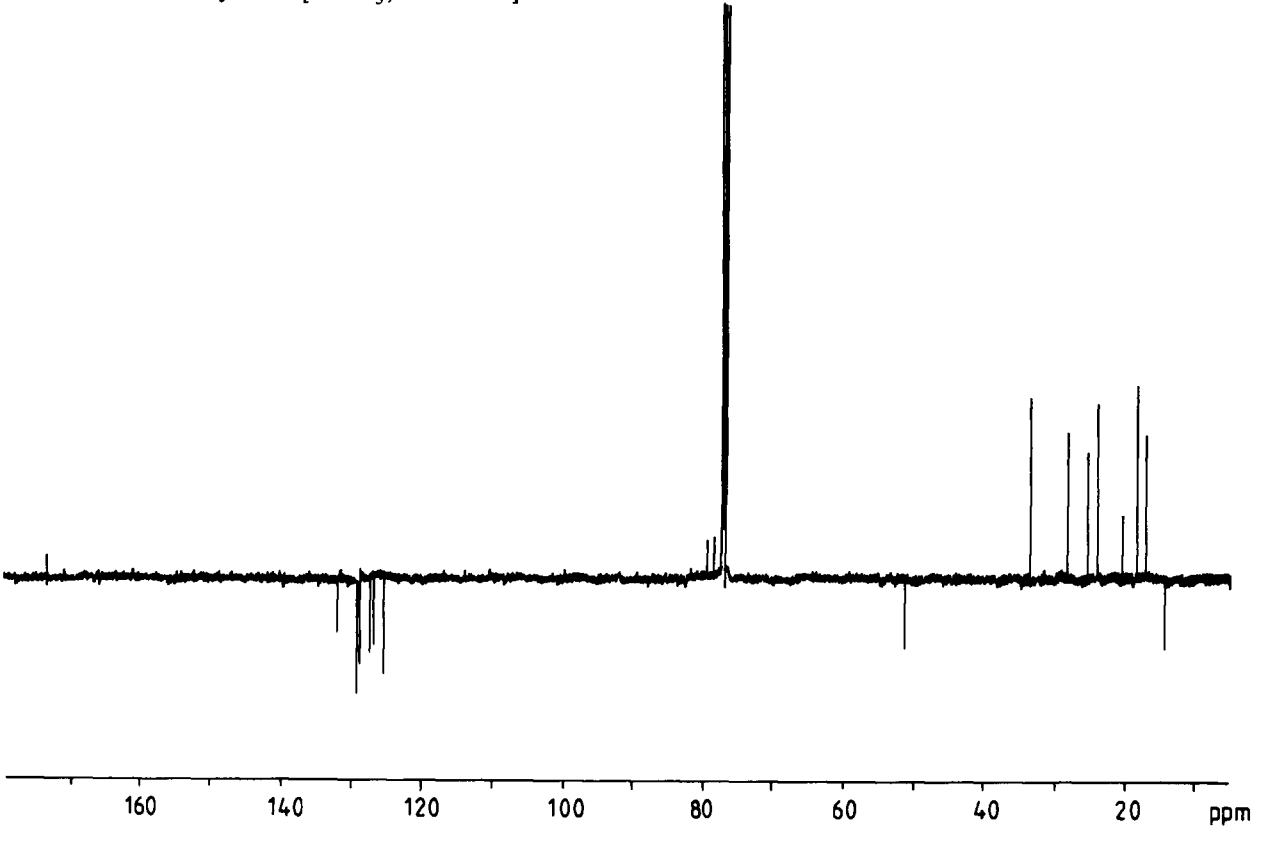
**Mass spectrum as methylester** [70 eV]



**$^1$H-NMR as methylester** [$CDCl_3$, 400 MHz]




**$^{13}$C-NMR as methylester** [$CDCl_3$, 400 MHz]

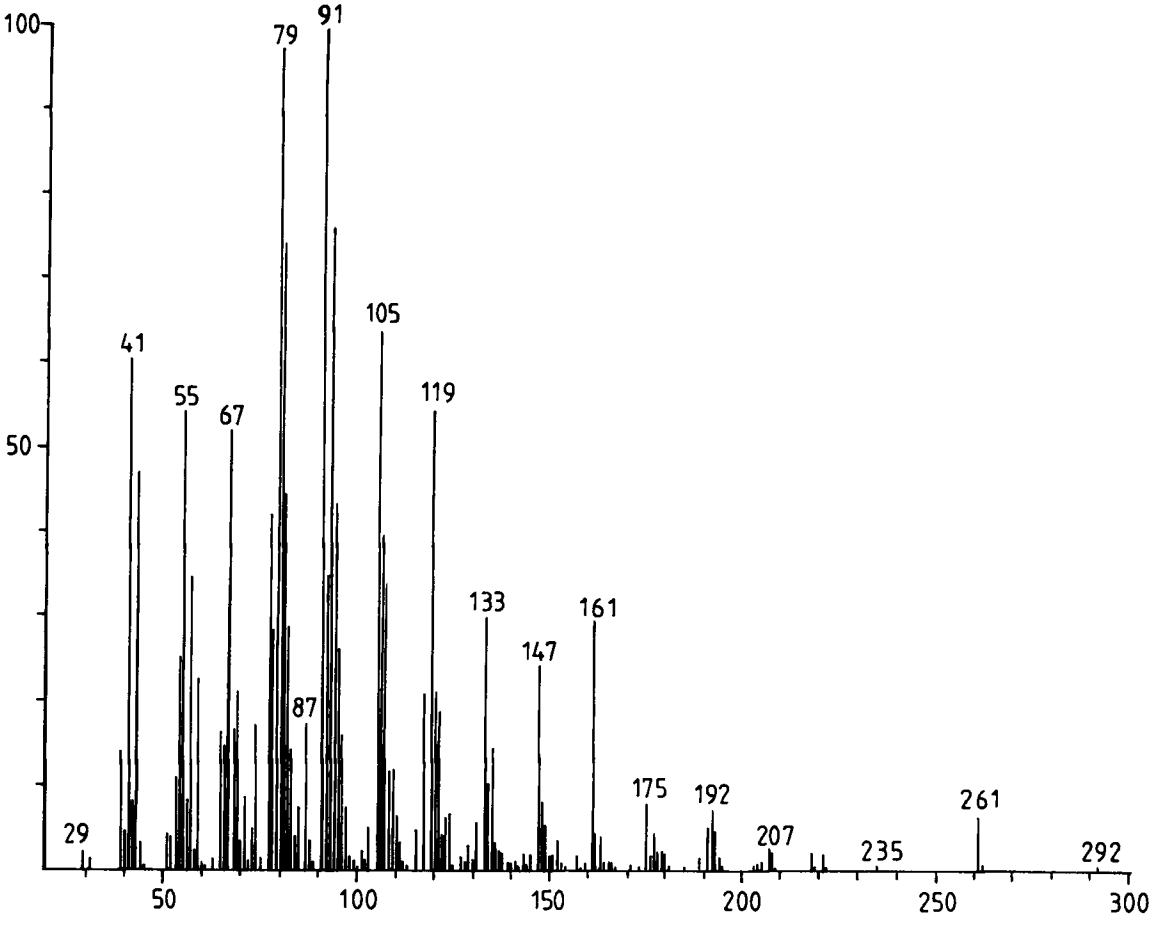



9,12,15-Octadecatrien-6-ynoic acid

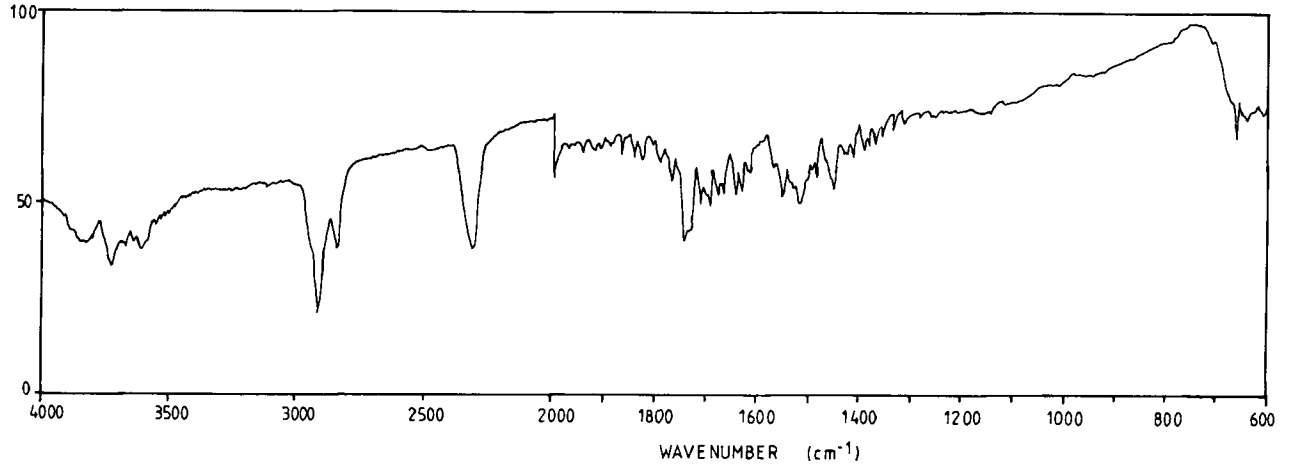
# 9-Octadecen-6-ynoic acid

**$C_{18}H_{30}O_2$** $HOOC-(CH_2)_4-C\equiv C-CH_2-CH=CH-(CH_2)_7-CH_3$

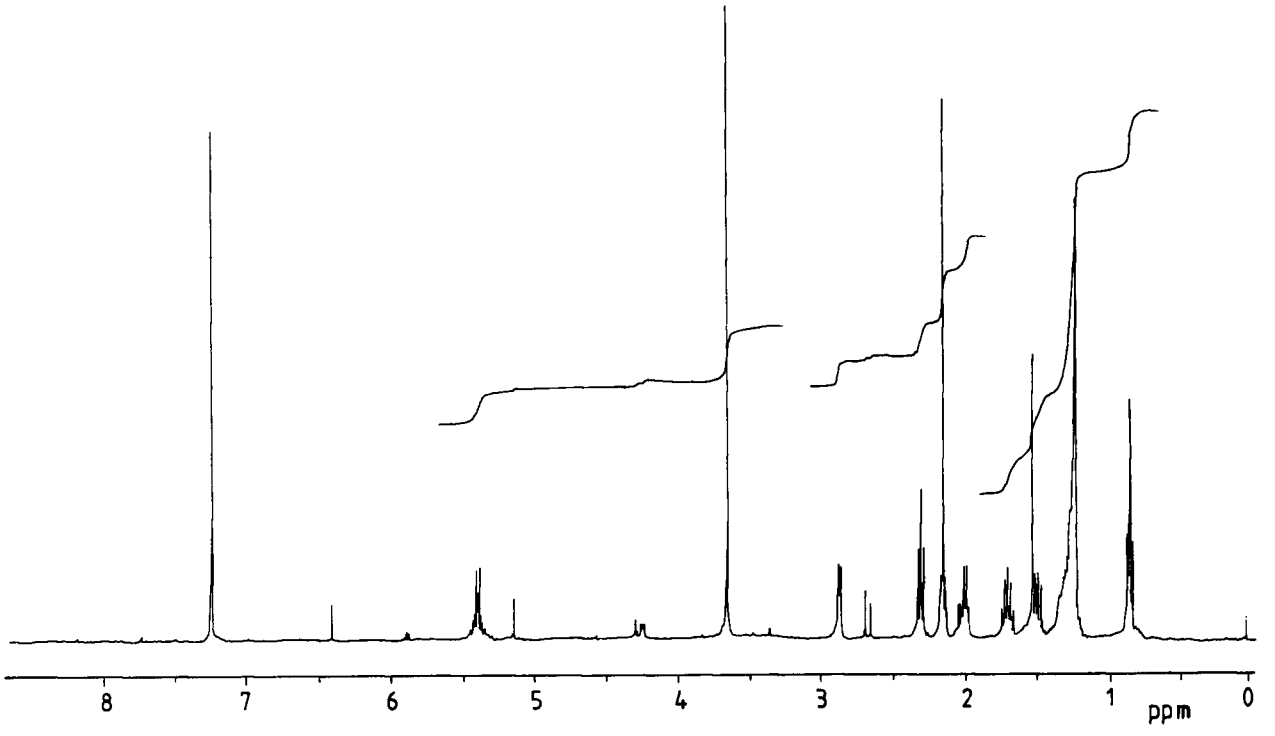
**Mass spectrum as methylester** [70 eV]



