Robert Martin · Jean-Pierre Buisson

Aromatic Hydroxyketones: Preparation & Physical Properties

Aromatic Hydroxyketones from Butanone (C4) to Dotriacontanone (C32)



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Robert Martin (deceased) Formerly Institut Curie Paris V, Antony, France Jean-Pierre Buisson Institut Curie - Paris V Sartrouville, France

Dr Martin wrote this book with Dr Buisson further to their cooperation while at Institut Curie, Paris, France, and could complete it a few months before passing away.

ISBN 978-3-319-14184-8 ISBN 978-3-319-14185-5 (eBook) DOI 10.1007/978-3-319-14185-5

Library of Congress Control Number: 2014958588

Springer Cham Heidelberg New York Dordrecht London © Springer International Publishing Switzerland 2015

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

Robert Martin

Foreword

Two centuries of organic chemistry have already yielded millions of molecules, either synthesized or isolated as natural products created by biosynthesis, but much still remains to be done. Therefore, from time to time, it is probably useful to gather and classify the scattered data concerning this or that class of compounds in order to save time for chemists planning new syntheses or natural products isolations.

In this work, special effort has been made to select material suitable to meet the needs of chemists who do not benefit from unlimited time for specialized research in the field of hydroxyketones.

These compounds are precursors of substituted aromatic derivatives which are often not straightforwardly obtained but have many potential applications in fine and medicinal chemistry.

In this book, Aromatic Hydroxyketones, Preparation and Physical Properties the reader will find more than 5,200 hydroxyketones from butanone C_4 to dotriacontanone C_{32} .

This work will primarily be of great value to professional chemists, from physicists to pharmacists, who are often called upon to solve problems about the synthesis of this kind of aromatic compounds.

Jean-Claude Florent

Institut Curie Research Unit "Conception, Synthesis and Targeting of Biomolecules", Institut Curie Paris, France

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Preface

Aromatic hydroxyketones are widely used as starting materials in organic synthesis to obtain medicines, dyes, perfumes, etc., and many of them have also specific uses.

This Handbook contains information on aromatic hydroxyketones from C_4 to C_{32} , collecting more than 5,200 ketones of which an indicated preparation as well as their physicochemical and spectrochemical data are presented.

This book is presented in dictionary style, with a logical classification of the ketones, which makes the information easily available for consultation. Ketones are classified methodically. They are easily accessible to the reader from three tables provided at the end of the book:

- The molecular formula index
- The Chemical Abstracts Registry Numbers
- The usual names index

Since hydroxyketones are widely utilized, themselves or as intermediates, in numerous interesting syntheses, among others, pharmaceuticals, dyes, agrochemicals, perfumes and plastic preservatives, this handbook will be of great value for both academic and industrial research chemists. The multiple ways of hydroxybenzophenones syntheses herein described will certainly help chemists, as most of these methods can be applied to prepare analogues in aromatic and even in some heterocyclic series.

This compilation will supply helpful and easy-to-read informations for the organic chemist.

Antony, France Sartrouville, France Robert Martin Jean-Pierre Buisson

Acknowledgements

I wish to express my heartily thanks to Dr. Pierre Demerseman who accepted me in his laboratory at Institut Curie in 1987.

My thanks are also directed to Prof. Claude Monneret, Head of the Chemical Department at Institut Curie, who has always been so benevolent to me, and all his collaborators for their warm welcome at each of my visits.

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

The author also thanks Prof. Jean Paul Guetté for all his good advices.

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

In this connection, thanks are due to Mrs. Feiga Weisbuch for her precious assistance as regards Rumanian and Russian texts, as well as Dr. Daniel Dauzonne. I wish to express my thanks to Mrs. Mireille Guyonneau, Mrs. Elisabeth Matarasso, Mrs. Françoise Rémy and Mrs. Simonne Rissé for their keen contribution to my bibliographic research, as well as the Orsay University Library for their helpful kindness towards me for 30 years now.

I am also grateful to Mrs. Colette Ledoux for judicious advice in the field of scientific edition.

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

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

Robert Martin

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Short Biography

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

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

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

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

In 1992, he published a review on Fries reaction in the *Organic Preparations* and *Procedures International*. This was followed by the publishing of two books dealing with aromatic hydroxyketones, published by KLUWER in 1997 and 2000.

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

Jean-Pierre Buissson is also doctor es-science (Ph.D.) from Paris University. The subject of his thesis was "Phenolic ether desalcoylation with pyridinium hydrochloride".

Chemist at the CNRS, he worked all his career in the chemistry lab of the Institut Curie in Paris with, successively, Drs. Royer, Demerseman, Monneret and Florent.

His research concerns the synthesis of heterocyclic compounds in the benzofuran, naphthofuran and oxaphenalene series.

The major product obtained was the R7000, the most mutagenic compound on bacterial strains, which is now an international reference for biologists.

Chapter 1 Butanones: Monoketones

1 Aromatic Hydroxyketones Derived from 1-Butanoic Acid

1.1 Unsubstituted Hydroxyketones

1-(2-Hydroxyphenyl)-1-butanone

[2887-61-8]

 $C_{10}H_{12}O_2$

mol. wt. 164.20

OH CO(CH₂)₂CH₃

Syntheses

-Phenol was energetically mixed with finely divided aluminium chloride (1 mol) and heated to 100° for 30 min (1 mol of hydrochloric acid is evolved); 1 mol of n-butyryl chloride was then added and heated to 125–130° for 1 h (45 %) [2700].

-Also obtained by Fries rearrangement of phenyl butyrate with aluminium chloride, *without solvent [3382], at 150° [2045], at 160–180° for 2 h (60 %) [726];

*in nitrobenzene at 50° for 18 h (18 %) [776];

*in tetrachloroethane at 95° for 6 h (50 %) [244] or at 70–80° for 7 h [3169];

*in heptane at 80–90° for 6 h (40 %) [244];

*in durene at reflux for 3 h (54 %) [1303].

-Also obtained by Fries rearrangement of phenyl butyrate,

*in the presence of polyphosphoric acid for 10 min at 100° (13 %) [2238];

*in the presence of zirconium halides or hafnium halides in o-dichlorobenzene over 3 h at 120° (85 %) [2096].

-Also obtained by photo-Fries rearrangement of phenyl butyrate in cyclohexane, heptane, benzene or n-amyl alcohol in a sealed tube with an unfiltered Hanovia 450 w medium pressure lamp [2067].

-Also obtained by diazotization of 2-aminobutyrophenone [2147].

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R. Martin, J.-P. Buisson, Aromatic Hydroxyketones: Preparation & Physical Properties, DOI 10.1007/978-3-319-14185-5_1

-Also obtained by treatment of 1-(2-hydroxyphenyl)-1-butanol with manganese dioxide in methylene chloride for 7 h at r.t. (46 %) [77].

-Also refer to: [173, 375, 932 (36 %), 970, 1078, 1172–1174, 1572, 1620, 2081, 2120–2124, 2848, 2920, 3028, 3477].

Isolation from natural sources

-Detection of the gel of Aloe vera (L.) burm. [1624].

colourless oil [77]; pale yellow, viscous liquid [2147]. b.p._{0.1} 64° [932], b.p._{0.01} 67–68° [3318], b.p.₅ 91–98° [2314], b.p.₉ 119° [2700], b.p.₁₄ 124–126° [726], b.p.₁₈ 129° [3169], b.p.₁₉ 130–132° [1762], b.p.₁₂ 135–138° [3477]; m.p. 10.5–10.6° [2700], 10–11° [3318], 8° [726]; ¹H NMR [77, 2067], ¹³C NMR [77], IR [77, 1762, 2067], UV [1996, 2067], MS [77, 173, 2067]; ESR [2940]; TLC [1994]; paper chromatography [1183]; GLC [2067]; GC [2067]. $n_D^{20.5} = 1.539$ [3318], $n_D^{20} = 1.5379$ [932].

N.B.: Photodegradation of o-(1-butanoyl)phenol in waste water [1385].

USE: Fungicide [547, 2044, 2045]; Hair dyes and method for highlighting or streaking hair [2785]; Oxidative hair dyes containing diazolium and triazolium compounds in combination with reactive carbonyl compounds [1301].

Oxime	[21667-43-6]	$C_{10}H_{13}NO_2$	mol. wt. 179.22		
-Refer to: [603].					
2,4-Dinitropheny m.p. 203° [932	·	$C_{16}H_{16}N_4O_5$	mol. wt. 344.33		
Acetate -Refer to: [2493];	[21550-10-7] UV [2493].	$C_{12}H_{14}O_3$	mol. wt. 206.24		
Benzoate C ₁₇ H ₁₆ O ₃ mol. wt. 268.31 m.p. 65–66° [3477].					
Methyl ether	[13404-83-6]	$C_{11}H_{14}O_2$	mol. wt. 178.23		
-Obtained by reaction of dimethyl sulfate with 2-butyrylphenol in the presence of aqueous sodium hydroxide for 4 h at reflux [2478].					

-Also refer to: [1084, 2300 (72 %), 2493].

b.p._{0.8} 83° [2478], b.p.₁₇ 146° [2300]; ¹H NMR [2848], UV [2493], MS [42]; Phosphorescence spectroscopy [2493].

Semicarbazone of the	e methyl ether	$C_{12}H_{17}N_3O_2$	mol. wt. 235.29
m.p. 146° [2300].			
Phenylhydrazone	C	$_{16}H_{18}N_2O$	mol. wt. 254.33
m.p. 85–87° [726].			
1-(3-Hydroxyphenyl)	-1-butanone		
[103323-29-1]	C ₁	$_{0}H_{12}O_{2}$	mol. wt. 164.20
OH CO(CH ₂) ₂ CH ₃	organocadmiu -Preparation b	3-hydroxybutyrophenon m derivatives (75 %) [258 by diazotization of 3-am is of the obtained diazon [47].	36]. inobutyrophenone
-Also refer to: [1614,	1618, 1822, 212	5].	
b.p. ₂ 155–157° [253 ¹ H NMR [1614, 16		° [3002], 63° [2147, 2586]	;
p-Nitrophenylhydraz	one	$C_{16}H_{17}N_3O_3$	mol. wt. 299.33
orange-yellow need	lles [2147]; m.	p. 160° [2147].	
Acetate [2	1999-97-3]	$C_{12}H_{14}O_3$	mol. wt. 206.24
b.p. ₁ 116–118° [258	86]; UV [2493].	

-Obtained by hydrolysis of m-methoxybenzoylethylketene, itself prepared from ethylketene dimethylacetal and m-benzoyl chloride at reflux for 5 h (83 %) [2028]. -Also obtained by reaction of m-benzoyl chloride with dipropylcadmium (78 %) [571].

 $C_{11}H_{14}O_{2}$

mol. wt. 178.23

[21550-06-1]

-Also refer to: [73, 1614, 1618, 2493].

Methyl ether

N.B.: Regioselective directed *meta*-acylation of aromatic compounds *via* cycloaddition of nitriles to benzyne-zirconocene complexes [73].

b.p.₂ 105–110° [571], b.p.₁₀ 121° [3002], b.p₁₁ 142–146° [2028], b.p. 265–270° [1067]; ¹H NMR [73, 1618], ¹³C NMR [73], IR [73], UV [2493], MS [73]; Phosphorescence spectroscopy [2493].

2,4-Dinitrophenylhydrazone of the methyl ether $C_{17}H_{18}N_4O_5$ mol. wt. 358.35

m.p. 172-173° [2028].

1-(4-Hydroxyphenyl)-1-butanone

[1009-11-6]

 $C_{10}H_{12}O_2$

mol. wt. 164.20



Syntheses

-Phenol was energetically mixed with finely divided aluminium chloride (1 mol) and heated to 100° (1 mol of hydrochloric acid is evolved); 1 mol of n-butyryl chloride was then added and heated to $125-130^{\circ}$ for 1 h (50 %) [2700].

-Also obtained by reaction of butyryl chloride,

*with phenol in the presence of aluminium chloride in nitrobenzene first at $5-10^{\circ}$, then at r.t. for some hours (76 %) [2970] or at r.t. overnight (67 %) [1769];

*with phenol in the presence of aluminium chloride in methylene chloride for 1 h at 0° , then at r.t. overnight (80 %) [114] or for 14 h at r.t. (43 %) [1910];

*with anisole in the presence of aluminium chloride in nitrobenzene [2923].

-Also obtained by Fries rearrangement of phenyl butyrate with aluminium chloride,

*without solvent [3382], at 150° [2045], for 2 h at 160–180° (19 %) [726];

*in nitrobenzene [2923], [2947] (66 %), for 18 h at 50° (72 %) [776];

*in tetrachloroethane at 95° for 6 h (43 %) [244] or at 70–80° for 7 h [3169];

*in heptane at 80–90° for 6 h (50 %) [244];

*in durene at reflux for 3 h (14 %) [1303].

-Also obtained by Fries rearrangement of phenyl butyrate,

*in the presence of polyphosphoric acid for 10 min at 100° (45 %) [2238];

*in the presence of boron trifluoride for 3 h at 70–75° (65.2 %) [1938].

-Also obtained by photo-Fries rearrangement of phenyl butyrate in cyclohexane, heptane, benzene or n-amyl alcohol in a sealed tube with an unfiltered Hanovia 450 w medium pressure lamp [2067].

-Also obtained by reaction of butyric acid with phenol,

*in the presence of boron trifluoride [511], for 2 h at 70° (81 %) [1685];

*in the presence of polyphosphoric acid for 5 min in a boiling water bath (54 %) [2240] or for 10 min at 100° (76 %) [2238].

-Also obtained by reaction of butyronitrile with phenol in the presence of triflic acid, first 15 days at r.t., then 1 h at reflux (78 %) [425].

-Also obtained by treatment of its methyl ether with boiling pyridinium chloride for 15 min (70 %) [221].

-Also obtained by anaerobic co-metabolic oxidation of 4-butylphenol by *Thauera* sp. strain R5 [2861].

-Also obtained by anaerobic biodegradation of 4-butylphenol in Kamajima paddy soil microcosm supplemented with nitrate [2862].

-Also refer to: [62, 131, 254, 274, 494, 513, 556, 597, 663, 664, 932 (34 %), 1070, 1087, 1510, 1531, 1615, 1616, 1620, 1650, 1651, 1791, 1792, 1805, 1872, 2031, 2032, 2125, 2457, 2711, 2772, 2943, 3418, 3452, 3454, 3471, 3477 (14 %)].

b.p._{0.01} 144–152° [2970], b.p.₁ 171° [932], b.p._{5.3} 171–174° [1769], b.p.₉ 187–188° [2700], b.p.₁₅ 192–200° [2947], b.p.₁₅ 200° [726];

white solid [1910]; m.p. 99.6–100° [1910], 93.5° [1685], 92° [2240], 91.5–92.5° [3169], 91–92° [3477], 91–91.5° [2700], 91° [726, 776, 932, 2457], 90.5–92° [425], 90.5–91.5° [1938], 89–90° [2238], 86–89° [1769]; ¹H NMR [114, 425, 1910, 2067], ¹³C NMR [114, 1910], IR [425, 1910, 2067], UV [1995, 2067], MS [425, 1910, 2067, 2861, 2862]; X-ray data [3347]; GC/MS [3131]; paper chromatography [1183]; GLC [2067]; GC [2067]; TLC [1910, 1994].

Isolation from natural sources

-From essential oil extracted from fruit of Zanthoxylum rhetsa (Roxb.) DC. in Vietnam (6.07 %) [3131].

N.B.: Photodegradation of p-(1-butanoyl)phenol in waste water [1385].

USE: Preparation of 3-phenylpropanoic acids derivatives as antidiabetic agents [3397].

BIOLOGICAL ACTIVITY: Inhibition of $17-\beta$ hydroxysteroid dehydrogenase 3 [1910].

-Also refer to: [597, 1510].

Oxime	$C_{10}H_{13}N_{1$	NO_2	mol. wt. 179.22
m.p. 83–84° [34	77].		
Semicarbazone	C ₁₁	$H_{15}N_{3}O_{2}$	mol. wt. 221.26
m.p. 167–169°	[3477].		
2,4-Dinitrophenyl	hydrazone	$C_{16}H_{16}N_4O_5$	mol. wt. 344.33
m.p. 217° [3169	9], 215° [932].		
O-Ethyloxime	[791065-70-8]	$C_{12}H_{17}NO_2$	mol. wt. 207.27
-Refer to: [638].			
Acetate	[13210-98-5]	$C_{12}H_{14}O_3$	mol. wt. 206.24
-Refer to: [2493].	UV [2493].		
Benzoate	C ₁₇ H ₁	₆ O ₃	mol. wt. 268.31
m.p. 107–107.5	° [2700], 106–107°	[726, 3477]; GC [1922]	GC/MS [1244].
Sulfate	[114] (87 %);	¹ H NMR [114], ¹³ C NMR	[114].

Benzyl ether [26945-71-1] C₁₇H₁₈O₂ mol. wt. 254.33

-Preparation by reaction of benzyl chloride with p-hydroxybutyrophenone in the presence of potassium carbonate and potassium iodide in refluxing 90 % ethanol for 5 h (86 %) [556].

-Also refer to: [147, 673, 2480, 2958].

m.p. 67° [556], 63–65° [147, 673], 59–64° [2958].

Methyl ether [4160-51-4] $C_{11}H_{14}O_2$ mol. wt. 178.23

Syntheses

-Preparation by reaction of n-butyryl chloride with anisole,

- *in the presence of aluminium chloride in carbon disulfide, first at 0° for 2 h, then between 10 and 25° for 3 h (81 %) [3062];
- *in the presence of aluminium chloride in 1,2-dichloroethane [2942], first at 0° for 40 min, then at r.t. for 8–15 h (65 %) [2243];
- *in the presence of titanium tetrachloride at 60° for 3 h (89 %) [777];
- *in the presence of cobalt (II) acetylacetonate in nitromethane and acetonitrile under mild conditions (excellent yields) [3055];
- *in the presence of Si-Fe catalyst at 25° (49 %) [427];
- *using moisture insensitive InCl₃ impregnated mesoporous Si-MCM-41 catalyst in dichloroethane at 80° for 3 h (87 %) [658];
- *over Ga_2O_3 (20 %)/Si-MCM-41 catalyst in dichloroethane at 80° for 3 h (77 %) [657].
- -Also obtained by Friedel-Crafts reaction of butyric anhydride with anisole in a chlorobenzene/GALDEN SV 135 mixture at 90° for 1 h,
- *in the presence of $Hf[N(SO_2C_8F_{17})_2]_4$ (71–73 %) [1243];
- *in the presence of $Hf(OSO_2CF_3)_4$ (44 %) [1243].
- -Also obtained by Friedel-Crafts reaction of butyric anhydride with anisole,
- *in the presence of large molecular sizes on mesoporous silica catalyst at 453 K for 4 h (82 %) [1466];
- *in the presence of $Hf[N(SO_2C_8H_{17})_2]_4$ at 90° for 1 h (88 %) or with $Hf(OSO_2CF_3)_4$ in the same conditions (57 %) [1244];
- *in the presence of microcrystalline beta zeolite-II for 3 h at 130° under argon (98 %) [1594].
- -Also obtained by acylation of anisole with butyric acid,
- *over the HPNbW-WO₃-Nb₂O₅ catalyst at 428 K for 3 h (98 %) [2332];
- *on the solid surface of alumina in the presence of trifluoroacetic anhydride for 20 min at r.t. (92 %) [2563];
- *over HZSM-5 catalyst for 48 h at 423° K (31 %) [3265];
- *in the presence of polyphosphoric acid for 1.5 h at 80° (91 %) [869].
- -Also obtained by direct acylation of 4-bromoanisole with butyraldehyde by palladium catalysis (81 %) [2668].
- -Also obtained by cross coupling reaction of butyryl chloride with $(4-CH_3OC_6H_5)_3Bi$ in the presence of Pd(o) as catalyst (65 %) [2565].

-Also obtained by 4-methoxyphenylboronic acid coupling with the benchmark electron-rich olefin n-butyl vinyl ether catalyzed by Pd-dppp (91 %) [2667].

-Also obtained by reaction of butyraldehyde N-tert-butylhydrazone with 4-bromoanisole in the presence of $Pd_2(dba)_3$, DPEphos and NaOtBu in dioxane at 80° for 24 h (94 %) [3042].

- -Also obtained in two steps by reaction of p-anisaldehyde with propylmagnesium bromide followed by pyridinium chlorochromate oxydation (46–54 %) [2557].
- -Also obtained by oxidation of 1-p-methoxyphenyl-1-butanol with CrO₃ [2924].
- -Also obtained by reaction of butyronitrile with anisole in the presence of triflic acid, first 14 days at r.t., then 1 h at reflux (40 %) [425].
- -Also obtained by reaction of butyric acid with anisole in the presence of $Cs_{2.5}H_{0.5}PW_{12}O_{40}$ at 110° for 5 h (59 %) [1636].
- -Also obtained by irradiation of mixture cyclopropyl 4-methoxyphenyl ketone and trichlorosilane at 23° (87 %) [957].
- -Also obtained (low yield) by isomerization of 1-(4-methoxyphenyl)-3-buten-1-ol under the catalysis of $RuCl_2(PPh_3)_3$ in water for 1.5 h at 90–100° [3261].
- -Also refer to: [131, 221, 251, 698 (60 %), 864, 895 (68 %), 1009, 1114, 1398, 1489, 1897 (58 %), 2166, 2279, 2292, 2493, 2840, 2901, 3000, 3001, 3015, 3254, 3412, 3472].

Isolation from natural sources

-Of essential oil of in Ocimum basilicum from Guangxi [1922].

-In leaf oil of *Persea americana* Mill. var. *drymifolia* CV. Duke (Lauraceae) [2488].

pale yellow oil [2243]; viscous oil [2557]; b.p.₃ 115° [3254], b.p.₂ 116–120° [3412], b.p. 123° [3062], b.p._{0.8} 123–124° [869], b.p.₁₀ 135–136° [425], b.p.₉ 144° [2240], b.p._{14.5} 155° [2924], b.p.₂₀ 160° [777], b.p.₁₄ 165–167° [2901], b.p. 285–286° [2924]; m.p. 26° [2240, 2901], 23° [2924], 21–22° [251], 21° [777], 20° [425], 19–21° [895], 16° [869]; ¹H NMR [425, 698, 1244, 2243, 2557, 2563, 2565, 3261], ¹³C NMR [698, 1244, 2557, 2565], IR [425, 698, 2563, 2565], UV [869], MS [425, 698, 1922, 2557, 2565, 3254]; TLC [2557]; GC [1922]; GC/MS [1244, 2488].

Oxime of the methyl ether [423115-90-6] $C_{11}H_{15}NO_2$ mol. wt. 193.25

liquid [2924]; m.p. 58° [452].

Semicarbazone of the methyl ether [91646-56-9] C₁₂H₁₇N₃O₂ mol. wt. 235.29 m.p. 183° [251], 181° [2924], 179–181° [895], 173.5° [3178], 172–173° [869].

2,4-Dinitrophenylhydrazone of the methyl ether $C_{17}H_{18}N_4O_5$ mol. wt. 358.35

m.p. 165-166° [869].

Ethyl ether [35031-73-3] $C_{12}H_{16}O_2$ mol. wt. 192.26

-Obtained by reaction of butyric acid with phenetole in the presence of aluminium chloride (76 %) [3477].

b.p.₅ 116–120° [3435], b.p. 129° [3477], b.p.₁₄ 162–164° [903], b.p.₂₃ 173–174° [1698].

Oxime of the ethyl ether C₁₂H₁₇NO₂ mol. wt. 207.27

m.p. 103–104° [3477].

Semicarbazone of the ethyl ether [88858-34-8] C₁₃H₁₉N₃O₂ mol. wt. 249.31

m.p. 181° [3477].

1-(2,3-Dihydroxyphenyl)-1-butanone

$$[103324-17-0] C_{10}H_{12}O_3 mtext{mol. wt. } 180.20$$

Syntheses CO(CH₂)₂CH₃ -Obtained by treatment of 2,3-dimethoxy-butyrophenone with hydriodic acid in refluxing acetic acid (38 %) [199].

-Also obtained by deprotection of its dimethyl ether with boron tribromide in methylene chloride at 0° .

Then, the mixture was stirred overnight at r.t. (91 %) [82].

m.p. 61° [199], 59° [82]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82].

USE: Reactant for preparation of lithium gallium/titanium ketone complexes [82].

$C_{12}H_{16}O_{3}$ **Dimethyl ether** [34052-09-0] mol. wt. 208.26

-Preparation by reaction of propylmagnesium iodide with 2,3-dimethoxybenzaldehyde in ethyl ether, then treated the obtained carbinol with potassium dichromate in dilute sulfuric acid (72 %) [199].

-Also obtained by treatment of 1-(2,3-dimethoxyphenyl)-1-butanol in acetone with potassium dichromate in dilute sulfuric acid (Jones reagent) at r.t. for 3 h (65 %) [82].

-Also prepared from 2,3-dimethoxybenzoyl chloride and di-n-propylcadmium by the general procedure [566], (72 %) [2300].

colourless liquid [199], colourless oil [82]; b.p._{0.2} 104° [2300], b.p._{0.6} 112–113° [199]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82].

1-(2,4-Dihydroxyphenyl)-1-butanone

(Resbutyrophenone)

[4390-92-5]

C₁₀H₁₂O₃ mol. wt. 180.20

-Obtained by reaction of butyric acid with resorcinol in the presence of,

*fused zinc chloride [2256] at reflux (about 165°) [449, 1128, (76 %) 1527, 2384, (68–78 %) 2501, 2702, 3243];

*boron trifluoride, first at 15° , then at 70° for 2 h (81 %) [643];

Syntheses

*polyphosphoric acid in boiling water bath for 10 min (44 %) [2239];

*cation-exchange resin at 160° for 2-3 h (73.4 %) [2523].

-Also obtained by reaction of butyric anhydride with resorcinol in the presence of cation-exchange resins, sulfonic acid type, Amberlite IR-120 (or Zeokarb) at 160° (82.3 %) [2523].

-Also obtained by reaction of butyronitrile with resorcinol in the presence of,

*trifluoromethanesulfonic acid at r.t. for 14 days (77 %) [425];

*zinc chloride (Hoesch reaction) [1608].

-Also obtained by Fries rearrangement of resorcinol dibutyrate with aluminium chloride (3 mol) at $180-185^{\circ}$ for 3 h (20 %) [855].

-Also obtained by reaction of butyryl chloride with resorcinol in the presence of aluminium chloride in nitrobenzene (60 %) [58].

-Also refer to: [22, 385 (57 %), 449, 1024, 1025, 1469, 1508, 2312 (81 %), 2662, 2946].

m.p. 73° [2239], 70° [1608, 2312], 69–70° [1527, 3243], 68–70° [449, 2523], 68° [385, 1469], 65–65.5° [425], 63–65° [1128], 59–60° [58]; ¹H NMR [58, 425], ¹³C NMR [1508], IR [425], MS [425].

BIOLOGICAL ACTIVITY: Bactericide [2662]; Antiseptic and germicidal product [2734]; Anthelmintic [1248]; Toxicity [1248].