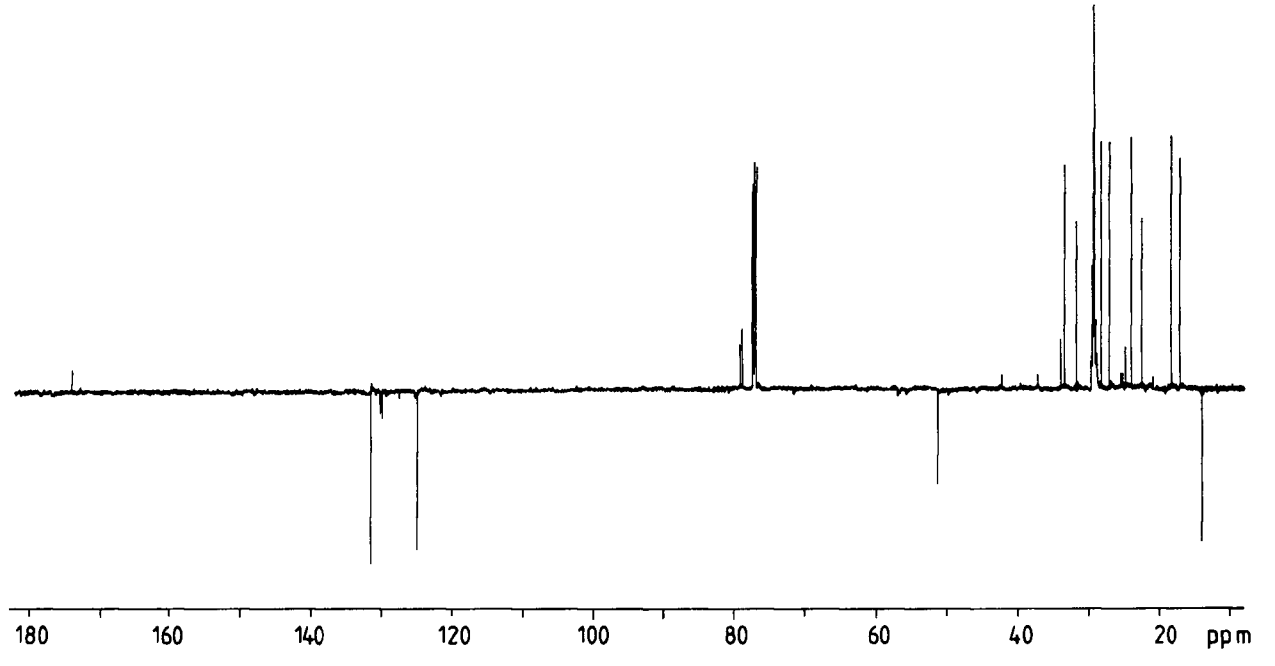

**IR spectrum as methylester** [KBr]



**$^1$H-NMR as methylester** [$CDCl_3$, 400 MHz]

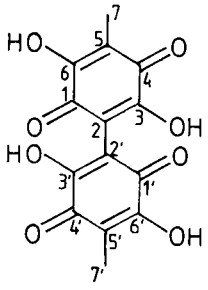



**$^{13}$C-NMR as methylester** [$CDCl_3$, 400 MHz]

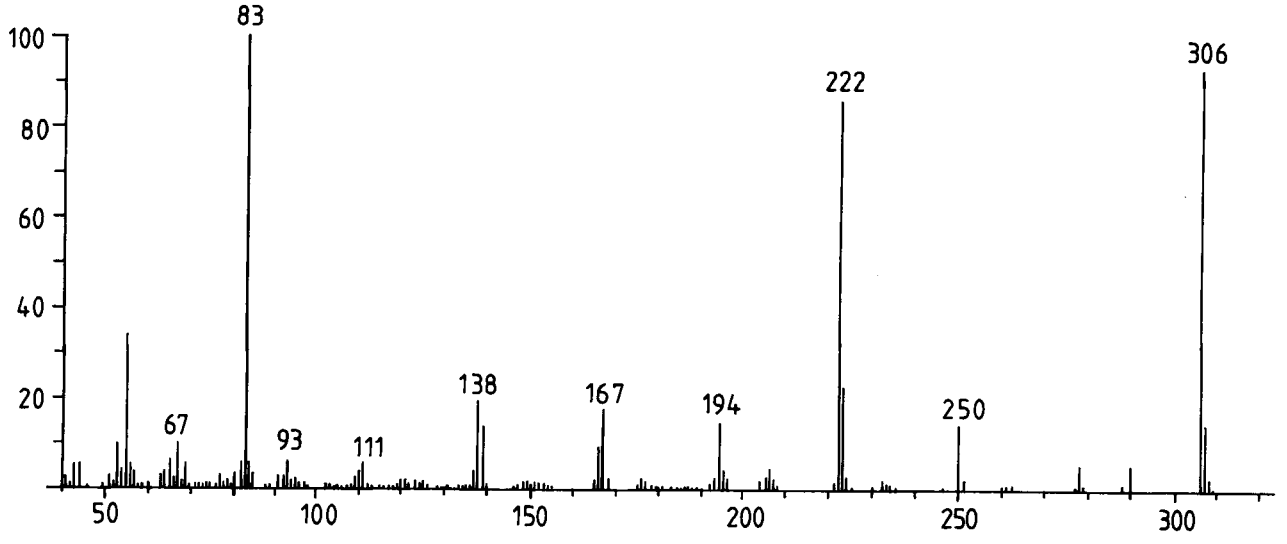


# Oosporein

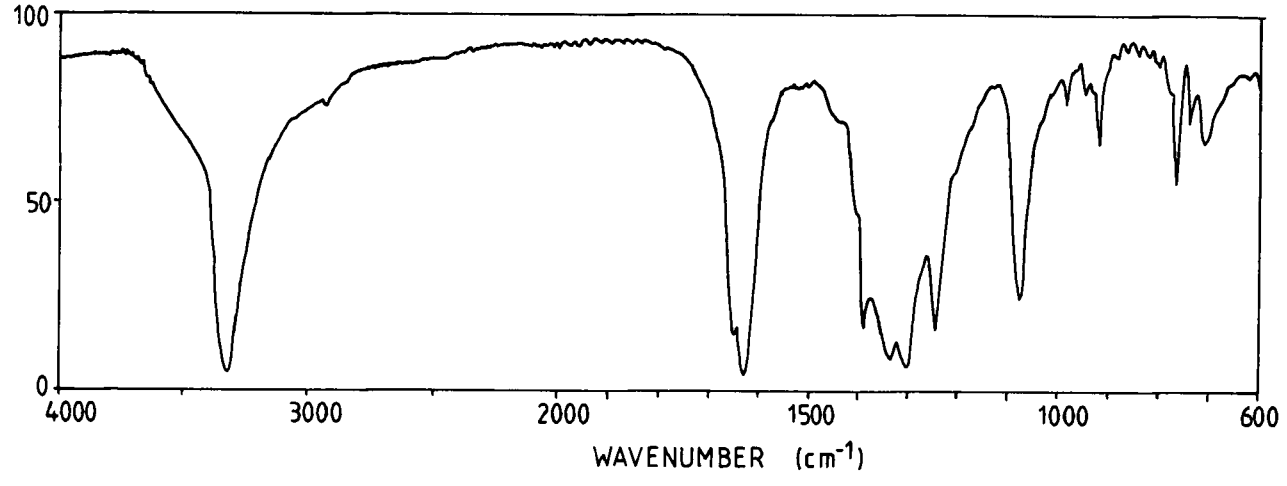
$C_{14}H_{10}O_8$



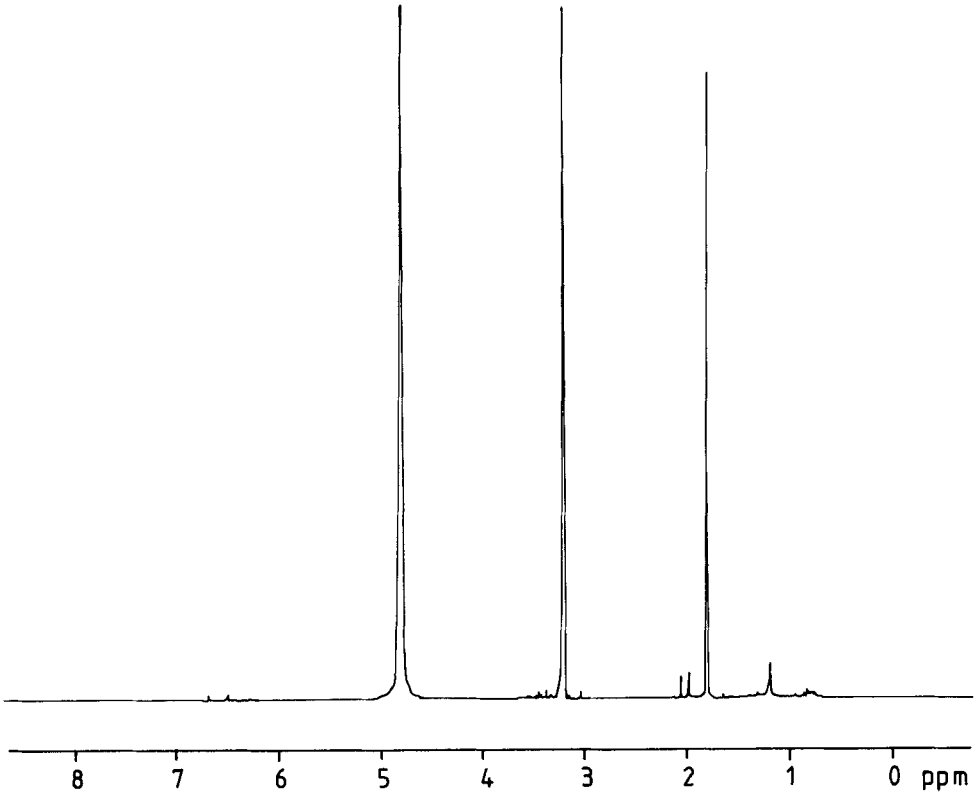

**Mass spectrum** [70 eV]



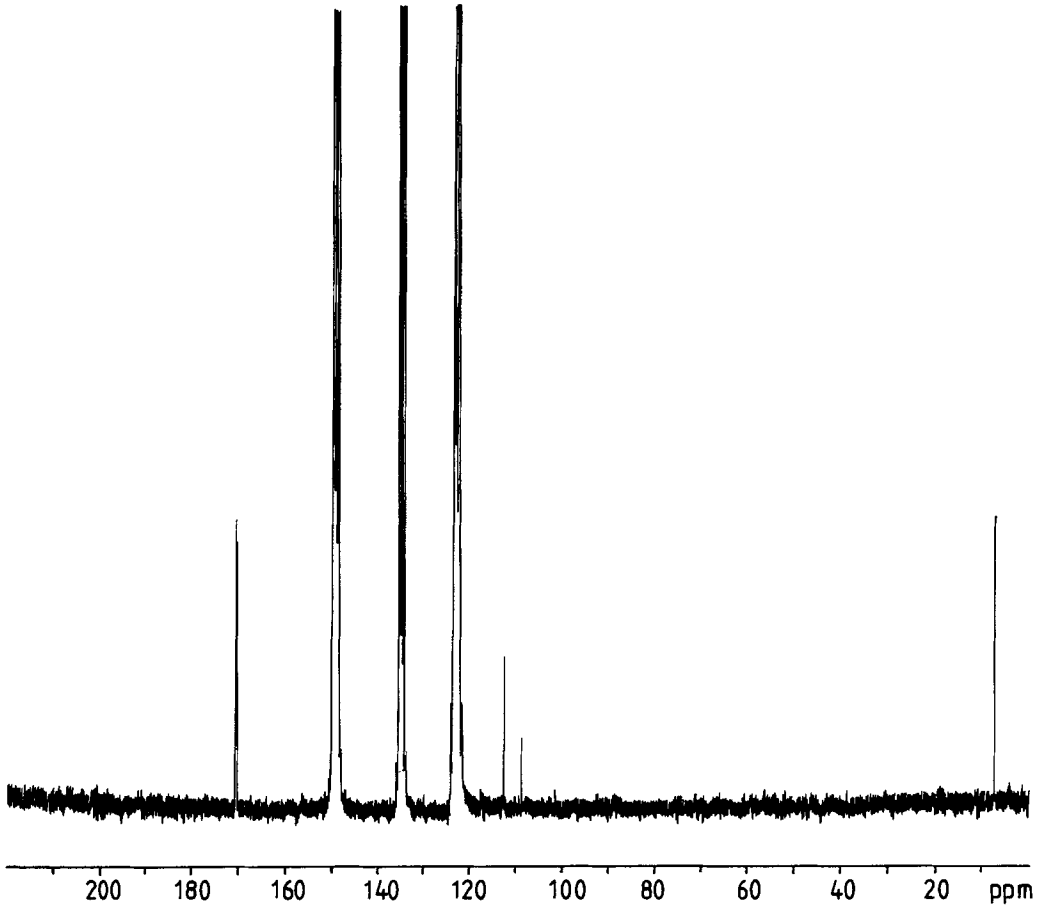

**IR spectrum** [KBr]



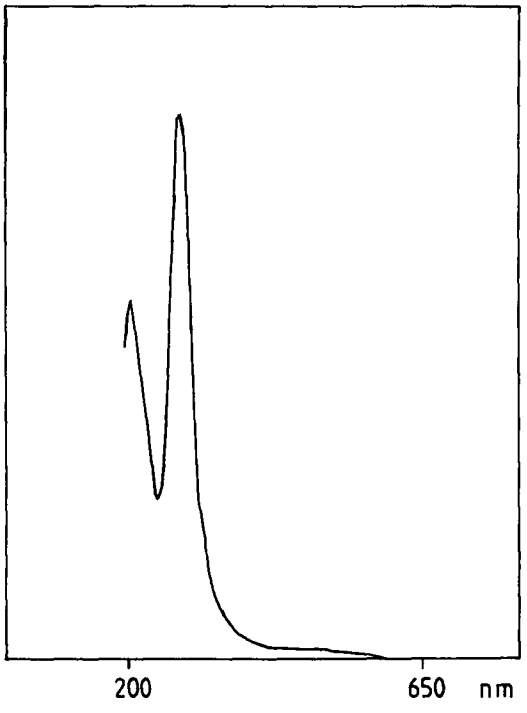
**$^1$H-NMR** [$CD_3OD$, 400 MHz]




**$^{13}$C-NMR** [$d_5$-Pyridin, 50 MHz]



**UV spectrum** [methanol]



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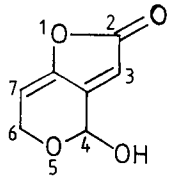
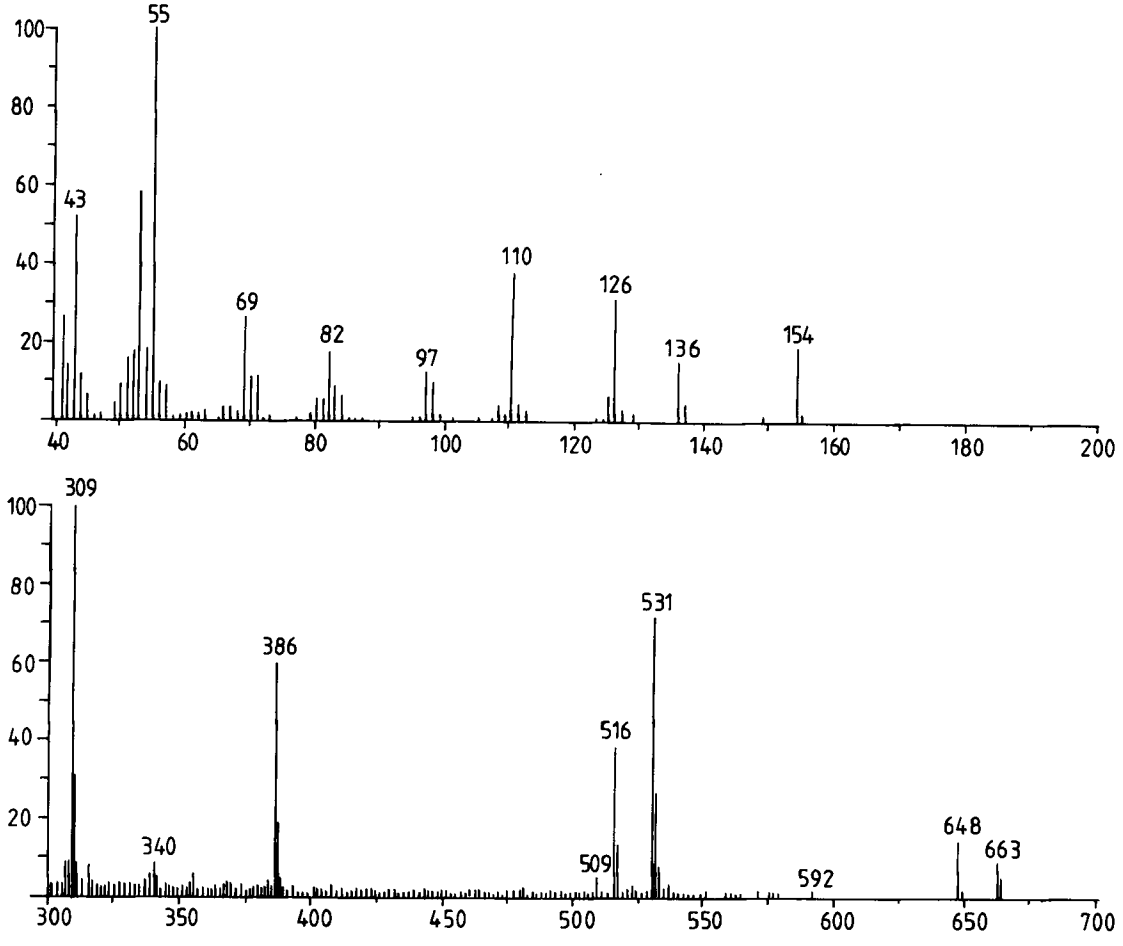
# Patulin

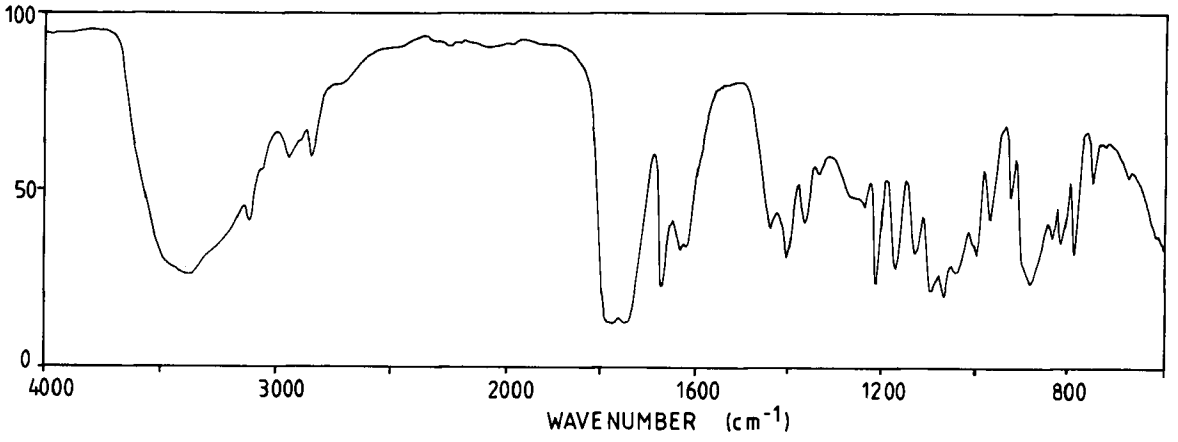
$C_7H_6O_4$



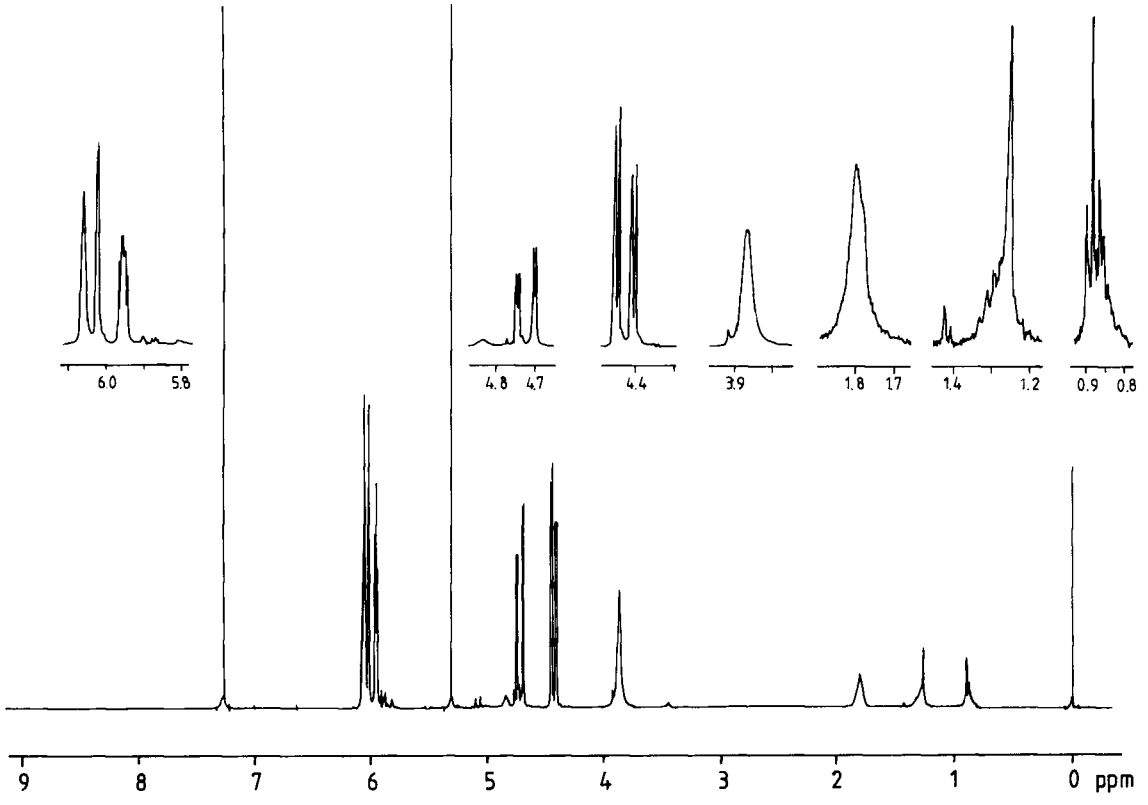

**Mass spectrum** [70 eV]



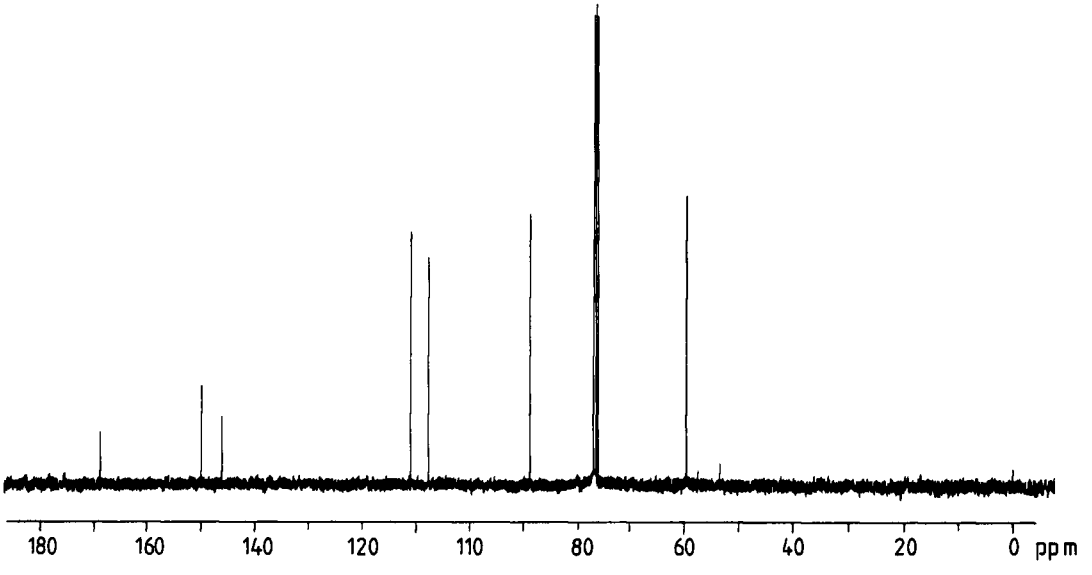

**IR spectrum** [liquid film]



**$^1$H-NMR** [$CDCl_3$, 400 MHz]

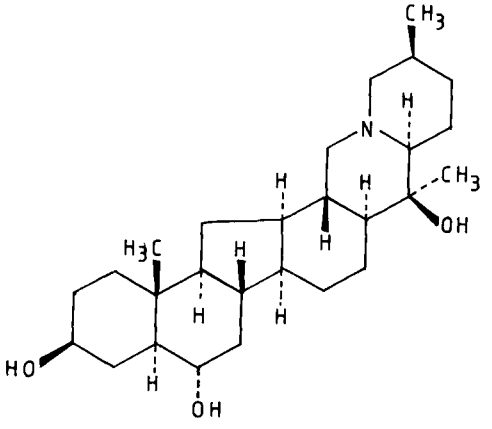



**$^{13}$C-NMR** [$CDCl_3$, 100 MHz]

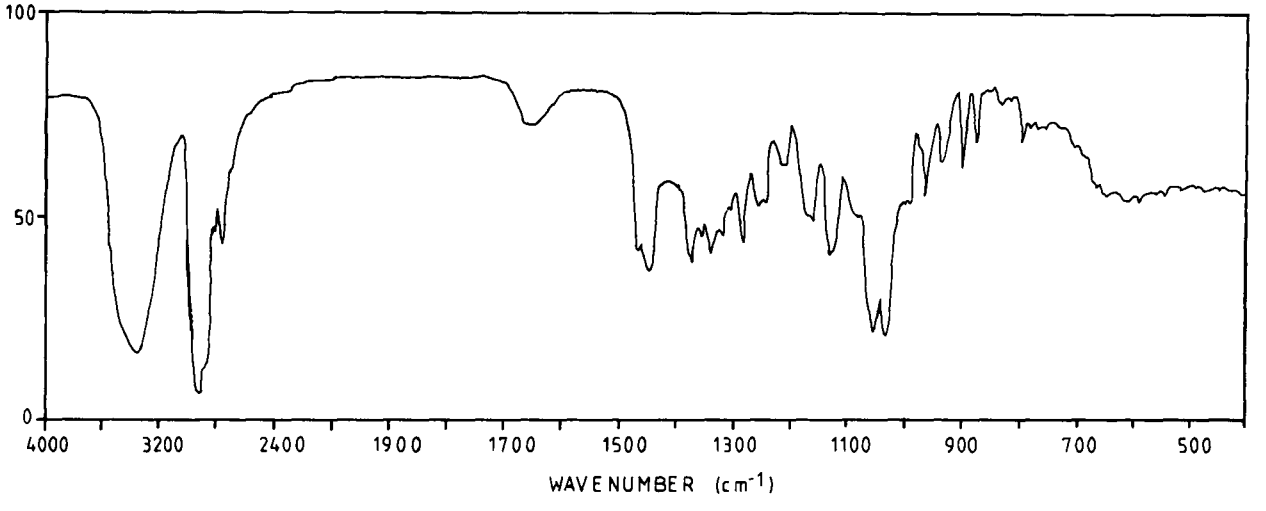


# Peimine

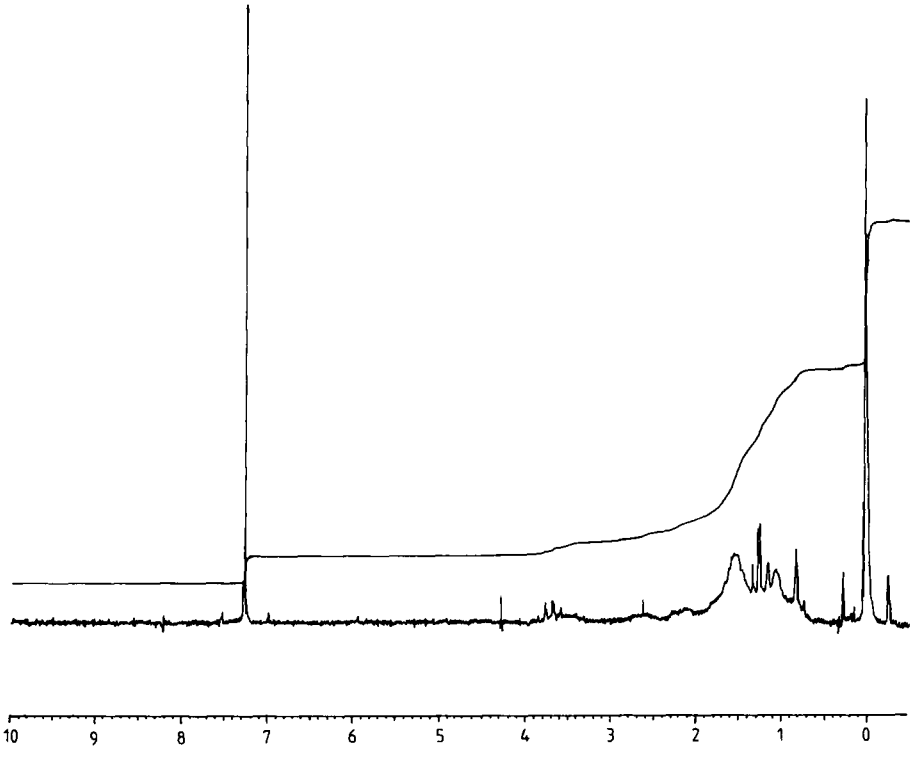
## $C_{27}H_{45}NO_3$




**IR spectrum** [KBr]




**$^1$H-NMR** [$CDCl_3$, 80 MHz]



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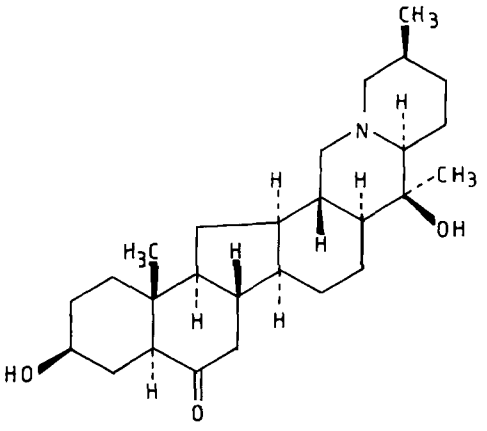
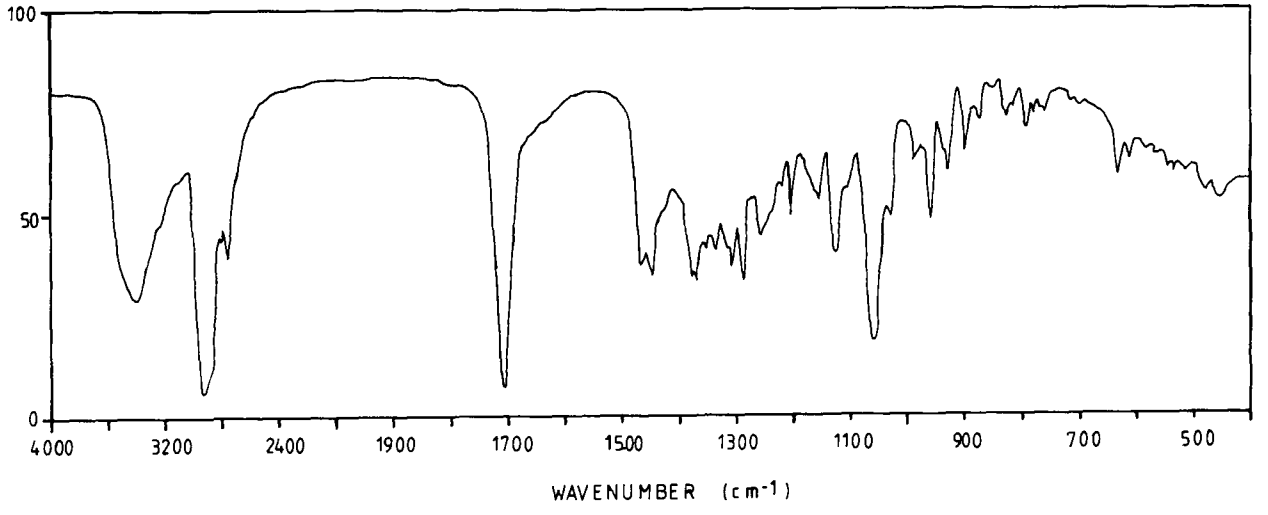
# Peiminine

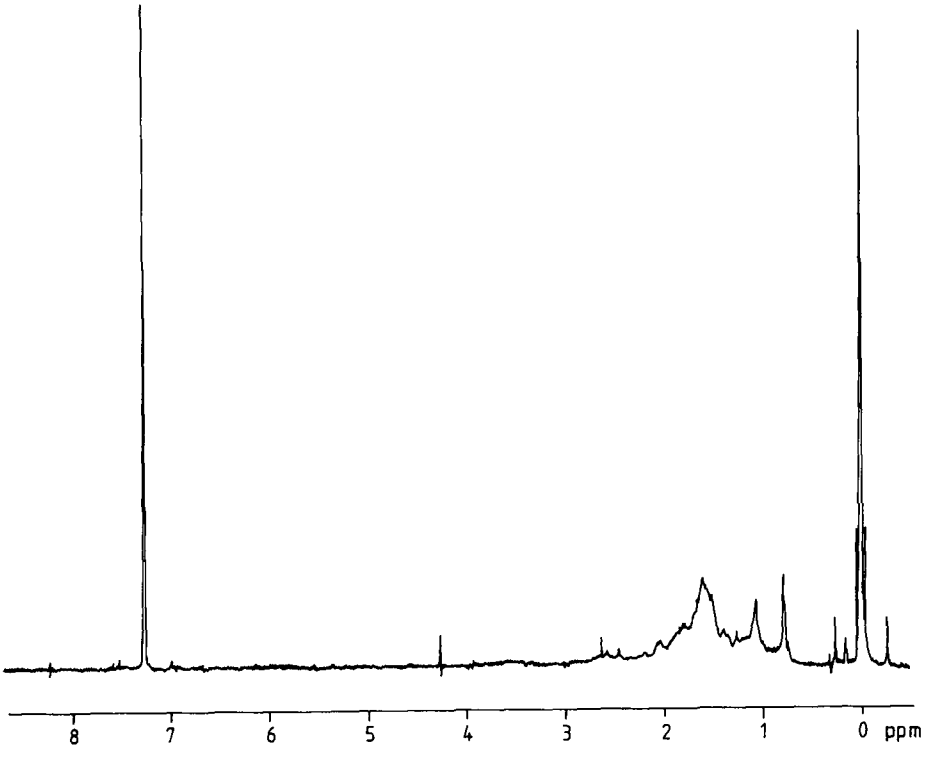
$C_{27}H_{43}NO_3$




**IR spectrum** [KBr]




**$^1$H-NMR** [$CDCl_3$, 80 MHz]



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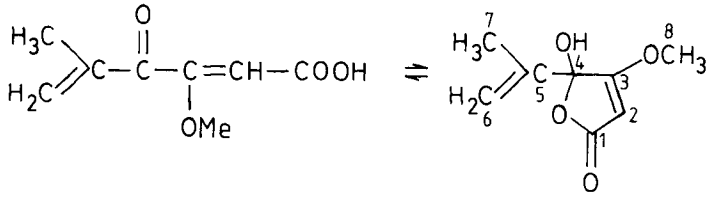
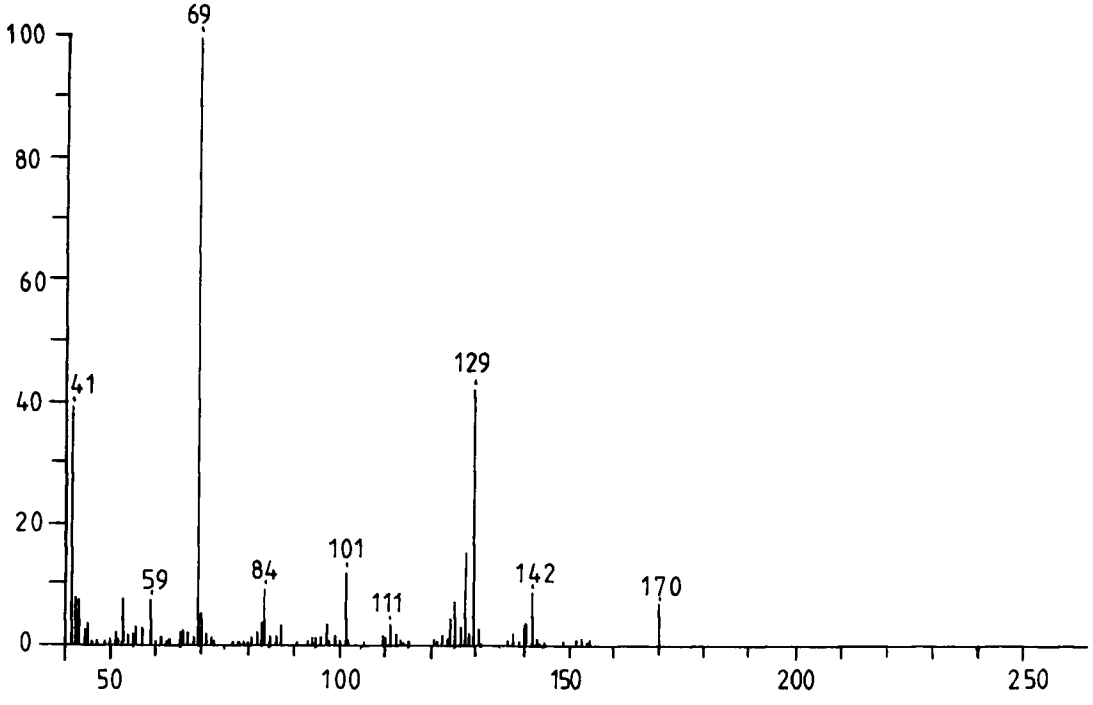
# Penicillic acid

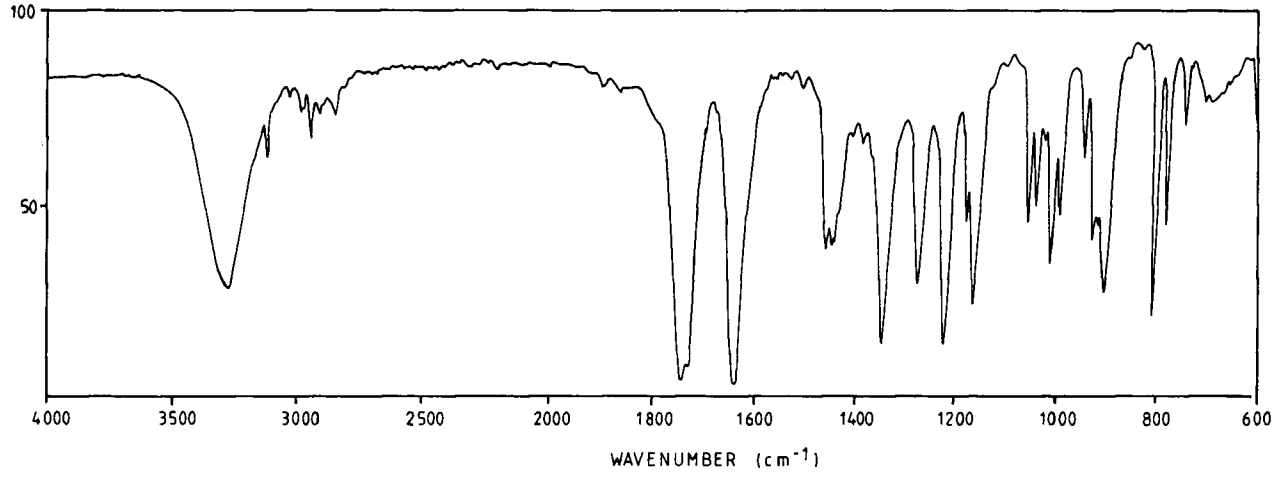
**$C_8H_{10}O_4$**



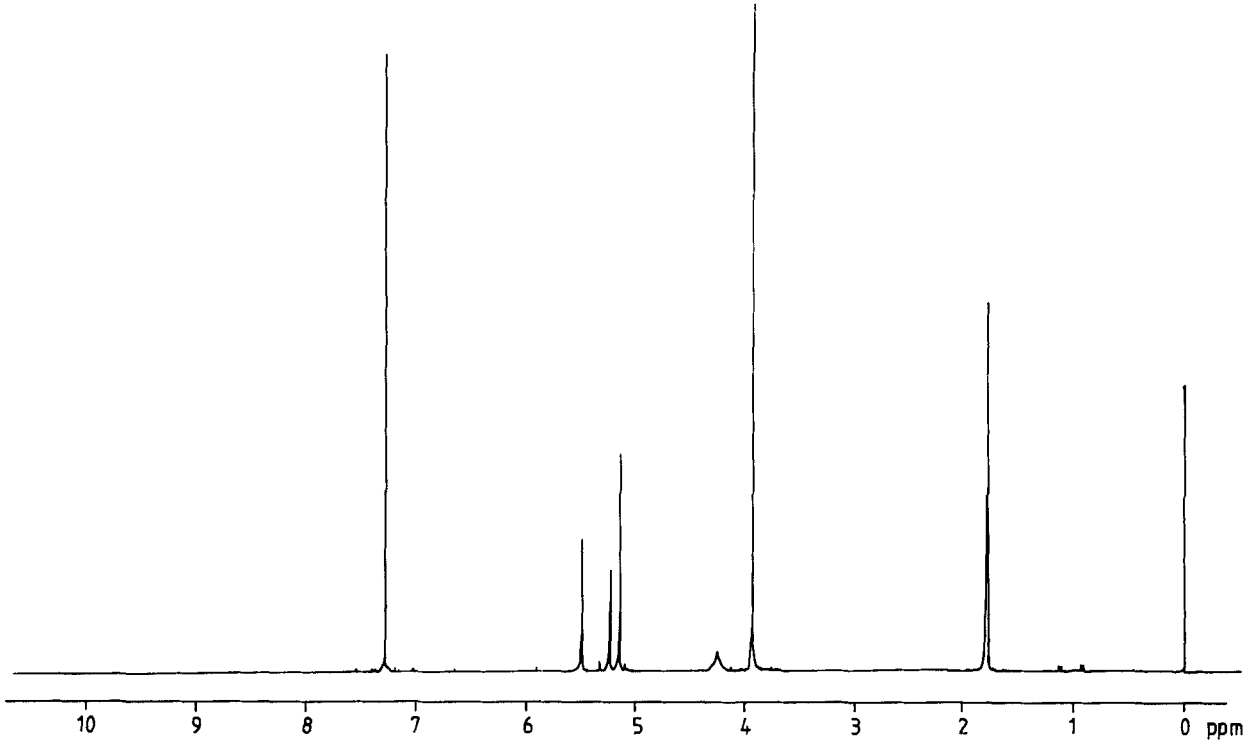
**Mass spectrum** [70 eV]