Hemihydrate	$C_{10}H_{12}O_3, 0.5 H_2O$	mol. wt. 189.01
m.p. 51–52° [1608, 3243].	
Oxime [22919-59 m.p. 190° [808], 189–190	- 10 13 - 3	mol. wt. 195.22
Phenylhydrazone m.p. 191–193° (d) [1128]	$C_{16}H_{18}N_2O_2$].	mol. wt. 270.33
2,4-Dinitrophenylhydrazon m.p. 245° [166].	he $C_{16}H_{16}N_4O_6$	mol. wt. 360.33

Diacetate

C₁₄H₁₆O₅ mol. wt. 264.28

-Obtained by reaction of acetic anhydride with resbutyrophenone in the presence of pyridine [2384].

viscous liquid [2384]; b.p. 15 170–175° [2384].

Dibenzoate C₂₄H₂₀O₅ mol. wt. 388.42

-Obtained by reaction of benzoyl chloride with resbutyrophenone in the presence of pyridine. The mixture was heated on a boiling water bath for 3 h [2384].

oily liquid [2384]; b.p.₂₅ 210° [2384].

C₂₄H₂₄O₃ mol. wt. 360.45

m.p. 61–62° [2181].

Dibenzyl ether

- -Obtained by Friedel-Crafts reaction of butyric anhydride with resorcinol dimethyl ether in the presence of $Hf[N(SO_2C_8H_{17})_2]_4$ in chlorobenzene and SV135 at 70° for 2 h (96 %) [1245].
- -Also obtained by Friedel-Crafts reaction of butyryl chloride with resorcinol dimethyl ether,
- *in the presence of $Hf[N(SO_2C_8H_{17})_2]_4$ in chlorobenzene and SV135 at 70° for 2 h (94 %) [1245];
- *in the presence of HY-zeolite. Before reaction, the zeolites were activated at 500° for 5 h in nitrogen flow [352].
- -Also obtained by reaction of butyronitrile with resorcinol dimethyl ether in the presence of trifluoromethanesulfonic acid at r.t. for 14 days (76 %) [425].
- -Also obtained by reaction of butanoic acid with resorcinol dimethyl ether in the presence of polyphosphoric acid and heated on a water bath for 30 min (70 %) [2424].

-Also refer to: [94, 413, 1388].

yellow oil [425]; b.p.₁ 140–142° [425], b.p.₂ 146–147° [2424], b.p. 156–158° [372]; m.p. 70–71° [57]; ¹H NMR [425], IR [425], MS [425].

Di(β -D-glucoside) C₂₂H₃₂O₁₂ mol. wt. 504.50

-Obtained by treatment of its tetraacetate below with 0.2 M sodium methoxide in methanol for 3 min (30 %) [3243].

 $(\alpha)_{\rm D}^{21} = -109^{\circ}$ (water) [3243].

Di(tetraacetyl)-\beta-D-glucoside $C_{38}H_{48}O_{21}$ mol. wt. 840.80

-Obtained by reaction of α -acetobromoglucose (α -ABG) with resbutyrophenone in the presence of silver oxide in quinoline for 4 h (15–20 %) [3243].

m.p. 152–153° [3243]; $(\alpha)_{D}^{21} = -36.3^{\circ}$ (chloroform) [3243].

1-(2,5-Dihydroxyphenyl)-1-butanone

$$[4693-16-7] C_{10}H_{12}O_3 mol. wt. 180.20$$

OH	Syntheses
CO(CH ₂) ₂ CH ₃	-Preparation by reaction of butyric acid with hydroquinone
	[2102] in the presence of boron trifluoride,
\mathbf{Y}	*on a water bath (73 %) [1797];
ÓН	*at 60° for 2 h in a sealed tube (72 %) [2312].

-Also obtained by reaction of butyryl chloride with hydroquinone [1759] in the presence of aluminium chloride [2374] in nitrobenzene by heating for 3 h in a water bath [1442].

-Also obtained by Fries reaction of hydroquinone dibutyrate at $150-160^{\circ}$ for 5 h [1442].

-Also obtained by treatment of 2,5-dimethoxybutyrophenone with hydrobromic acid in refluxing acetic acid for 6 h [1442].

-Also obtained by photoacylation of hydroquinone with butyraldehyde in benzene under nitrogen (82 %) [1759, 1991].

-Also obtained by irradiation of 1,4-benzoquinone and butyraldehyde mixture in benzene containing 5 % ethanol for 3 days (60 %) [2374].

-Also obtained by Fries rearrangement of 4-methoxyphenyl butyrate with aluminium chloride (5 part)/sodium chloride (2 part) mixture at 180–200° (30 %) [1796]. -Also refer to: [1182, 1312, 2781].

b.p.₁₆ 193° [1442]; m.p. 175° [1796], 101° [1442], 96° [1797], 94–96° [1759, 1991], 91° [2102], 87–89° [1182], 87–88° [2312], 85° [3204]. **N.B.**: One of the reported melting point is obviously wrong.

BIOLOGICAL ACTIVITY: Anthelmintic [2781]; Antiprotozoal [1312].

Dimethyl ether [54419-64-6] $C_{12}H_{16}O_3$ mol. wt. 208.26

-Obtained by reaction of butyryl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride,

*in nitrobenzene at r.t. overnight (70 %) [1442];

*in methylene chloride at r.t. for 1 h (70 %) (with carbon disulfide, 61 %) [2878].

mol. wt. 388.42

-Also obtained by reaction of butanoyl chloride with hydroquinone dimethyl ether in the presence of HY-zeolite. Before reaction, the zeolites were activated at 500° for 5 h in a nitrogen flow [352].

-Also obtained by reaction of butyric anhydride with p-dimethoxybenzene in the presence of aluminium chloride in carbon disulfide at -5° , then at r.t. overnight [1159].

-Prepared by Friedel-Crafts acylation (90 %) [1755].

-Also obtained by catalytic hydrogenation of 2,5-dimethoxyphenyl 2-piperidinopropyl ketone over platinum in ethanol under atmospheric pressure at r.t. (50 %) [1217].

-Also refer to: [425, 1159, 1217, 1442, 1755, 1760, 1761, 2874, 2878, 3271].

b.p.₃ 121–123° [1755], b.p._{0.001} 136–138° [1159], b.p.₂₀ 170–178° [2878], b.p.₂₅ 172–175° [1442], b.p.₃ 178° [2874]; ¹H NMR [1755], IR [1217, 1755], MS [1217].

USE: First step of syntheses of Frenolicin B (anticoccidial agent) and Kalafungin (antifungal agent) [1761].

2,4-Dinitrophenylhydrazone of the dimethyl ether

[54419-69-1]	$C_{18}H_{20}N_4O_6$	mol. wt. 388.38
m.p. 174–175° [1442], 154–156° [1217].		

C24H20O5

Dibenzoate

m.p. 110° [1442].

1-(2,6-Dihydroxyphenyl)-1-butanone

[10121-26-3]	$C_{10}H_{12}O_3$	mol. wt. 180.20
OH CO(CH ₂) ₂ CH ₃	Syntheses -Obtained by treatment of 8-butyry 2H-1-benzopyran-2-one, *with 12 % sodium hydroxide on (80 %) [2670];	

*with N sodium hydroxide at reflux for 30 min (55 %) [1882].

-Preparation by treating resorcinol-4,6-dicarboxylic acid dimethyl ester with butyryl chloride in the presence of aluminium chloride, followed by hydrolysis and decarboxylation of the compound obtained (30 %) [862].

-Also obtained from 2,6-dihydroxyacetophenone (59 %) [385].

Isolation from natural sources

-From the culture extract of the endophytic fungus *Nodulisporium* sp. isolated from the plant *Erica arborea*, from Gomera [791].

-Of Rhizophora apiculata endophytic fungus 3920 from South China Sea [1391].

-From the extracts of cultures of the estuarine fungus [1390]. -From the extracts of cultures of *D. concentrica* strain 26 A1 [91].

m.p. 120° [1882], 116–118° [91], 116–117° [385], 106–107° [862], 106° [2670]; ¹H NMR [1390, 1391], ¹³C NMR [792], IR [792], UV [91], MS [792, 1391]; X-ray data [1390, 1391].

N.B.: Other names of 8-butyryl-7-hydroxy-4-methyl-2*H*-1-benzopyran-2-one: *8-butyryl-4-methylumbelliferone [1882]; *8-butyryl-7-hydroxy-4-methylcoumarin [2670].

C12H16O3 **Dimethyl ether** mol. wt. 208.26 b.p.₁₀ 150° [1882], 287–290° [2736], 289–291° [1882]; ¹H NMR [1887], ¹³C NMR [1887], MS [1887].

1-(3,4-Dihydroxyphenyl)-1-butanone

[17386-89-9]

OH

OH

C10H12O3

mol. wt. 180.20

Syntheses

-Obtained by reaction of butyric acid with pyrocatechol, *in the presence of zinc chloride at reflux [726]; *in the presence of boron trifluoride for 2.5 h at 150° (61 %) $\dot{CO}(CH_2)_2CH_3$ [2312] or for 2–3 h between 65 and 85° [503].

-Also obtained by Fries rearrangement of pyrocatechol dibutyrate,

*in the presence of aluminium chloride [726] in nitrobenzene for 30 min at 100° (35 %) [2646];

*in the presence of aluminium chloride and pyrocatechol (1 mol) in nitrobenzene for 2 h at 80° (70 %) [2646].

-Also obtained by reaction of butyryl chloride with pyrocatechol in the presence of aluminium chloride in nitrobenzene (27 %) [2646].

-Also obtained by treatment of guaiacol butyrate with aluminium chloride in carbon disulfide at 90° for 50 min, then at $135-140^{\circ}$ for 2 h after solvent elimination [2075].

-Also obtained by treatment of 4-hydroxy-3-methoxybutyrophenone with boiling pyridinium chloride [505].

-Also refer to: [2127, 3183].

b.p.₁₅ 220–230° [726];

m.p. 149° [505], 147° [503], 146–147° [2646], 146° [2312], 139° [2075].

BIOLOGICAL ACTIVITY: Central nervous system depressant [2127].

Dimethyl ether [54419-21-5] C₁₂H₁₆O₃ mol. wt. 208.26

-Obtained by Friedel-Crafts reaction of butyric anhydride with veratrole in the presence of $Hf[N(SO_2C_8H_{17})_2]_4$ in chlorobenzene and SV135 at 90° for 1 h (97 %) or with $Hf(OSO_2CF_3)_4$ in the same conditions (66 %) [1244, 1245].

-Also obtained by Friedel-Crafts reaction of butyryl chloride with veratrole,

- *in the presence of $Hf[N(SO_2C_8H_{17})_2]_4$ in chlorobenzene and SV135 at 110° for 1 h (94 %) or with $Hf(OSO_2CF_3)_4$ in the same conditions (70 %) [1245];
- *in the presence of zinc chloride in refluxing carbon disulfide for 4 h (36.6 %) [1565];
- *in the presence of aluminium chloride in carbon disulfide [1602], [1800] (43 %), first at r.t. for 5 h, then heating on a water bath for 30 min (14 %) [1565];
- *in the presence of HY-zeolite. Before reaction, the zeolites were activated at 500° for 5 h in a nitrogen flow [352].
- -Also obtained by reaction of butyric acid with veratrole in the presence of polyphosphoric acid for 2.5 h at 60° (91 %) [1364].
- -Also obtained in two steps by reaction of 3,4-dimethoxybenzaldehyde with propylmagnesium bromide followed by pyridinium chlorochromate oxydation (46–54 %) [2557].

-Also obtained by hydrogenation of 3,4-dimethoxyphenyl 2-piperidinopropyl ketone at pH 4.2 with Raney nickel (14 %) [1217].

-Also refer to: [851, 895 (74 %), 1242, 2581, 3056].

White crystalline solid [2557], colourless needles [1565]; b.p.₄ 160–165° [1565], b.p.₉ 175° [3056], b.p.₁₅ 192–196° [1602]; m.p. 63–66° [1602], 61–62° [2557], 59–61° [895], 54.4–55.2° [1364], 54° [1565], 53–54° [1800], 52–53° [1217, 3056]; ¹H NMR [1217, 1244, 1800, 2557], ¹³C NMR [1244, 2557], IR [1217, 1800], UV [2815], MS [2557]; TLC [2557]; GC/MS [1244].

Oxime of the dimethyl ether C	$C_{12}H_{17}NO_3$	mol. wt. 223.27
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m.p. 73.3–74.5° [1364].

Fine colourless needles [1565]; m.p. 178–180° [895], 135° [1565]. **N.B.**: One of the reported melting point is obviously wrong.

1-(3,5-Dihydroxyphenyl)-1-butanone

[103323-62-2]	$C_{10}H_{12}O_3$	mol. wt. 180.20
HO CO(CH ₂) ₂ CH ₃	Synthesis -Obtained by treatment of sodium hydroxide at reflux m.p. 107° [1406].	f its diacetate with 5 % x for 4–5 h (54 %) [1406].

2,4-Dinitrophenylhydrazone	$C_{16}H_{16}N_4O_6$	mol. wt. 360.33
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m.p. 232° [1406].

Diacetate [1008	84-40-0] C ₁ .	$_{4}H_{16}O_{5}$ mol.	wt. 264.28
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-Preparation by reaction of dipropylcadmium with 3,5-diacetoxybenzoyl chloride in refluxing benzene for 1 h (72 %) [1406].

b.p._{0.6} 168-174° [1406].

2,4-Dinitrophenylhydrazone of the diacetate

 $[102158-27-0] C_{20}H_{20}N_4O_8 mtext{mol. wt. } 444.40$

m.p. 146° [1406].

Dimethyl ether [39911-73-4] C₁₂H₁₆O₃ mol. wt. 208.26

-Preparation by reaction of propylmagnesium bromide with 3,5-dimethoxybenzamide in refluxing ethyl ether for 5 h (88 %) [2990], or for 70 h under argon (58 %) [2470].

colourless oil [2470]; b.p.₁₁ 145° [2470], b.p.₇ 157–158° [2990]; m.p. 33.5–34° [2990]; ¹H NMR [2470], IR [2470], MS [2470].

1-(2,3,4-Trihydroxyphenyl)-1-butanone

[2437-61-8]	$C_{10}H_{12}O_4$	mol. wt. 196.20
ОН	Syntheses	
HO_{1} \downarrow $CO(CH_{2})$ CH_{2}	-Obtained by reaction of by	utyric acid with pyrogallol,

-Obtained by reaction of butyric acid with pyrogallol, *in the presence of zinc chloride [214, 2678], at 135–140° for 2 h (Nencki reaction) (60 %) [1283];

*in the presence of boron trifluoride in ethyl ether at 0° for 1 h (79 %) [538, 540];
*in the presence of 70 % perchloric acid on boiling during 30 min (25 %) [2070];
*in the presence of cation-exchange resins, sulfonic acid type, Amberlite IR-120 (or Zeokarb) at 160° (70.8 %) [2523];

*in the presence of strongly acidic ion exchanger Amberlyst-15 at 120° for 24 h (52 %) [231].

-Also obtained by reaction of butyric anhydride,

- *in the presence of polyphosphoric acid or concentrated sulfuric acid (1 drop) at reflux for 10–15 min, according to the method [1470], (64.5 %) [2523];
- *in the presence of Amberlite IR-120, (a cation exchange resin, sulfonic acid type), at 160° for 2–3 h (80.5 %) [2523], although Zeokarb 225 was found to be as effective.

-Also obtained by reaction of butyryl chloride with pyrogallol in the presence of aluminium chloride in nitrobenzene (50 %) [58].

-Also refer to: [1260 (36 %), 1361, 1527].

m.p. 109–110° [58], 102° [538, 540], 101–102° [1527], 101° [1260], 100° [214, 215, 2678], 98–99° [2523], 95° [2070], 90–91° [1283]; ¹H NMR [58, 231], ¹³C NMR [231], UV [540].

Trimethyl ether [108401-78-1] C₁₃H₁₈O₄ mol. wt. 238.28

1-(2,4,5-Trihydroxyphenyl)-1-butanone

[1421-63-2]

 $C_{10}H_{12}O_4$

mol. wt. 196.20



HO OH OH OH

-Obtained by reaction of 1,2,4-trihydroxybenzene with butyric anhydride,

*in the presence of a catalyst (ZnCl₂, AlCl₃ or SOCl₂) using a mixture of aromatic hydrocarbon and water as binary solvent (80–86 %) [3074];

*in the presence of aluminium chloride in nitrobenzene first at 25° , then at 60° for 45 min (61 %) [292].

-Also obtained by Fries rearrangement of 1,2,4-tributyryloxybenzene (b.p. $_{0.08}$ 153–155°) with aluminium chloride in nitrobenzene [291].

-Also refer to: [78, 165, 333, 347, 376, 495, 666, 899, 961, 1102, 1152, 1310, 1311, 1508, 1570, 1666, 1707, 1896, 2126, 2127, 2257, 2271, 2402, 2712, 2722, 2808, 2809, 2863, 3047, 3229, 3240, 3279, 3425].

Isolation from natural sources

-From urine of rats or dogs [166].

m.p. 151–153° [291, 292], 147–148° [166];

¹³C NMR [1508], UV [166], MS [1874, 3355];

LC/MS [3047]; HPLC [1874, 2712]; paper chromatography [166].

USE: Preparation of 7-oxabicyclo[4.1.0]heptane *via* epoxidation of cyclohexene catalyzed by vanadium bromoperoxidase from Corallina officinalis (red algae) [3425]; Candesartan cilexetil formulations [2806]; Continuous multimicroencapsulation process for improving stability and storage life of biologically active ingredients in foods, cosmetics and drugs [562]; Excipients in drug delivery vehicles for depot gel [625]; Reducing agent for thermographic imaging composition containing silver behenate and second reducing agent [3279]; Thermostability of synthetic antioxidant for food [3182]. BIOLOGICAL ACTIVITY: Effective inhibitor of the sn-glycerol-3-phosphate oxidase of *Trypanosoma brucei brucei* [1152]; Antioxidant in oils and fats [1874, 2568, 3426]; Antioxidant for fats, oils and paraffin waxes [292]; Antioxidant in foods [2712]; Antioxidant and pesticide [165]; Antioxidant for fats and oils [289, 291]; Antioxidant in estrogen derivatization [3355]; Antioxidant [666, 2295, 3032]; Central nervous system depressant [2127]; Non-permitted phenolic antioxidant [3047]; Antibacterial agent [2722]; Bilirubin stabilization in control serums and calibrators [495]; Antimelanoma activity and skin depigmentation by, *in vitro* method for screening of, [899]; Toxicity [1152]; Cytotoxicity [1393].

Triacetate [145747-19-9] C₁₆H₁₈O₇ mol. wt. 322.31

-Obtained by reaction of acetic anhydride with 2,4,5-trihydroxyphenol in the presence of pyridine (8) [376].

-Also refer to: [2410].

m.p. 108–110° [376]; ¹H NMR [376], IR [376], MS [376].

Trimethyl ether [2020-73-7] $C_{13}H_{18}O_4$ mol. wt. 238.28

-Obtained by heating dimethyl sulfate with 2,4,5-trihydroxybutyrophenone [1250] in the presence of aqueous sodium hydroxide at reflux for 90 min (83 %) [2127]. -Prepared by Friedel-Crafts acylation (88 %) [1755].

- -Also obtained by reaction of butyric anhydride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride in methylene chloride at 60° for 1 h (74 %) [772]. -Also obtained by reaction of diazomethane with 4-hydroxy-2,5-dimethoxybutyr-ophenone in ethyl ether [166].
- -Also obtained by reaction of butyryl chloride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride,
- *in ethyl ether between 0 and 5° for 7 h 15 min (32 %) [1250];
- *without solvent (60–74 %) [771].
- -Also obtained by treatment of 1-(2,4,5-trimethoxyphenyl)butane with DDQ in wet dioxane in the presence of silica gel (59 %) [1546].

-Also obtained by reaction of butyric acid with 1,2,4-trimethoxybenzene in the presence of polyphosphoric acid for 4 h at $45-50^{\circ}$ [2695].

-Also refer to: [1800 (48 %), 2696].

white crystals [2127], yellow prisms [166], white solid [1546], white powder [772]; m.p. 79° [1755], 78.5–80° [765, 2127], 77° [766, 1250], 76–77.5° [2695], 76–77° [166], 75–77° [772, 1800], 75–76° [1546];

- ¹H NMR [772, 1546, 1800, 2695], ¹³C NMR [772],
- IR [772, 1800, 2695], MS [772, 2695].
- BIOLOGICAL ACTIVITY: Central nervous system depressant [2127].

Triethyl ether [63213-31-0] C₁₆H₂₄O₄ mol. wt. 280.36

-Obtained by reaction of butyric acid with 1,2,4-triethoxybenzene in the presence of PPA (60–80 %) [2196].

m.p. 50-51° [2196].

OH

HO

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

1-(2,4,6-Trihydroxyphenyl)-1-butanone

$$[2437-62-9] C_{10}H_{12}O_4 mol. wt. 196.20$$

Syntheses

-Preparation by reaction of n-butyronitrile with phloroglucinol (Houben-Hoesch reaction) (64 %) [83, 965, (71.7 %) 1375, 1608, 1610].

-Also obtained by reaction of butyryl chloride with phloroglucinol in the presence of aluminium chloride,

*in nitrobenzene [2646], (60–70 %) [421], (53 %) [2618];

*in nitrobenzene and carbon disulfide mixture (56 %) [2113], (53 %) [2620].

-Also obtained by reaction of butyric acid with phloroglucinol in the presence of boron trifluoride etherate [3019, 3020].

-Also obtained by reaction of butyric anhydride with phloroglucinol,

*in the presence of Amberlite IR-120, (a cation exchange resin, sulfonic acid type), at 160° for 2–3 h (32.2 %) [2523];

*in the presence of polyphosphoric acid or concentrated sulfuric acid (1 drop) at reflux for 30 min, according to the method [1470], (32 %) [2523];

*in the presence of boron trifluoride etherate (70–80 %) [2014].

-Also obtained by reaction of butyronitrile with phloroglucinol in the presence of trifluoromethanesulfonic acid at r.t. for 16 days (45 %) [425].

-Also refer to: [180, 205, 544, 763, 1026, 1439, 1916 (**XIIb**), 1942, 2616, 2771, 2860, 3297, 3391].

Isolation from natural sources

-From Dryopteris lacera [3299].

-From Dryopteris sacrosancta [3299].

m.p. 185–186° [2523, 3297], 183° [544], 182–184° [3391], 181.5–183° [425], 180–181° [1375, 1439], 180° [2113, 2620, 2646], 179–180° [421, 1608, 1610, 2616, 2618], 176° [83]; ¹H NMR [83, 421, 425, 3019], ¹³C NMR [205, 3019], IR [421, 425, 3019], UV [540, 3019], MS [421, 425]; GLC [2531].

BIOLOGICAL ACTIVITY: Antimicrobial activity against *Bacillus subtilis* [3020]; Antimicrobial for *Staphylococcus aureus* [3372]; Antagonist both thromboxane A_2 and Leukotriene D_4 [3019]; Antifungal [2113]; Anthelmintic [1248]; For hepatic and nephritic colic [1813]; Antioxidant in food [2024]; Toxicity [1248].

Monohydrate $C_{10}H_{12}O_4, H_2O$ mol. wt. 214.22

-Refer to: [1608, 1610, 2646].

m.p. 110° [1610].

-Obtained by reaction of butyronitrile with phloroglucinol trimethyl ether in the presence of trifluoromethanesulfonic acid at r.t. for 13 days (67 %) [425].

-Also obtained by reaction of butyryl chloride with 1,3,5-trimethoxybenzene in the presence of stannic chloride in methylene chloride at -15 to -10° for 2 h (96 %) [3068].

-Also refer to: [3070].

yellow oil [425]; b.p.₁ 158–160° [425]; ¹H NMR [425], IR [425], MS [425].

1-(3,4,5-Trihydroxyphenyl)-1-butanone

 $[3329-02-0] C_{10}H_{12}O_4 mol. wt. 196.20$ OH Syntheses -Refer to: [151, 3280]. $Trimethyl ether [170489-31-3] C_{13}H_{18}O_4 mol. wt. 238.28$

-Obtained by treatment of ethyl 3-(3,4,5-trimethoxyphenyl)-3-oxo-2-propyl-1-propanoate with 5 % alc. KOH for 1 h at 60° [151]. -Also refer to: [392, 1424–1426].

b.p.₂₀ 218–220° [392]; long needles [392]; m.p. 51–52.5° [392]; ¹H NMR [1894], ¹³C NMR [1894], IR [1894], MS [1894].

p-Nitrophenylhydrazone of the trimethyl ether C₁₉H₂₃N₃O₅ mol. wt. 373.41

dark brownish red needles [392]; m.p. 160° [392].

1.2 Substituted Hydroxyketones

1-(3-Bromo-2-chloro-4-hydroxyphenyl)-1-butanone

[1134-09-4]

Br

C1

OH

 $C_{10}H_{10}BrClO_2$

mol. wt. 277.54

Syntheses

-To n-butyryl chloride, 2-bromo-3-chloroanisole and carbon disulfide was added in small portions at 25°, aluminium chloride; the mixture stirred 1 h at r.t. and 45 min at 55°; pentane and $CO(CH_2)_2CH_3$ aluminium chloride added, heated 3 h at 80° [2056].

-Also refer to: [2047, 2048, 2766, 2767 (77 %)].

m.p. 107-108.5° [2047, 2048, 2056, 2766, 2767].

1-(3-Bromo-5-fluoro-2-hydroxyphenyl)-1-butanone

[1957-56-8]	$C_{10}H_{10}BrFO_2$	mol. wt. 261.09
ОН	Synthesis	
Br CO(CH ₂) ₂ CH ₃	-Obtained by Fries rearrangement of 2	2-bromo-4-fluoro-
	phenyl butyrate with aluminium chl	oride at 130–140°
Ŷ	for 3 h (63 %) [1550].	
F	b.p. ₂ 125–130° [1550].	

2,4-Dinitrophenylhydrazone [1995-70-6] C₁₆H₁₄BrFN₄O₅ mol. wt. 441.21

m.p. 187–189° [1550].

1-(3-Bromo-5-fluoro-4-hydroxyphenyl)-1-butanone

[586-03-8]	$C_{10}H_{10}BrFO_2$	mol. wt. 261.09
F Br $CO(CH_2)_2CH_3$	Synthesis -Obtained by reaction of bromine with butyrophenone in acetic acid [516]. m.p. 97° [516].	3-fluoro-4-hydroxy-

1-(5-Bromo-2-hydroxy-3-iodophenyl)-1-butanone

[883566-10-7]	$C_{10}H_{10}BrIO_2$	mol. wt. 368.99
$I \xrightarrow{OH} CO(CH_2)_2CH_3$ Br	Synthesis -Obtained by iodination of 2-hydrox phenone in the presence of iodine 95 % ethanol at 35–40° for 1.5 h (78	and iodic acid in

m.p. 127° [2422]; ¹H NMR [2422], IR [2422], MS [2422].

1-(3-Bromo-2,4-dihydroxy-5-nitrophenyl)-1-butanone

[103274-59-5]	$C_{10}H_{10}BrNO_5$	mol. wt. 304.10
Br, CO(CH ₂) ₂ CH ₃ HO, NO ₂	Synthesis -Obtained by reaction of bromine w 5-nitrobutyrophenone in hot acetic m.p. 120–121° [799].	

1-(5-Bromo-2,4-dihydroxy-3-nitrophenyl)-1-butanone

[103273-98-9]	$C_{10}H_{10}BrNO_5$	mol. wt. 304.10
OH	Synthesis -Obtained by reaction of	of broming with
	2,4-dihydroxy-3-nitrobutyroph	enone in acetic acid
HO	at r.t. for 2 h [799].	
Br	yellow needles [799]; m.p. 10)4–105° [799].

1-(3,5-Dibromo-2-hydroxyphenyl)-1-butanone

[22362-68-1]	$C_{10}H$	$_{10}\mathrm{Br_2O_2}$	mol. wt. 322.00
Br CO(C	ium chlori 150° for 2 -Also obta 2,4-dibron	hophenyl butyrate in de at 160–165° for 3 h (43 %) [659]. ined by reaction of	rearrangement of the presence of alumin- 0 min (68 %) [647] or at butyric anhydride with ence of aluminium chlo- 559].
-Also refer to: [3	375, 998].		
b.p. _{3.5} 155–16	60° [659]; m.p. 71–7	2° [647].	
Benzoate	[101602-22-6]	$C_{17}H_{14}Br_2O_3$	mol. wt. 426.10
m.p. 56–58° [[659].		
Oxime	[99070-35-6]	$C_{10}H_{11}Br_2NO_2$	mol. wt. 337.01
m.p. 178° (d) [659].			
Semicarbazone	[105041-43-8]	C ₁₁ H ₁₃ Br ₂ N ₃ O	mol. wt. 379.05
m.p. $> 280^{\circ}$	[659].		

mol. wt. 336.02

mol. wt. 428.12

mol. wt. 216.64

1-(3,5-Dibromo-4-hydroxyphenyl)-1-butanone

[2904-87-2]	$C_{10}H_{10}Br_2O_2$	mol. wt. 322.00
Br, Br CO(CH ₂) ₂ CH ₃	Syntheses -Obtained by reaction of bromine with phenone in dilute acetic acid [516]. -Also obtained by adding an aqueous so and potassium bromide to a solution of phenone in acetone at r.t. [2001].	olution of bromine

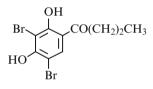
 $C_{11}H_{12}Br_2O_2$

m.p. 117° [516, 1762, 2001]; UV [1995].

-Refer to: [516]; m.p. 53° [516].

1-(3,5-Dibromo-2,4-dihydroxyphenyl)-1-butanone

 $C_{10}H_{10}Br_2O_3$ mol. wt. 338.00



Methyl ether

Syntheses
-Obtained by direct bromination of 2,4-dihydroxy-
butyrophenone with bromine,
*in acetic acid for 2 h at r.t. [1128] or in 80 % acetic
acid solution (10 %) [449];
*in chloroform [860].

yellow needles [860]; m.p. 113° [1128], 108–109° [449], 100° [860].

Phenylhydrazone

[2585-70-8]

m.p. 155° [1128].

1-(3-Chloro-5-fluoro-2-hydroxyphenyl)-1-butanone

•	1	
	ŌН	Syn
Cl	CO(CH ₂) ₂ CH ₃	-Ob
	(J	phe
	Ý	for
	F	b.p.

Synthesis -Obtained by Fries rearrangement of 2-chloro-4-fluorophenyl butyrate with aluminium chloride at 130–140° for 3 h (94 %) [1550]. b.p._{0.5} 122° [1550].

2,4-Dinitrophenylhydrazone [1995-74-0] $C_{16}H_{14}ClFN_4O_5$ mol. wt. 396.76

 $C_{16}H_{16}Br_2N_2O_2$

 $C_{10}H_{10}ClFO_2$

m.p. 187–189° [1550].

1-(5-Chloro-2-hydroxy-3-iodophenyl)-1-butanone

[883566-09-4]	$C_{10}H_{10}CIIO_2$	mol. wt. 324.54
OH I CO(CH ₂) ₂ CH ₃	Synthesis -Obtained by iodination of 2-hydro phenone in the presence of iodine a ethanol at 35–40° for 1.5 h (76 %) m.p. 163° [2422]; ¹ H NMR [2422], IR [2422], MS [2	nd iodic acid in 95 % [2422].

1-(2,3-Dichloro-4-hydroxyphenyl)-1-butanone

[2350-46-1]

$$C_{10}H_{10}Cl_2O_2$$
 mol. wt. 233.10



Syntheses

-To n-butyryl chloride, 2,3-dichloroanisole and carbon disulfide was added aluminium chloride, in small portions at 25° . The mixture stirred 1 h at r.t. and 45 min at 55° . Pentane and aluminium chloride added, heated 3 h at 80° (**IVa**) (78 %) [2056], (**IV**) (69 %) [2059].

-Also refer to: [940, 1339, 2047, 2049, 2050, 2054, 2055, 2060, 2061, 2767 (85 %), 2929].

b.p._{0.5} 140–142° [2929]; m.p. 109–110.5° [2047, 2049, 2050, 2056, 2767], 109–110° [2054, 2055, 2059–2061], 105–107° [940], 85–86° [2929].

Methyl ether [41715-70-2] C₁₁H₁₂Cl₂O₂ mol. wt. 247.12

-Preparation by reaction of butyryl chloride with 2,3-dichloroanisole in the presence of aluminium chloride (77 %) [3333], (90 %) [742]. -Also refer to: [732, 734–736, 738, 739, 2052].

m.p. 43-44° [3333], 42-44° [732, 734-736, 738, 739, 2052].

1-(2,4-Dichloro-6-hydroxyphenyl)-1-butanone

[1133-34-2]	$C_{10}H_{10}Cl_2O_2$	mol. wt. 233.10
ОН	Syntheses	
CO(CH ₂) ₂ CH ₃	-Refer to: [2060, 2767 (51 %)].	
	b.p. _{0.15} 101–102° [2047, 2767];	
Cl	m.p. 47–48.5° [2047, 2048, 2058, 20	50, 2766, 2767].

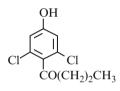
1-(2,6-Dichloro-3-hydroxyphenyl)-1-butanone

Methyl ether
$$[5862-11-3]$$
 $C_{11}H_{12}Cl_2O_2$ mol. wt. 247.12

yellowish oil [2767].

1-(2,6-Dichloro-4-hydroxyphenyl)-1-butanone

[1133-33-1]



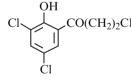
Syntheses -To n-butyryl chloride, 3,5-dichloroanisole and carbon disulfide was added aluminium chloride, in small portions at 25° . The mixture stirred 1 h at r.t. and 45 min at 55° . Pentane and aluminium chloride added, heated 3 h at 80° [2056].

-Also refer to: [2047, 2048, 2766, 2767 (10 %)].

b.p._{0.10} 162–163° [2047, 2767]; m.p. 75.5–76.5° [2047, 2048, 2056, 2766, 2767].

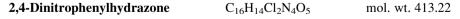
1-(3,5-Dichloro-2-hydroxyphenyl)-1-butanone

[81141-14-2]	$C_{10}H_{10}Cl_2O_2$	mol. wt. 233.10
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Syntheses .CO(CH₂)₂CH₃ -Obtained by Fries rearrangement of 2,4-dichlorophenyl butyrate with aluminium chloride at 170° for 40 min (68 %) [646]. -Also refer to: [563 (70 %), 564, 1159, 1372, 2956].

m.p. 49–50° [646], 47–48° [1159], 47° [563]; ¹H NMR [563], IR [563, 2957], UV [2956].



m.p. 226-227° [1159].

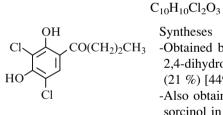
1-(3,5-Dichloro-4-hydroxyphenyl)-1-butanone

[129527-09-9]	C ₁	$_0H_{10}Cl_2O_2$	mol. wt. 233.10
CI CI CO(CH ₂	•	n aluminium chloride].	of 2,6-dichlorophenyl for 1 h at 140–150°
Na salt	[129527-10-2]	C ₁₀ H ₉ Cl ₂ O ₂ Na	mol. wt. 255.07

1-(4,5-Dichloro-2-hydroxyphenyl)-1-butanone

[71290-02-3]	$C_{10}H_{10}Cl_2O_2$	mol. wt. 233.10
OH CI CI CI	Synthesis -Obtained by condensation 3,4-dichlorophenol [2958]. m.p. 75–76° [2958].	of butyryl chloride on

1-(3,5-Dichloro-2,4-dihydroxyphenyl)-1-butanone



CO(CH₂)₂CH₃ -Obtained by reaction of an excess of chlorine with 2,4-dihydroxybutyrophenone in 80 % acetic acid (21 %) [449]. -Also obtained by treatment of 4-chloro-6-butyrylresorcinol in ether with a slow stream of chlorine gas

m.p. 124–125° [2141], 110.5–111° [449].

1-(4-Fluoro-2-hydroxy-5-nitrophenyl)-1-butanone

[120259-64-5]	$C_{10}H_{10}FNO_4$	mol. wt. 227.19
$\begin{array}{c} OH \\ F \\ NO_2 \end{array} CO(CH_2)_2CH_3 \end{array}$	Synthesis -Refer to: [958].	

for 1 h (36 %) [2141].

mol. wt. 249.10

1-(4-Hydroxy-3,5-diiodophenyl)-1-butanone

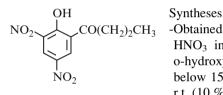
[340317-30-8]	$C_{10}H_{10}I_2O_2$	mol. wt. 415.99
OH I I CO(CH ₂) ₂ CH ₃	Syntheses -Obtained by reaction of iodine with none in ethanol in the presence oxide [516]. -Also refer to: [2741]. m.p. 115° [2741], 106° [516].	

Methyl ether	$C_{11}H_{12}I_2O_2$	mol. wt. 430.02
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colourless prisms [516]; m.p. 79° [516].

1-(2-Hydroxy-3,5-dinitrophenyl)-1-butanone

 $C_{10}H_{10}N_2O_6$ mol. wt. 254.20



 $\rm CO(\rm CH_2)_2\rm CH_3$ -Obtained by slowly adding a cold solution of 90 % HNO3 in acetic anhydride to a cold solution of o-hydroxy-butyrophenone in acetic anhydride at below 15°. The mixture was then stirred for 1 h at r.t. (10 %) [116].

-Also refer to: [1078].

yellow needles [116]; m.p. 118–120° [116]; ¹H NMR [116], IR [116]; TLC [116, 1078]; HPLC [1078].

1-(3-Bromo-4-hydroxyphenyl)-1-butanone

OH Synthesis -Obtained (by-product) by reaction of butyryl chloride with Br 2-bromoanisole or 2-bromophenetole in the presence of aluminium chloride (XIX) [1334]. CO(CH₂)₂CH₃ m.p. 122° [1334].

Methyl ether C11H13BrO2 mol. wt. 257.13

-Preparation by reaction of butyryl chloride with 2-bromoanisole in the presence of aluminium chloride (X) [1334].

m.p. 71° [1334].

Ethyl ether $C_{12}H_{15}BrO_2$ mol. wt. 271.15

-Preparation by reaction of butyryl chloride with 2-bromophenetole in the presence of aluminium chloride (**XIII**) [1334].

m.p. 57° [1334].

1-(4-Bromo-2-hydroxyphenyl)-1-butanone

	$C_{10}H_{11}BrO_2$		mol. wt. 243.10
OH CO(CH ₂) ₂ CH ₃	Synthesis -Refer to: [132]. m.p. 31–32° [132];	¹ H NMR [132].	

1-(5-Bromo-2-hydroxyphenyl)-1-butanone

 $\begin{array}{cccc} [105211-80-1] & C_{10}H_{11}BrO_2 & \text{mol. wt. } 243.10 \\ \\ OH & Syntheses \\ -Obtained by Fries rearrangement of 4-bromophenyl buty-rate with aluminium chloride [1701, 2797], [1640] (56 \%). \\ b.p._{0.35} 104-106^{\circ} [1640], b.p._{3} 127-132^{\circ} [1701]; \\ m.p. 53.6^{\circ} [1701]; & IR [1640]. \end{array}$

1-(3-Bromo-2,4-dihydroxyphenyl)-1-butanone

[1204738-04-4]	$C_{10}H_{11}BrO_3$	mol. wt. 259.10
Br, CO(CH ₂)CH ₃ HO	Synthesis -Refer to: [1672]. MS [1672].	

1-(3-Bromo-2,6-dihydroxyphenyl)-1-butanone

[99070-24-3]	$C_{10}H_{11}BrO_3$	mol. wt. 259.10
	Synthesis -Obtained by decarboxylation 2,4-dihydroxy-3-(1-oxobutyl)]benzoic	
Y `OH Br	yellow fibrous needles [2811]; m.p. 108–109° [2811].	

075 10

1-(4-Bromo-2,5-dihydroxyphenyl)-1-butanone

 $\begin{array}{cccc} [52376-23-5] & C_{10}H_{11}BrO_3 & \text{mol. wt. } 259.10 \\ & OH & Synthesis \\ & OH & Obtained by Fries rearrangement of 2-bromohydroquinone dibutyrate with aluminium chloride at 170–180° for 2 h [1105]. \\ & \text{m.p. } 98–99° [1105]. \end{array}$

Semicarbazone [52376-24-6] $C_{11}H_{14}BrN_3O_3$ mol. wt. 316.15

m.p. 198° [1105].

1-(5-Bromo-2,3,4-trihydroxyphenyl)-1-butanone

	$C_{10}H_{11}BrO_4$			mol. wt. 2	2/5.10
HO HO Br	Synthesis -Obtained 4-butyroylp m.p. 137° [5	oyroga			with

1-(2-Chloro-3-hydroxyphenyl)-1-butanone

[1201-04-3]	$C_{10}H_{11}ClC$	D_2	mol. wt. 198.65
OH Cl CO(CH ₂) ₂ CH ₃		48, 2766, 2767 (88 %) 47, 2048, 2766, 2767	
Methyl ether	[1133-58-0]	$C_{11}H_{13}ClO_2$	mol. wt. 212.68

 $\begin{array}{l} b.p._{22} \ 174{-}180^\circ \ [2047, \ 2048, \ 2767]; \\ n_D^{23} = 1.5375 \ [2047, \ 2048, \ 2767]. \end{array}$

1-(2-Chloro-4-hydroxyphenyl)-1-butanone



Syntheses -To n-butyryl chloride, 3-chloroanisole and carbon disulfide was added aluminium chloride, in small portions at 25°. The mixture stirred 1 h at r.t. and 45 min at 55°. Pentane and aluminium chloride added, heated 3 h at 80° [2056]. -Also obtained from its 2-propynyl ether by palladium-catalyzes deprotection [2378].

-Also refer to: [2047, 2048, 2055, 2058, 2060, 2765-2767].

b.p._{0.1} 100–110° [2061], b.p._{0.2} 155–175° [2054], b.p._{0.03} 160–178° [2767]; m.p. 82.5–84° [2047, 2048, 2055, 2056, 2058, 2060, 2765–2767].

Methyl ether[4070-68-2] $C_{11}H_{13}ClO_2$ mol. wt. 212.68

-Refer to: [2047, 2767].

b.p._{1.5-2.9} 122–138° [2047].

2-Propynyl ether [500127-72-0] $C_{13}H_{13}ClO_2$ mol. wt. 236.70 -Obtained by reaction of propargyl bromide with 2-chloro-4-hydroxybutyrophenone [2378].

1-(3-Chloro-2-hydroxyphenyl)-1-butanone

[60474-43-3]	$C_{10}H_{11}ClO_2$	mol. wt. 198.65	
CI CO(CH ₂) ₂ CH ₃	Syntheses -Obtained by Fries rearrangement of o-chlorophenyl butyrate with aluminium chloride for 2 h at 110° (36.5 %) [2799]. -Also refer to: [2423].		
m.p. 118° [2799].			
Semicarbazone [1	.00130-01-6] C ₁₁ H ₁₄ C	$M_{3}O_{2}$ mol. wt. 255.70	

m.p. 142° [2799].

1-(3-Chloro-4-hydroxyphenyl)-1-butanone

 $[500127-73-1] C_{10}H_{11}ClO_2 mol. wt. 198.65$



Syntheses -Obtained (by-product) by reaction of butyryl chloride with 2-chloroanisole or 2-chlorophenetole in the presence of aluminium chloride (**XVI**) [1334].

 $CO(CH_2)_2CH_3$ -Also refer to: [2767].

m.p. 123.5–124° [2378], 122° [1334], 82.5–84° [2061]; ¹H NMR [2378], IR [2378].

Methyl ether [868075-01-8] C₁₁H₁₃ClO₂ mol. wt. 212.68

-Preparation by reaction of butyryl chloride with 2-chloroanisole in the presence of aluminium chloride (**III**) [1334].

m.p. 71° [1334].

Ethyl ether

$C_{12}H_{15}ClO_2$ mol. wt. 226.70

-Preparation by reaction of butyryl chloride with 2-chlorophenetole in the presence of aluminium chloride (VI) [1334].

m.p. 72° [1334].

1-(4-Chloro-2-hydroxyphenyl)-1-butanone

[4133-95-3]	$C_{10}H_{11}ClO_2$	mol. wt. 198.65
ОН	Syntheses	
$CO(CH_2)_2CH_3$	-Preparation by Fries rearrangement	of m-chlorophenyl
	butyrate with aluminium chloride,	
	*without solvent for 2 h at 130° (80 G	%) [2802] or for 3 h

at 140–150° (87 %) [2432]; *in nitrobenzene at 25° for 6 h (83 %) [2802]. -Also refer to: [2428, 2429, 2767].

b.p._{0.03} 145° [2047, 2767], b.p._{0.03} 160–178° [2055]; m.p. 51° [2432, 2802]; ¹H NMR [2432], IR [2432].

Methyl ether	[4070-69-3]	$C_{11}H_{13}ClO_2$	mol. wt. 212.68
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-Obtained by methylation of the above ketone in the usual way (90 %) [2802]. -Also refer to: [2767].

 $C_{10}H_{11}ClO_2$

b.p.₄₅ 135° [2802].

1-(5-Chloro-2-hydroxyphenyl)-1-butanone

[51978-33-7]

OH

Syntheses

CO(CH₂)₂CH₃ -Preparation by Fries rearrangement of 4-chlorophenyl butyrate with aluminium chloride [3170], (59.5 %) [1640], *without solvent [3325] at 155° for 30 min [1702] or at 150–160° for 30 min [1701];

mol. wt. 198.65

*in nitrobenzene, first at r.t. overnight, then at $50-60^{\circ}$ for 8 h [1702].

-Also obtained by reaction of butyric acid with 4-chlorophenol in the presence of boron trifluoride at 150° for 5 h (86 %) [1684]. -Also refer to: [1686, 2956].

b.p.₃ 108–112° [1702], b.p._{0.8} 115–118° [1640], b.p.₃ 127–132° [1701], b.p.₁₈ 151–152° [1684]; m.p. 55° [1684], 53.6° [1701], 50.5° [1702, 3170], 49–50° [3325]; IR [1640, 1684, 2957], (Sadtler standard N° 8982), UV [2956, 3170].

Methyl ether

$C_{11}H_{13}ClO_2$

mol. wt. 212.68

b.p. 98-105° [1166].

1-(3-Chloro-2,6-dihydroxyphenyl)-1-butanone

[99070-83-4]	$C_{10}H_{11}ClO_3$	mol. wt. 214.65
ОН	Syntheses	
CO(CH ₂) ₂ CH ₃	-Obtained by decarboxylation	of 1-[5-chloro-
	2,4-dihydroxy-3-(1-oxobutyl)]benzoic	acid [2811].
ОН	-Also obtained by hydrolysis of 8	-butyryl-7-hydroxy-
Cl	6-chloro-4-methylcoumarin with	12 % sodium
	hydroxide [2811].	

yellow needles [2811]; m.p. 115° [2811].

Dibenzoate		[102665-28-1]		$C_{24}H_{19}ClO_5$	mol.	wt. 422.86
1 1	1.	[0011]	1000 5001	4.7		

colourless plates [2811]; m.p. 123° [2811].

1-(5-Chloro-2,4-dihydroxyphenyl)-1-butanone

 $[90919-46-3] C_{10}H_{11}ClO_3 mol. wt. 214.65$ $OH CO(CH_2)_2CH_3 OD CO(CH_2)_2CH$

*at 140–145° for 3 min [586]. -Also refer to: [1052].

b.p.₂₂ 180–195° [2141]; m.p. 96° [586], 84–85° [2141].

BIOLOGICAL ACTIVITY: Bactericide [1052].

$\textbf{2,4-Dinitrophenylhydrazone} \quad [92907-10-3] \quad C_{16}H_{15}ClN_4O_6 \quad mol. \ wt. \ 394.77$

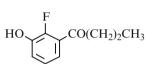
m.p. 235° [586].

mol. wt. 182.20

1-(3-Chloro-2,4,6-trihydroxyphenyl)-1-butanone

[1245818-23-8]	$C_{10}H_{11}ClO_4$	mol. wt. 230.65
CI CO(CH ₂) ₂ CH ₃ HO OH	Synthesis -Obtained by reaction of 2,4,6-trihy with sulfuryl dichloride in ethan 0° for 0.5 h [2141]. ¹ H NMR [1676], ¹³ C NMR [1676];	ol/chloroform at

1-(2-Fluoro-3-hydroxyphenyl)-1-butanone



Synthesis CO(CH₂)₂CH₃ -Refer to: [1516]. m.p. 85–87° [1516].

 $C_{10}H_{11}FO_2$

1-(2-Fluoro-4-hydroxyphenyl)-1-butanone

$$\begin{array}{c} C_{10}H_{11}FO_2 & \text{mol. wt. 182.20} \\ F & Synthesis \\ -Refer to: [2233]. \\ HO & HO \end{array}$$

1-(3-Fluoro-2-hydroxyphenyl)-1-butanone

[949902-14-1]	$C_{10}H_{11}FO_2$	mol. wt. 182.20
$CO(CH_2)_2CH_3$	Syntheses -Refer to: [310, 311].	
	Methyl ether [949902-16-3]	
\sim	$C_{11}H_{13}FO_2$	mol. wt. 196.22

-Refer to: [310, 311].

1-(3-Fluoro-4-hydroxyphenyl)-1-butanone



Syntheses

-Obtained by total demethylation of 3-fluoro-4-methoxy-butyrophenone by treatment in boiling pyridinium chloride (62 %) [517].

CO(CH₂)₂CH₃ -Also obtained by reaction of butyryl chloride with 2-fluoroanisole in the presence of aluminium chloride [918].

m.p. 91° [918], 90° [517]; ¹H NMR [918].

Methyl ether [347-65-9] C₁₁H₁₃FO₂ mol. wt. 196.22

-Obtained by reaction of butyryl chloride with o-fluoroanisole in the presence of aluminium chloride,

*in carbon disulfide (90 %) [517]; *in nitrobenzene at 110° [671].

-Also refer to: [672, 3158].

b.p.₁₆ 161–162° [517]; m.p. 57–58° [671, 672], 55° [517].

2,4-Dinitrophenylhydrazone $C_{17}H_{17}FN_4O_6$ mol. wt. 392.34 of the methyl ether

m.p. 188° [517].

1-(5-Fluoro-2-hydroxyphenyl)-1-butanone

[575-67-7]	$C_{10}H_{11}FO_2$	mol. wt. 182.20
CO(CH ₂) ₂ CH ₃	Syntheses Obtained by Fries rearrangement rate with aluminium chloride, *without solvent at 155° for 30 m *in 1,2-dichloroethane at 100° for 2	in (83 %) [2991];

N.B.: Industrial preparation from 500 kg of the ester (Sanofi industries, Aramon, Fr.). -Also obtained by reaction of butyric acid with 4-fluorophenol in the presence of boron trifluoride in a sealed tube for 3 h at 125° (80 %) [1684].

-Also obtained by reaction of p-fluorophenetole, first treated with aluminium chloride in benzene, with butyryl chloride in the presence of aluminium chloride at $150-160^{\circ}$ for 60-90 min (25 %) [2991].

-Also refer to: [1686].

b.p.₈ 105–114° [2991], b.p.₁₄ 116–118° [2991], b.p.₂₀ 130–131° [1684]; m.p. 39° [1998], 38–39° [1684, 2991]; ¹H NMR [1998], IR [1998], UV [1998], MS [1998].

2,4-Dinitrophenylhydrazone [100697-02-7] C₁₆H₁₅FN₄O₅ mol. wt. 362.32

m.p. 216° [1998].

1-(3-Hydroxy-2-iodophenyl)-1-butanone

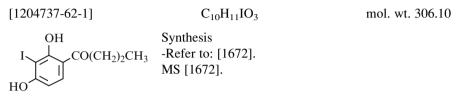
 $\begin{array}{c} C_{10}H_{11}IO_2 & \text{mol. wt. } 290.10 \\ \hline OH & Synthesis & \\ \hline I & -Refer to: [73]. \\ \hline Methyl ether & [213387-03-2] \\ CO(CH_2)_2CH_3 & C_{11}H_{13}IO_2 & \text{mol. wt. } 304.13 \\ \hline \end{array}$

-Refer to: [73] (76 %).

N.B.: Regioselective directed *meta*-acylation of aromatic compounds *via* cycloaddition of nitriles to benzyne-zirconocene complexes.

reddish orange oil [73]; ¹H NMR [73], ¹³C NMR [73], IR [73], MS [73].

1-(2,4-Dihydroxy-3-iodophenyl)-1-butanone



1-(2-Hydroxy-3-nitrophenyl)-1-butanone

 $[91992-00-6] C_{10}H_{11}NO_4 mol. wt. 209.20$ OH Syntheses -Obtained by treatment of 3-ethyl-8-nitrochromone with potassium hydroxide [785].

-Also obtained by slowly adding a cold solution of 90 % HNO_3 in acetic anhydride to a cold solution of o-hydroxybutyrophenone in acetic anhydride at below 15°. The mixture was then stirred for 1 h at r.t. (30 %) [116]. -Also refer to: [116, 786, 1078].

yellow needles [116]; m.p. 108–110° [785, 786], 43–45° [116]; **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [116], IR [116]; TLC [116, 1078]; HPLC [1078].

1-(2-Hydroxy-4-nitrophenyl)-1-butanone

[90922-78-4]	$C_{10}H_{11}NO_4$
OH	Syntheses
$CO(CH_2)_2CH_3$	-Obtained by Fries r
	butyrate with alumi
NO ₂	150 min (3.5–5 %) [
	-Also refer to: [2921]

m.p. 63.5-64° [3009].

C₁₀H₁₁NO₄ mol. wt. 209.20

-Obtained by Fries rearrangement of 3-nitrophenyl butyrate with aluminium chloride at $135-140^{\circ}$ for 150 min (3.5–5 %) [3009]. -Also refer to: [2921].

Phenylhydrazone	[94708-70-0]	$C_{16}H_{17}N_3O_3$	mol. wt. 299.33
m.p. 179–180° [3009)].		

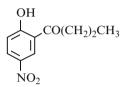
Benzyl ether	[1192824-04-6]	$C_{17}H_{17}NO_4$	mol. wt. 299.33
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-Obtained by reaction of benzyl chloride with 2-butanoyl-5-nitrophenol [2921].

1-(2-Hydroxy-5-nitrophenyl)-1-butanone

Syntheses

$$C_{10}H_{11}NO_4$$
 mol. wt. 209.20



-Obtained by slowly adding a cold solution of 90 % HNO_3 in acetic anhydride to a cold solution of o-hydroxy-buty-rophenone in acetic anhydride at below 15°. The mixture was then stirred for 1 h at r.t. (60 %) [116].