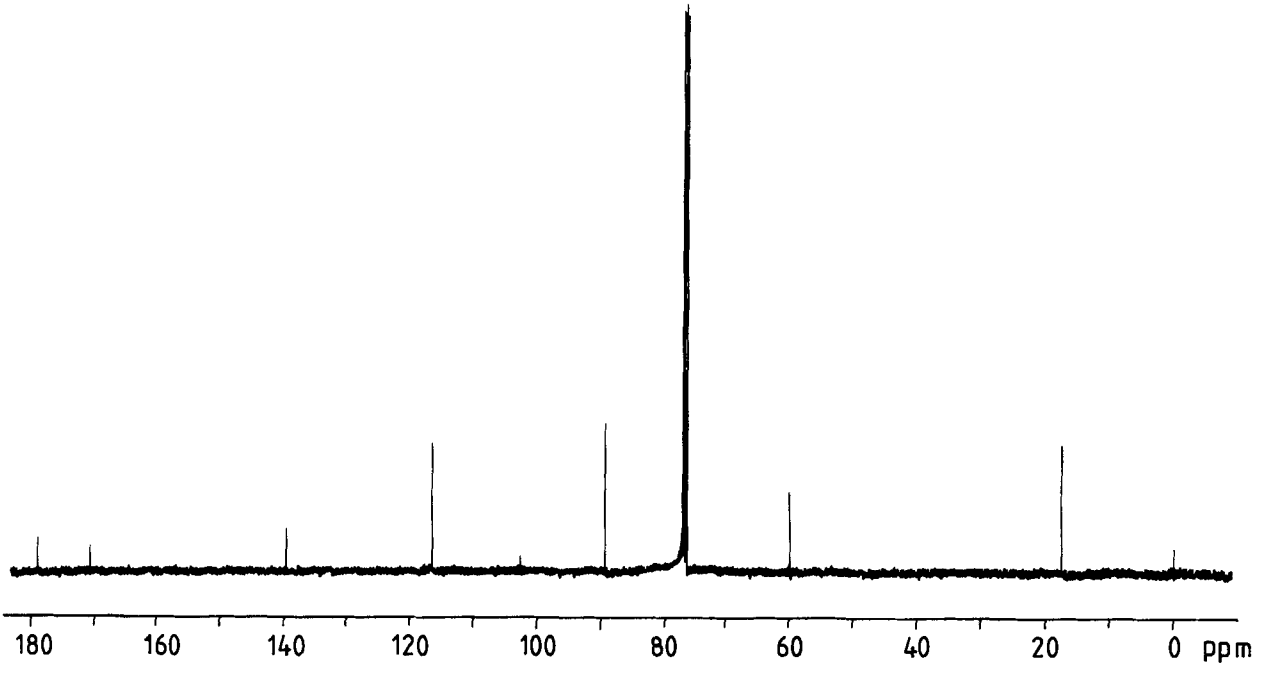

**IR spectrum** [KBr]



**$^1$H-NMR** [$CDCl_3$, 400 MHz]

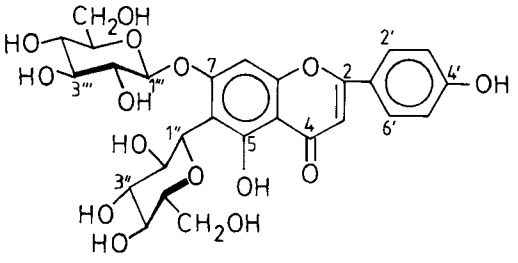



**$^{13}$C-NMR** [$CDCl_3$, 100 MHz]

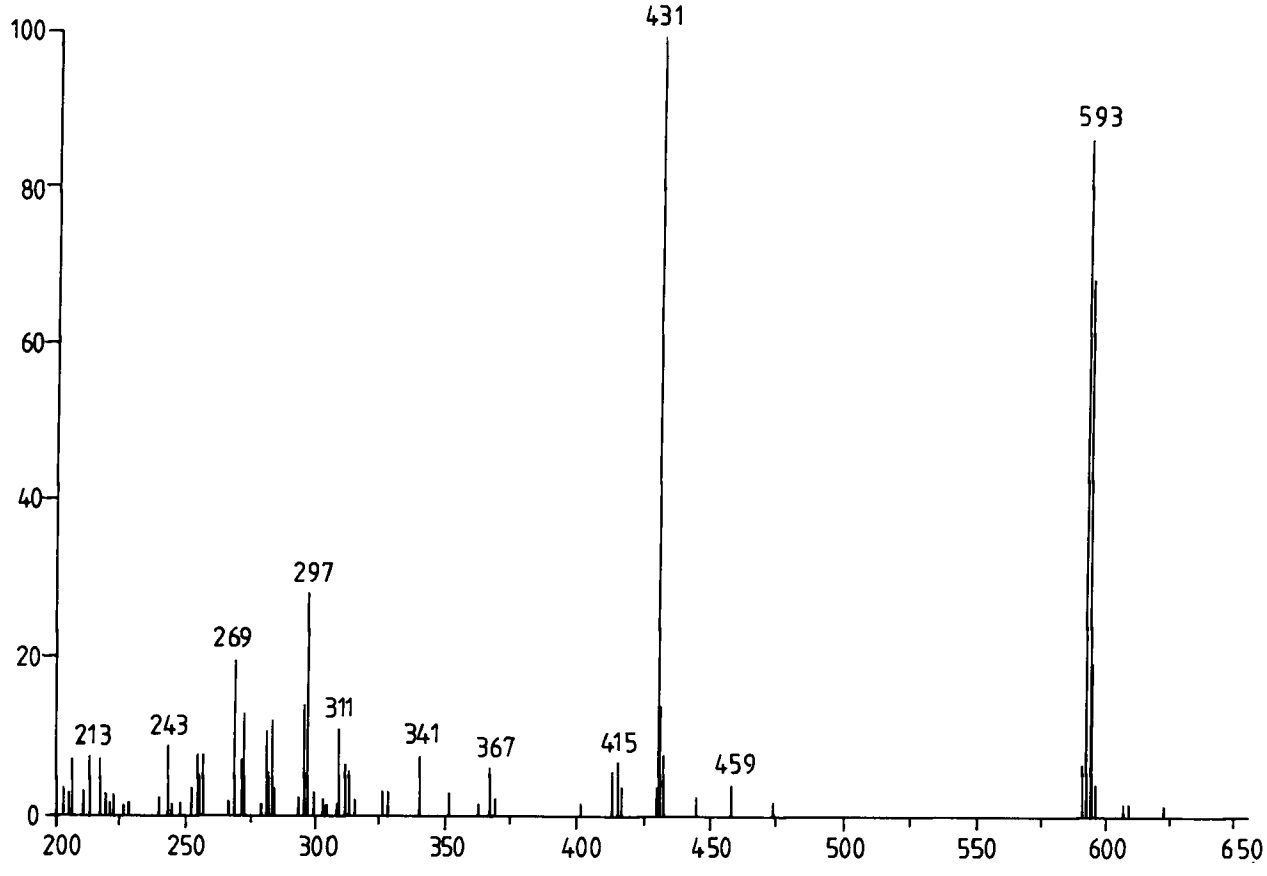


# Saponarin

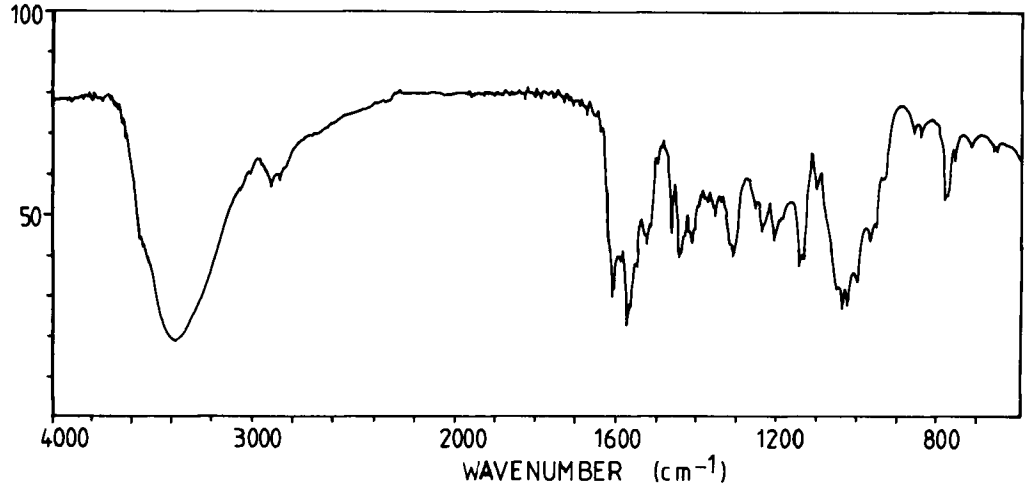
$C_{27}H_{30}O_{15}$



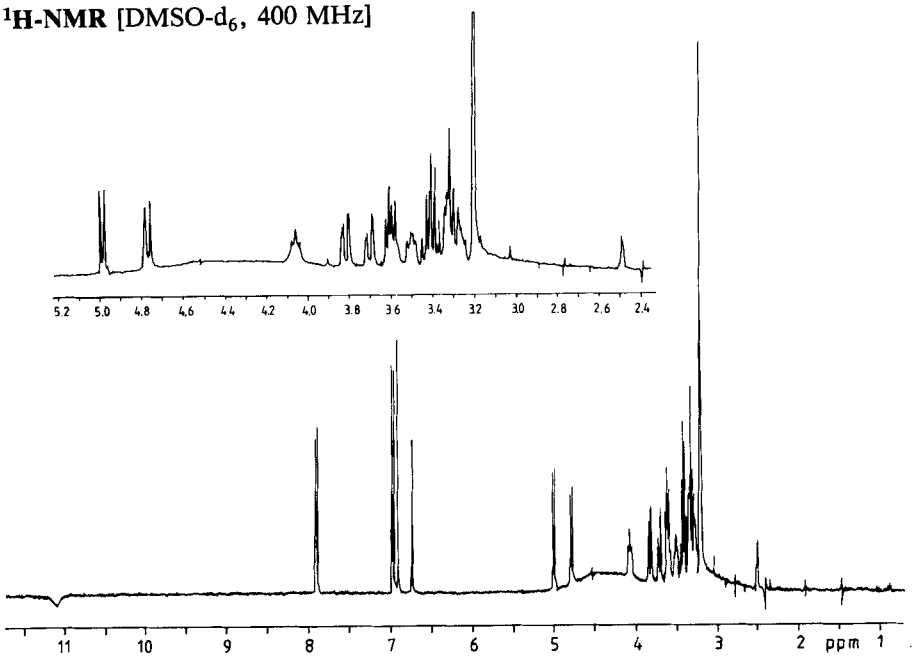
**Mass spectrum** [4.3 keV]



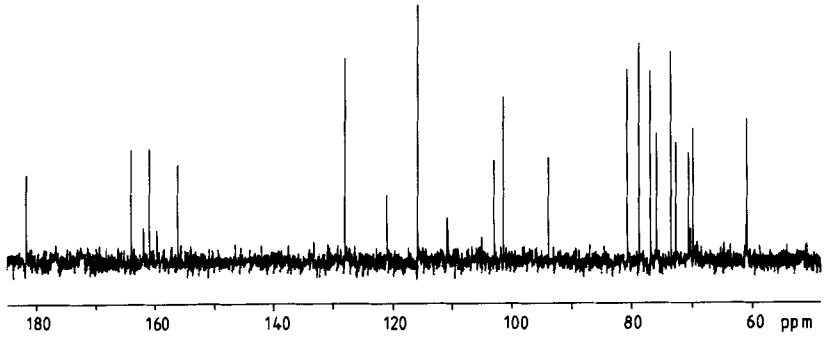

**IR spectrum** [KBr]



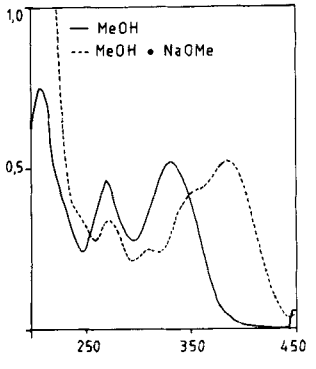
**$^1$H-NMR** [DMSO-$d_6$, 400 MHz]

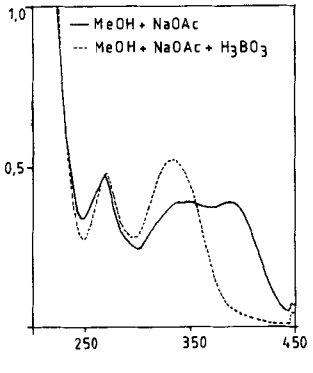


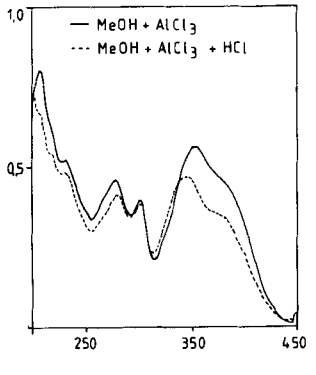

**$^{13}$C-NMR** [DMSO-$d_6$, 400 MHz]




**UV spectra**



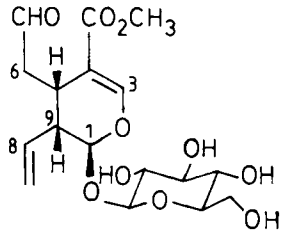



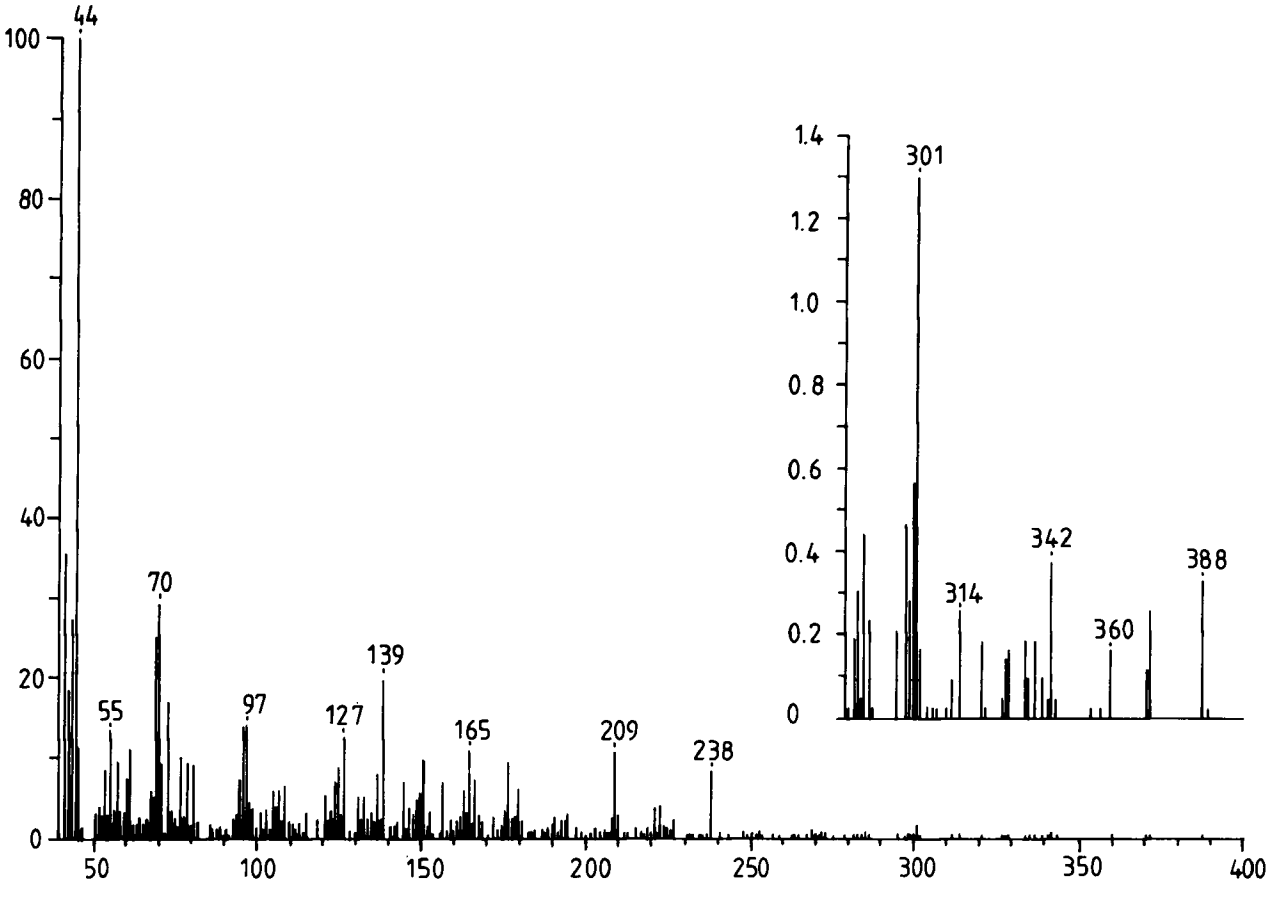

Saponarin

# Secologanin

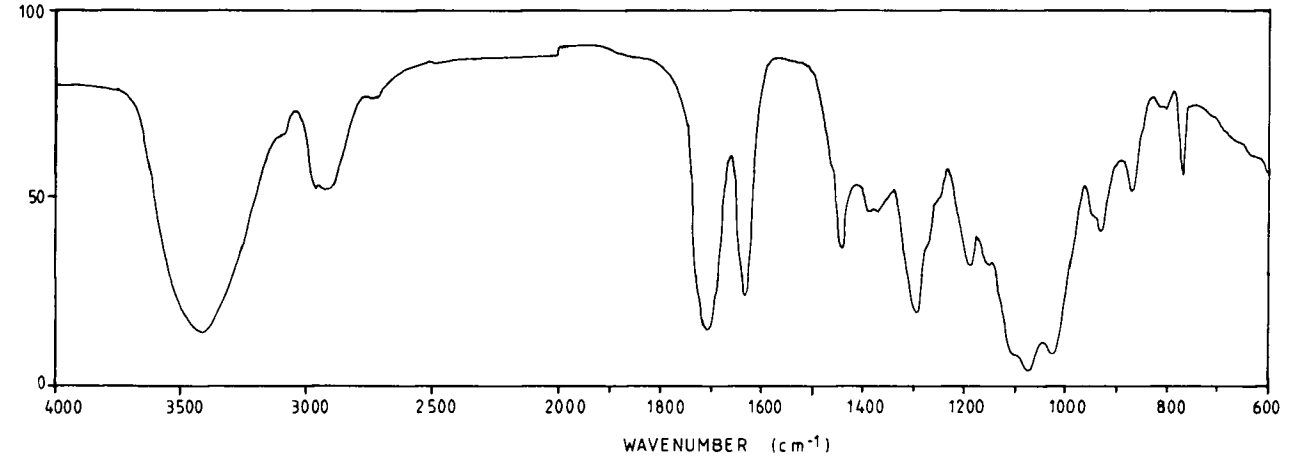
$C_{17}H_{24}O_{10}$



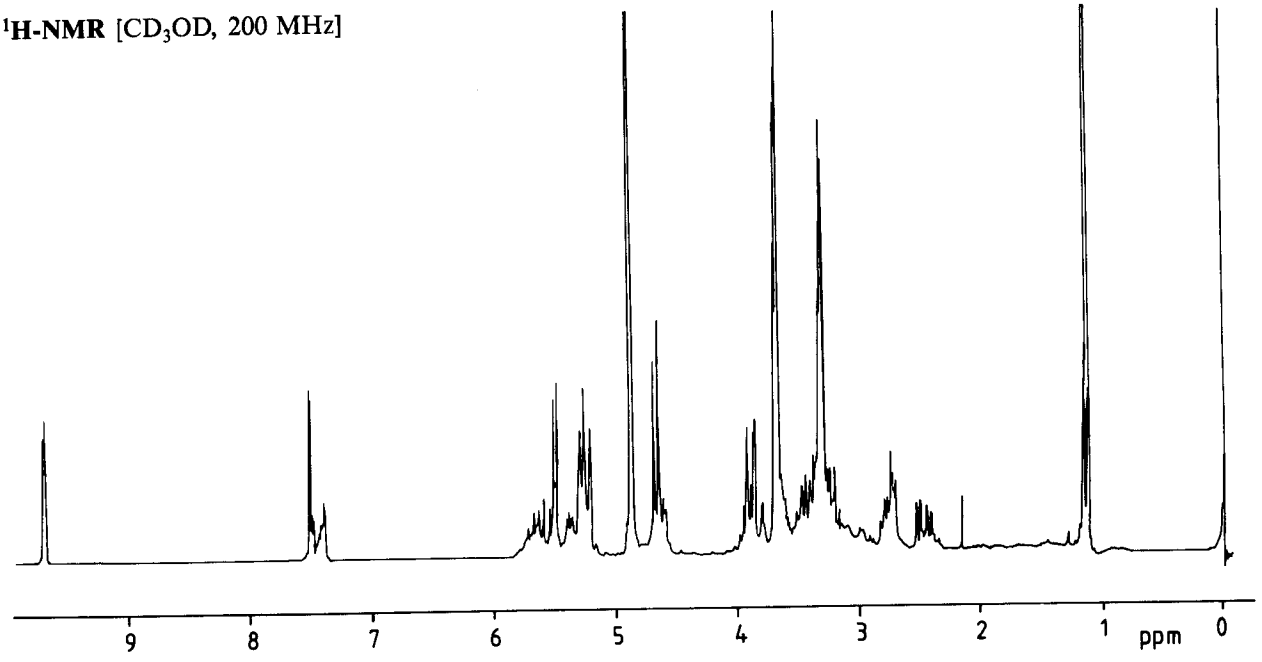

**Mass spectrum** [70 eV]



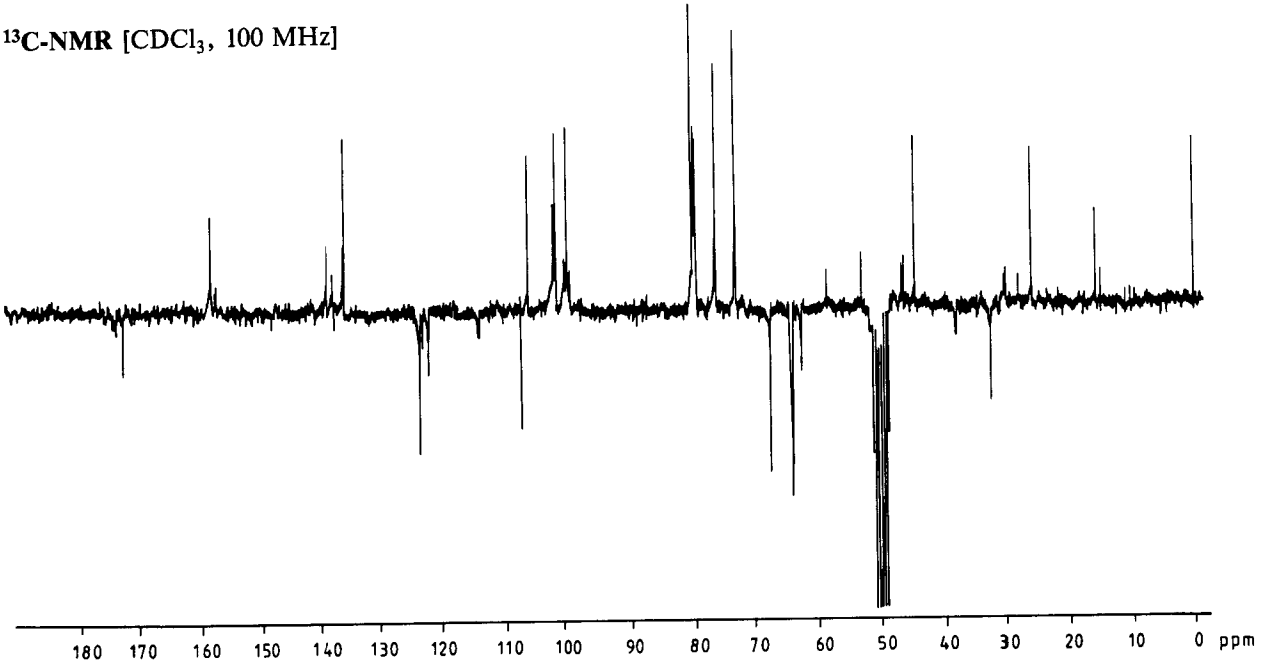

**IR spectrum** [KBr]



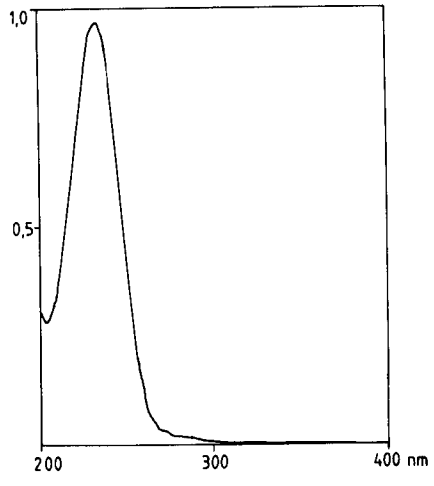
**$^1$H-NMR** [$CD_3OD$, 200 MHz]