-Also refer to: [1078, 2415].

yellow needles [116]; m.p. 108–110° [116]; ¹H NMR [116], IR [116]; TLC [116, 1078]; HPLC [1078].

USE: Formation of chelates with Cu(II), Ni(II), Co(II) and Zn(II) [2415].

Diphenylmethyl ether [508210-78-4] $C_{23}H_{21}NO_4$ mol. wt. 375.42 -Refer to: [2020]; ¹H NMR [2019].

1-(4-Hydroxy-3-nitrophenyl)-1-butanone

[82350-83-2] C₁₀H₁₁NO₄ mol. wt. 209.20



Syntheses -Obtained by treatment of 4-chloro-3-nitrobutyrophenone with boiling 6 % aqueous potassium hydroxide [2147]. -Also obtained by reaction of butyryl chloride with o-nitro-

phenol in the presence of aluminium chloride in nitrobenzene for 2.5 h at $55-60^{\circ}$, then at r.t. overnight (43 %) [465].

pale yellow needles [2147]; m.p. 47.6–48.2° [465], 46° [2147].

2,4-Dinitrophenylhydrazone	$C_{16}H_{15}N_5O_7$	mol. wt. 389.32

m.p. 192.4–192.8° [465].

4-Nitrophenylhydrazone $C_{16}H_{16}N_4O_5$ mol. wt. 344.33

masses of orange crystals [2147]; m.p. 203–204° [2147].

Methyl ether
$$[1032174-10-9]$$
 $C_{11}H_{13}NO_4$ mol. wt. 223.23

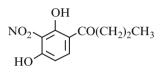
-Obtained by adding potassium nitrate to an ice-cold solution of 4-methoxybutyrophenone in sulfuric acid, then the reaction was run at r.t. for 8-15 h (61 %) [2243].

white solid [2243]; m.p. 69–70° [2243]; ¹H NMR [2243].

Syntheses

1-(2,4-Dihydroxy-3-nitrophenyl)-1-butanone

$$[103205-61-4]$$
 C₁₀H₁₁NO₅ mol. wt. 225.20



-Obtained by reaction of butyric anhydride (1.1 mol) with 2-nitroresorcinol (1 mol) in the presence of aluminium chloride (3.3 mol) in nitrobenzene at 100° [105].

-Also obtained by reaction of butyric anhydride with 2-nitroresorcinol in the presence of aluminium chloride in nitrobenzene on a boiling water bath for 3 h (46 %) [799].

m.p. 90–91° [799], 90° [105].

1-(2,4-Dihydroxy-5-nitrophenyl)-1-butanone

 $[103204-42-8] C_{10}H_{11}NO_5$ mol. wt. 225.20 OH CO(CH₂)₂CH₃ Syntheses -Obtained by reaction of nitric acid (d = 1.40–1.42) with resbutyrophenone, *without solvent, first at 0°, then at r.t. (92 %) [166]; *in acetic acid, first at 0°, then at 45° (40 %) [799].

pale yellow needles [799], yellow needles [166]; m.p. 121.5–122° [166], 120–121° [799].

Dimethyl ether	[100137-55-1]	$C_{12}H_{15}NO_5$	mol. wt. 253.25
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-Obtained by reaction of dimethyl sulfate with 2,4-dihydroxy-5-nitrobutyrophenone in the presence of potassium carbonate in refluxing acetone for 12 h (70 %) [166].

m.p. 113–114° [166].

1-(2,6-Dihydroxy-3-nitrophenyl)-1-butanone

 $\begin{bmatrix} 103204-43-9 \end{bmatrix} & C_{10}H_{11}NO_5 & \text{mol. wt. } 225.20 \\ OH & Syntheses \\ OH & OH & OH & OH \\ OH & OH & CO(CH_2)_2CH_3 & Obtained by Fries rearrangement of 4-nitroresorcinol butyrate (1 mol) with aluminium chloride (3.3 mol) in nitrobenzene at 110–120° for 2 h or at r.t. for 72 h [104]. \\ \end{bmatrix}$

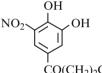
-Also obtained by heating a mixture of butyric anhydride and 4-nitroresorcinol in the presence of aluminium chloride in nitrobenzene for 3 h in a water bath [2224]. -Also obtained by reaction of nitric acid (d = 1.42) with 2,6-dihydroxybutyrophenone at 0° for 10 min [2224].

m.p. 78° [104, 2223, 2224].

1-(3,4-Dihydroxy-5-nitrophenyl)-1-butanone

 $C_{10}H_{11}NO_5$

mol. wt. 225.20



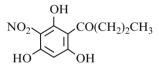
-Obtained by treatment of 4-hydroxy-3-methoxy-5-nitrobutyrophenone with pyridinium chloride at 200° for 40 min [324].

CO(CH₂)₂CH₃ m.p. 88–90° [324].

Synthesis

1-(2,4,6-Trihydroxy-3-nitrophenyl)-1-butanone

 $[119691-93-9] C_{10}H_{11}NO_6 mtext{mol. wt. } 241.20$



CO(CH₂)₂CH₃
 Obtained by adding hexane, then a mixture of concentrated sulfuric acid and fuming nitric acid at 0° to a solution of phlorobutyrophenone in concentrated sulfuric acid below 0° (70–80 %) [3414].

bright yellow needles [3414]; m.p. 93–94° [3414]; ¹H NMR [3414], IR [3414], MS [3414].

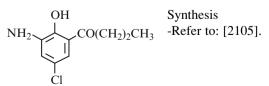
BIOLOGICAL ACTIVITY: Germination inhibitory activity [3414]; PET inhibition [3414].

1-(3-Amino-5-chloro-2-hydroxyphenyl)-1-butanone

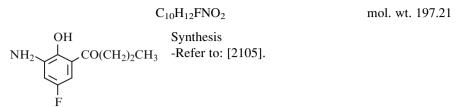
$C_{10}H_{12}CINO_2$

Synthesis

mol. wt. 213.66



1-(3-Amino-5-fluoro-2-hydroxyphenyl)-1-butanone



1-[2,4-Dihydroxy-5-(sulfooxy)phenyl]-1-butanone

	$C_{10}H_{12}O_7S$	mol. wt. 276.27
HO HO OSO ₃ H	HCl at 95° for 30 min	sis of its potassium salt with 3 N n (41 %) [166]. m.p. 147–148° [166].

Isolation from natural sources

-From urines of dogs and rats [166].

K salt

Potassium 5-butyryl-2,4-dihydroxyphenylsulfate

[116956-62-8]

C10H11O7SK

mol. wt. 314.36

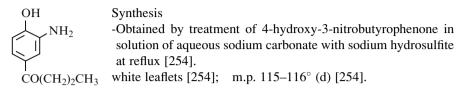
colourless plates [166]; m.p. 230–232° [166]; UV [166]; paper chromatography [166].

1-(2-Amino-5-hydroxyphenyl)-1-butanone

[404919-01-3]	$C_{10}H_{13}NO_2$	mol. wt. 179.22
NH ₂ CO(CH ₂) ₂ CH ₃	Synthesis -Refer to: [3331].	
OH	USE: Preparation of indazoles havin that of a thyroid hormone and method	od for the production

that of a thyroid hormone and method for the production thereof, and their use in medicaments [3331].

1-(3-Amino-4-hydroxyphenyl)-1-butanone



BIOLOGICAL ACTIVITY: Antibacterial [254].

1-(4-Amino-2-hydroxyphenyl)-1-butanone

$$\begin{array}{c} C_{10}H_{13}NO_2 & \text{mol. wt. 179.22} \\ OH & Syntheses \\ -Obtained by hydrolysis of 1-(4-acetylamino-2-hydroxy-phenyl)-1-butanone (SM) (m.p. 102°) \\ \text{with boiling 50 \% HCl [1552]. SM was prepared,} \end{array}$$

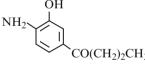
*by Friedel-Crafts reaction of n-butyryl chloride with m-acetylaminoanisole in the presence of an excess of aluminium chloride in refluxing 1,2-dichloroethane for 2 h (58 %) [1552];

*or by Fries rearrangement of m-acetylaminophenyl n-butyrate (m.p. 85°) with aluminium chloride (2 mol) for 4 h at 180° (46 %) [1552].

m.p. 139° [1552].

1-(4-Amino-3-hydroxyphenyl)-1-butanone

[123172-47-4]	$C_{10}H_{13}NO_2$	mol. wt. 179.22
OH	Syntheses	



-Obtained by treatment [424] of 6-butyrylbenzoxazolinone with boiling 10 % aqueous sodium CO(CH₂)₂CH₃ hydroxide for 4 h (60 %) [2161]. -Also refer to: [2162].

m.p. 118-120° [2161].

USE: Preparation of acylbenzoxazinon acetates [2162].

1-(5-Amino-2-hydroxyphenyl)-1-butanone

[99075-34-0]	$C_{10}H_{13}NO_2$	mol. wt. 179.22
OH CO(CH ₂) ₂ CH ₃ NH ₂	Syntheses -Obtained by hydrolyzing 5-acetamid phenone with boiling 50 % HCl [144 -Also refer to: [2029]. m.p. 53° [1449].	

mol. wt. 221.26

Acetate

m.p. 134° [1449].

BIOLOGICAL ACTIVITY: Glycosuric [1449]; Toxicity [1449].

C₁₂H₁₅NO₃

1-(5-Amino-2,4-dihydroxyphenyl)-1-butanone

$$\begin{array}{cccc} & & C_{10}H_{13}NO_3 & & mol. \ wt. \ 195.22 \\ & & \\ & & \\ & & \\ HO & & \\$$

-Obtained from 2,4-dimethoxy-5-nitrobutyrophenone by treatment with clean mossy zinc and HCl on the water bath (52 %) [166].

m.p. 75–76° [166].

1-[5-Bromo-2,4-dihydroxy-3-(1-oxobutyl)]benzoic acid

[99853-34-6] C₁₁H₁₁BrO₅ mol. wt. 303.11

Syntheses

$$CH_{3}(CH_{2})_{2}CO \xrightarrow{OH} CO_{2}H$$

$$HO \xrightarrow{Br}$$

-Obtained by adding butyric anhydride to a cold solution of methyl 2,4-dihydroxy-5-bromobenzoate and aluminium chloride in nitrobenzene. The mixture was left overnight at r.t., and then heated at $100-105^{\circ}$ for 4 h [2811].

-Also obtained by Friedel-Crafts butyrylation of 5-bromo- β -resorcylic acid [2811]. -Also obtained by hydrolysis of the methyl ester with 10 % NaOH on a water bath for 1 h and then kept overnight at r.t. [2811].

-Also obtained by heating 2-hydroxy-4-butyroxy-5-bromobenzoic acid (m.p. 146°) with aluminium chloride at $170-175^{\circ}$ for 1 h [2811].

needles [2811]; m.p. 211° (d) [2811].

 $\label{eq:methylester} {\mbox{Methyl ester}} \ \ [104780-38-3] \ \ \ C_{12}H_{13}BrO_5 \ \ \ mol. \ wt. \ 317.14$

-Obtained at the same time in the first reaction above mentioned (21 %) [2811].

yellow needles [2811]; m.p. 107° [2811].

Oxime of the methyl ether [100116-12-9] $C_{12}H_{14}BrNO_5$ mol. wt. 332.15

colourless granules [2811]; m.p. 166° (d) [2811].

5-(1-Oxobutyl)-2-hydroxybenzoyl chloride

$$\begin{array}{ccc} C_{11}H_{11}ClO_3 & \mbox{mol. wt. } 226.66 \\ OH & Synthesis & \\ \hline COCl & -Refer to: [523]. & \\ \hline Methyl \ ether & [64808-72-6] \\ C_{12}H_{13}ClO_3 & \mbox{mol. wt. } 240.68 \\ \hline CO(CH_2)_2CH_3 & \\ \end{array}$$

-Obtained by chlorination of 5-butyryl-2-methoxybenzoic acid [523].

m.p. 65° [523]; IR [523].

1-[5-Chloro-2,4-dihydroxy-3-(1-oxobutyl)]benzoic acid

 $[99854-28-1] C_{11}H_{11}ClO_5 mol. wt. 258.66$

 $CH_3(CH_2)_2CO$ CO_2H CO_2H Obtained $CH_3(CH_2)_2CO$ CO_2H CO_2H Cold soCold so5-chlorobbenzene

-Obtained by adding butyric anhydride to a cold solution of methyl 2,4-dihydroxy-5-chlorobenzoate and aluminium chloride in nitrobenzene. The mixture was left overnight at r.t., and then heated at $100-105^{\circ}$ for 4 h [2811].

-Also obtained Fries rearrangement of 2-hydroxy-4-butyroxy-5-chlorobenzoic acid (m.p. 145°) with aluminium chloride at 145° for 1 h [2811].

-Also obtained by hydrolysis of the methyl ester with 10 % NaOH on a water bath for 1 h and then kept overnight at r.t. [2811].

pale yellow needles [2811]; m.p. 203° (d) [2811].

Methyl ester [104851-85-6] C₁₂H₁₃ClO₅ mol. wt. 272.68

-Obtained at the same time in the first reaction above mentioned [2811].

yellow silky needles [2811]; m.p. 100° [2811].

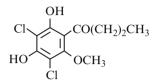
Oxime of the methyl ester [100116-82-3] $C_{12}H_{14}CINO_5$ mol. wt. 287.70 shining needles; m.p. 167° (d) [2811].

Dibenzoate of the methyl ester [102949-16-6] $C_{26}H_{21}ClO_7$ mol. wt. 480.90

colourless truncated needles; m.p. 101° [2811].

1-(3,5-Dichloro-2,4-dihydroxy-6-methoxyphenyl)-1-butanone

$$C_{11}H_{12}Cl_2O_4$$
 mol. wt. 279.12



Synthesis -Obtained by reaction of chlorine with 2,4-dihydroxy-6-methoxybutyrophenone in water [2012]. ¹H NMR [2012], MS [2012].

BIOLOGICAL ACTIVITY: Differentiation-inducing factor from the slime mould *Dictyostelium discoideum* [2012].

1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-1-butanone (*DIF-1*) (-2)

[118222-70-1]

CH₃O

OH

 $C_{11}H_{12}Cl_2O_4$ mol. wt. 279.12

Syntheses

-Obtained by reaction of sulfuryl chloride (1.5 equiv.) with 2,6-dihydroxy-4-methoxybutyr-ophenone in a methylene chloride/ethanol mixture at r.t. [1129].

-Also obtained by reaction of chlorine with 2,6-dihydroxy-4-methoxybutyrophenone in water [2012].

-Also refer to: [1772, 1773, 2341].

OH

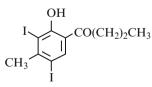
CO(CH₂)₂CH₃

Yellow amorphous solid [1129]; ¹H NMR [2012], MS [1129, 2012].

BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of *Dictyostelium* differentiation-inducing factors for their stalk-cell-inducing activity in *Dictyostelium* cells and antiproliferative activity in K562 human leukemic cells [1129]; Differentiation-inducing factor from the slime mould *Dictyostelium discoideum* [2012].

1-(2-Hydroxy-3,5-diiodo-4-methylphenyl)-1-butanone

$$C_{11}H_{12}I_2O_2$$
 mol. wt. 430.02



[883566-08-3]

Synthesis -Obtained by iodination of 2-hydroxy-4-methylbutyrophenone in the presence of iodine and iodic acid in 95 % ethanol at 35–40° for 1.5 h (81 %) [2422]. m.p. 98° [2422];

¹H NMR [2422], ¹³C NMR [2422], IR [2422], MS [2422].

3-Butyryl-4-hydroxybenzoic acid

[25065-15-0]	$C_{11}H_{12}O_4$	mol. wt. 208.21
$\bigcup_{CO_2H}^{OH} CO(CH_2)_2CH_3$	Synthesis -Obtained by hydrolysis of its ethyl ester m.p. 202° [967]. Acetate [23298-90-0] C ₁₃ H ₁₄ O ₅	[967]. mol. wt. 250.24

-Obtained by acetylation of 3-butyryl-4-hydroxybenzoic acid [967].

m.p. 113° [967].

Methyl ether	[258273-25-5]	$C_{12}H_{14}O_4$	mol. wt. 222.24
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-Obtained by methylation of 3-butyryl-4-hydroxybenzoic acid [967].

m.p. 185° [967].

Ethyl ether	[258273-42-6]	$C_{13}H_{16}O_4$	mol. wt. 236.27
Ethyl ester	C ₁₃ H ₁₆	O ₄	mol. wt. 236.27

-Obtained by reaction of butyryl chloride with ethyl 4-hydroxybenzoate in the presence of aluminium chloride in tetrachloroethane, then the reaction mixture was heated at 120° for 3–4 h [967].

b.p.₃₅ 180° [967]; $n_D^{34} = 1.5268$ [967].

5-Butyryl-2-hydroxybenzoic acid

[106393-53-7]	$C_{11}H_{12}O_4$	mol. wt. 208.21
OH COOH CO(CH ₂) ₂ CH ₃	Syntheses -Obtained by hydrolysis of methyl benzoate, *with boiling 20 % solution of potassium *with boiling dilute solution of sodium (81 %) [107].	hydroxide [730];

-Also obtained by alkaline hydrolysis of 5-butyrylsalicylamide [1158].

m.p. 168–170° [107], 153° [1158], 152–153° [730].

Methyl ester [36481-17-1] C₁₂H₁₄O₄ mol. wt. 222.24

-Obtained by Fries rearrangement of methyl 2-(butyryloxy)benzoate with aluminium chloride [523] in boiling carbon disulfide for 2 h, then the reaction mixture heated for a few min after solvent elimination [730]. -Also refer to: [107, 689 (91 %), 700] (79 %)].

m.p. 73° [730], 70.5–71.5° [689], 70–71° [700], 70° [523]; IR [523].

Methyl ether of the methyl ester [64808-71-5] $C_{13}H_{16}O_4$ mol. wt. 236.27

-Obtained by methylation of the above methyl ester [523].

m.p. 72° [523]; IR [523].

-Obtained by saponification of its above methyl ester [523].

m.p. 105–106° [523]; IR [523].

1-(3-Bromo-2-hydroxy-5-methylphenyl)-1-butanone

[727687-84-5]	$C_{11}H_{13}BrO_2$	mol. wt. 257.13
$\begin{array}{c} OH\\ Br \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\$	Synthesis -Obtained by reaction of bron 5-methyl-butyrophenone in rr acid (69 %) [375]. yellow crystals [375]; m.p. 71	efluxing dilute acetic

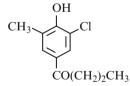
¹H NMR [375], IR [375].

1-(3-Chloro-2-hydroxy-5-methylphenyl)-1-butanone

	$C_{11}H_{13}ClO_2$	mol. wt. 212.68
$\begin{array}{c} OH\\ Cl & \downarrow\\ & \downarrow\\ & \downarrow\\ & CH_3 \end{array} CO(CH_2)_2CH_3$		rearrangement of 2-chloro- ate with aluminium chloride at titative yield) [2647].

1-(3-Chloro-4-hydroxy-5-methylphenyl)-1-butanone

 $C_{11}H_{13}ClO_2$ mol. wt. 212.68



Synthesis

-Obtained from wastes in manufacturing of 4-chloro-2-methylphenol [2762].

1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-butanone

[408309-74-0]	$C_{11}H_{13}ClO_2$	mol. wt. 212.68
CH ₃ CH ₃ CH ₃ CH ₃ CI	Syntheses -Obtained by reaction of buty 3-methylphenol in the presen *at 100° for 1 h in a sealed tu *at 70–80° for 2 h (80 %) [16	ce of boron trifluoride, be (82 %) [1684];

-Also obtained by Fries rearrangement of 4-chloro-3-methylphenyl butyrate in the presence of aluminium chloride at 120° for 10 min (92 %) [2647].

m.p. $61-62^{\circ}$ [2647], 61° [1684]; IR (Sadtler standard N° 8987) [1684].

1-[(3-Chloromethyl)-4-hydroxyphenyl]-1-butanone

[56490-86-9]

 $C_{11}H_{13}ClO_2$

mol. wt. 212.68



OH CH₂Cl CO(CH₂)₂CH₃

-Obtained by adding 4-hydroxybutyrophenone in dioxane to a 37 % formaldehyde solution and 11 N hydrochloric acid. Then, the mixture was heated at 50–60° for 1 h (55 %) [1573]. m.p. 121–123° [1573]; ¹H NMR [1573].

1-(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)-1-butanone (DIF-3)(-2)

[861889-88-5]

CH₃O

C11H13ClO4 mol. wt. 244.67

Syntheses

-Obtained by reaction of sulfuryl chloride (1.5 $CO(CH_2)_2CH_3$ equiv.) 2,6-dihydroxy-4-methoxybutyrwith ophenone in a methylene chloride/ethanol mixture at r.t. [1129].

-Also refer to: [1772].

OH

OH

colourless amorphous solid [1129]; MS [1129].

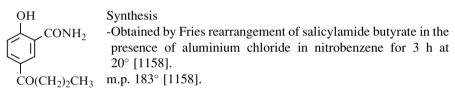
BIOLOGICAL ACTIVITY: Structural requirements of Dictyostelium factors differentiation-inducing for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

1-(2-Hydroxy-5-iodo-3-methylphenyl)-1-butanone

[883566-11-8]	$C_{11}H_{13}IO_2$	mol. wt. 304.13
CH ₃ I CO(CH ₂) ₂ CH ₃	Syntheses -Obtained by iodination butyrophenone in the pres acid in 95 % ethanol (80 %) [2422]. -Also refer to: [374].	sence of iodine and iodic
4 4 0 0 50 4003		

m.p. 119° [2422]; ¹H NMR [2422], IR [2422], MS [2422].

2-Hydroxy-5-butyrylbenzamide



1-(2-Hydroxy-5-methyl-3-nitrophenyl)-1-butanone

[70978-45-9]	$C_{11}H_{13}NO_4$	mol. wt. 223.23
ОН	Synthesis	
NO ₂ CO(CH ₂) ₂ CH ₃	-Preparation by nitration	
	butyrophenone at -20°	using standard reagents
\mathbf{i}	(55 %) [1017].	
ĊH ₃	m.p. 72–74° [1017].	

1-(4-Hydroxy-2-methyl-3-nitrophenyl)-1-butanone

[1210-91-9]

 $C_{11}H_{13}NO_4$

mol. wt. 223.23



Syntheses

-To n-butyryl chloride, 3-methyl-2-nitroanisole and carbon disulfide was added aluminium chloride, in small portions at 25°. The mixture stirred 1 h at r.t. and 45 min at 55°. Pentane and aluminium chloride added, heated 3 h at 80° [2056].

-Also refer to: [2047, 2048, 2766, 2767 (16.5 %)].

m.p. 133-134° [2047, 2048, 2056, 2766, 2767].

1-(4-Hydroxy-3-methoxy-5-nitrophenyl)-1-butanone

1-(2-Hydroxy-3-methylphenyl)-1-butanone

 $[36375-38-9] C_{11}H_{14}O_2 mol. wt. 178.23$ $CH_3 \qquad OH CO(CH_2)_2CH_3 \qquad Obtained by Fries rearrangement of 2-methylphenyl butyrate with aluminium chloride, *at 160–180° for 30 min (60 %) [726], (45 %) [1644]; *at 100° (40 %) [726].$

-Also obtained by reaction of butyric acid with o-cresol in the presence of zinc chloride at reflux for 2 h (Nencki reaction) (8 %) [317]. -Also refer to: [195].

b.p.₁₀ 142–143° [317], b.p.₁₁ 143° [726, 1644]; IR [2777].

Phenylhydrazone

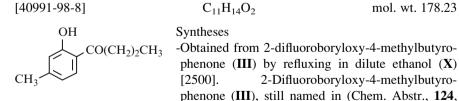
m.p. 157-158° [726].

2,4-Dinitrophenylhydrazone	$C_{17}H_{18}N_4O_5$	mol. wt. 358.35
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 $C_{17}H_{20}N_2O$

m.p. 190-192° [317].

1-(2-Hydroxy-4-methylphenyl)-1-butanone



145544 u):

Difluoro[1-(2-hydroxy-4-methylphenyl)-1-butanonato-O,O'] boron (T-4) [173380-47-7] $C_{11}H_{13}BF_2O_2$ mol. wt. 226.03

mol. wt. 268.36

-Compound (III) was obtained by reaction of butyric acid with m-cresol in the presence of boron trifluoride etherate at r.t. for 30 min (39 %) [2500].

m.p. 75–76° [2500]; IR [2500].

-Also obtained by reaction of butyric acid with m-cresol,

*in the presence of zinc chloride, then boil for five min (17 %) [726];

*in the presence of boron trifluoride for 2 h at 70° (84 %) [1685].

-Also obtained by Fries rearrangement of m-tolyl butyrate with aluminium chloride,

-without solvent,

*for 2 h at 160° (88 %) [726];

*for 10-20 min at 120-140° (75 %) [243];

*for 30 min at 120–148° (48 %) [1644];

*at 140-150° [906].

-in the presence of solvent,

*in nitrobenzene for 24 h at 25° (66 %) [243], for 66 h at 25–30° (70 %) [244] or for 10 days at + 2° (72 %) [243];

*first in carbon disulfide, then at $60-70^{\circ}$ for 1 h and 20° for 24 h after carbon disulfide elimination (71 %) [515, 2089].

-Also obtained by reaction of butyryl chloride with m-cresol methyl ether in the presence of aluminium chloride in carbon disulfide [1602].

-Also obtained (8) by irradiation of 2-methoxy-5-methylphenyl butyrate in 90 % ethanol solution with the American Hanovia 450 W high pressure mercury arc amp in a water-cooled immersion photo-chemical reactor for 2.5 h (10 %) [599]. -Also refer to: [2633].

pale yellow liquid [599]; b.p._{0.4} 45° [599], b.p.₁ 92–93° [244], b.p.₄ 112–115° [243], b.p.₄ 114° [906], b.p.₁₇ 130–131° [2500], b.p.₁₀ 136° [1602], b.p.₁₅ 141–143° [1685], b.p.₁₅ 142–144° [726, 1644], b.p.₁₇ 144–146° [515, 2089]; m.p. 17° [726, 1644], 14.5° [1685], 12–13° [1602]; ¹H NMR [599], IR [599, 2500], UV [2500], MS [599]; ESR [2940].

Methyl ether

 $C_{12}H_{16}O_2 \qquad \qquad \text{mol.}$

mol. wt. 192.26

-Obtained by reaction of butyryl chloride with 3-methylanisole in the presence of aluminium chloride in carbon disulfide at r.t. for 24 h [2088].

Oxime	$C_{11}H_{15}NO_2$	mol. wt. 193.25
m.p. 74–75° [726].		
Phonylhydrozono	CUNO	
Phenylhydrazone	$C_{17}H_{20}N_2O$	mol. wt. 268.36

1-(2-Hydroxy-5-methylphenyl)-1-butanone

[24323-47-5] $C_{11}H_{14}O_2$ mol. wt. 178.23

OH CO(C

CH₃

Syntheses

 $\begin{array}{rl} -\text{Obtained by Fries rearrangement of 4-methylphenyl buty-rate with aluminium chloride for 2 h at 170° (90 \%) \\ [1348], for 3 h at 120–130° [194], for 1 h at 100° (60 \%) \\ [1644] or for 2 h at 150° [726]. \end{array}$

-Also obtained by dealkylation of its ethyl ether with aluminium chloride in carbon disulfide for 8 h at $60-70^{\circ}$ [191].

-Also obtained by reaction of butyric acid with p-cresol in the presence of,

*zinc chloride at reflux for 2 h (27 %) [317];

*zinc chloride under microwave irradiation with 600 W for 3 min (95 %) [2211];

*stannic chloride under microwave irradiation with 700 W for 2 min at 50° and atmospheric pressure conditions (95 %) [2212];

*boron trifluoride etherate under microwave irradiation for 2 min at r.t. (95 %) [2210].

-Also obtained by reaction of butyryl chloride with p-cresol in the presence of aluminium chloride in ethylene chloride at $110-120^{\circ}$ for 8 h (66 %) [1769].

-Also obtained by adding aluminium chloride, then butyryl chloride to a solution of aluminium p-cresylate in benzene. After standing 15 h, the mixture was refluxed 2 h (84.3 %) [1802].

-Also refer to: [375, 1822, 2516, 2932, 3028].

oil [2210–2212]; b.p.₁₅ 132–133° [194], b.p.₁₅ 132° [317], b.p. 246° [2516]; m.p. 34° [191, 1644], 33° [1802], 32–33° [194], 30–31.5° [1769]; ¹H NMR [2210–2212], IR [2210–2212]; TLC [2210, 2211]; GLC [1348]. Cryoscopic study [182].

STUDIES: Stability constants of bivalent metal complexes with 2-hydroxy-5-methyl-butyrophenone Cu(II), Ni(II), Co(II), UO₂(II), Zn(II) and Mn(II) [2516].

 Oxime
 [103582-37-2]
 C11H15NO2
 mol. wt. 193.25

 m.p. 97.5–98.5° [1769];
 1
 1
 NMR [1769], 13°C
 NMR [1769], IR [1769], UV [1769], MS [1921].

USE: Extraction of copper [1769].

$\label{eq:2.4-Dinitrophenylhydrazone} \ensuremath{ [127699-71-2] C_{17}H_{18}N_4O_5 \ensuremath{ mol. wt. 358.35} \ensuremath{ simple straight and simple straight a$

m.p. 218-219° [1769].

p-Nitrophenylhydrazone	$C_{17}H_{19}N_3O_3$	mol. wt. 313.36
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m.p. 184–186° [194].

Methyl ether	[5340-05-6]	$C_{12}H_{16}O_2$	mol. wt. 192.26
wieuryi cuici	19940-09-01	$C_{12} I_{16} O_2$	11101. wt. 192.20

-Obtained by reaction of butyric anhydride with 4-methylanisole in the presence of aluminium chloride in boiling carbon disulfide for 30 min (84 %) [2297]. -Obtained by methylation of 2-hydroxy-5-methylbutyrophenone [1602].

b.p.₃ 123° [2297], b.p.₂₁ 166–168° [1602, 2296], b.p.₇₃₉ 276.5° [2297]; m.p. 86° [2297].

Ethyl ether $C_{13}H_{18}O_2$ mol. wt. 206.28

-Obtained by Friedel-Crafts reaction of butyryl chloride with 4-methylphenetole in the presence of aluminium chloride [191].

C16H24O2

oil [191]; b.p.₁₀₀ 205° [191].

isoAmyl ether

b.p.₁₃ 150° [823].

1-(2-Hydroxy-6-methylphenyl)-1-butanone

[1200-95-9]	$C_{11}H_{14}O_2$	mol. wt. 178.23
OH CO(CH ₂) ₂ CH ₃	Syntheses -Refer to: [2047, 2048, 2766, 2767]. b.p. ₂₀ 155–158° [2047, 2048, 2766, 2767]	

C12H16O2

Methyl ether -Refer to: [2767].

1-(4-Hydroxy-2-methylphenyl)-1-butanone

[104174-31-4]		-41	1	-3	74	41	0	[1	
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OH

$C_{11}H_{14}O_2$

mol. wt. 178.23

mol. wt. 192.26

mol. wt. 248.37

Syntheses

-Obtained by Fries rearrangement of 3-methylphenyl butyrate with aluminium chloride,

 $CO(CH_2)_2CH_3$

*without solvent for 2 h at 160° [726];

*in carbon disulfide and diphenyl oxide, at $100-110^{\circ}$ for 15–20 min, after elimination of carbon disulfide ($\leq 40 \%$) [730];

*in nitrobenzene at + 2° for 10 days (3 %) [243], at r.t. (2 %) [726] or at 25–30° for 66 h (11 %) [244].

-Also obtained by reaction of butyric acid with m-cresol in the presence of boron trifluoride for 2 h at 70° (8 %) [1685].

-Also obtained by reaction of butyryl chloride with 3-methylanisole in the presence of aluminium chloride in carbon disulfide [1602] at r.t. for 24 h (2 %) [2088].

-Also obtained by treatment of its methyl ether with boiling pyridinium chloride for 15 min (79 %) [221].

-Also obtained by treatment of 4-hydroxy-2-methyl-5-isopropylbutyrophenone with aluminium chloride in chlorobenzene, first at r.t. for 20 h, then at 50° for 4 h (55 %) [1523].

b.p.₁₅ 175–200° [730]; m.p. 115° [1602], 104° [1523], 102° [1685], 98–99° [243], 98° [2088], 97–98° [726], 88° [730].

Methyl ether [54696-06-9] C₁₂H₁₆O₂ mol. wt. 192.26

-Obtained by reaction of butyryl chloride with 3-methylanisole in the presence of aluminium chloride in carbon disulfide for 24 h at r.t. (70 %) or without solvent first 1 h at 70°, then 24 h at r.t. (52 %) [2088].

-Also obtained by reaction of butyric anhydride with 3-methylanisole (78 %) [2297].

-Also obtained by reaction of dimethyl sulfate with 4-hydroxy-2-methylbutyrophenone in the presence of sodium hydroxide (50 %) [1602].

C14H20O2

C15H22O2

-Also refer to: [221].

b.p.₁₂ 156° [1602], b.p.₁₅ 161–163° [2088], b.p. 270° [823]; m.p. 13–14° [1602]; $n_D^{21} = 1.5376$ [2088].

Propyl ether

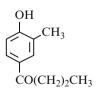
b.p.₁₃ 197–199° [823].

Butyl ether

 $b.p._{18}\ 205^{\circ}\ [823].$

1-(4-Hydroxy-3-methylphenyl)-1-butanone

[52780-68-4]	$C_{11}H_{14}O_2$	mol. wt. 178.23
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Syntheses -Obtained by Fries rearrangement of o-tolyl butyrate in the presence of aluminium chloride (47 %) [184], at 100° (55 %) [726] or at 160–180° for 30 min (23 %) [1644].

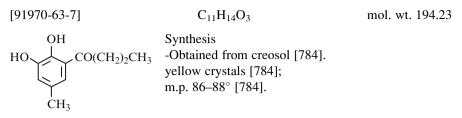
-Also obtained by reaction of butyric acid with o-cresol in the presence,

mol. wt. 220.31

mol. wt. 234.34

 *of boron trifluoride (84 %) [1685]; *of zinc chloride at reflux for 1 h (10 %) [317]. -Also obtained by reaction of butyronitrile with o-cresol in the presence of trifluoromethane-sulfonic acid for 21 days at r.t. (75 %) [425]. -Also obtained by treatment of 4-hydroxy-3-methyl-6-isopropylbutyrophenone with aluminium chloride in chlorobenzene, first at r.t. for 20 h, then at 50° for 4 h (64 %) [1522, 1523]. b.p.₁₅ 195–200° [726, 1644], b.p.₂₀ 220–240° [184]; m.p. 133° [1522, 1523], 132–133° [317, 726], 130–131° [184], 130° [1685], 129–130° [1967], 127–129° [425]; 			
¹ H NMR [425, 1967	7], IR [425, 1967], N	AS [425, 1967].	
Methyl ether	[29665-52-9]	$C_{12}H_{16}O_2$	mol. wt. 192.26
-Refer to: [683, 718, 82	23, 1602, 3049].		
b.p. _{0.08} 105–107° [7 m.p. 48° [1602]; r	(18], b.p. ₁₁ 162–163 $p_D^{20} = 1.5392$ [718].	° [1602], b.p. ₁₃ 168	–171° [823];
Semicarbazone of the	methyl ether	$C_{13}H_{19}N_3O_2$	mol. wt. 249.31
m.p. 168° [823].			
Ethyl ether	$C_{13}H_{18}C_{13}$) ₂	mol. wt. 206.28
b.p. 284–286° [823]		-	
Semicarbazone of the	ethvl ether	$C_{14}H_{21}N_{3}O_{2}$	mol. wt. 263.34
m.p. 174° [823].	·	11 21 3 2	
Butyl ether	$C_{15}H_{22}C_{15}$	\mathbf{D}_2	mol. wt. 234.34
b.p. ₁₅ 200° [823].			
Semicarbazone of the	butyl ether	C ₁₆ H ₂₅ N ₃ O ₂	mol. wt. 291.39
m.p. 160° [823].			
isoAmyl ether	$C_{16}H_{24}$	02	mol. wt. 248.37
b.p. ₁₃ 197° [823].			
Semicarbazone of the m.p. 139° [823].	isoamyl ether	$C_{17}H_{27}N_3O_2$	mol. wt. 305.42

1-(2,3-Dihydroxy-5-methylphenyl)-1-butanone



1-(2,4-Dihydroxy-3-methylphenyl)-1-butanone

[93970-93-5] $C_{11}H_{14}O_3$ mol. wt. 194.23 OH Syntheses -Obtained by treatment of 2-hydroxy-4-methoxy-3-methyl-butyrophenone with hydriodic acid in refluxing acetic anhydride at 125–135° for 2 h (43 %) [2822].

-Also obtained by reaction of n-butyronitrile with 2-methylresorcinol (Houben-Hoesch reaction) [2821, 2822].

-Also obtained by reaction of butyryl chloride with 2-methylresorcinol in the presence of aluminium chloride,

*in methylene chloride first at 0°, then at r.t. for 16 h [2486];

*in nitrobenzene (50 %) [58].

-Also refer to: [773, 1240, 1957].

straw [2821]; m.p. 155–157° [2821, 2822], 154–155° [1515], 113–114° [58]; **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [58, 1515], ¹³C NMR [1240, 1515], MS [1515].

1-(2,4-Dihydroxy-6-methylphenyl)-1-butanone

[154921-40-1]	$C_{11}H_{14}O_3$	mol. wt. 194.23
OH CO(CH ₂) ₂ CH ₃	6-methyl-butyrophe	2-difluoroboryloxy-4-hydroxy- enone by refluxing in dilute etha-
но СН3	nol (70 %) [2938].	

-Compound 2-difluoroboryloxy-4-hydroxy-6-methylbutyrophenone, still named difluoro[1-(2,4-dihydroxy-6-methylphenyl)-1-butanonato-O,O'] boron (70 %).

m.p. 118° [2938]; IR [2938], UV [2938].

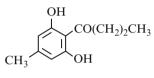
1-(2,5-Dihydroxy-4-methylphenyl)-1-butanone

-Prepared by Friedel-Crafts acylation (96 %) [1755].

b.p.₃ 129–130° [1755]; m.p. 46–46.5° [1755]; ¹H NMR [1755], IR [1755].

1-(2,6-Dihydroxy-4-methylphenyl)-1-butanone

 $[4390-93-6] C_{11}H_{14}O_3 mol. wt. 194.23$



Syntheses

-Obtained by treatment of 2,4-dibutyrylorcinol with 85 % sulfuric acid for 4 h (quantitative yield) [855]. -Also obtained by reaction of butyric anhydride with orcinol,

*in the presence of aluminium chloride (40 %) [854];

*in the presence of Amberlite IR-120, (a cation exchange resin, sulfonic acid type), at 160° for 2–3 h [2523];

*in the presence of polyphosphoric acid or concentrated sulfuric acid (1 drop) at reflux for 30 min, according to the method [1470], (16 %) [2523].

C17H19N3O4

m.p. 120-122° [2523], 120-121° [854].

4-Nitrophenylhydrazone

m.p. 180° [854].

OH

1-(2-Hydroxy-4-methoxyphenyl)-1-butanone

CH₃O

 $C_{11}H_{14}O_3$

mol. wt. 194.23

mol. wt. 329.36

Syntheses

CO(CH₂)₂CH₃ -Preparation by reaction of butyric anhydride with resorcinol monomethyl ether in the presence of concentrated sulfuric acid (2 drops) at 130° for few min [1469].

-Also obtained by reaction of butyryl chloride with resorcinol dimethyl ether in the presence of aluminium chloride in cooled ethyl ether for 4 h [57].

-Also obtained by partial methylation of resbutyrophenone [1469],

*with dimethyl sulfate in the presence of sodium hydroxide solution by heating the mixture on water bath for about half an hour (60 %) [799];

*with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 4–6 h (85–90 %) [2501];

*with methyl bromide in refluxing acetone [284].

-Also refer to: [60, 1787].

yellow oil [57]; b.p.₂ 124–125° [787], b.p.₅ 143–145° [674], b.p.₈ 162–164° [284, 799]; m.p. 32.5° [1469], 32–33° [674], 30–31° [787]; ¹H NMR [57].

Oxime	[143286-57-1]	C ₁₁ H ₁₅ NO ₃	mol. wt. 209.25
	L	- 11 15 - 5	

m.p. 64–67° [284]

1-(2-Hydroxy-5-methoxyphenyl)-1-butanone

[57314-81-5]

C₁₁H₁₄O₃ mol. wt. 194.23

ОН 	CO(CH ₂) ₂ CH ₃
	co(cm ₂) ₂ cm ₃
I ОСН	3

Synthesis -Obtained by reaction of butyryl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride in methylene chloride at r.t. for 1 h (0.4 %) [2878]. oil [2878].

1-(2-Hydroxy-6-methoxyphenyl)-1-butanone

 $C_{11}H_{14}O_3$ mol. wt. 194.23

OH	Synthesis
CO(CH ₂) ₂ CH ₃	-Refer to: [1882].
	m.p. 44–45° [1882]; ¹ H NMR [792], ¹³ C NMR [792],
OCH3	IR [792], MS [792].

BIOLOGICAL ACTIVITY: Phytotoxicity [792]; Antifungal [792].

1-(3-Hydroxy-4-methoxyphenyl)-1-butanone

[91970-65-9]	$C_{11}H_{14}O_3$	mol. wt. 194.23
CH ₃ O CO(CH ₂) ₂ CH ₃	Syntheses -Obtained by reaction of butyric in the presence of phosphorou steam bath for 3 h [726].	Ų

-Also obtained by treatment of 3-hydroxy-4-methoxy-phenyl monochloroacetate with sodium acetate in refluxing methanol for 3 h [3163].

-Also obtained by treatment of 3-butyloxy-4-methoxybutyrophenone with sodium methoxide in methanol at r.t. overnight (82 %) [3220].

m.p. 81-82° [726, 3220], 78-79° [3163].

-Obtained by reaction of butyric anhydride with guaiacol in the presence of polyphosphoric acid by heating at reflux for 5 h (21 %) [3220].

1-(4-Hydroxy-2-methoxyphenyl)-1-butanone

$$C_{11}H_{14}O_3$$
 mol. wt. 194.23

Syntheses

-Preparation by reaction of butyric anhydride with resorcinol monomethyl ether in the presence of concentrated sulfuric acid (2 drops) at 130° for few min [1469].

 $CO(CH_2)_2CH_3$ -Also obtained by partial methylation of resbutyrophenone [1469].

m.p. 69° [1469].

DCH₃

1-(4-Hydroxy-3-methoxyphenyl)-1-butanone

[64142-23-0]

OH

 $C_{11}H_{14}O_3$

mol. wt. 194.23

Syntheses

CO(CH₂)₂CH₃

-Obtained by reaction of butyric acid with guaiacol, *in the presence of fused zinc chloride for 3 h at reflux (Nencki reaction) [726];

*in the presence of a boron trifluoride and hydrogen fluoride mixture in xylene at $60-70^{\circ}$ [505].

-Also obtained by Fries rearrangement of 2-methoxyphenyl butyrate with aluminium chloride in nitrobenzene [1990], at 80° for 30–60 min (50 %) [726]. -Also obtained by reaction of butyric anhydride with guaiacol in the presence of

zinc chloride at 155° for 3 min [324].

-Also obtained by adding DDQ to 1-(4-hydroxy-3-methoxyphenyl)-1-butanol in benzene and the reaction mixture stirred for 4 h at r.t. [2989].

-Also obtained by hydrolysis of its benzoate (90 %) [1377].

-Also refer to: [526, 527, 978, 1616].

Isolation from natural sources

-From 1988 to 1989 vintage Chardonnay juices (butyrovanillone N° 98) [614, 2783].

-Varietal aroma compounds of some grapes grown in Southern Italy [3054].

-Volatile compound evolution in Spanish oak wood (Quercus petraea and Quercus pyrenaica) during natural seasoning. [528].

-In oak wood chips [3222].

-Volatile of oak heartwood [832], volatile compound in grapes from 18 Italian Malvasia grapevine cultivars [430].



OН

-Aroma precursors of non-floral grapes of Vitis vinifera L. Bombino n. and Uva di Troia varieties [3053].

-In oak wood used for aging of wines and spirits [2454].

-Identification of volatile compounds with a "Toasty" aroma in heated Oak used in Barrel making [780].

monoclinic and triclinic crystals [1377]; b.p.₁₅ 185–195° [726, 1990], b.p.₁₂ 202° [505]; m.p. 62.9° [1377], 55° [2989], 54–55° [726, 1990], 45° [505], 40–41° [324]; ¹H NMR [2989], MS [2783, 3222]; GC-MS [780, 2454, 2783, 3222].

BIOLOGICAL ACTIVITY: Choleretic [2989].

Phenylhydrazone	$C_{17}H_{20}N_2O_2$	mol. wt. 284.36
m.p. 91–92° [726], 7	79° [1377].	
Benzoate	$C_{18}H_{18}O_4$	mol. wt. 298.34

-Refer to: [1129, 1377 (92 %)].

shiny triclinic crystals [1377]; m.p. 88.1° [1377], 75–77° [726].

Phenylhydrazone of the benzoate	$C_{24}H_{24}N_2O_3$	mol. wt. 388.47

m.p. 175.5° [1377].

1-(2,4-Dihydroxy-6-methoxyphenyl)-1-butanone

(o-Desaspidinol)

[21185-39-7]	$C_{11}H_{14}O_4$	mol. wt. 210.23
OH	Syntheses	

\triangleleft	CO(CH ₂) ₂ CH ₃
HO	OCH3

H₃ -Preparation by reaction of n-butyronitrile with phloroglucinol monomethyl ether (Houben-Hoesch reaction) (**XIX**) [1610].

-Also obtained by acylation of methyl 2,6-dihydroxy-4-methoxybenzoate with butyric acid and boron trifluoride and hydrolysis and decarboxylation [3296]. -Also refer to: (II) [205, 2448].

m.p. 130° [1610], 127–128° [3296]; ¹³C NMR [205], UV [2448]; GLC [2531].

mol. wt. 390.48

1-(2,5-Dihydroxy-4-methoxyphenyl)-1-butanone

[2015-80-7]	$C_{11}H_{14}O_4$	mol. wt. 210.23
CH ₃ O OH CH ₃ O OH	Synthesis -Preparation by Fries ream hydroquinone with alumi benzene (64 %) [1250]. m.p. 109–110° [1250].	

-Obtained by reaction of benzyl chloride with title ketone in the presence of potassium hydroxide (74 %) [1250].

 $C_{25}H_{26}O_4$

m.p. 92° [1250].

OН

1-(2,6-Dihydroxy-4-methoxyphenyl)-1-butanone

[2017-96-1]

(Desaspidinol B)

Dibenzyl ether

$$[437-72-9]$$
 C₁₁H₁₄O₄ mol. wt. 210.23

Syntheses

-Preparation by reaction of butyryl chloride with phloroglucinol monomethyl ether in the presence of aluminium chloride in methylene chloride at r.t. [1129].

-Preparation by reaction of n-butyronitrile with phloroglucinol monomethyl ether (Houben-Hoesch reaction) (**XX**) [1610].

-Also obtained by partial methylation of phlorobutyrophenone with diazomethane [3296].

-Also refer to: [205, 567, 1045, 2303, 3447, 3448].

Isolation from natural sources

-From the roots of Dryopteris championii [3480].

-From kochia (Kochia scoparia L.) Schrad. KOCSC [817].

-From giant foxtail (Setaria faberi) SEFTA [817].

-From yellow foxtail (Setaria glauca L.) Beauv. SETLU [817].

-From field pennycress (Thlaspi arvense L.) THLAR [817].

-From the fern of *Elaphoglossum spathulatum* [2917].

-Of Populus tritis bud exudate [955].

-In bud exudate of *Populus koreana*, *Populus maximowiczii* and *Populus suaveolens* (Salicaceae) [1164].

m.p. 127–128° [2444, 3301], 125–127° [1151], 121–123° [37], 113° [1610]; ¹³C NMR [205], UV [37]; GC-MS [1164]; GLC [2531]. BIOLOGICAL ACTIVITY: Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

1-(2,4,6-Trihydroxy-3-methylphenyl)-1-butanone

(Aspidinol-B)

$$[1509-06-4] C_{11}H_{14}O_4 mol. wt. 210.23$$

Syntheses

CH₃ HO HO OH

CO(CH₂)₂CH₃ -Preparation by reaction of n-butyronitrile with 2-methyl-phloroglucinol (Houben-Hoesch reaction) OH (**XVI**) [1610].

-Also obtained by mild alkaline hydrolysis of the flavaspidic acid AB (m.p. 102–106°) (**VI**) from *Dryopteris goldiana* (Hook.) A. Gray [3294] according to [2449].

-Also obtained by reaction of butyryl chloride with 2-methylphloroglucinol in the presence of aluminium chloride in carbon disulfide/nitrobenzene mixture [2618], (41 %) [2617, 2620].

-Also refer to: [35, 205, 1916 (XIII), 1917, 2033, 2305, 2911 (19%), 3297, 3372].

Isolation from natural sources

-From absolute oil of Lysimachia foenum-graecum [3429].

-From the roots of Dryopteris championii [3480].

-From Dryopteris caucasica (A. Br.) [3298].

-From Dryopteris bissetiana [3299].

-From Dryopteris lacera [3299].

-From Dryopteris sacrosancta [3299].

m.p. 166–167° [1917, 3297], 162° [2617], 160–162° [3296], 155° [2620], 154–155° [1610];
¹³C NMR [205], UV [540], MS [1917, 3429];
TLC [3294, 3298]; paper chromatography [2449]; GC [3429];
GC/MS [2305]; GLC [2531].

BIOLOGICAL ACTIVITY: Insecticide [3429]; Antimicrobial for *Staphylococcus aureus* [3372]; Toxicity [3429].

Monohydrate $C_{11}H_{14}O_4, H_2O$ mol. wt. 228.2	Monohydrate	$C_{11}H_{14}O_4, H_2O$	mol. wt. 228.25
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m.p. 154–155° [1610].

1-(2-Hydroxy-4-methoxy-6-methylphenyl)-2-(methylsulfinyl)-1-ethanone

$$[104783-89-3] C_{11}H_{14}O_4S mol. wt. 242.30$$

$$OH \\ CH_3O CH_2 - S - CH_3 OCCH_2 - S - CH_3 OCCH_2$$

m.p. 146–148° [1157]; ¹H NMR [1157], IR [1157].

1-(2-Amino-3-methoxyphenyl)-1-butanone

$C_{11}H_{15}NO_2$		mol. wt. 193.25
CH ₃ O CO(CH ₂) ₂ CH ₃	Synthesis -Refer to: [81]. b.p. _{0.02} 82–84° [81].	

1-(3-Amino-2-hydroxy-5-methylphenyl)-1-butanone

[70978-64-2]	$C_{11}H_{15}NO_2$	mol. wt. 193.25
OH NH ₂ CH ₃ OH CO(CH ₂) ₂ CH ₃	Synthesis -Preparation by hydrogenation 5-methyl-3-nitrobutyrophenone us catalyst in ethanol (77 %) [1017]. m.p. 79–81° [1017].	

1-(3-Hydroxy-4-methylaminophenyl)-1-butanone

[123172-48-5]	$C_{11}H_{15}NO_2$	mol. wt. 193.25
OH CH ₃ NH	Syntheses -Obtained by treatment	[424] of 6-butyryl-
	3-methylbenzoxazolinone	with boiling 10 %

 $CO(CH_2)_2CH_3$ aqueous sodium hydroxide for 4 h (70 %) [2161]. -Also refer to: [2160].

m.p. 130° [2160, 2161].

[4,6-Dihydroxy-5-(1-oxobutyl)phenyl]-1,3-dicarboxylic acid

$$\begin{array}{cccc} & & & C_{12}H_{12}O_7 & & mol. \ wt. \ 268.22 \\ & & CO(CH_2)_2CH_3 & Synthesis \\ & HO & & -Refer \ to: \ [862]. \\ & & Dimethyl \ ester & \ [13937-24-1] \\ & & CO_2H & C_{14}H_{16}O_7 & & mol. \ wt. \ 296.28 \end{array}$$

-Preparation by treating resorcinol-4,6-dicarboxylic acid dimethyl ester with butyryl chloride in the presence of aluminium chloride (75 %) [862].

m.p. 115–116° [862].

OH

CH₃CH₂

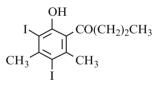
1-(3-Bromo-4-ethyl-5-fluoro-2-hydroxyphenyl)-1-butanone

 $C_{12}H_{14}BrFO_{2} \qquad \text{mol. wt. } 289.15$ Synthesis $CO(CH_{2})_{2}CH_{3} \qquad -Refer \text{ to: } [2002].$ $m.p. 50^{\circ} \text{ (Sadtler standard N^{\circ} 76413K);}$ $^{1}H \text{ NMR (Sadtler standard N^{\circ} 49340M),}$ $IR \text{ (Sadtler standard N^{\circ} 76413K).}$

1-(2-Hydroxy-3,5-diiodo-4,6-dimethylphenyl)-1-butanone

[883566-12-9] C₁₂H₁₄I₂O₂ mc OH Synthesis

mol. wt. 444.04



CO(CH₂)₂CH₃ -Obtained by iodination of 2-hydroxy-4,6-dimethylbutyrophenone in the presence of iodine and iodic acid in 95 % ethanol at $35-40^{\circ}$ for 1.5 h (75 %) [2422].

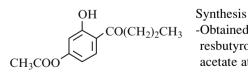
m.p. 181° [2422]; ¹H NMR [2422], IR [2422], MS [2422].

1-[4-(Acetyloxy)-2-hydroxyphenyl]-1-butanone

[91497-29-9]

 $C_{12}H_{14}O_4$

mol. wt. 222.24



m.p. 47.5–48° [3243].

CO(CH₂)₂CH₃ -Obtained by reaction of acetic anhydride with resbutyrophenone in the presence of sodium acetate at r.t. for 24–30 h (70–80 %) [3243].

5-Hydroxy-6-(1-oxobutyl)-1,4-benzodioxane

$$CH_{3}(CH_{2})_{2}CO \xrightarrow{O}_{OH} O \xrightarrow{O}_{O$$

m.p. 82.5–83° [801]; UV [801].

Oxime

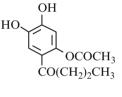
C12H15NO4

mol. wt. 237.26

USE: Photometric determination of titanium [2558].