**$^{13}$C-NMR** [$CDCl_3$, 100 MHz]

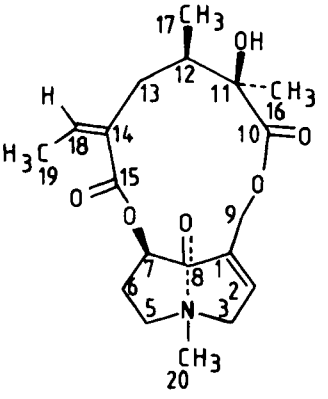



**UV spectrum** [methanol]



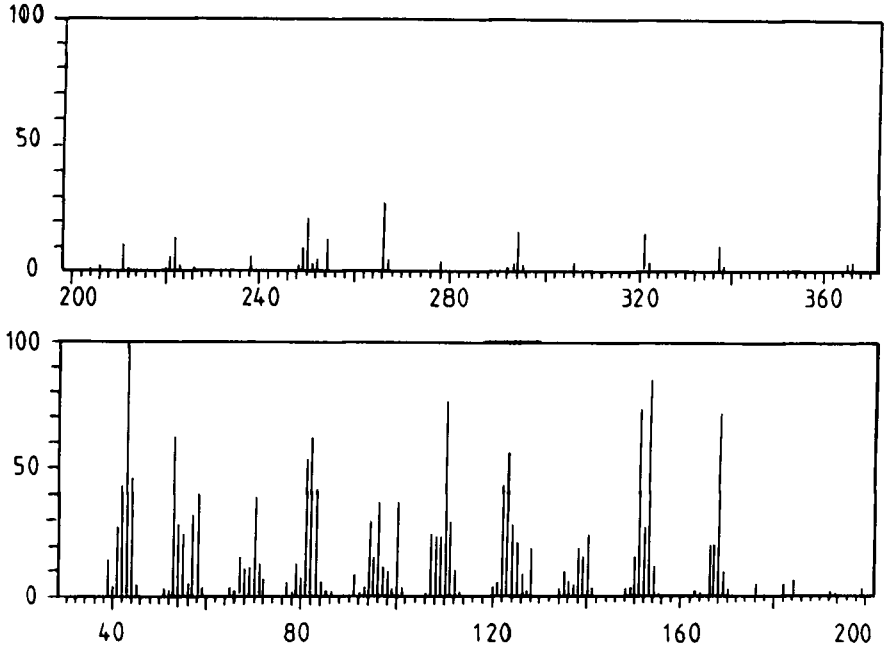

Secologanin

# Senkirkine

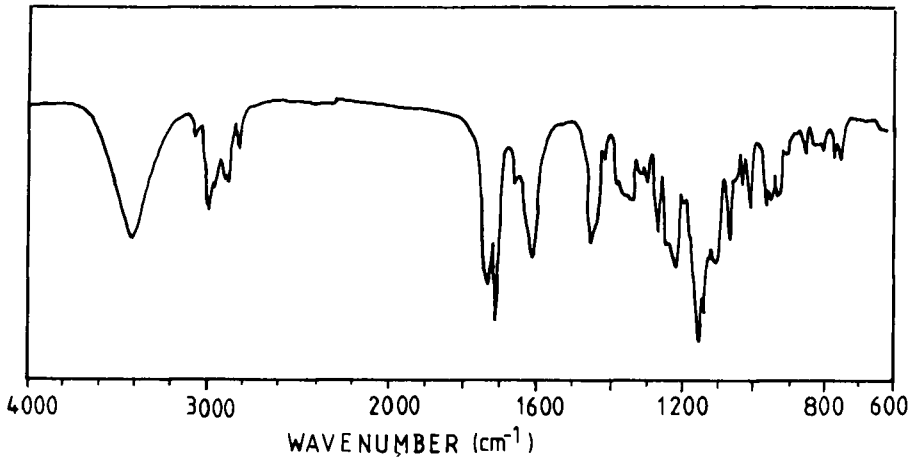



**$C_{19}H_{27}NO_6$**

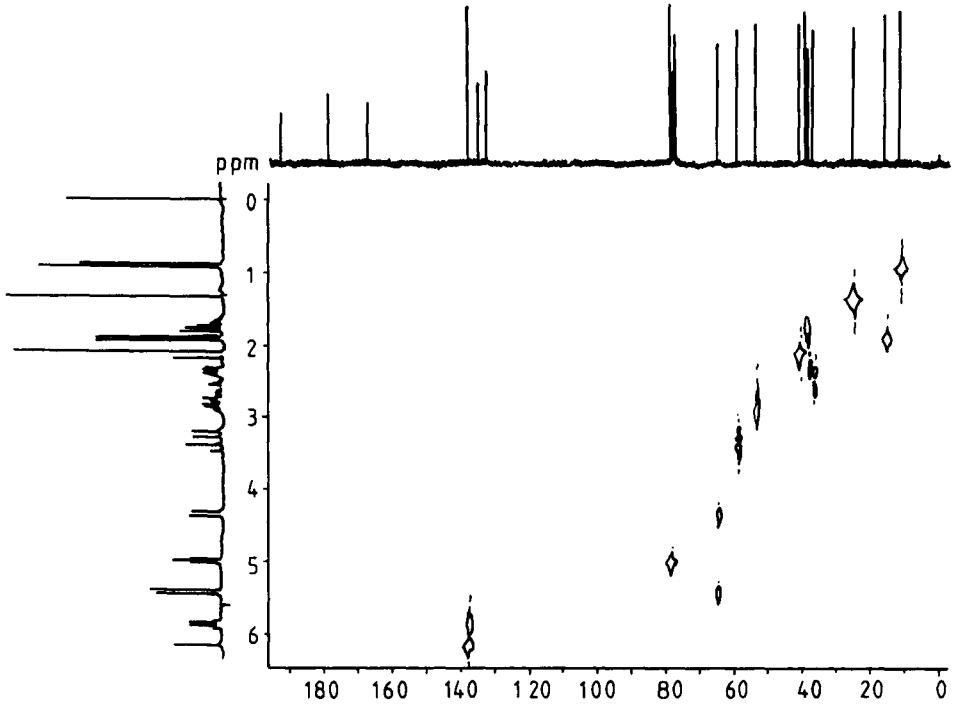
**Mass spectrum** [70 eV]



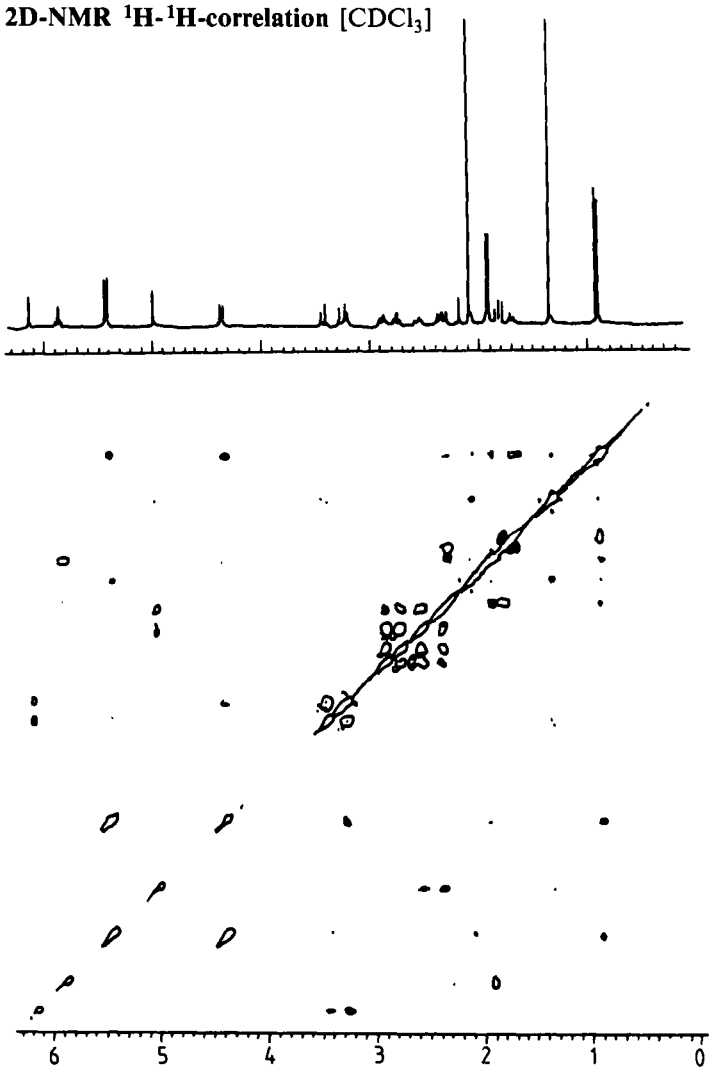

**IR spectrum** [KBr]



**2D-NMR $^{13}C$-$^{1}H$-correlation** [$CDCl_3$]

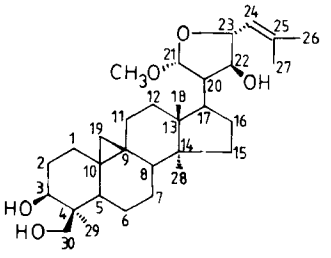



**2D-NMR $^{1}H$-$^{1}H$-correlation** [$CDCl_3$]

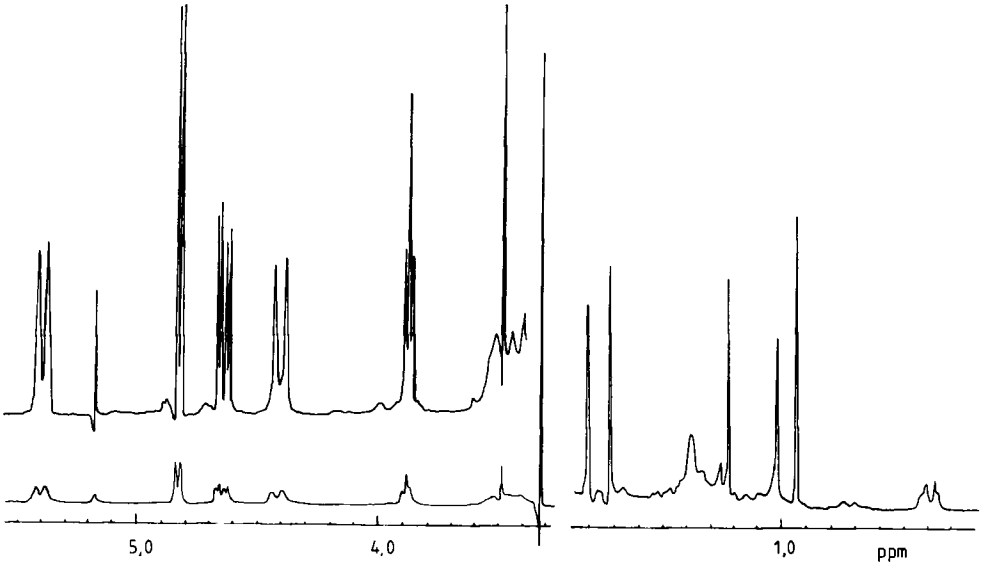


# Squarrogenin 1

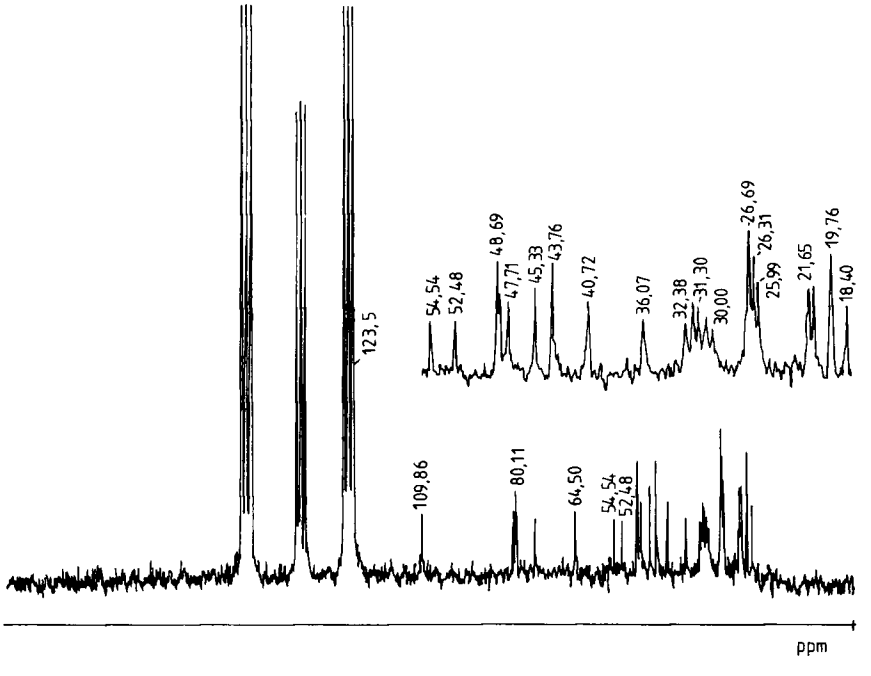
$C_{31}H_{50}O_5$



**$^1$H-NMR** [$CDCl_3$, 200 MHz]




**$^{13}$C-NMR** [$C_5D_5N$, 22.49 MHz]



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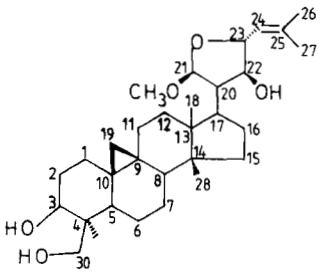
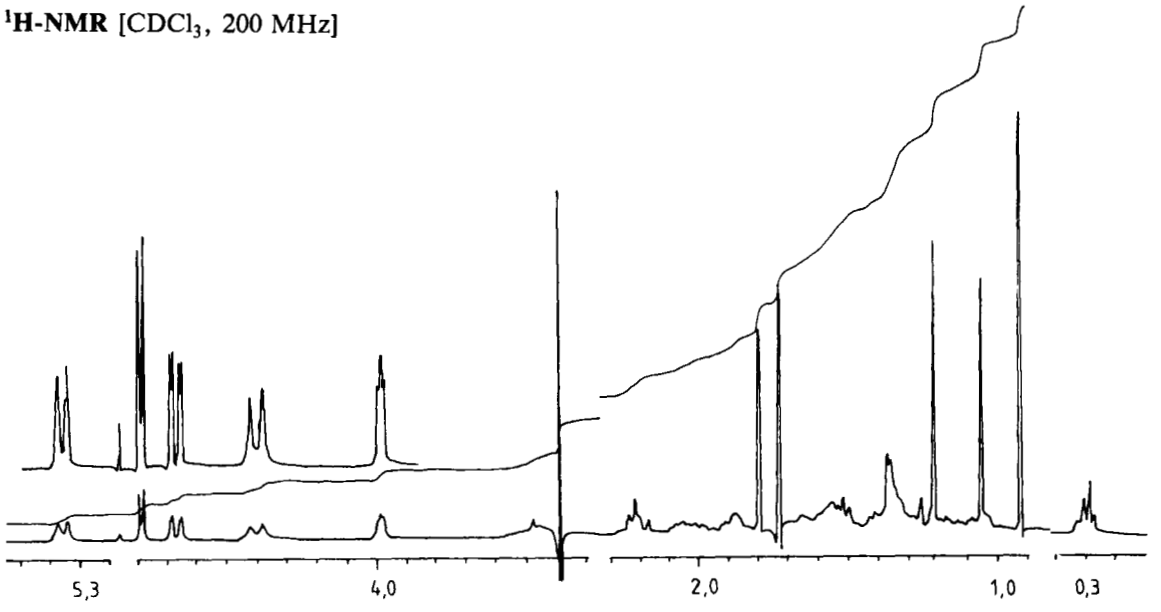
# Squarrogenin 2

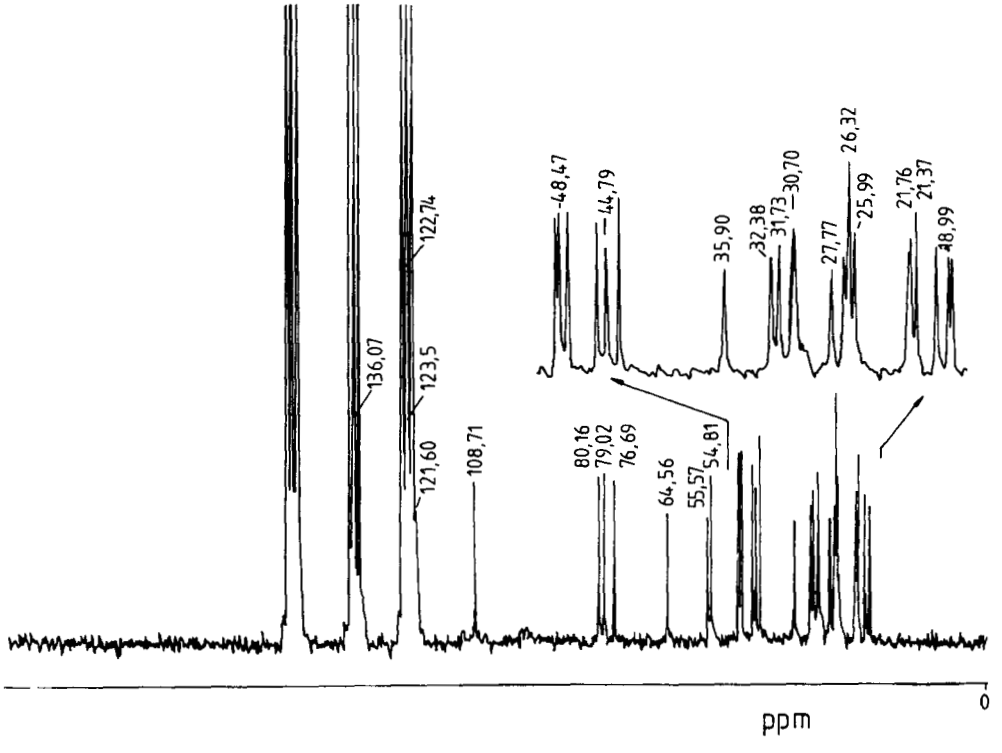
$C_{31}H_{50}O_5$




**$^1$H-NMR** [$CDCl_3$, 200 MHz]




**$^{13}$C-NMR** [$C_5D_5N$, 22.49 MHz]



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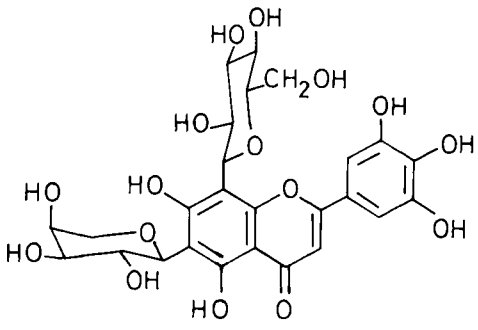
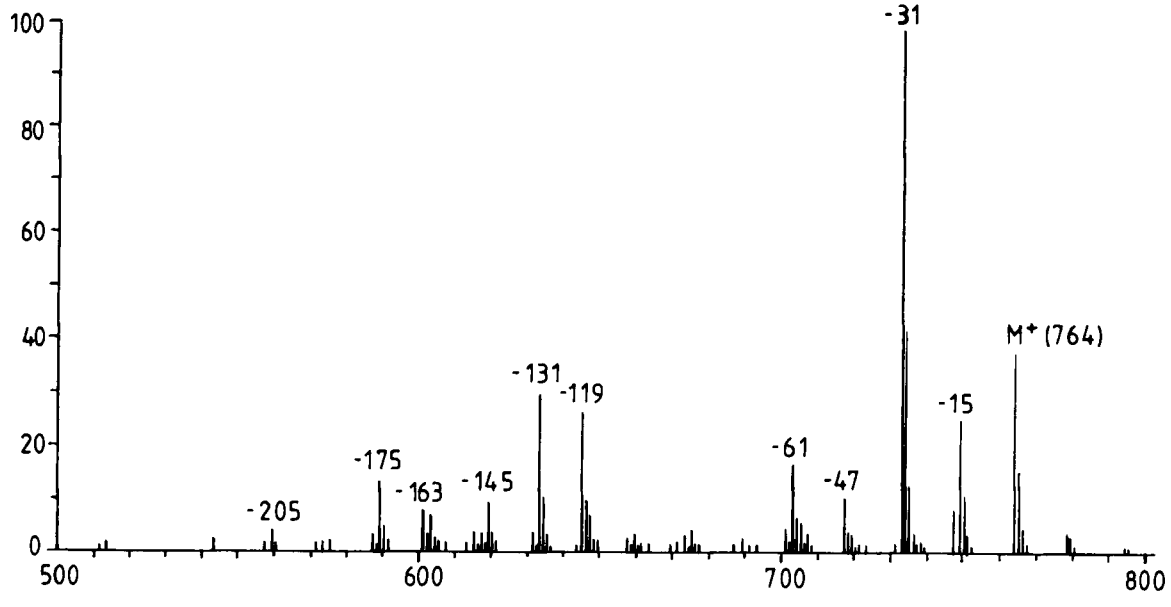
# Tricetin-6-C-arabinoside-8-C-glucoside

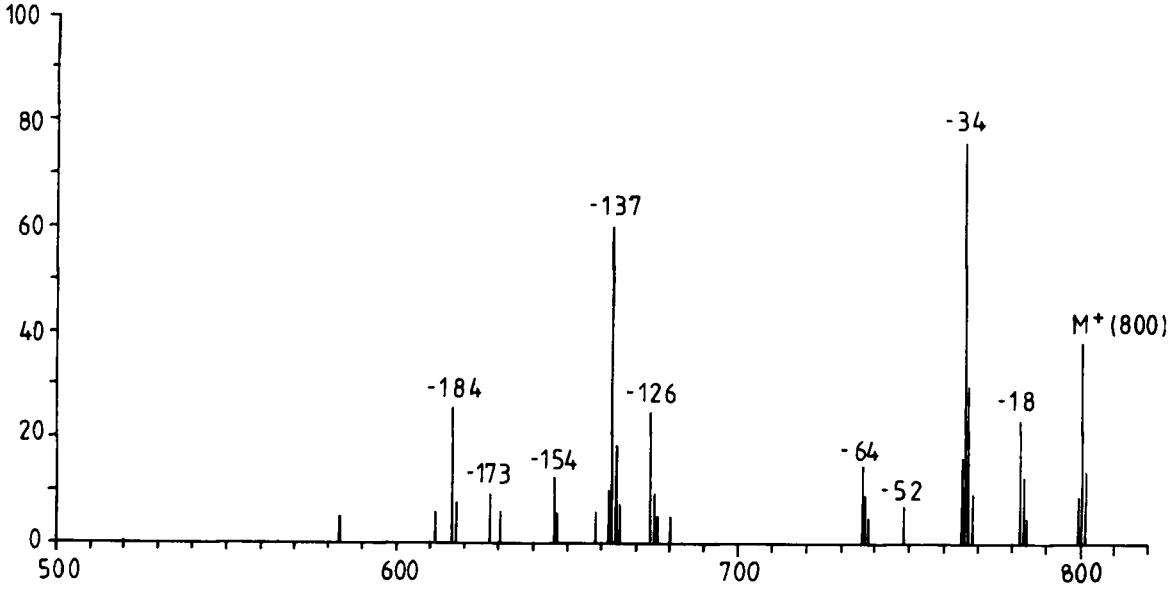
$C_{26}H_{28}O_{16}$



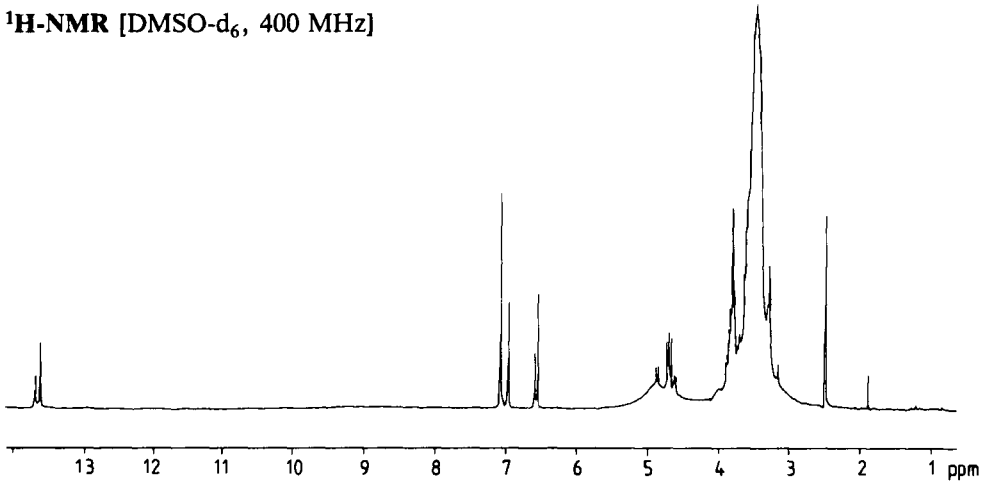

**Mass spectrum of PM-derivative** [90 eV]



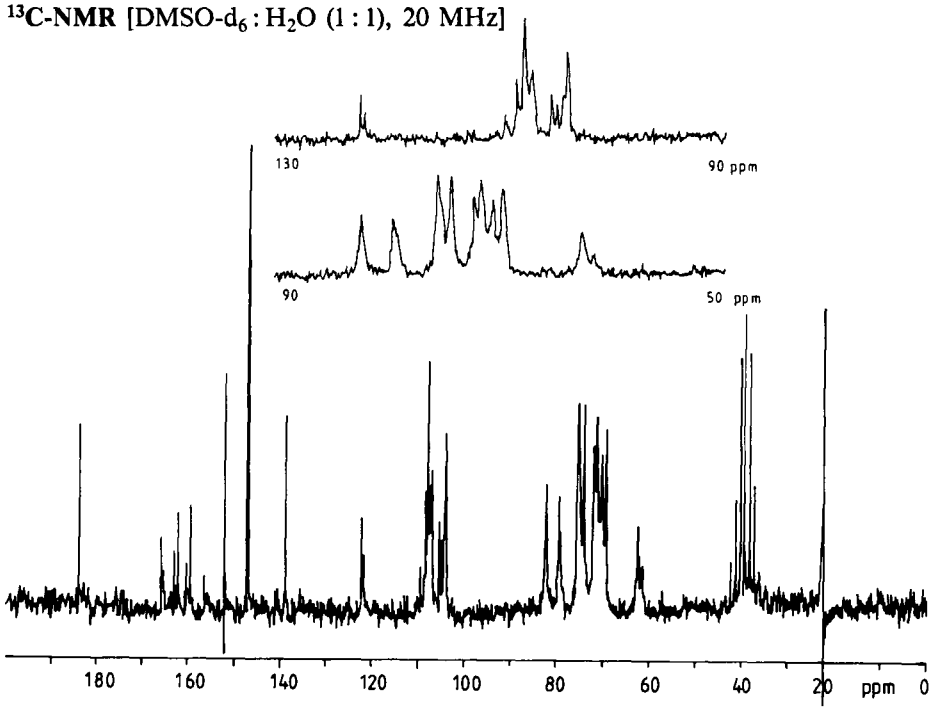

**Mass spectrum of PDM-derivative** [90 eV]



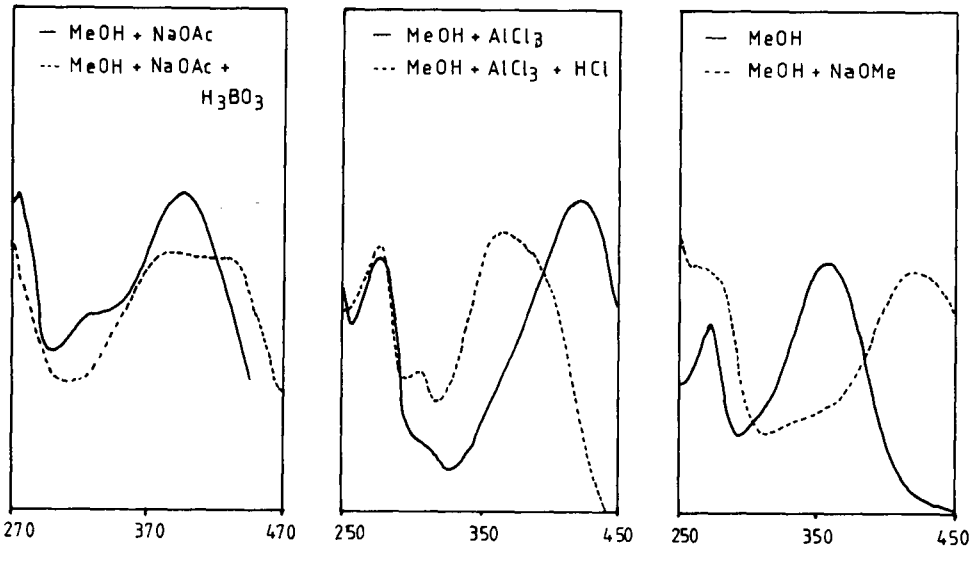
**$^1$H-NMR** [DMSO-$d_6$, 400 MHz]




**$^{13}$C-NMR** [DMSO-$d_6$ : $H_2O$ (1 : 1), 20 MHz]



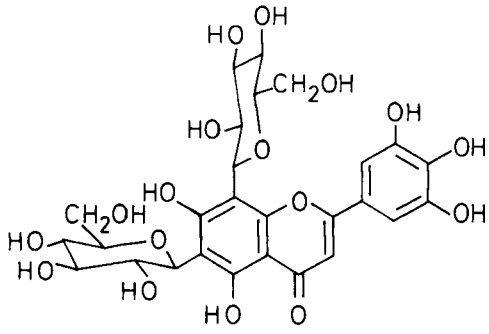

**UV spectra**



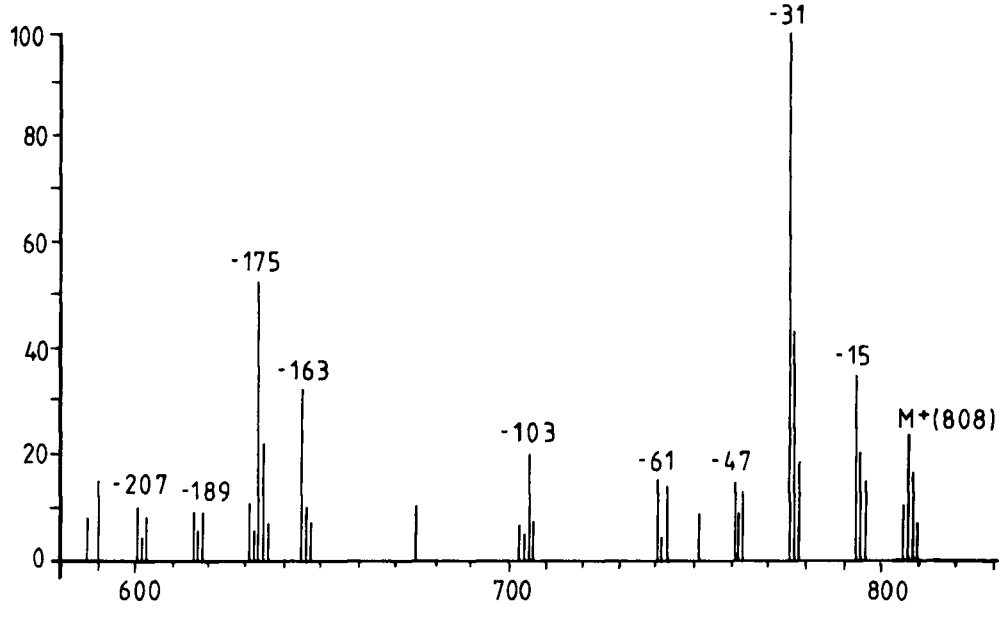

Tricetin-6-C-arabinoside-8-C-glucoside

# Tricetin-6,8-di-C-glucoside

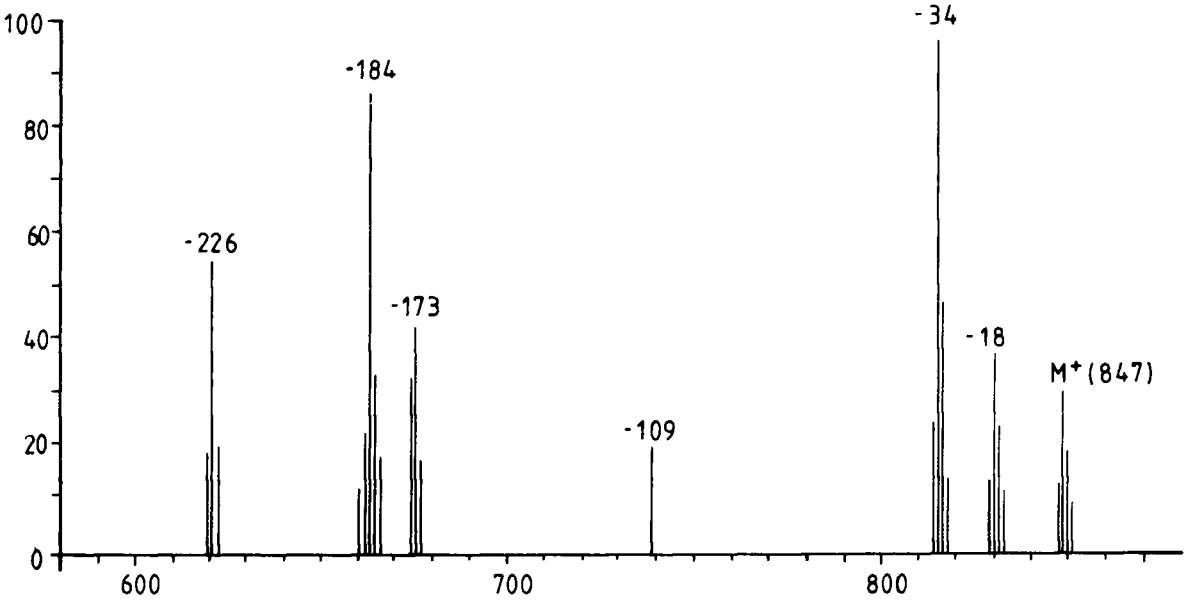
$C_{27}H_{30}O_{17}$



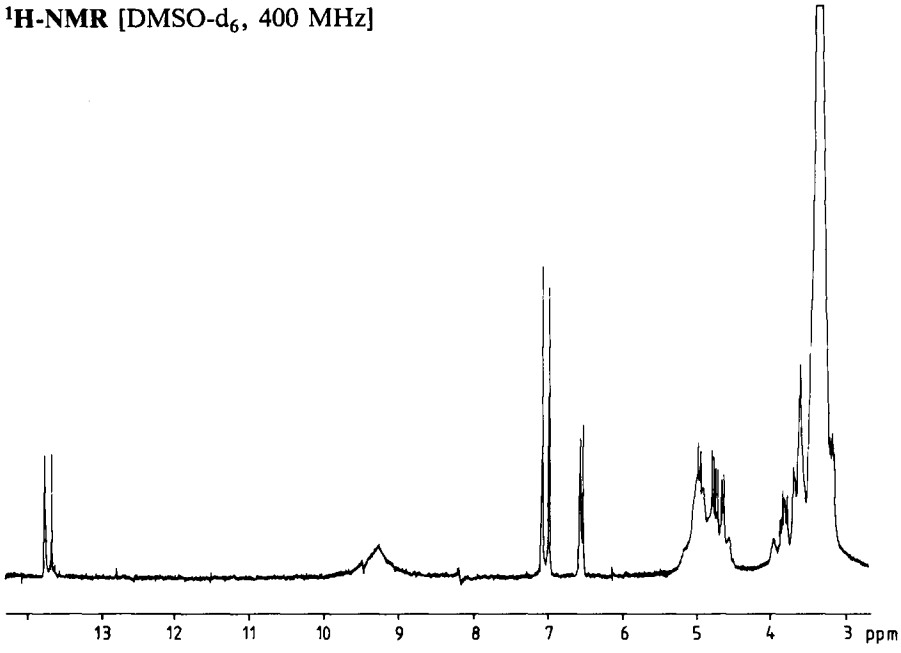

**Mass spectrum of PM-derivative** [90 eV]



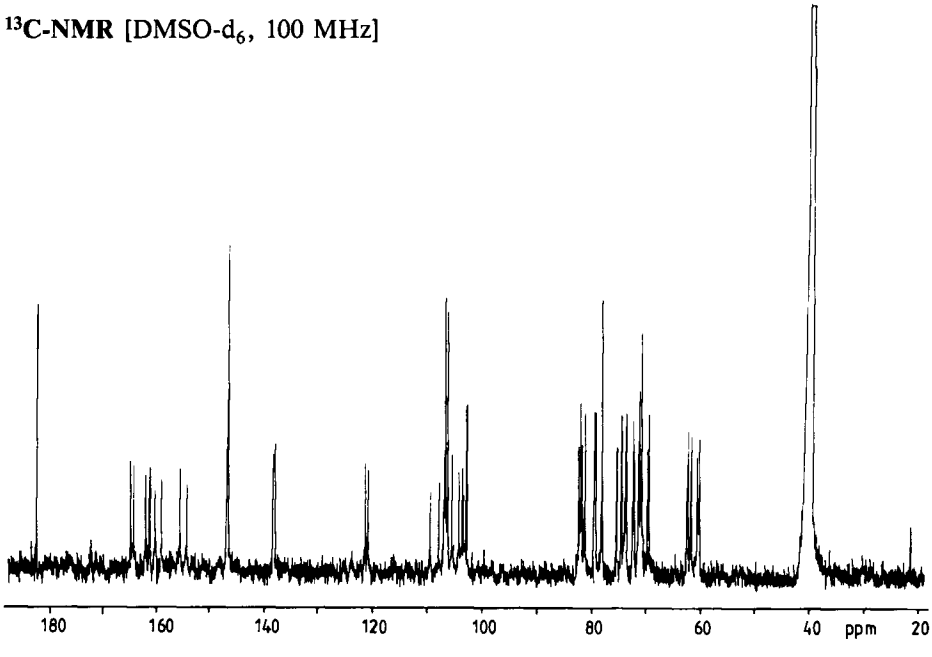

**Mass spectrum of PDM-derivative** [90 eV]



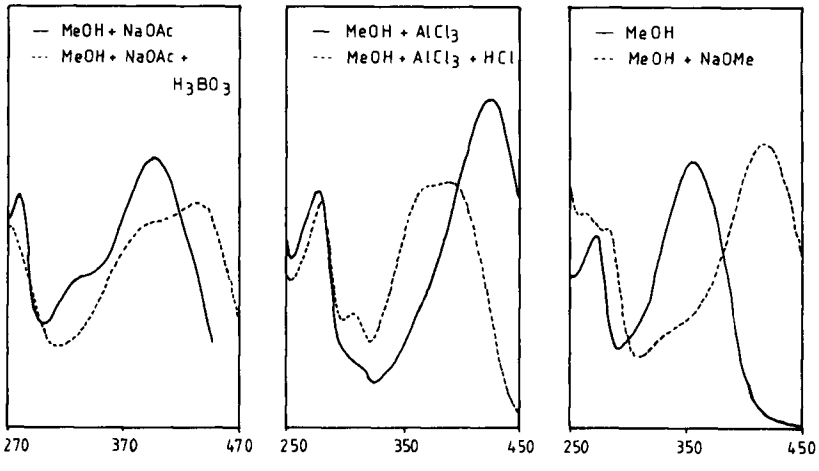
**$^1$H-NMR** [DMSO-$d_6$, 400 MHz]