1-(2-Acetyloxy-4,5-dihydroxyphenyl)-1-butanone

[145747-23-5] $C_{12}H_{14}O_5$ mol. wt. 238.24



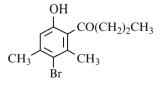
Syntheses -Obtained by partial enzymatic deacylation of 2,4,6-triacetoxybutyrophenone (8) at 42–45° for 40 h H₃ [376, 2410], H₃ *with **PPL** (porcine pancreatic lipase), -in THF (tetrahydrofuran) (10 %) (17) [376]; -in DIPE (diisopropyl ether) (15 %) (17) [376]; -in acetone (15 %) (17) [376].

*with **CCL** (Candida cylindracea lipase), -in THF (tetrahydrofuran) (20 %) (**17**) [376], (20 %) (**14**) [2412]; -in a THF and n-butanol mixture (20 %) (**20**) [2410]; -in DIPE (diisopropyl ether) (10 %) (**17**) [376]; -in acetone (10 %) (**17**) [376].

m.p. 118–120° [376]; ¹H NMR [376], MS [376], IR [376], UV [376]; TLC [376].

1-(3-Bromo-6-hydroxy-2,4-dimethylphenyl)-1-butanone

$$C_{12}H_{15}BrO_2$$
 mol. wt. 271.15



Syntheses -Obtained by reaction of bromine (1 mol) with 2-hydroxy-4,6-dimethylbutyrophenone in carbon disulfide [189].

-Also obtained by reaction of butyryl chloride with 4-bromo-3,5-dimethylanisole in the presence of aluminium chloride in carbon disulfide [189].

b.p.₁₂ 170–182° [189]; m.p. 106–106.5° [189].

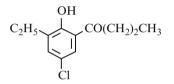
1-(3-Chloro-6-hydroxy-2,4-dimethylphenyl)-1-butanone

[100388-73-6]	$C_{12}H_{15}ClO_2$	mol. wt. 226.70
CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃	3,5-dimethylphenyl but ride in carbon disulfide	rearrangement of 4-chloro- tyrate with aluminium chlo- at 80° for 2 h, then at 110° mination (96 %) [3114].

m.p. 76° [3114].

1-(5-Chloro-3-ethyl-2-hydroxyphenyl)-1-butanone

$$[53347-08-3] C_{12}H_{15}ClO_2 mol. wt. 226.70$$

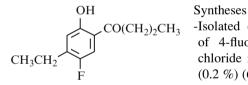


Synthesis -Obtained by Fries rearrangement of 4-chloro-2-ethylphenyl butyrate in the presence of aluminium chloride at 120° for 1.5 h (86.4 %) [2763].

b.p.₁₈ 173.5–174° [2763]; $n_D^{20} = 1.5470$ [2763].

1-(4-Ethyl-5-fluoro-2-hydroxyphenyl)-1-butanone

[98841-70-4] C12H15FO2 mol. wt. 210.25



CO(CH₂)₂CH₃ -Isolated on Fries rearrangement (by-product) of 4-fluorophenyl butyrate with aluminium chloride in 1,2-dichloroethane at 100° for 2 h (0.2 %) (6) [1998].

N.B.: In tar of the industrial preparation from 500 kg of the ester (Sanofi industries, Aramon, Fr.).

-Obtained (6) by Fries rearrangement of 4-fluorophenyl butyrate with aluminium chloride in bromoethane, first at 0° , then at r.t. for 2 days (30 %) [1998]. -Refer to: [2002].

m.p. 50° (Sadtler standard N° 68953K) [1998]; H NMR (Sadtler standard N° 41516M) [1998], IR (Sadtler standard N° 68953K) [1998], UV [1998], MS [1998].

1-[2-(Acetylamino)-4-hydroxyphenyl]-1-butanone

[1211-07-0] C₁₂H₁₅NO₃ mol. wt. 221.26 OH Syntheses

-To n-butyryl chloride, 3-methoxy-N-phenylacetamide and carbon disulfide was added in small portions at 25° , aluminium chloride; the mixture stirred 1 h at r.t. and 45 min at 55° ; pentane and aluminium chloride added, heated 3 h at 80° [2056].

-Also refer to: [2047, 2048, 2766, 2767 (61 %)].

m.p. 94–96° [2047, 2048, 2056, 2766, 2767].

1-[4-(Acetylamino)-2-hydroxyphenyl]-1-butanone

[28583-62-2]

NHCOCH₃

CO(CH₂)₂CH₃

 $C_{12}H_{15}NO_3 \\$

mol. wt. 221.26

Syntheses

H₂)₂CH₃ -Obtained by *Friedel-Crafts reaction of butyryl chloride with m-acetylaminoanisole in the presence of an excess of aluminium chloride in refluxing 1,2-dichloroethane for 2 h (58 %) [1552];

*or by Fries rearrangement of m-acetylaminophenyl butyrate (m.p. 85°) with aluminium chloride (2 mol) for 4 h at 180° (46 %) [1552]. -Also refer to: [2117].

m.p. 115–117° [2117], 102° [1552]; ¹H NMR [767], ¹³C NMR [767].

1-[5-(Acetylamino)-2-hydroxyphenyl]-1-butanone

[28583-76-8] $C_{12}H_{15}NO_3$ mol. wt. 221.26 OH Syntheses -CO(CH₂)₂CH₃ -Obtained by Fries rearrangement of p-acetamidophenyl

-Obtained by Fries rearrangement of p-acetamidophenyl butyrate with aluminium chloride, first at 110–120°, then for 3 h at 160° [1449]. -Also refer to: [2117].

m.p. 100.5–102° [2117], 99.5–99.8° [2117].

CH₃CONH

1-(3-Ethyl-2-hydroxyphenyl)-1-butanone

$$\begin{array}{c} C_{12}H_{16}O_2 \\ OH \\ C_{2}H_5 \\ \hline \\ CO(CH_2)_2CH_3 \\ \hline \\ CO(CH_2)_2CH_3 \\ \hline \\ CO(CH_2)_2CH_3 \\ \hline \\ CO(CH_2)_2CH_3 \\ \hline \\ CO(CH_2)_2CH_3 \\ \hline \\ CO(CH_2) \\ \hline \\ CO(CH_2) \\ \\ CO(CH_2) \\ \hline \\ C$$

b.p.₁ 170–175° [2955]; m.p. 91–92° [2955].

1-(4-Ethyl-2-hydroxyphenyl)-1-butanone

$$C_{12}H_{16}O_2$$
 mol. wt. 192.26
OH
 $C_{2}H_5$ $CO(CH_2)_2CH_3$ $Obtained by Fries rearrangement of 3-ethylphenyln-butyrate (1 equiv.),*in the presence of aluminium chloride (1.3 equiv.)in nitrobenzene at 25° for 6 h (89 %) [2801];$

*in the presence of aluminium chloride (2.8 M), first in refluxing carbon disulfide for 2 h, then for 2 h at 130° after solvent elimination (88 %) [2801];

*in the presence of aluminium chloride (1.3 equiv.) without solvent at 140-150° [906].

b.p.₄ 126–128° [906], b.p.₁₅ 180° [2801]; ESR [2940].

Methyl ether

-Obtained by reaction of dimethyl sulfate with 1-(4-ethyl-2-hydroxyphenyl)-1-butanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating

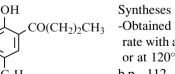
b.p.35 150° [2801].

1-(5-Ethyl-2-hydroxyphenyl)-1-butanone

on the water bath for 5 h (80 %) [2801].

$$C_{12}H_{16}O_2$$
 mol.

wt. 192.26



CO(CH₂)₂CH₃ -Obtained by Fries rearrangement of 4-ethylphenyl butyrate with aluminium chloride at 100° for 2 h (75 %) [2800] or at 120° (50 %) [2955].

b.p.₁ 112–113° [2955], b.p.₁₀ 145° [2800].

$$C_{13}H_{18}O_2$$

mol. wt. 206.28

Methyl ether [3781-76-8] C₁₃H₁₈O₂ mol. wt. 206.28

-Obtained by reaction of butyryl chloride with p-ethylanisole in the presence of aluminium chloride in carbon disulfide at r.t. for 1 h (75.5 %) [845].

b.p.₂₄ 170–172° [845];
$$n_D^{24} = 1.5239$$
 [845].

2,4-Dinitrophenylhydrazone
$$C_{18}H_{20}N_4O_5$$
 mol. wt. 372.38

m.p. 184° [2801].

1-(2-Hydroxy-3,5-dimethylphenyl)-1-butanone

[873989-36-7]	$C_{12}H_{16}O_2$	mol. wt. 192.26
CH ₃ CH ₃ CH ₃ CH ₃	2,4-dimethylphenyl buty *with aluminium chlorid 6.5 h (84 %) [1016];	ries rearrangement of yrate, de (2 mol) at 110–115° for de at 120° (45 %) [2955].

oil [2955]; b.p.₂ 121° [2955], b.p.₃₀ 145–150° [1016]; m.p. 57–58° [2061], 30° [1016].

Phenylhydrazone $C_{18}H_{22}N_2O$ mol. wt. 282.39

m.p. 168-170° [2955].

1-(2-Hydroxy-4,5-dimethylphenyl)-1-butanone

[91667-38-8]	$C_{12}H_{16}$	O_2		mol. wt. 1	92.26
ОН	Syntheses				
$CO(CH_2)_2CH_3$	-Obtained	by	Fries	rearrangement	of
		ylpheny	l butyrate	with aluminium	chlo-
CH ₃	ride at 110°	[°] withou	t solvent	(85 %) [3117].	
ĊH ₃	-Also obtain	ned by	reaction	of butyric acid	with

-Also obtained by reaction of butyric acid with 3,4-dimethylphenol in the presence of boron trifluoride etherate (74 %) [2939, 2956].

b.p.₁₀ 116° [3117]; m.p. 52–53° [2939], 40–41° [2955]; ¹H NMR [2939], IR [2939], UV [2939, 2956].

Methyl ether	$C_{13}H_{18}O_2$	mol. wt. 206.28
b.p. ₁₃ 171° [824];	m.p. 54° [824].	
isoAmyl ether	$C_{17}H_{26}O_2$	mol. wt. 262.39
b.p. ₁₃ 182° [824];	m.p. 54° [824].	

1-(2-Hydroxy-4,6-dimethylphenyl)-1-butanone

[1639-85-6]	$C_{12}H_{16}C_{12}$	D_2		mol. wt. 19	2.26
ОН	Syntheses				
$CO(CH_2)_2CH_3$	-Obtained	by	Fries	rearrangement	of
	3,5-dimethy	lpheny	yl n-butyra	te (1 equiv.),	
CH ₃ CH ₃	*in the prese	ence of	f aluminiu	m chloride (1.3 equ	uiv.)
	in nitroben:	zene at	25° for 6	h (90 %) [2801];	

*in the presence of aluminium chloride (2.8 M), first in refluxing carbon disulfide for 2 h, then for 2 h at 130° after solvent elimination (86 %) [2801].

-Also obtained by reaction of butyryl chloride with 3,5-dimethylanisole in the presence of aluminium chloride [189].

-Also obtained from 2-difluoroboryloxy-4,6-dimethylbutyrophenone (VII) by refluxing in dilute ethanol (XIV). VII was obtained by reaction of butyric acid with 3,5-dimethylphenol in the presence of boron trifluoride etherate at r.t. for 30 min ($C_{12}H_{15}BF_2O_2$, mol. wt. 240.06, 60 %, m.p. 121–122°, IR) [2500].

-Also obtained by reaction of butyric acid with 3,5-dimethylphenol in the presence of boron trifluoride for 2 h at 70° (85 %) [1685].

-Also refer to: [2767].

b.p.₁₅ 157.5–158° [1685]; colourless solid [146]; m.p. 60° [1685], 59° [189], 57–58° [2061, 2500, 2767], 51° [2801]; IR [146, 2500], UV [2500].

4-Nitrophenylhydrazone	$C_{18}H_{21}N_3O_3$	mol. wt. 327.38
m.p. 162–162.5° [189].		
2,4-Dinitrophenylhydrazone	$C_{18}H_{20}N_4O_5$	mol. wt. 372.38
1050 [2001]		

m.p. 185° [2801].

Acetate	$C_{14}H_{18}O_3$	mol. wt. 234.30

-Obtained by reaction of acetyl chloride with 2-hydroxy-4,6-dimethylbutyrophenone [189].

oil; b.p.₁₇ 173–175° [189].

Methyl ether $C_{13}H_{18}O_2$ mol. wt. 206.28

-Obtained by reaction of dimethyl sulfate with 1-(2-hydroxy-4,6-dimethylphenyl)-1-butanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating on the water bath for 5 h (85 %) [2801].

-Also obtained by reaction of butyryl chloride with 3,5-dimethylanisole in the presence of aluminium chloride [189].

oil; b.p.₁₇ 162° [189]; m.p. 44° [2801].

mol. wt. 206.28

1-(4-Hydroxy-2,3-dimethylphenyl)-1-butanone

[5862-05-5]	$C_{12}H_{16}O_2$	mol. wt. 192.26
OH CH ₃ CH ₃ CO(CH ₂) ₂ CH ₃	Synthesis -Obtained by treatment of 4-methoxy-2,3-dim none with aluminium chloride (2 mo heptane [2060]. m.p. 100–102° [2060].	• • •

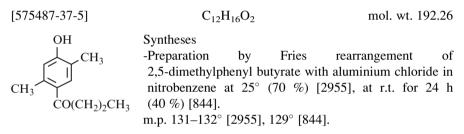
 $C_{13}H_{18}O_{2}$

Methyl ether

-Obtained by reaction of butyryl chloride with 2,3-dimethylanisole in the presence

of aluminium chloride in petroleum ether at r.t. [2060].

1-(4-Hydroxy-2,5-dimethylphenyl)-1-butanone



 $C_{15}H_{22}O_{2}$

C₁₆H₂₄O₂

Propyl ether

b.p.₁₂ 187–190° [824].

Butyl ether

b.p.₁₃ 198° [824]; m.p. 40° [824].

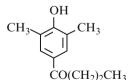
1-(4-Hydroxy-3,5-dimethylphenyl)-1-butanone

 $C_{12}H_{16}O_2$

mol. wt. 192.26

mol. wt. 234.34

mol. wt. 248.37



Syntheses -Obtained by Fries rearrangement of 2,6-dimethylphenyl n-butyrate (*vic-m-Xylenyl butyrate*) (b.p. 248–250°) in the presence of aluminium chloride (67 %) [184] in nitrobenzene at r.t. for 24 h [2008].

m.p. 127–127.5° [2008], 124–125° [184].

Methyl ether

 $C_{13}H_{18}O_2$ mol. wt. 203.29

¹H NMR [1267].

1-(5-Hydroxy-2,4-dimethylphenyl)-1-butanone

[1203-88-9]	$C_{12}H_{16}O_2$	mol. wt. 192.26
CH ₃ CH ₃ CH ₃ CO(CH ₂) ₂ CH ₃		

1-(2,4-Dihydroxy-5-ethylphenyl)-1-butanone

[859939-56-3]	$C_{12}H_{16}O_3$	mol. wt. 208.26
HO CH ₂ CH ₂ CH ₃	Synthesis -Obtained by Fries rearrange dibutyrate (1 mol) i 3-ethylresorcinol (1 mol) y (2 mol) in nitrobenzene at 5	n the presence of with aluminium chloride
m.p. 100° [2651].		
Phenylhydrazone	$C_{18}H_{22}N_2O_2$	mol. wt. 298.38

m.p. 142.5° [2651].

1-(4-Ethyl-2,5-dihydroxyphenyl)-1-butanone

C ₁₂	$H_{16}O_3$	mol. wt. 208.26
CH ₃ CH ₂ OH OH OH	Synthesis -Refer to: [2441]. Dimethyl ether [1 $C_{14}H_{20}O_3$	153756-51-5] mol. wt. 236.31

-Obtained by acylation of 2-ethyl-1,4-dimethoxybenzene with butyric acid in the presence of trifluoroacetic anhydride [2441].

1-(2,6-Dihydroxy-3-ethylphenyl)-1-butanone

[100256-99-3]	$C_{12}H_{16}O_3$	mol. wt. 208.26
ОН	Syntheses	
\downarrow CO(CH ₂) ₂ CH ₃	-Obtained by decarboxylation of 1-[5-e	thyl-2,4-dihydroxy-
	3-(1-oxobutyl)]benzoic acid with conc.	HCl (few drops) in
OH	boiling acetic acid for 20 h (25 %) [28	511].
C ₂ H ₅	-Also obtained by hydrolysis of 8-	butyryl-7-hydroxy-
	6-ethylcoumarin [2811].	

m.p. 80° [2811].

1-(2,5-Dihydroxy-3,4-dimethylphenyl)-1-butanone

$$C_{12}H_{16}O_{3}$$
 mol. wt. 208.26

$$CH_{3} \rightarrow CO(CH_{2})_{2}CH_{3}$$
 -Refer to: [1755].

$$Dimethyl \ ether$$

$$CH_{3} \rightarrow OH$$
 mol. wt. 236.31

-Prepared by Friedel-Crafts acylation (88 %) [1755].

b.p.₃ 140–143° [1755], m.p. 48–48.5° [1755]; ¹H NMR [1755], IR [1755].

1-(4-Ethoxy-2-hydroxyphenyl)-1-butanone

-Also obtained by reaction of butanoyl chloride with m-diethoxybenzene in the presence of aluminium chloride in dichloroethane at 0° . Then, the mixture was stirred for 1 h at 10° , and then for 6 h at $20-25^{\circ}$.

The temperature was then raised to 80° and stirring was continued at that temperature for 5 h (60 %) [1194].

-Also refer to: [852].

b.p.₅ 155° [1587]; m.p. 51.5–52.5° [1194]; UV [1194].

USE: UV absorber [1194]; Light-stabilizer of polymeric materials [1194].

OXINC $[1/3/3/2+4/3]$ $C_{13}I_{17}I_{10}O_{3}$ IIIOI. WL 233.20	Oxime	[173959-45-0]	$C_{13}H_{17}NO_{3}$	mol. wt. 235.28
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USE: As analytical reagent for palladium determination by gravimetry and spectrophotometry [852].

2,4-Dinitrophenylhydrazone	$C_{18}H_{20}N_4O_6$	mol. wt. 388.38
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m.p. 187° [1587].

1-(2-Hydroxy-3-methoxy-6-methylphenyl)-1-butanone

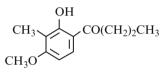
[40991-99-9]	$C_{12}H_{16}O_3$	mol. wt. 208.26
OH	Synthesis	

CO(CH₂)₂CH₃ -Obtained (7) by irradiation of 2-methoxy-5-methylphenyl butyrate in 90 % ethanol solution with the American Hanovia 450 W high pressure mercury arc amp in a water cooled immersion photo-chemical reactor for 2.5 h (23 %) [599].

pale yellow liquid [599]; b.p._{0.6} 71° [599]; ¹H NMR [599], IR [599], UV [599], MS [599].

1-(2-Hydroxy-4-methoxy-3-methylphenyl)-1-butanone





Syntheses

-Obtained by reaction of methyl iodide with resbutyrophenone in the presence of potash in methanol first at r.t. overnight, then refluxing for 6 h (24 %) [2822].

-Also obtained by adding butyryl chloride to a cooled solution of 1,3-dimethoxy-2-methylbenzene in ethyl ether and aluminium chloride for 1 h. The stirring continued for a further 4 h (70 %) [60].

m.p. 82–84° [2822], 65–66° [60]; ¹H NMR [60].

1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1-butanone

$$\begin{array}{ccc} [92755-96-9] & C_{12}H_{16}O_3 & \text{mol. wt. } 208.26\\ OH & Synthesis \end{array}$$

CH₃O CO(CH₂)₂CH

CO(CH₂)₂CH₃ -Obtained by adding butyryl chloride to a cooled solution of 1,3-dimethoxy-5-methylbenzene in ethyl ether and aluminium chloride for 1 h. The stirring continued for a further 4 h (50 %) [60].

m.p. 68–69° [60]; ¹H NMR [60].

1-(4-Hydroxy-5-methoxy-2-methylphenyl)-1-butanone

 $\begin{array}{cccc} [41082-97-7] & C_{12}H_{16}O_3 & \mbox{mol. wt. } 208.26 \\ \\ CH_3O & & \\ CH_3O & & \\ CH_3O & & \\ CH_3 & \\ CO(CH_2)_2CH_3 & \\ \end{array} \begin{array}{c} CH_{16}O_3 & \mbox{mol. wt. } 208.26 \\ \\ Syntheses & \\ -Obtained (6) by Fries rearrangement of 2-methoxy-5-methylphenyl butyrate with titanium tetrachloride in nitrobenzene at 60-67^{\circ} for 4 h (43 \%) [599]. \end{array}$

-Also obtained (6) by irradiation of 2-methoxy-5-methylphenyl butyrate in 90 % ethanol solution with the American Hanovia 450 W high pressure mercury arc amp in a water cooled immersion photochemical reactor for 2.5 h (16 %) [599].

pale yellow crystals [599]; m.p. 63–64° [599]; ¹H NMR [599], IR [599], UV [599], MS [599].

Methyl ether	[3307-03-7]	$C_{13}H_{18}O_3$	mol. wt. 222.28
b.p. ₇ 180° [305];	m.p. 45° [305].		

1-(2,4-Dihydroxy-6-methoxy-3-methylphenyl)-1-butanone

(Isoaspidinol B)

[479-96-9] $C_{12}H_{16}O_4$ mol. wt. 224.26 OH Syntheses CH_3 -Preparation by reaction of n-butyronitrile with $CO(CH_2)_2CH_3$ -Preparation by reaction of n-butyronitrile with 2-methyl-5-methoxyresorcinol (Houben-Hoesch reaction) (**XXI**) [1610]. -Also refer to: [205, 325, 326, 1609, 2448 (**IX**)].

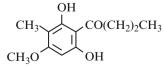
m.p. 152–153° [3301], 151.5° [1609, 1610]; ¹³C NMR [205], UV [2448], MS [2531]; GLC [2531].

BIOLOGICAL ACTIVITY: Inhibitor of angiogenesis using human antiangiogenic assay [326]; Inhibition of endothelial cell functions by novel potential cancer chemopreventive agents [325].

1-(2,6-Dihydroxy-4-methoxy-3-methylphenyl)-1-butanone

(Aspidinol) (Aspidinol B)

$$[519-40-4] C_{12}H_{16}O_4 mol. wt. 224.26$$



CO(CH₂)₂CH₃ -Obtained by reaction of butyryl chloride with 5-methoxy-4-methylresorcinol in the presence OH of aluminium chloride in carbon disulfide/ nitrobenzene mixture (71.5 %) [2620].

-Also obtained from 5-methoxy-4-methylresorcinol (I) [1609, 2611].

Syntheses

-Also obtained by partial methylation of 3-methyl-2,4,6-trihydroxybutyrophenone with diazomethane [3296].

-Also refer to: [35, 205, 2393, 3302].

Isolation from natural sources

-Occurs in extracts of male fern [388, 1276].

-From Dryopteris austriaca (Jacq.) Woynar (Polypodiaceae) [36].

-From Dryopteris fragrans [1471, 2851, 2854, 2855, 3453].

-From industrial Eucalyptus globulus kraft black liquor [2258].

-In essential oil from aromatic plants [2922].

-From the rose cell liquid [1187].

-From the leaves and twigs of Calyptranthes pallens [1898].

-From the fern of Elaphoglossum spathulatum [2917].

-From the leaves of *Dryopteris villarii* and *Dryopteris arguta* (Pteridaceae) [3328].

-From the leaves of Currania robertiana (Pteridaceae) [3328].

-From the rhizomes of Dryopteris crassirhizoma (Dryopteridaceae) [2208].

-Also refer to: [386, 1270, 1610, 2414, 2612, 2713, 3102, 3297].

yellow needles [1898];

m.p. 156–161° [3102], 144–145° [1898], 143° [386], 142–143° [2713, 3297], 141–143° [3302], 141° [2620], 140–141° [36, 1609]; ¹H NMR [1898, 2208, 3328], ¹³C NMR [204, 205, 1898, 3328], IR [1898, 3302], UV [35, 1898], MS [1898, 2208, 2258, 3328]; GLC [2531]; HPLC [3328].

N.B.: There are two different representations of the Aspidinol:

*1-(2,6-Dihydroxy-4-methoxy-3-methylphenyl)-1-butanone (847) [3102] page 140. *1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)-1-butanone (I) [1610] page 466.

BIOLOGICAL ACTIVITY: Antiinfective ointment containing Dryopteris fragrans. The Dryopteris fragrans extract comprises dryofragin, aspidin AB, aspidin PB, aspidin BB, aspidinol and albicanol [2852]; QSAR vasodilatory activity relationship of resveratrol-coumarin hybrids [3225]; Cytotoxicity [1898]; Antitumor [1471]; Fatty acid synthase inhibitory activity [2208].

Diacetate

 $C_{16}H_{20}O_{6}$

mol. wt. 308.33

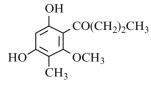
m.p. 68° [2630], 64–66° [2133].

1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)-1-butanone

(Pseudoaspidinol B)

[478-48-8]

C₁₂H₁₆O₄ mol. wt. 224.26



Syntheses CO(CH₂)₂CH₃ -Obtained by reductive alkaline cleavage of Aspidin BB (VII) from *Dryyoteris assimilis* [1916] (IVd). -Also obtained by reaction of butyryl chloride with methyl 2,6-dihydroxy-4-methoxy-3-methylbenzoate in the presence of aluminium chloride [2436].

-Also obtained by reaction of butyronitrile with 5-methoxy-4-methylresorcinol (Hoesch reaction) [1609].

-Also obtained by hydrolysis of 1-[4,6-bis(1,1-dimethylethoxy)-2-methyl-3-methoxyphenyl]-1-butanone with hydrochloric acid in ethanol [1213]. -Also refer to: [2448 (**X**), 2534, 2612 (**VIII**), 2860, 3302].

Isolation from natural sources

-From the roots of Dryopteris championii [3480].

colourless plates; m.p. 116.5° [1609], 79–80° [2534], 72–73° [3302], 70–72° [1916, 2436]. **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [1916, 2436, 2534, 3302], ¹³C NMR [205], IR [2436, 3302], UV [2436, 2448], MS [1916, 2534]; GLC [2531].

Monohydrate $C_{12}H_{16}O_4, H_2O$ mol. wt. 242.27

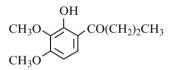
-Refer to: [2611, 2612 (VIII)].

4,6-Dimethylethyl ether [172219-19-1] C₂₀H₃₂O₄ mol. wt. 336.47

-Obtained by oxidation of 1-[4,6-bis(1,1-dimethylethoxy)-2-methoxy-3-methylphenyl]-1-butanol with NACAA (nicotinic-chromic anhydride) in methylene chloride in the presence of pyridine [1213].

1-(2-Hydroxy-3,4-dimethoxyphenyl)-1-butanone

 $[75218-94-9] C_{12}H_{16}O_4 mol. wt. 224.26$



Syntheses

 $CO(CH_2)_2CH_3$ -Preparation by reaction of butyryl chloride with pyrogallol trimethyl ether in the presence of aluminium chloride in cooled ethyl ether for 4 h (30 %) [57].

-Also refer to: [60, 1787].