**$^{13}$C-NMR** [DMSO-$d_6$, 100 MHz]



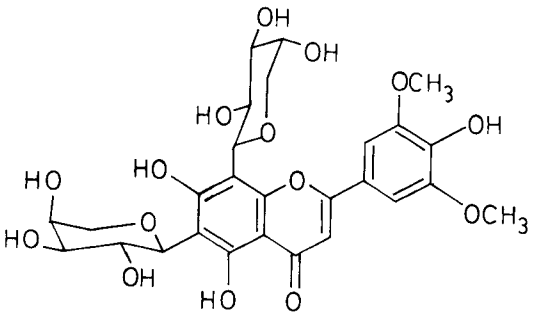

**UV spectrum**



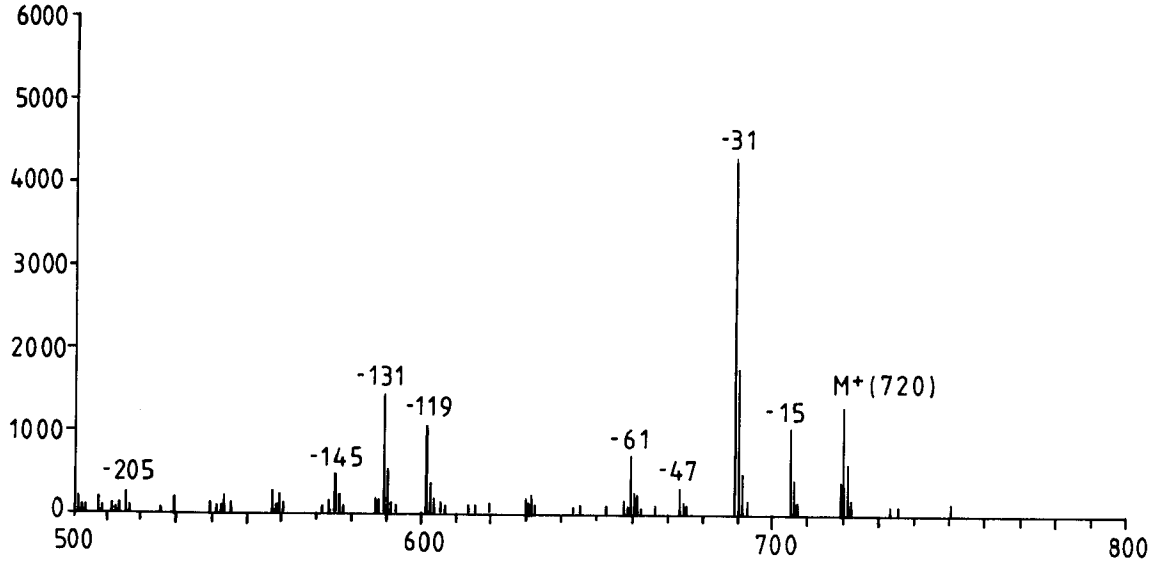

Tricetin-6,8-di-C-glucoside

# Tricin-6,8-di-C-arabinoside

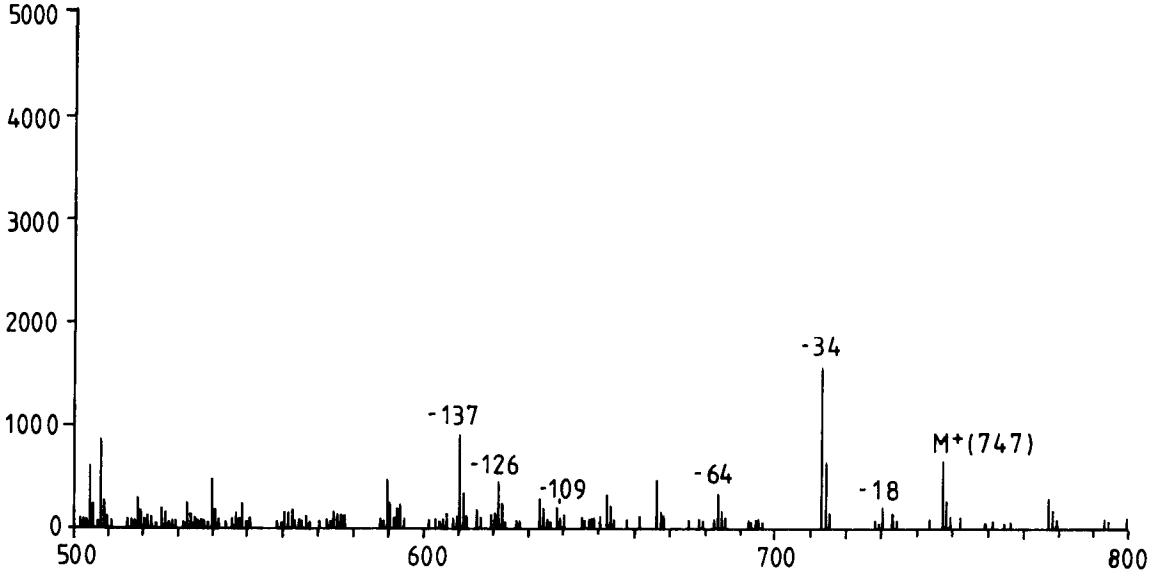
$C_{27}H_{30}O_{15}$



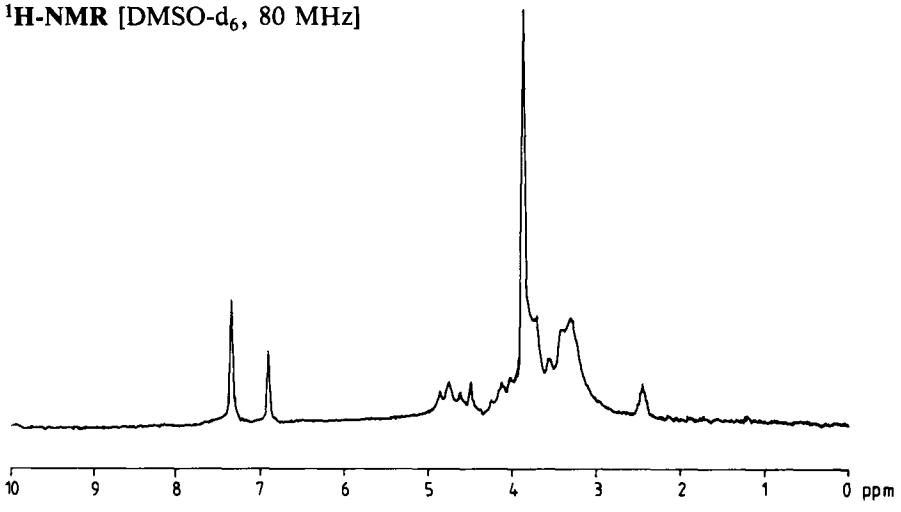

**Mass spectrum of PM-derivative** [90 eV]



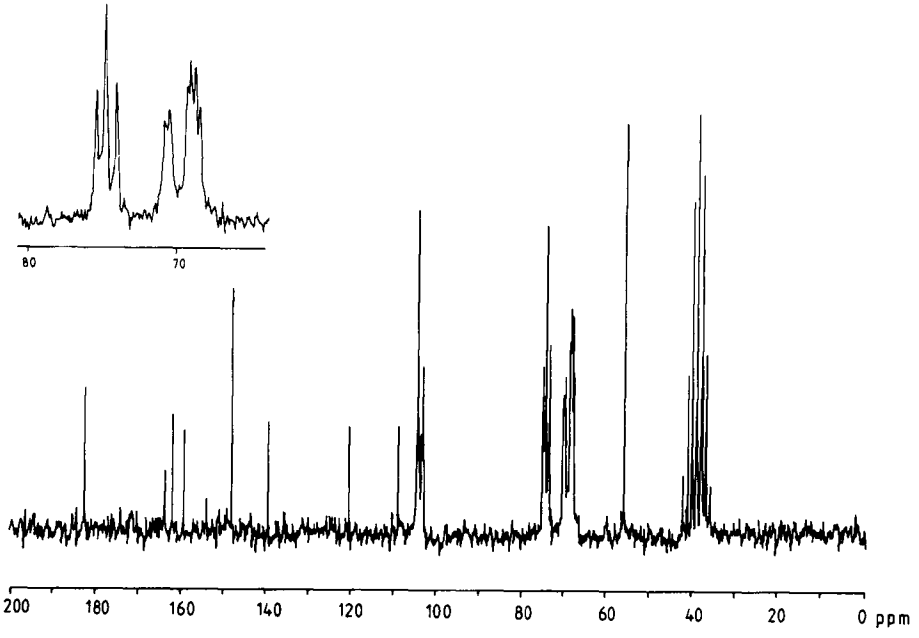

**Mass spectrum of PDM-derivative** [90 eV]



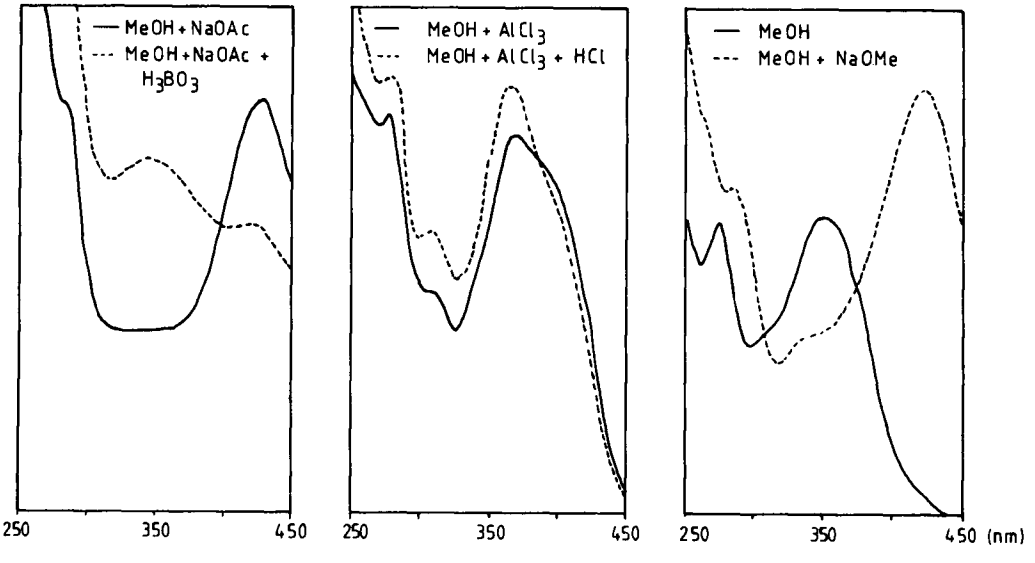
**$^1$H-NMR** [DMSO-$d_6$, 80 MHz]




**$^{13}$C-NMR** [DMSO-$d_6$, 20 MHz]



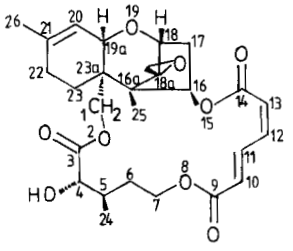

**UV spectra**



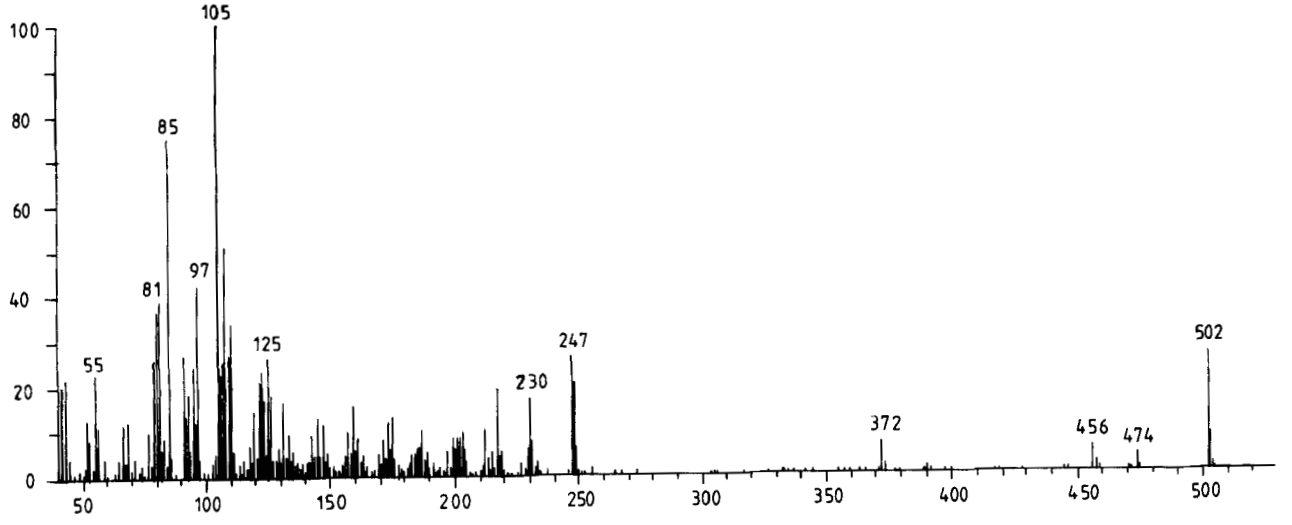

Tricin-6,8-di-C-arabinoside

# Verrucarin A

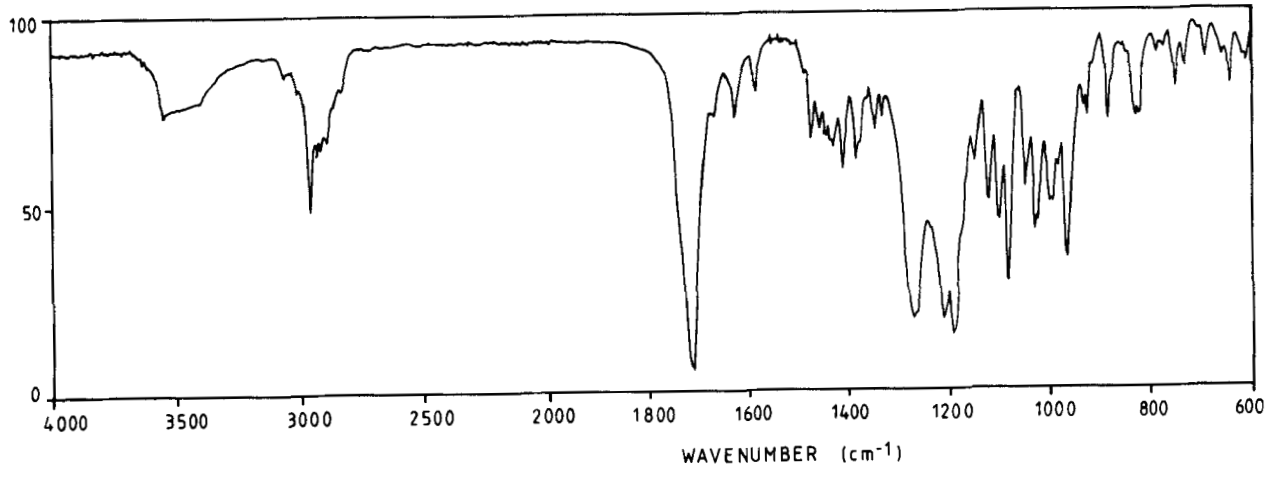
$C_{27}H_{34}O_9$



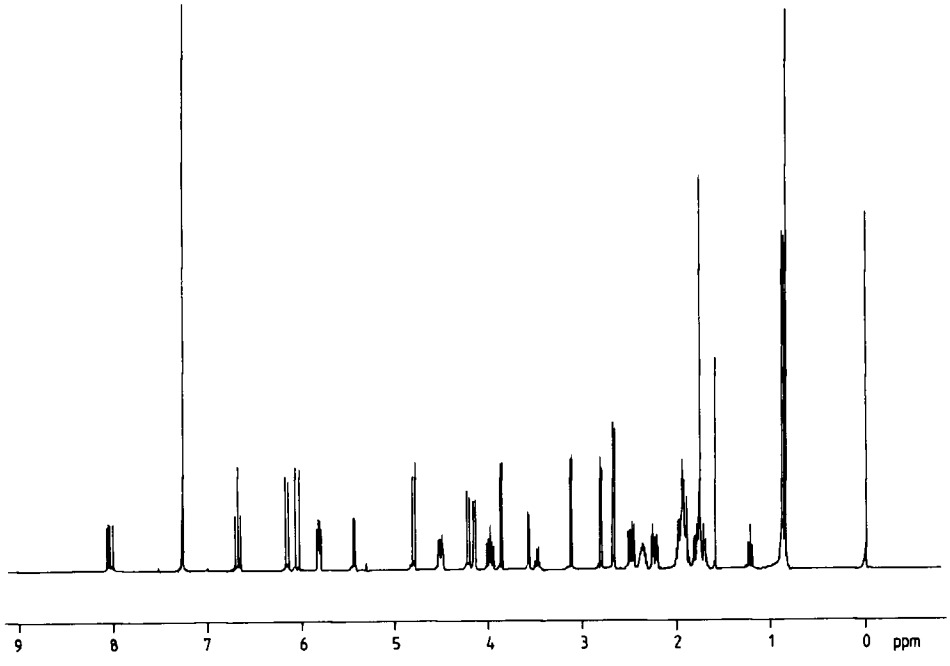
**Mass spectrum** [70 eV]



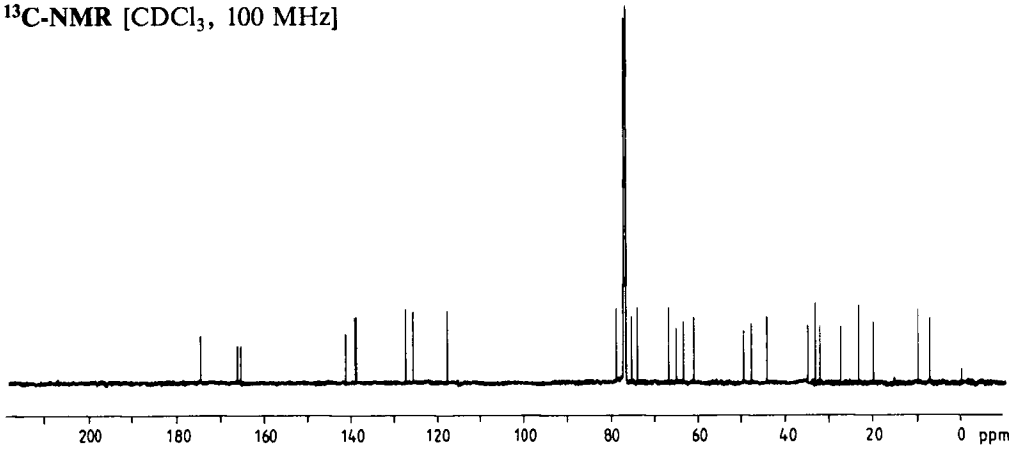

**IR spectrum** [KBr]



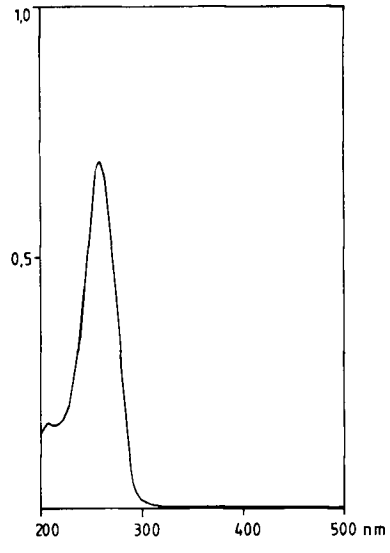
**$^1$H-NMR** [$CDCl_3$, 400 MHz]