OCH3

pale yellow plates [57]; m.p. 53–54° [57]; ¹H NMR [57].

1-(2-Hydroxy-4,5-dimethoxyphenyl)-1-butanone

$$[91555-34-9] C_{12}H_{16}O_4 mol. wt. 224.26$$
OH
$$CO(CH_2)_2CH_3$$

$$CH_2O$$

$$CH_2O$$

$$CH_2O$$

$$CH_2O$$

$$CO(CH_2)_2CH_3$$

$$CO(CH_2)_2CH_3$$

$$CH_2O$$

-Also obtained by reaction of n-butyronitrile with 3,4-dimethoxyphenol in the presence of zinc chloride (Houben-Hoesch reaction) (poor yield) [1537]. -Also refer to: [166, 1787].

15.5 h (25 %) [2127].

white crystals [2127], nadeln [1537]; m.p. 81° [166, 1537], 77–78° [2127]; ¹H NMR [2127], IR [2127].

BIOLOGICAL ACTIVITY: Central nervous system depressant [2127].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-1-butanone

 $\label{eq:constraint} [2999-37-3] \qquad \qquad \text{C}_{12}\text{H}_{16}\text{O}_4 \qquad \qquad \text{mol. wt. } 224.26$

Syntheses

CH₃O OH CH₃O OCH₃

 $CO(CH_2)_2CH_3$ -Preparation by reaction of butyryl chloride with phloroglucinol trimethyl ether in the presence of aluminium chloride in cooled ethyl ether for 4 h (53 %) [57].

-Also obtained by reaction of butyronitrile with 3,5-dimethoxyphenol [544, 1547]. -Also obtained by reaction of dimethyl sulfate with phlorobutyrophenone in the presence of potassium carbonate in refluxing acetone for 30–40 min (80–90 %) [2014].

-Also obtained by treatment of 2,4,6-trimethoxybutyrophenone with boron trichloride in methylene chloride at $0-5^{\circ}$ for 2.5 h (85 %) [3068].

-Also obtained by degradation of dimethyllathodoratin (5,7-dimethoxy-3-ethylchromone) with potassium hydroxide in refluxing 50 % aqueous ethanol for 3 h (51 %) [83].

-Also refer to: [60, 545, 1464, 3069].

Isolation from natural sources

-From *Dysophylla stellata* Benth. (Labiatae) [1547]. -From *Dysophylla tomentosa* (Labiatae) [2242].

yellow flakes [57]; m.p. 72–74° [2242], 72–73° [1547], 70–71° [57, 83], 70° [544], 68° [203]; ¹H NMR [57, 83, 1547, 2242], ¹³C NMR [204], IR [1547, 2242], UV [1547, 2242], MS [1547, 2242]; GLC [2531].

1-(4-Hydroxy-2,5-dimethoxyphenyl)-1-butanone

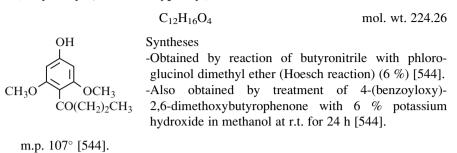
[105476-01-5]	$C_{12}H_{16}O_4$	mol. wt. 224.26
ОН	Syntheses	

CH₃O OCH₃ CO(CH₂)₂CH₃

-Obtained by reaction of n-butyronitrile with 2,5-dimethoxyphenol (Hoesch reaction) (4 %) [166]. -Also obtained from 4-butyryl-2,5-dihydroxyphenyl glucosiduronic acid (compound **A**) [166].

elongated plates [166]; b.p._{0.1} 120–140° [166]; m.p. 86–88° [166], 85–86° [166], IR [166], UV [166].

1-(4-Hydroxy-2,6-dimethoxyphenyl)-1-butanone



Benzoate

C19H20O5 mol. wt. 328.36

m.p. 86° [544].

1-(4-Hydroxy-3,5-dimethoxyphenyl)-1-butanone

(Butyrylsyringone)

[69271-91-6]

	ОН
CH ₃ O	JOCH ₃
Ĩ	
	Ý

Syntheses -Refer to: [1180, 1514, 2738, 2887]. Isolation from natural sources -In oak wood chips [3222]. CO(CH₂)₂CH₃ -In liquid smoke flavoring preparations [1179].

m.p. 80-82° [2887]; MS [1179, 2887, 3222]; GC-MS [1179, 3222].

BIOLOGICAL ACTIVITY: Antioxidant and organoleptic properties [1179]. -Also refer to: [2738].

 $C_{12}H_{16}O_{4}$

1-(5-Hydroxy-2,4-dimethoxyphenyl)-1-butanone

[105475-57-8] C12H16O4 mol. wt. 224.26 OН Syntheses

-Obtained by treatment of potassium 5-butyryl-2,4-di-hydroxyphenylsulphate (compound \mathbf{B}) in methanol with an excess of ethereal diazomethane for 12 h (23 %) [166].

-Preparation by diazotization of 5-amino-2,4-dimethoxy-butyrophenone (22 %) [166].

pink plates [166]; m.p. 87–88° [166]; UV [166].

mol. wt. 224.26

1-(2,4,6-Trihydroxy-3,5-dimethylphenyl)-1-butanone

[84633-27-2]	$C_{12}H_{16}O_4$	mol. wt. 224.26
$\begin{array}{c} OH\\ CH_3 \\ HO \\ CH_3 \\ HO \\ CH_3 \\ OH \\ CH_3 \end{array} OH$	Syntheses -Preparation by reaction 2,4-dimethylphloroglucino tion) [539, 1610] (XVII)]. -Also refer to: [724, 2414,	ol (Houben-Hoesch reac-

Isolation from natural sources

Benzyl ether

-From Dryopteris abbreviata (DC) NEWMAN (Aspidiaceae) [724].

yellow needles [724]; m.p. 140–141° [2451], 140° [539, 1610], 135–137° [724]; ¹H NMR [724], IR [724], UV [540, 724], MS [724]; TLC [724]; HPLC [724].

1-[4-Hydroxy-3-(methylsulfonylmethyl)phenyl]-1-butanone

[56490-73-4]

[56490-66-5]	$C_{12}H_{16}O_4S$	mol. wt. 256.32
OH CH ₂ SO ₂ CH ₃ CO(CH ₂) ₂ CH ₃	Synthesis -Obtained by reaction of 3-chloromethy phenone with magnesium methylsulfina methanol for 18 h (50 %) [1573]. m.p. 130–132° [1573].	

-Obtained by treatment of the title ketone with benzyl chloride in the presence of potassium carbonate and sodium iodide in refluxing acetone for 18 h (61 %) [1573].

 $C_{19}H_{22}O_4S$

mol. wt. 346.45

m.p. 120–122° [1573]; ¹H NMR [1573].

1-(4-Chloro-8-hydroxy-3-quinolinyl)-1-butanone

[125500-45-0]	$C_{13}H_{12}CINO_2$	mol. wt. 249.70
OH N CO(CH ₂) ₂ CH ₃	Synthesis -Refer to: [1099]. Methyl ether [115607-76-6] $C_{14}H_{14}CINO_2$	mol. wt. 263.72

-Refer to: [170, 1099, 1420, 1579, 1841].

m.p. 114–116° [1420, 1841]; ¹H NMR [1420].

4-Hydroxy-3-(1-oxobutyl)-2H-1-benzopyran-2-one

 $[4139-74-6] C_{13}H_{12}O_4 mtext{mol. wt. } 232.24$

OH CO(CH₂)₂CH₃

Syntheses -Obtained by Fries rearrangement of 4-butyryloxycoumarin in the presence of several metal halides as catalysts at 100–150° for 30– 60 min. Similarly the conversion was accomplished in nitrobenzene [1705].

The yields of 3-acyl-4-hydroxycoumarin were as follows:

- TiCl₄ (74 %); AlBr₃ (50 %); AlCl₃ (43 %); SnCl₄ (25 %); FeCl₃ (23 %), CrCl₃ (21 %).
- N.B.: SnCl₂, SiCl₄, HgPh₂; AlPh₃, HgCl₂, ZnCl₂, Ph₃SnCl and PhHgCl were unreactive [1705].

-Also obtained by reaction of butyryl chloride with 4-hydroxycoumarin in pyridine containing one drop of piperidine for 3 h on a water bath (42 %) [3174]. -Also refer to: [525, 610, 2306, 3144].

m.p. 120-121° [3174].

1-(5-Bromo-6-hydroxy-3-methyl-7-benzofuranyl)-1-butanone

[106320-28-9]

 $C_{13}H_{13}BrO_3$

mol. wt. 297.15

CO(CH₂)₂CH₃ HO Br CH₃ Synthesis -Obtained by decarboxylation of 5-bromo-6-hydroxy-3-methyl-7-(1-oxobutyl)-2-benzofurancarboxylic acid on heating for 10 min at 255° [2827].

m.p. 103° [2827].

Oxime	[100380-49-2]	$C_{13}H_{14}BrNO_3$	mol. wt. 312.16

-Refer to: [2827].

m.p. 196° [2827].

1-(5-Chloro-6-hydroxy-3-methyl-7-benzofuranyl)-1-butanone

[100712-42-3]	$C_{13}H_{13}ClO_3$	mol. wt. 252.70

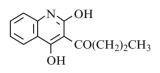
$CO(CH_2)_2CH_3$	Synthesis
HO	-Obtained
ĬŢ ĬŢ Ĭ	3-methyl
Cl CH ₃	heating for

-Obtained by decarboxylation of 5-chloro-6-hydroxy-3-methyl-7-(1-oxobutyl)-2-benzofurancarboxylic acid on heating for 10 min at 255° [2827].

m.p. 102° [2827].

1-(2,4-Dihydroxy-3-quinolinyl)-1-butanone

C ₁₃ H ₁₃ NO ₃	mol. wt. 231.24
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OH Synthesis -Obtained by reaction of butyryl chloride with 2,4-dihydroxyquinoline in the presence of aluminium chloride in nitrobenzene on the steam bath for 3 h (25 %) [3123].

m.p. 218–219° [3123]; UV [3123].

BIOLOGICAL ACTIVITY: Antibacterial properties (Staphylococcus aureus and Escherichia coli) [3123].

C₁₃H₁₄O₃

1-(6-Hydroxy-3-methyl-2-benzofuranyl)-1-butanone

[99245-46-2]

^cH₃ Syntheses
 -Obtained by Fries rearrangement of 6-butyryloxy-3-methylbenzofuran (m.p. 45°) with aluminium chloride in nitrobenzene at r.t. overnight [2825].

-Also refer to: [2826].

m.p. 124° [2825, 2826].

Acetate	[100976-33-8]	$C_{15}H_{16}O_4$	mol. wt. 260.29
m.p. 85–86°	[2825, 2826].		

Methyl ether	[56397-48-9]	$C_{14}H_{16}O_{3}$	mol. wt. 232.28
00.040.50005	20263		

m.p. 93–94° [2825, 2826].

1-(6-Hydroxy-3-methyl-7-benzofuranyl)-1-butanone

[107623-54-1]		$C_{13}H_{14}O_3$	mol. wt. 218.25
CO(CII) CII	Sunthagag		

CH_3	Syntheses
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-Obtained by decarboxylation of 6-hydroxy-7-butyryl-3-methylcoumarilic acid [2826].

-Also obtained by alkaline hydrolysis of 3-bromo-7-hydroxy-8-butyryl-4-methylcoumarin (m.p. 146°) [2826].

m.p. 83° [2826].

$CO(CH_2)_2CH$	-3
$HO_{1} \downarrow 0$	
💛 `сн	2

mol. wt. 218.25

N-(3-Butanoyl-2-hydroxy-5-methylphenyl)-1H-tetrazole-5-carboxamide

1-[4-Hydroxy-3-(2-propenyl)phenyl]-1-butanone

$$[97023-54-6] C_{13}H_{16}O_2 mol. wt. 204.27$$

$$OH Syntheses -Obtained by Claisen rearrangement of 4-allyloxy-butyro-phenone in boiling dimethylaniline for 5-6 h [511]. -Also refer to: [467]. b.p._{16} 206-207^{\circ} [511]; m.p. 64^{\circ} [511].$$

USE: As intermediate for squalene synthase inhibitors [467].

1-[2,4-Dihydroxy-3-(2-propenyl)phenyl]-1-butanone

[194792-30-8] C13H16O3 mol. wt. 220.27

$$CH_2 = CHCH_2 \xrightarrow{I} CO(CH_2)_2CH_3$$
 -Refer to: [22].

1-[2-Hydroxy-4-(2-propenyloxy)phenyl]-1-butanone

[194792-29-5] C₁₃H₁₆O₃ mol. wt. 220.27 Synthesis OH $CO(CH_2)_2CH_3$ -Refer to: [22]. $CH_2 = CHCH_2O$

1-[2,4,6-Trihydroxy-3-(2-propenyl)phenyl]-1-butanone 2-Butyryl-4-(propen-2-yl)phloroglucinol (16) [1026]

 $C_{13}H_{16}O_4$

Syntheses

-Obtained by adding cuprous chloride, a saturated aqueous solution of sodium carbonate and allyl chloride to a solution of phlorobutyrophenone in ethyl ether at r.t., then to keep the mixture for 12 h at this temperature [1026].

$$CH_2 = CHCH_2 \xrightarrow{OH} CO(CH_2)_2CH_3$$

$$HO \xrightarrow{OH} OH$$

mol. wt. 236.27

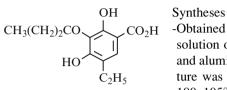
-Also refer to: [1501, 1502].

¹H NMR [1026], ¹³C NMR [1026], IR [1026].

BIOLOGICAL ACTIVITY: Antimicrobial [1026, 1501, 1502].

1-[5-Ethyl-2,4-dihydroxy-3-(1-oxobutyl)]benzoic acid

[106214-14-6]



CO₂H -Obtained by adding butyric anhydride to a cold solution of methyl 2,4-dihydroxy-5-ethylbenzoate and aluminium chloride in nitrobenzene. The mixture was left overnight at r.t., and then heated at 100–105° for 4 h (16 %) [2811].

-Also obtained by hydrolysis of the methyl ester with 10 % NaOH on a water bath for 1 h and then kept overnight at r.t. [2811].

m.p. 181° [2811].

Methyl ester [108125-65-1] $C_{14}H_{18}O_5$ mol. wt. 266.29

-Obtained at the same time in the first reaction above mentioned (20 %) [2811].

m.p. 66° [2811].

Dibenzoate of the methyl ester [103098-69-7] $C_{28}H_{26}O_7$ mol. wt. 474.51

m.p. 108° [2811].

1-(3-Chloro-2,6-dihydroxy-5-propylphenyl)-1-butanone

[120058-71-1] $C_{13}H_{17}ClO_3$ mol. wt. 256.73 OH Synthesis $C_{3}H_7$ CO(CH₂)₂CH₃ -Refer to: [1836 (1 h)]. OH Cl

1-(2,4,6-Trihydroxy-3-nitro-5-propylphenyl)-1-butanone

[119691-99-5]	$C_{13}H_{17}NO_6$	mol. wt. 283.28
$\begin{array}{c} OH\\ NO_2 \\ HO \\ C_3H_7 \end{array} CO(CH_2)_2CH_3 \\ OH \\ C_3H_7 \end{array}$	Synthesis -Obtained by adding a mixture and acetic acid to the solution droxy-3-propylphenyl)-1-butar 60° for 30 min (30–40 %) [34]	on of 1-(2,4,6-trihy- none in acetic acid at

m.p. 67–69° [3414]; ¹H NMR [3414], IR [3414], MS [3414].

BIOLOGICAL ACTIVITY: Germination inhibitory activity [3414]; PET inhibition [3414].

1-(2-Ethyl-6-hydroxy-4-methylphenyl)-1-butanone

	$C_{13}H_{18}O_2$		mol. wt. 206.28
OH CO(CH ₂) ₂ CH ₃ CH ₃ CH ₂ CH ₃	5-methyl-phe	enyl butyra	rearrangement of 3-ethyl- te with aluminium chloride, o for 2 h (79 %) [2802];

*in nitrobenzene at 25° for 6 h (80 %) [2802].

m.p. 58° [2802].

Methyl ether

 $C_{14}H_{20}O_{2}$

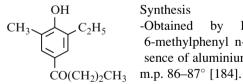
mol. wt. 220.31

-Obtained by methylation of the above ketone in the usual way [2802].

b.p.₂₈ 175° [2802].

1-(3-Ethyl-4-hydroxy-5-methylphenyl)-1-butanone

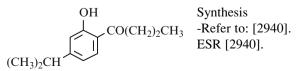
 $C_{13}H_{18}O_{2}$ mol. wt. 206.28



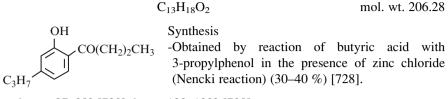
Synthesis -Obtained by Fries rearrangement of 2-ethyl-6-methylphenyl n-butyrate (b.p. 258–261°) in the presence of aluminium chloride (53 %) [184].

C13H18O2

mol. wt. 206.28



1-(2-Hydroxy-4-propylphenyl)-1-butanone



C14H21N3O2

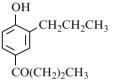
b.p.₀₇ 87–88° [728], b.p.₁₉ 130–132° [728].

Semicarbazone

vellow solid [728]; m.p. 175° [728].

1-(4-Hydroxy-3-propylphenyl)-1-butanone

C13H18O2 mol. wt. 206.28



HO

Synthesis

CH₂CH₂CH₃ -Obtained by reaction of butyryl chloride with 2-propylphenol in the presence of aluminium chloride in nitrobenzene at r.t. overnight [2648]. b.p.₁₄ 210° [2648]; m.p. 54° [2648].

1-(2,4-Dihydroxy-3-propylphenyl)-1-butanone

[194792-31-9] C13H18O3 mol. wt. 222.28 Syntheses OH CO(CH₂)₂CH₃ -Refer to: [20, 22, 307]. C₃H₇.

1-(2,4-Dihydroxy-5-propylphenyl)-1-butanone

	$C_{13}H_{18}O_3$			mol. wt. 22	2.28
HO CH ₂ CH ₂ CH ₂ CH ₃	4-propylres	orcinol h alumii [2651].	nium chlo	rearrangement yrate/4-propylresord pride in nitrobenzer [2651].	

C19H24N2O2

m.p. 139–140° [2651].

Phenylhydrazone

mol. wt. 263.34

mol. wt. 312.41

1-(5-Ethyl-2-hydroxy-4-methoxyphenyl)-1-butanone

[178754-67-1]	$C_{13}H_{18}O_3$	mol. wt. 222.28
CH ₃ O CH ₃ O C ₂ H ₅ O	butyric acid in the pres	of 4-ethylresorcinol with sence of zinc chloride and de, followed by methyla- hydroxy group [2397].

1-(2-Hydroxy-4-propoxyphenyl)-1-butanone

$C_{13}H_{18}O_3$		mol. wt. 222.28
OH	Synthesis	
$CO(CH_2)_2CH_3$	-Refer to: [851].	
	Oxime [168978-08-3]	
C ₃ H ₇ O	C ₁₃ H ₁₉ NO ₃	mol. wt. 237.30

USE: Reagent for copper determination by gravimetry and spectrophotometry [851].

1-(2,6-Dihydroxy-4-methoxy-3,5-dimethylphenyl)-1-butanone

(Methylaspidinol) (V)

[4069-47-0]	$C_{13}H_{18}O_4$	mol. wt. 238.28
$CH_{3} \xrightarrow{OH} CO(CH_{2})_{2}CH_{3}$ $CH_{3}O \xrightarrow{H} OH CH_{3}OH$	Syntheses -Obtained by alkaline cleavage of with zinc dust in 5 % sodium hydrogenetic bath for 5 min [2445]. -Also refer to: [37, 2442, 2451, 3	droxide on a water
m.p. 111° [2445, 3113], 109	9–111° [2451], 108–110° [37];	UV [37].

1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-1-butanone

[69480-08-6]	$C_{13}H_{18}O_4$	mol. wt. 238.28
CH ₃ CH ₃ O CH ₃ O CH ₃ O OCH ₃ O	Syntheses -Refer to: [203, 2531, 2630]. m.p. 111–112° [2630], 108–110° ¹³ C NMR [203]; GLC [2531].	[203];

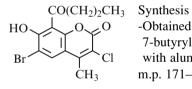
1-[2,4-Dihydroxy-3-(2-hydroxyethyl)-6-methoxyphenyl]-1-butanone (*Phomalone*)

USE: Wood and lawn fungicide [202].

1-[3-[(Dimethylamino)methyl]-2,4,5-trihydroxyphenyl]-1-butanone

6-Bromo-3-chloro-7-hydroxy-4-methyl-8-(1-oxobutyl)-2H-1-benzopyran-2-one

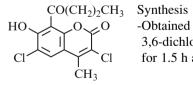
$C_{14}H_{12}BrClO_4$	mol. wt. 359.60
C141112D1C104	11101. wt. 557.00



Synthesis -Obtained by Fries rearrangement of 6-bromo-7-butyryloxy-3-chloro-4-methylcoumarin (m.p. 138°) with aluminium chloride for 1.5 h at 155° [2827]. m.p. 171–172° [2827].

3,6-Dichloro-7-hydroxy-4-methyl-8-(1-oxobutyl)-2H-1-benzopyran-2-one

$$C_{14}H_{12}Cl_2O_4$$
 mol. wt. 315.15

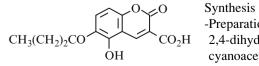


-Obtained by Fries rearrangement of 7-butyryloxy-3,6-dichloro-4-methylcoumarin with aluminium chloride for 1.5 h at 155° [2827].

5-Hydroxy-6-(1-oxobutyl)-2H-1-benzopyran-2-one-3-carboxylic acid

 $C_{14}H_{12}O_6$

mol. wt. 276.25



-Preparation by the condensation of the 2,4-dihydroxy-3-formylbutyrophenone with cyanoacetic acid [2821].

straw-coloured needles [2821]; m.p. 198–200° (d) [2821].

6-Bromo-7-hydroxy-4-methyl-8-(1-oxobutyl)-2H-1-benzopyran-2-one

[110029-30-6]	$C_{14}H_{13}BrO_4$	mol. wt. 325.16
CO(CH ₂) ₂ CH ₃ HO O O	Syntheses -Obtained by Pechmann condensat 3-bromophenyl)-1-butanone with	

presence of 80 % sulfuric acid [2811].
-Also obtained by Fries rearrangement of 7-butyryloxy6-bromo-4-methylcoumarin (m.p. 120°) with aluminium

chloride at 145–150° for an hour (40 %) [2811].

yellow needles [2811]; m.p. 157° [2811].

Semicarbazone	[109262-64-8]	$C_{15}H_{16}BrN_3O_4$	mol. wt. 382.21
colourless granules	; m.p. 231° (d) [281	1].	

Acetate	[109602-86-	0]	$C_{16}H_{15}BrO_{5}$	5 m	ol. wt. 367.20
colourless nee	edles [2811];	m.p. 128°	[2811].		

Benzoate	[112222-68-1]	$C_{21}H_{17}BrO_5$	mol. wt. 429.27
	1	1 5 1 0 5 0 0 1 1 3	

colourless lustrous cubes [2811]; m.p. 151° [2811].

5-Bromo-6-hydroxy-3-methyl-7-(1-oxobutyl)-2-benzofurancarboxylic acid

[108955-15-3]	$C_{14}H_{13}BrO_5$	mol. wt. 341.16
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CO($CH_2)_2CH_3$
HO	O_CO ₂ H
	I
Br	CH ₃

Synthesis -Obtained by Fries rearrangement of 5-bromo-6-butyryloxy-3-methylcoumarilic acid (m.p. 261°) with aluminium chloride for 1 h at 150–160° [2827].

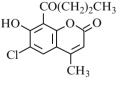
m.p. 268° [2827].

6-Chloro-7-hydroxy-4-methyl-8-(1-oxobutyl)-2H-1-benzopyran-2-one

[109103-09-5]

 $C_{14}H_{13}ClO_4$

mol. wt. 280.78



₃ Syntheses

-Obtained by Pechmann condensation of 1-(2,6-dihydroxy-3-chlorophenyl)-1-butanone with ethyl acetoacetate in presence of 80 % sulfuric acid [2811].

-Also obtained by Fries rearrangement of 7-butyryloxy-6-chloro-4-methylcoumarin (m.p. 119°) (33 %) [2811].

yellow fibrous needles [2811]; m.p. 153° [2811].

Oxime	$C_{14}H_{14}CINO_4$		mol. wt. 295.72
colourless no	eedles [2811]; m.p. 197	7° (d) [2811].	
Acetate colourless ne	C ₁₆ H ₁₅ C eedles [2811]; m.p. 139	5	mol. wt. 322.74
Benzoate colourless lu	[112222-67-0] Istrous cubes [2811]; m	C ₂₁ H ₁₇ ClO ₅ n.p. 144° [2811].	mol. wt. 384.82

4-(Chloromethyl)-5,7-dihydroxy-6-(1-oxobutyl)-2H-1-benzopyran-2-one

Syntheses

[98498-60-3]

$$C_{14}H_{13}ClO_5$$
 mol. wt. 296.71

HO CH₃(CH₂)₂CO OH CH₂Cl

-Obtained by condensation of 2-butyrylphloroglucinol with ethyl 4-chloro-3-oxobutanoate (61 %) [763].

-Also obtained from a Pechmann reaction (low yield) [766].

yellow solid [763]; m.p. 225–230° (d) [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

4-(Chloromethyl)-5,7-dihydroxy-8-(1-oxobutyl)-2H-1-benzopyran-2-one

ethyl 4-chloro-3-oxobutanoate (3 %) [763].

m.p. 210–212° (d) [763];

OH CH₂Cl ¹H NMR [763], IR [763], UV [763], MS [763].

5-Chloro-6-hydroxy-3-methyl-7-(1-oxobutyl)-2-benzofurancarboxylic acid

[109103-12-0]

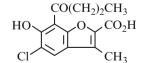
$$C_{14}H_{13}ClO_5$$

mol. wt. 296.71

Synthesis

 CO_2H -Obtained by Fries rearrangement of 5-chloro-6-butyryloxy-3-methylcoumarilic acid with aluminium CH₃ chloride for 1 h at 150–160° [2827].

m.p. 255° [2827].



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5-Hydroxy-4-methyl-6-(1-oxobutyl)-2H-1-benzopyran-2-one

[855159-27-2] C₁₄H₁₄O₄ mol. wt. 246.26

Syntheses -Obtained by condensation of resbutyrophenone with ethyl acetoacetate in the presence of aluminium chloride in nitrobenzene for 1 h at $120-130^{\circ}$ (37 %) [841].

-Also obtained by Fries transformation of 5-butyroxy-4-methylcoumarin (m.p. 100–101°) with aluminium chloride [841]. -Also refer to: [2824].

m.p. 141–142° [841, 2824].

Acetate $C_{16}H_{16}O_5$ mol. wt. 288.30

-Preparation by means of acetic anhydride in pyridine with the title ketone [841].

m.p. 167° [841].

Methyl ether $C_{15}H_{16}O_4$ mol. wt. 260.29

-Obtained by reaction of methyl iodide with the title ketone in the presence of potassium carbonate in refluxing acetone for 24 h [841].

m.p. 83–84° [841].