**$^{13}$C-NMR** [$CDCl_3$, 100 MHz]



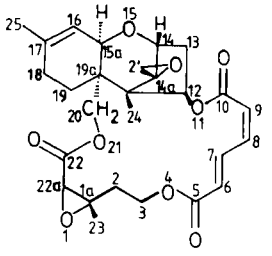

**UV spectrum** [methanol]



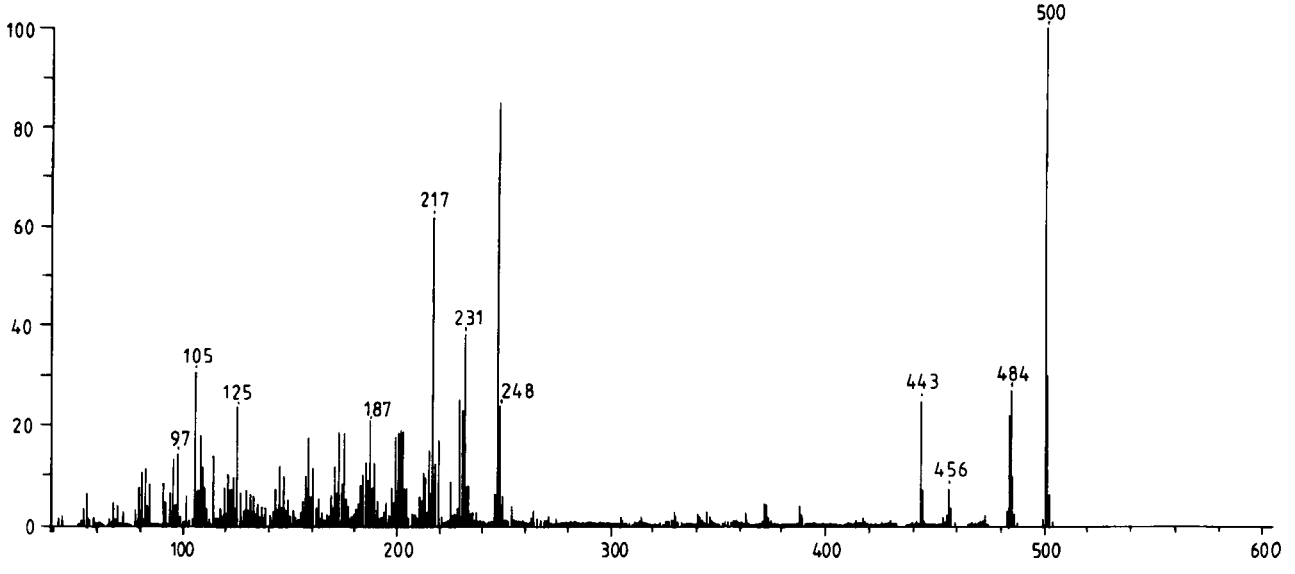

Verrucarin A

# Verrucarin B

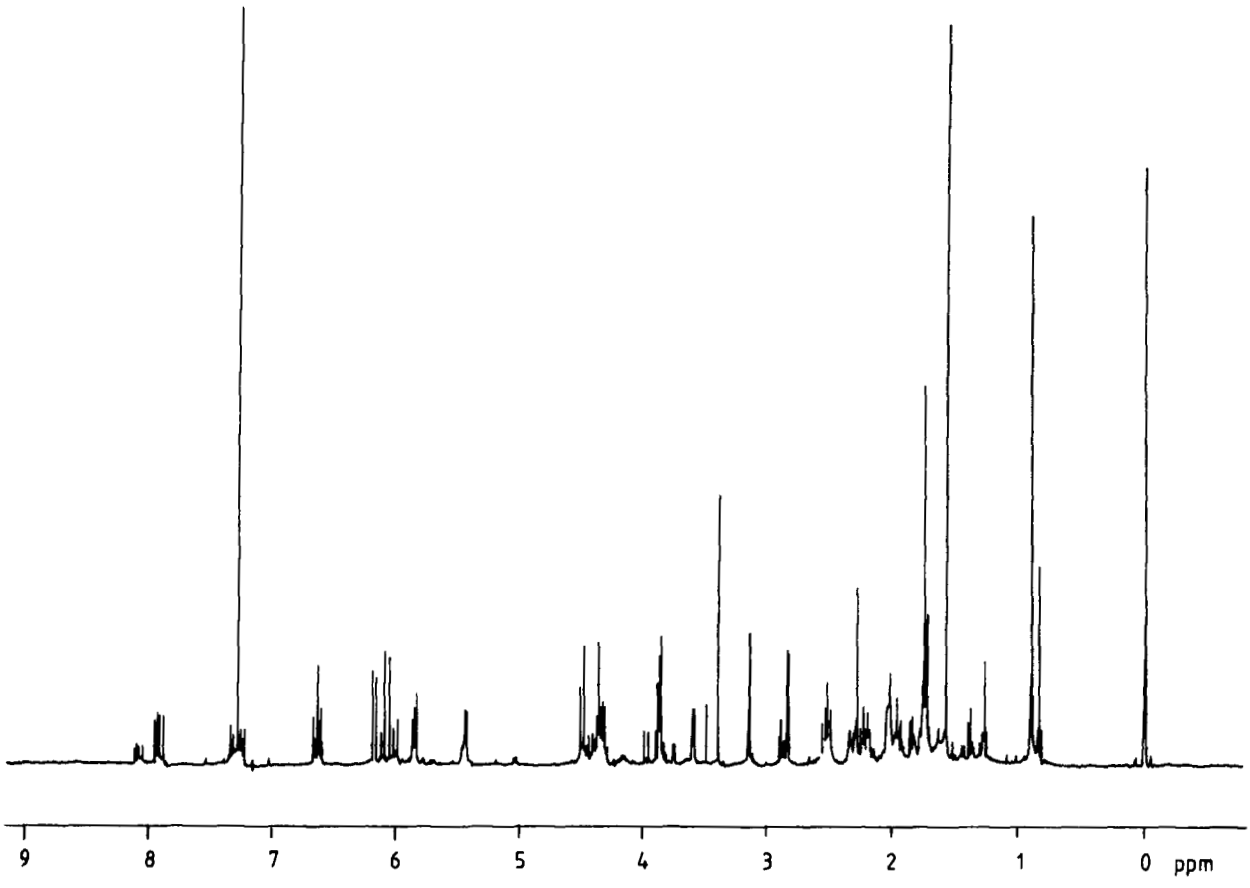
$C_{27}H_{32}O_9$



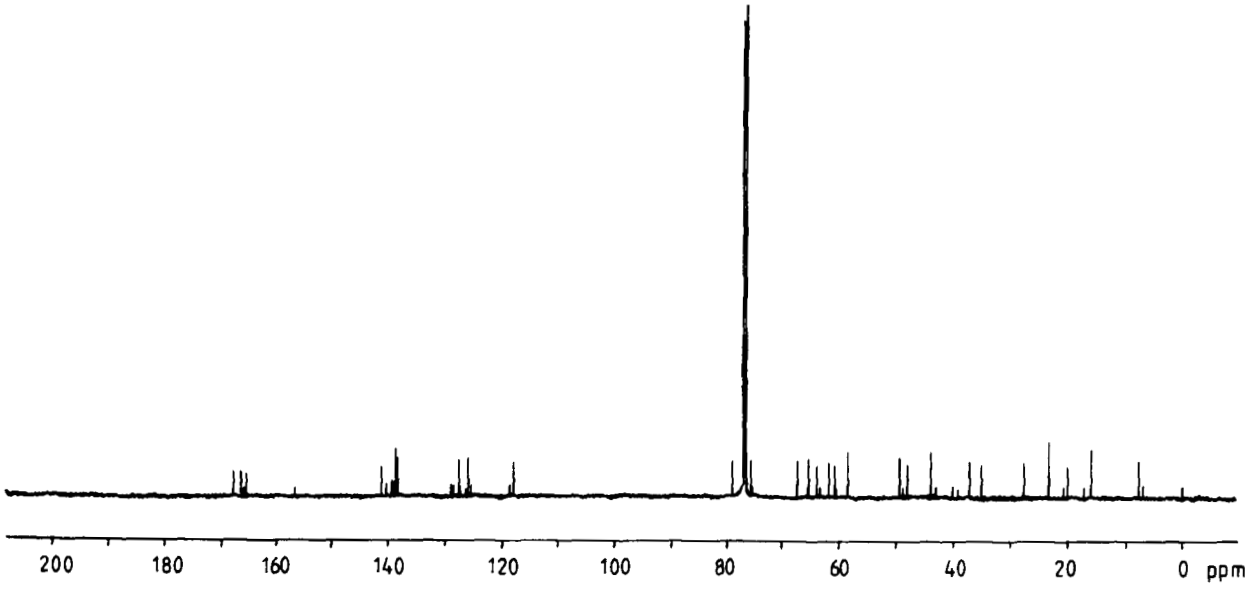
**Mass spectrum** [70 eV]



**$^1$H-NMR** [$CDCl_3$, 400 MHz]

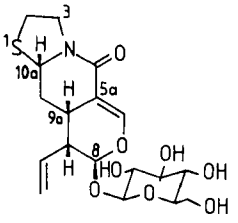



**$^{13}$C-NMR** [$CDCl_3$, 100 MHz]

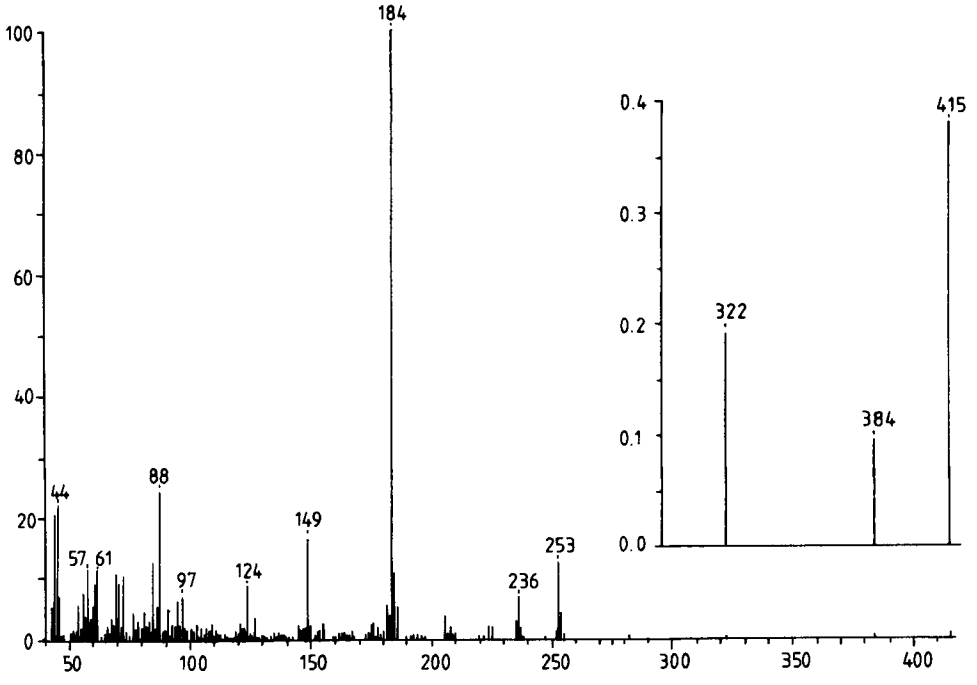


# Xylostosidine

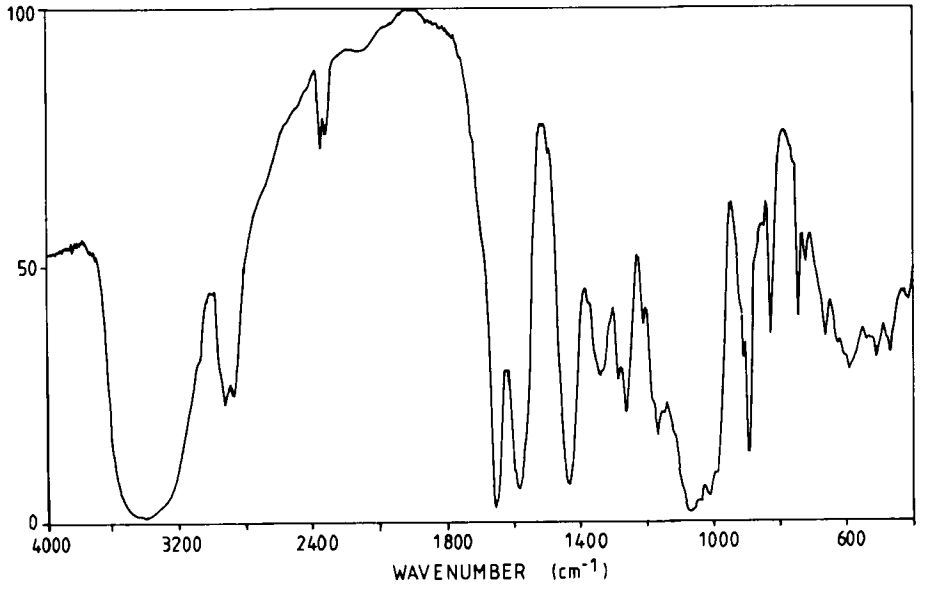
$C_{18}H_{25}NO_8S$



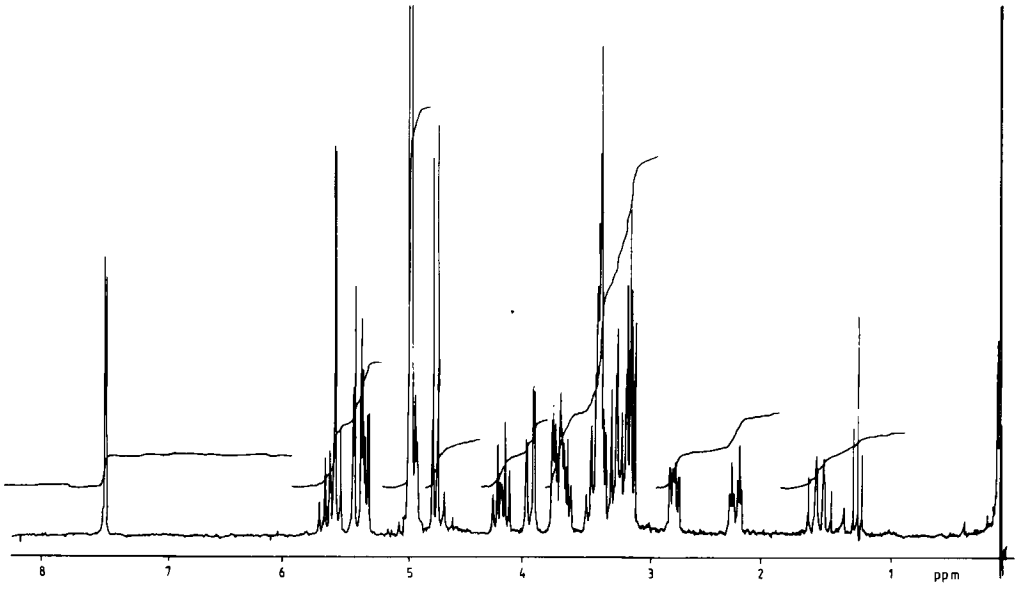
**Mass spectrum** [70 eV]



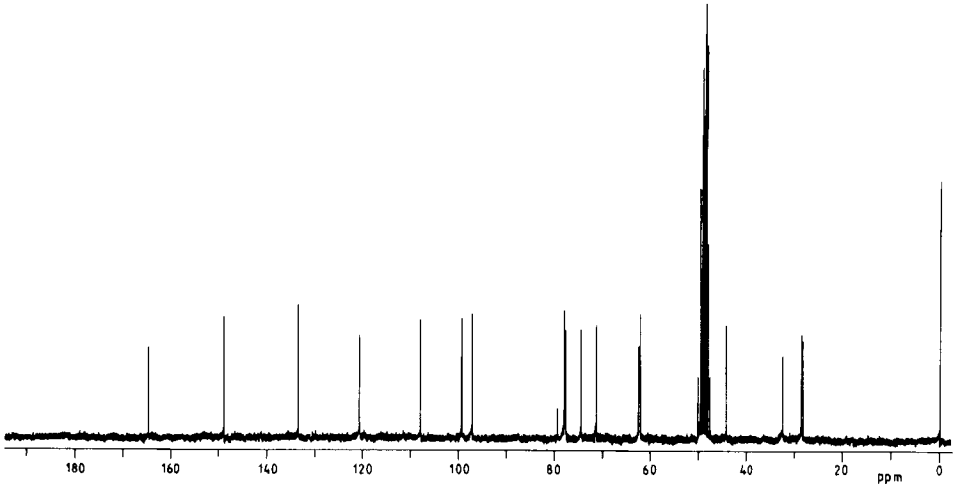

**IR spectrum** [KBr]



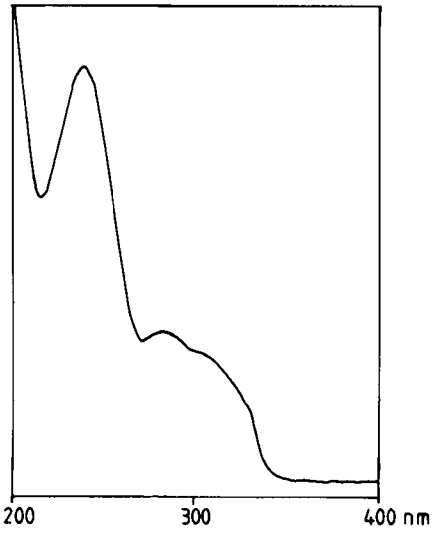
**$^1$H-NMR** [$CD_3OD$, 200 MHz]




**$^{13}$C-NMR** [$CD_3OD$, 50.3 MHz]



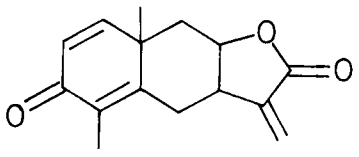

**UV spectrum** [methanol]



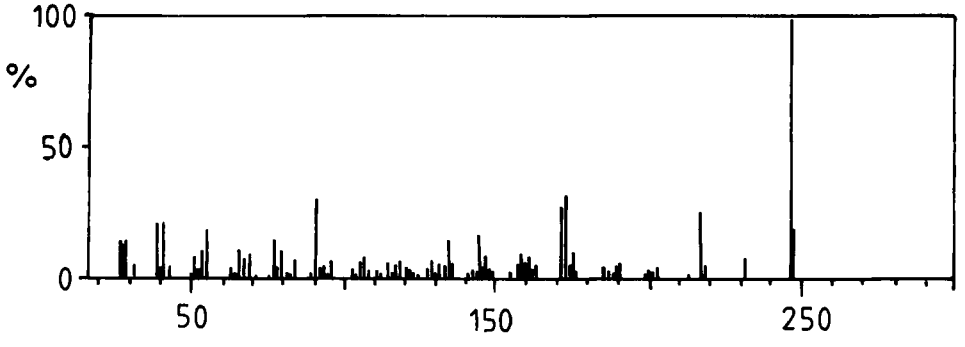

Xylostosidine

# Yomogin

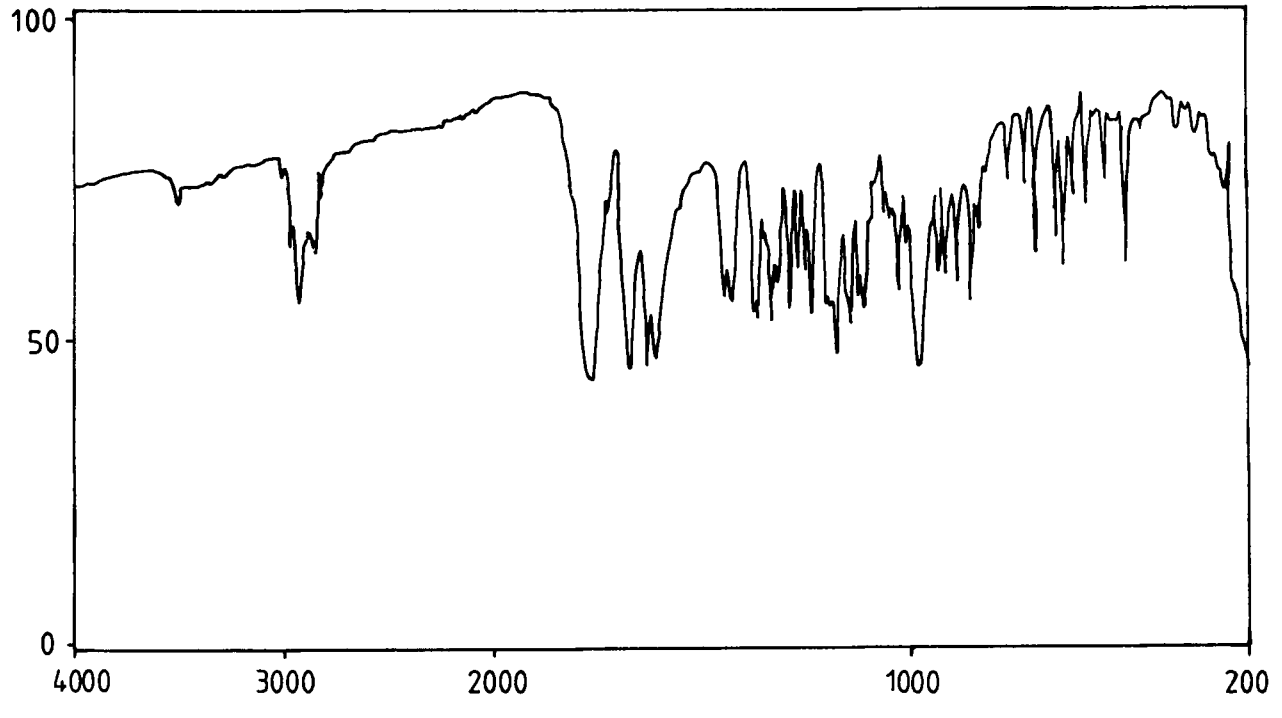
$C_{15}H_{16}O_3$



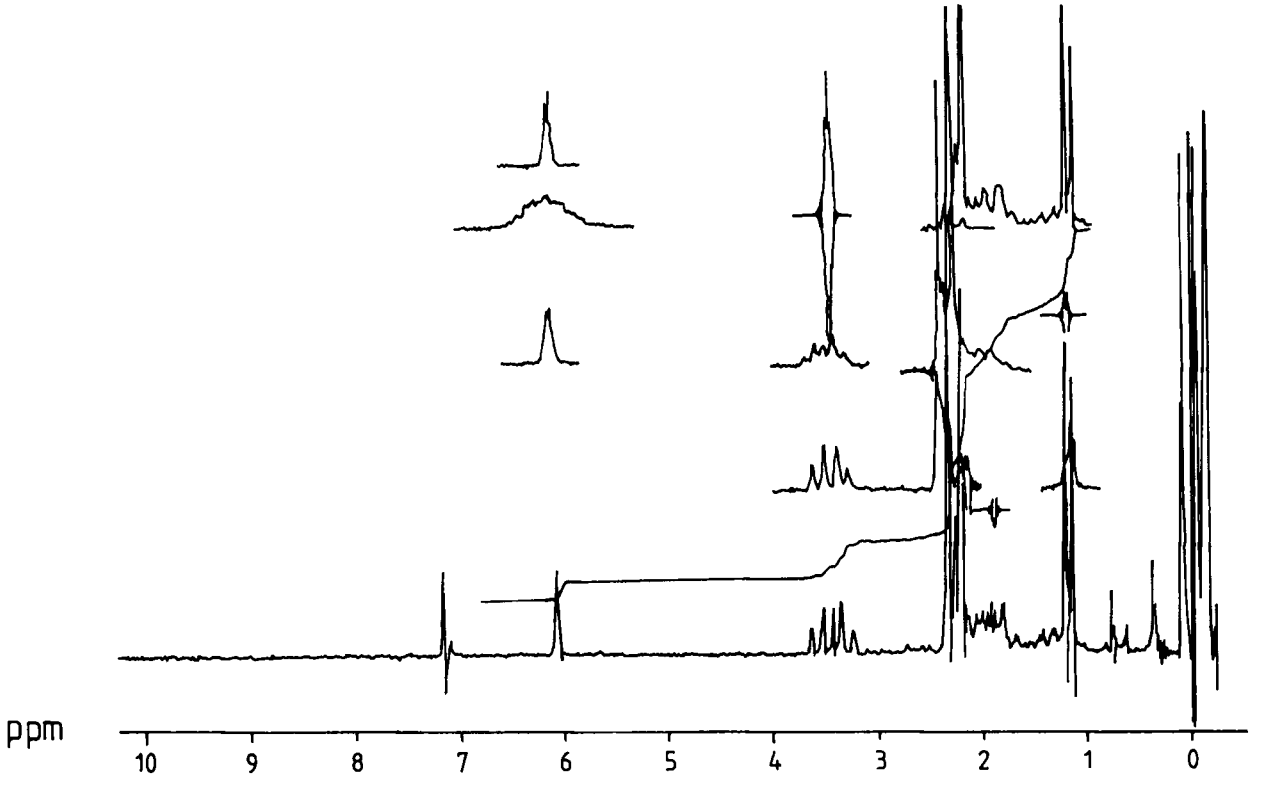
**Mass spectrum** [70 eV]




**IR spectrum** [KBr]



**$^1$H-NMR** [$CDCl_3$, 90 MHz]



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