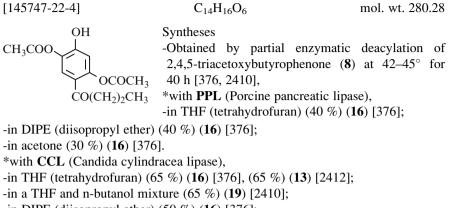
6-Hydroxy-7-(1-oxobutyl)-3-methylcoumarilic acid

[109564-87-6]	$C_{14}H_1$	₄ O ₅	mol. wt. 262.26
HO CO(CH ₂) ₂ CH ₃ HO CO ₂ H CH ₃	3-methyl-coum	U	nt of 6-butyryloxy- ^{77°} (d)] with alumin- 2826].
m.p. 218° [2826].			
Acetate [13 m.p. 211° [2826].	0907-67-4]	$C_{16}H_{16}O_{6}$	mol. wt. 304.30
Methyl ether	[109641-80-7]	$C_{15}H_{16}O_5$	mol. wt. 276.29

-Refer to: [2826].

m.p. 107-108° [2826].

1-[2,5-Bis(acetyloxy)-4-hydroxyphenyl]-1-butanone



-in DIPE (diisopropyl ether) (50 %) (16) [376];

-in acetone (30 %) (**16**) [376].

m.p. 142–143° [376]; ¹H NMR [376], MS [376], UV [376]; TLC [376].

1-[3-Bromo-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-butanone

[100792-29-8]	$C_{14}H_{19}BrO_2$	mol. wt. 299.21
$\begin{array}{c} OH\\ Br \\ \downarrow \\ C(CH_3)_3 \end{array} CO(CH_2)_2CH_3$	Synthesis -Obtained by Fries rearrangement of butylphenyl butyrate with aluminium for 2 h (65 %) [3113]. b.p. ₂ 150° [3113].	

2,4-Dinitrophenylhydrazone $C_{20}H_{23}BrN_4O_5$ mol. wt. 479.33

m.p. 175° [3113].

1-[3-Chloro-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-butanone

[106321-41-9]	$C_{14}H_{19}ClO_2$	mol. wt. 254.76
$\begin{array}{c} OH\\ Cl & \downarrow\\ & \downarrow\\ & \downarrow\\ & \downarrow\\ & C(CH_3)_3 \end{array}$	Synthesis -Obtained by Fries rearrangeme butylphenyl butyrate with alum (80 %) [3119]. b.p. ₁₀ 152° [3119].	

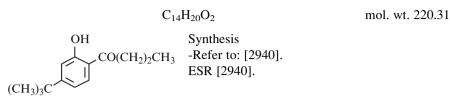
2,4-Dinitrophenylhydrazone

 $C_{20}H_{23}CIN_4O_5$

mol. wt. 434.88

m.p. 176° [3119].

1-[4-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-butanone



1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-butanone

[75060-53-6] $C_{14}H_{20}O_2$ mol. wt. 220.31

$$\bigcup_{C(CH_3)_3}^{OH}$$

Syntheses -Obtained by Fries rearrangement of 4-tert-butylphenyl butyrate with aluminium chloride in carbon disulfide at r.t., then at 120° for 1 h after solvent elimination (57 %) [2796].

-Also obtained by treatment of 2-methoxy-5-tert-butyl-butyrophenone with 47 % hydrobromic acid/57 % hydriodic acid mixture in refluxing acetic acid for 2 h (81 %) [1475].

b.p.₂₀ 150° [2796]; ¹H NMR [1475]; ESR [2940]; TLC [1475].

Methyl ether	[75060-46-7]	$C_{15}H_{22}O_2$	mol. wt. 234.34
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-Obtained by reaction of butyryl chloride with 4-tert-butylanisole in the presence of aluminium chloride in methylene chloride under nitrogen, first at 0°, then at 20° for 30 min (85 %) [1475].

¹H NMR [1475]; TLC [1475].

1-[2-Hydroxy-3-methyl-6-(1-methylethyl)phenyl]-1-butanone

	$C_{14}H_{20}O_2$		mol. v	wt. 22	20.31
CH ₃ CH ₃ CO(CH ₂) ₂ CH ₃ CH(CH ₃) ₂	Synthesis -Preparation by 1 butyrate with (70 %) [2798]. b.p. ₃ 142° [2798].	aluminium			

1-[2-Hydroxy-3-(1-methylethyl)-6-methylphenyl]-1-butanone

[106476-93-1]	$C_{14}H_{20}O_2$	mol. wt. 220.31
(CH ₃) ₂ CH (CH ₃) ₂ CH (CH ₂) ₂ CH ₃ (CH ₃)	Syntheses -Obtained by Fries butyrate with alu solvent at 120° (85	rearrangement of thymyl minium chloride without %) [2803].

-Also obtained by reaction of butyric acid with thymol in the presence of aluminium chloride on heating at reflux for 12 h (81 %) [2960]. -Also refer to: [2961].

b.p.₂ 190° [2803], b.p.₁₂ 120–121° [2960, 2961].

2,4-Dinitrophenylhydrazone $C_{20}H_{24}N_4O_5$ mol. wt. 400.43

m.p. 216° [2803].

1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-butanone

[95185-72-1]	$C_{14}H_{20}O_2$	mol. wt. 220.31
(CH ₃) ₂ CH CH ₃ CO(CH ₂) ₂ CH ₃	Syntheses -Obtained by Fries rearrang with aluminium chloride i *first 2 h at 30°, then at r.t. *for 12 h at 30° (86 %) [26	n nitrobenzene, for 24 h [2704];

-Also obtained by refluxing its methyl ether with pyridinium chloride $(205-215^{\circ})$ for 40 min (40 %) [2660].

b.p.₁₄ 204° [2648], b.p.₁₄ 203–205° [2660]; m.p. 94° [2660], 93–94° [2647].

Methyl ether [854870-33-0] C₁₅H₂₂O₂ mol. wt. 234.34

-Obtained by reaction of butyryl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (50 %) [2660]. -Also refer to: [1652, 2647].

b.p.₁₆ 175–178° [2660]; m.p. 50° [2647, 2660].

1-[4-Hydroxy-5-methyl-2-(1-methylethyl)phenyl]-1-butanone

 $(CH_{3})_{2}CH \xrightarrow{OH}_{CO(CH_{2})_{2}CH_{3}} CH_{3} \xrightarrow{OH}_{CO(CH_{2})_{2}CH_{3}} CO(CH_{2})_{2}CH_{3} CO(CH_{2})_$

-Also obtained by Fries rearrangement of carvacryl butyrate with aluminium chloride in nitrobenzene for 24 h at 25° (88 %) [2647].

b.p.₁₅ 200° [2650]; m.p. 66° [1522, 2647, 2650].

1-(3,5-Diethyl-4-hydroxyphenyl)-1-butanone

[104008-48-2]

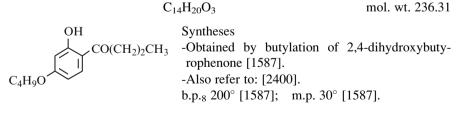
 $C_{14}H_{20}O_2$ mol. wt. 220.31 Syntheses

 $C_{2}H_{5} \underbrace{\downarrow}_{CO(CH_{2})_{2}CH_{3}}^{OH} C_{2}H_{5}$ Syntheses -Refer to: [3005, 3006].

1-[4-Hydroxy-3-(1-methylpropyl)phenyl]-1-butanone

 $[16648-70-7] C_{14}H_{20}O_2 mtext{mol. wt. } 220.31$

1-(4-Butyloxy-2-hydroxyphenyl)-1-butanone



Oxime[161138-02-9] $C_{14}H_{21}NO_3$ mol. wt. 251.33

m.p. 106° [1587].

USE: Nickel (II) and copper (II) simultaneous detn. in synthetic mixts. and alloy samples using this oxime as an extractive spectrophotometric reagent [2400]; Spectrophotometric and gravimetric reagent for Cu (II) [3048].

2,4-Dinitrophenylhydrazone	$C_{20}H_{24}N_4O_6$	mol. wt. 416.43
_,	-20244-0	

m.p. 187° [1587].

1-(4-Ethoxy-3-ethyl-2-hydroxyphenyl)-1-butanone

$$\begin{array}{cccc} C_{14}H_{20}O_3 & \text{mol. wt. 236.31} \\ OH & Synthesis \\ C_2H_5 & CO(CH_2)_2CH_3 & -Obtained & by & ethylation & of \\ 2,4-dihydroxybutyrophenone [1587]. \\ b.p._5 & 155^{\circ} [1587]; & m.p. 45^{\circ} [1587]. \end{array}$$

Oxime

m.p. 141° [1587].

2,4-Dinitrophenylhydrazone $C_{20}H_{24}N_4O_6$ mol. wt. 416.43

C₁₄H₂₁NO₃

m.p. 201° [1587].

1-[2-Hydroxy-4-(β-methoxyethoxymethoxy)-6-methylphenyl]-2-(methylsulfinyl)-1-ethanone

m.p. 106–107° [1157]. ¹H NMR [1157], IR [1157].

1-[3-Amino-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-butanone

$$C_{14}H_{21}NO_2$$

mol. wt. 235.33

cooled to 35° and treated dropwise with ethyl 2-hydroxy- $4-(\beta-methoxyethoxy)methoxy-$ 6-methylbenzoate (86 %) [1157].

 $\begin{array}{c} OH & Synthesis \\ NH_2 & & CO(CH_2)_2CH_3 & -Refer to: [2105]. \\ & & C(CH_3)_3 \end{array}$

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.

mol. wt. 251.33

1-[2-(N,N-Dimethylaminoethoxy)-4-hydroxyphenyl]-1-butanone

	$C_{14}H_{21}NO_3$	mol. wt. 251.33
OH I	Synthesis -Refer to: [787].	
	Methyl ether (hydrochloride)	
OCH ₂ CH ₂ N(CH ₃) ₂ CO(CH ₂) ₂ CH ₃	$C_{15}H_{23}NO_3$, HCl	mol. wt. 301.81

-Obtained by adding a solution of 2-hydroxy-4-methoxybutyrophenone and sodium ethoxide in ethanol to the N,N-dimethylaminoethyl chloride. Then, the mixture was refluxed for 3 h [787].

m.p. 101-102° [787].

BIOLOGICAL ACTIVITY: Local anaesthetic [787]; Antispasmodic [787]; Convulsions [787].

LD₅₀ 130 [787].

1-[4-(N,N-Dimethylaminoethoxy)-2-hydroxyphenyl]-1-butanone

OH CO(CH₂)₂CH₃ (CH₃)₂NCH₂CH₂O

-Obtained by adding a solution of resbutyrophenone and sodium ethoxide in ethanol to the N, N-dimethylaminoethyl chloride. Then, the mixture was refluxed for

Hydrochloride

C14H21NO3, HCl

mol. wt. 287.79

mol. wt. 251.33

m.p. 154–156° [787].

BIOLOGICAL ACTIVITY: Local anaesthetic [787]; Antispasmodic [787]; Convulsions [787]; Transient decrease of arterial blood pressure [787]; Increase of resistance of isolated heart to anoxia [787].

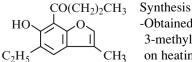
LD₅₀ 250 [787].

1-(5-Ethyl-6-hydroxy-3-methyl-7-benzofuranyl)-1-butanone

[101088-95-3]

C15H18O3

mol. wt. 246.31



-Obtained by decarboxylation of 5-ethyl-6-hydroxy-3-methyl-7-(1-oxobutyl)-2-benzofurancarboxylic acid on heating for 10 min at 255° [2827].

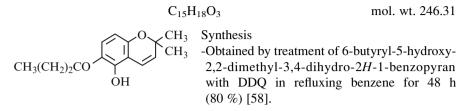
m.p. 57° [2827].

3 h [787].

 $C_{14}H_{21}NO_3$ Synthesis

1-(5-hydroxy-2,2-dimethyl-2H-1-benzopyran-6-yl)-1-butanone

6-Butyryl-5-hydroxy-2,2-dimethyl-2H-1-benzopyran



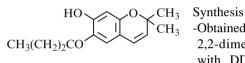
m.p. 65–66° [58]; ¹H NMR [58].

1-(7-hydroxy-2,2-dimethyl-2H-1-benzopyran-6-yl)-1-butanone

6-Butyryl-7-hydroxy-2,2-dimethyl-2H-1-benzopyran

 $C_{15}H_{18}O_3$

mol. wt. 246.31



-Obtained by treatment of 6-butyryl-5-hydroxy-2,2-dimethyl-3,4-dihydro-2H-1-benzopyran with DDQ in refluxing benzene for 48 h (80 %) [58].

m.p. 53–54° [58] ¹H NMR [58].

1-(5,7-Dihydroxy-2,2-dimethyl-2H-1-benzopyran-8-yl)-1-butanone

 $\begin{array}{c} C_{15}H_{18}O_4 & \text{mol. wt. } 262.31 \\ \hline CO(CH_2)_2CH_3 & \text{Synthesis} \\ HO & CH_3 & -\text{Refer to: } [3203]. \\ \hline Dimethyl \ ether & [924889-47-4] \\ C_{17}H_{22}O_4 & \text{mol. wt. } 290.36 \end{array}$

-Obtained by treatment of Malloapelta B with sodium borohydride in methanol, for 10 min at $10-15^{\circ}$, then 10 min at 60° (90 %) [3203].

white solid [3203]; m.p. 62.8° [3203]; ¹H NMR [3203], IR [3203].

BIOLOGICAL ACTIVITY: Exploration of essential structure of malloapelta B for the inhibitory activity against TNF-induced NF-kB activation [3203]; Cytotoxicity [3203].

1-(3,4-Dihydro-5-hydroxy-2,2-dimethyl-2H-1-benzopyran-6-yl)-1-butanone

6-Butyryl-5-hydroxy-2,2-dimethyl-3,4-dihydro-2*H*-1-benzopyran

[93970-91-3] C15H20O3 Syntheses CH₃ CH₂ -Obtained reaction by CH₃(CH₂)₂CC 2,4-dihydroxybutyrophenone with 2-methyl-OH 1,3-butadiene in petrol in the presence of polyphosphoric acid at 30-35° for 18 h (45 %) [58].

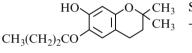
-Also refer to: [59].

m.p. 53–54° [58]; ¹H NMR [58].

1-(3,4-Dihydro-7-hydroxy-2,2-dimethyl-2H-1-benzopyran-6-yl)-1-butanone 6-Butyryl-7-hydroxy-2,2-dimethyl-3,4-dihydro-2H-1-benzopyran

C15H20O3

[93970-92-4]



Syntheses -Obtained reaction by of 2,4-dihydroxybutyrophenone with 2-methyl-1.3-butadiene in petrol in the presence of polyphosphoric acid at 30-35° for 18 h (40 %) [58].

-Also refer to: [59].

m.p. 95–96° [58]; ¹H NMR [58].

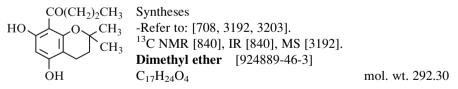
1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2H-1-benzopyran-8-yl)-1-butanone

[105306-66-9]

C15H20O4

mol. wt. 264.32

mol. wt. 248.32



-Obtained by treatment of Malloapelta B in methanol with hydrogen in the presence of 10 % Pd/C at r.t. for overnight (80 %) [3203].

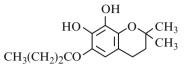
white solid [3203]; m.p. 48.8° [3203]; ¹H NMR [3203], IR [3203]; TLC [3203]. mol. wt. 248.32

of

1-(3,4-Dihydro-7,8-dihydroxy-2,2-dimethyl-2H-1-benzopyran-6-yl)-1-butanone

6-Butyryl-7.8-dihydroxy-2.2-dimethyl-3.4-dihydro-2H-1-benzopyran

[93970-90-2] $C_{15}H_{20}O_{4}$ mol. wt. 264.32



Synthesis -Obtained by reaction of 2,3,4-trihydroxybutyrophenone with 2-methyl-1,3-butadiene in petrol in the presence of polyphosphoric

acid at 30-35° for 18 h (80 %) [58].

m.p. 66–67° (page 360) and 82–83° (page 361) (?) [58]; ¹H NMR [58].

1-[2,6-Dihydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-1-butanone

[122585-49-3]

$$C_{15}H_{20}O_4$$

mol. wt. 264.32

 $(CH_3)_2C = CHCH_2C$

Isolation from natural sources $CO(CH_2)_2CH_3$ -From *Helichrysum* (Thunb.) Hilliard et asperum (Thunb.) Hilliard et Burtt. var. albidulum (DC) Hilliard [1488].

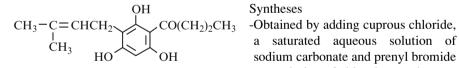
colourless crystals [1488]; m.p. 119° [1488]; ¹H NMR [1488], IR [1488], MS [1488].

1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-butanone

[69916-08-1]

 $C_{15}H_{20}O_4$

mol. wt. 264.32



Syntheses

sodium carbonate and prenyl bromide to a solution of phlorobutyrophenone

in ethyl ether at r.t., then to keep the mixture for 12 h at this temperature [1026]. -Also prepared by adding prenyl bromide to a suspension of the sodium salt of phlorobutyrophenone in benzene, then the mixture obtained was refluxed for 3 h [1026].

-Also prepared by adding at 5° a solution of sodium methoxide in methanol, followed by isopentenyl bromide in ethyl ether to a solution of phlorobutyrophenone in benzene and ethyl ether; then the mixture was stirred at r.t. for 6 h (16 %) [2113].

-Also obtained by adding prenyl chloride to a two-phase mixture consisting of phlorobutyrophenone in diethyl ether and saturated aqueous sodium carbonate. A catalytic amount of CuCl was added and the mixture was stirred or shaken vigorously for 3 h at r.t. (70 %) [838].

Isolation from natural sources

-From *Helichrysum asperum* (Thunb.) Hilliard et Burtt. var. *albidulum* (DC) Hilliard [1488].

m.p. 147° [2113], 140–143° [838]; ¹H NMR [1026, 1488], ¹³C NMR [838, 1026], IR [838, 1026, 1488], MS [838, 1488].

BIOLOGICAL ACTIVITY: Antifungal [2113].

1-[2,6-Dihydroxy-4-[(4-hydroxy-3-methyl-2-butenyl)oxy]phenyl]-1-butanone (*E*)

[122585-50-6] C15H20O5 mol. wt. 280.32 Isolation from natural sources OH -From Helichrysum asperum (Thunb.) Hil-CO(CH₂)₂CH₃ CH₂ liard Burtt. var. albidulum et (DC) Hilliard [1488]. C=CHCH₂O ĊH₂OH

¹H NMR [1488], IR [1488], MS [1488].

1-[2,6-Dihydroxy-4-[[4-hydroxy-3-(hydroxymethyl)-2-butenyl]oxy]phenyl]-1-butanone

[122616-67-5]	C	${}_{5}\text{H}_{20}\text{O}_{6}$		mol. wt. 296.32
СН2ОН С=СНСН2О	OH CO(CH ₂) ₂ CH ₃	liard et E (DC) Hilliard [s <i>um aspert</i> Burtt. v 1488].	um (Thunb.) Hil- var. albidulum
CH2OH		Tetraacetate*	[122585-	96-0]
0112011		$C_{23}H_{28}O_{10}$		mol. wt. 464.47

-Refer to: [1488].

¹H NMR [1488], IR [1488], MS [1488].

*1-[2,6-Bis(acetyloxy)-4-[[4-(acetyloxy)-3-[(acetyloxy)methyl]-2-butenyl]oxy] phenyl]-1-butanone

4-[3,5-Dihydroxy-4-(1-oxobutyl)phenoxy]-2-methyl-1-butanoic acid

[122585-56-2]

 $C_{15}H_{20}O_{6}$

mol. wt. 296.32

CH₃(CH₂)₂CO HO OCH₂CH₂-CH₃ CH₃

CH₃ OCH₂CH₂-CH-CO₂H Isolation from natural sources -From *Helichrysum asperum* (Thunb.) Hilliard et Burtt. var. *albidulum* (DC) Hilliard [1488].

¹H NMR [1488], IR [1488], MS [1488].

1-]2-Hydroxy-5-(1,1-dimethylpropyl)phenyl]-1-butanone

[854867-25-7]	$C_{15}H_{22}O_2$	mol. wt. 234.34
$CH_{3} - CO(CH_{2})_{2}CH_{3}$ $CH_{3} - C - CH_{3}$ $CH_{3} - C - CH_{3}$ $C_{2}H_{5}$	-	rearrangement of p-tert- 120° for 1 h (73 %) [2796].

2,4-Dinitrophenylhydrazone C₂₁H₂₆N₄O₅ m.p. 186° [2796].

1-[2-Hydroxy-6-methyl-3-(1,1-dimethylethyl)phenyl]-1-butanone C.-H.-O.

$$\begin{array}{c} C_{15}H_{22}O_2 \\ OH \\ (CH_3)_3C \\ CH_3 \\ CH_3 \\ CH_3 \end{array} \\ \begin{array}{c} OH \\ CH_3 \\ CH_3 \\ CH_3 \end{array} \\ \begin{array}{c} CO(CH_2)_2CH_3 \\ CH_3 \\ C$$

*in the presence of aluminium chloride (3 equiv.) without solvent at 110° for 2 h (78 %) [3118].

 $C_{21}H_{26}N_4O_5$

b.p.₈ 112° [3118].

2,4-Dinitrophenylhydrazone

m.p. 219° [3118].

1-(2-Hydroxy-5-pentylphenyl)-1-butanone

[101100-37-2] OH

C₁₅H₂₂O₂

C5H11

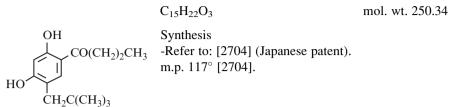
Synthesis CO(CH₂)₂CH₃ -Preparation by reaction of butyric acid with n-pentylphenol in the presence of boron trifluoride at 140–150° (88 %) [142]. b.p.₁₆ 185–192° [142]. $n_{\rm D}^{25} = 1.518$ [142].

mol. wt. 414.46

mol. wt. 414.46

mol. wt. 234.34

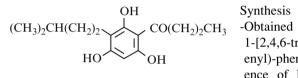
1-[2,4-Dihydroxy-5-(2,2-dimethylpropyl)phenyl]-1-butanone



USE: As colour developer [2704].

1-[2,4,6-Trihydroxy-3-(3-methylbutyl)phenyl]-1-butanone

[74477-97-7] mol. wt. 266.34 C15H22O4



Synthesis by hydrogenation 1-[2,4,6-trihydroxy-3-(3-methyl-2-butenvl)-phenvl]-1-butanone in the pres-

ence of PtO₂ in methanol under a hydrogen atmosphere at r.t. for 1 h (81 %) [2113].

of

m.p. 188° [2113].

BIOLOGICAL ACTIVITY: Antifungal [2113].

1-(2,4,6-Trihydroxy-3-pentylphenyl)-1-butanone

[74477-96-6]	$C_{15}H_{22}O_4$	mol. wt. 266.34
ОН	Syntheses	
C ₅ H ₁₁ CO(CH ₂) ₂ CH	H_3 -Obtained by adding	a solution of butanoyl
	chloride in nitrobenz	ene to a suspension of

chloride in nitrobenzene to a suspension, of 2,4,6-trihydroxypentyl-benzene and aluminium chloride in carbon disulfide at r.t., then stirring the mixture for 6 h at 30–35° (48 %) [2113].

-Also refer to: [1026, 1501, 1502].

m.p. 150° [2113].

но он

BIOLOGICAL ACTIVITY: Antimicrobial [1026, 1501, 1502]; Antifungal [2113].

1-[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-butanone

$$\begin{array}{cccc} C_{15}H_{23}NO_2 & \text{mol. wt. } 249.35\\ OH & Synthesis & \\ NH_2CH_2 & & CO(CH_2)_2CH_3 & -Refer to: [1475]. \\ Hydrochloride & [75060-71-8] \\ C_{15}H_{23}NO_2, HCl & \text{mol. wt. } 285.81 \end{array}$$

-Obtained by treatment of 1-[3-(N-chloroacetylaminomethyl)-5-(1,1-dimethylethyl)-2-hydroxy-phenyl]-1-butanone with conc. hydrochloric acid in refluxing ethanol for 20 h (56 %) [1475]. -Also refer to: [1289].

white amorphous crystals [1475]; m.p. 197–200° [1289, 1475]; ¹H NMR [1475]; TLC [1475].

1-(2-Hydroxy-3-phenoxyphenyl)-1-butanone

[479580-83-1]
$$C_{16}H_{16}O_3$$
 mol. wt. 256.30
OH Syntheses
 C_6H_5O CO(CH₂)₂CH₃ -Refer to: [1018–1022].

USE: Cosmetic compounds containing derivs of hydroxydiphenyl ether for inhibiting body odours [1018–1022].

1-(2-Hydroxy-4-phenoxyphenyl)-1-butanone

[307000-31-3] $C_{16}H_{16}O_3$ mol. wt. 256.30 OH Syntheses -Refer to: [1018–1022, 1345]. C_6H_5O

USE: Cosmetic compounds containing derivs of hydroxydiphenyl ether for inhibiting body odours [1018–1022].

BIOLOGICAL ACTIVITY: Antimicrobial [1345].

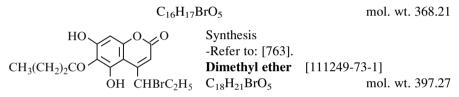
1-(4-Hydroxy-3-phenoxyphenyl)-1-butanone

[307000-52-8] $C_{16}H_{16}O_3$ mol. wt. 256.30 OH Syntheses -Refer to: [1018–1022, 1345]. CO(CH₂)₂CH₃

USE: Cosmetic compounds containing derivs of hydroxydiphenyl ether for inhibiting body odours [1018–1022].

BIOLOGICAL ACTIVITY: Antimicrobial [1345].

4-(1-Bromopropyl)-5,7-dihydroxy-6-(1-oxobutyl)-2H-1-benzopyran-2-one



-Preparation by refluxing a mixture of 6-butyryl-5,7-dimethoxy-4-propylcoumarin, N-bromo-succinimide and azobis(isobutyronitrile) in tetrachloromethane under nitrogen (quantitative yield) [766], (99 %) [763].

white solid [763]; m.p. 91–93° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

Diacetate [111249-88-8] C₂₀H₂₁BrO₇ mol. wt. 453.29

-Obtained by treatment of 5,7-diacetoxy-6-butyryl-4-propylcoumarin with N-bromosuccinimide [766] in tetrachloromethane. A trace of azobis(isobutyr-onitrile) was added and the mixture heated at reflux for 2 days (61 %) [763].

m.p. 84–85° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

4-(1-Bromopropyl)-5,7-dihydroxy-8-(1-oxobutyl)-2H-1-benzopyran-2-one

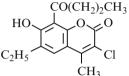
	$C_{16}H_{17}BrO_5$		mol. wt. 368.21
HO HO HO HO HO HO HO HO HO HO HO HO HO H	Synthesis -Refer to: [763]. Dimethyl ether $C_{18}H_{21}BrO_5$	[111249-74-2]	mol. wt. 397.27

-Preparation by refluxing a mixture of 8-butyryl-5,7-dimethoxy-4-propylcoumarin, N-bromosuccinimide and azo-isobutyronitrile in tetrachloromethane under nitrogen (86 %) [763].

white solid [763]; m.p. 85–86° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

3-Chloro-6-ethyl-7-hydroxy-4-methyl-8-(1-oxobutyl)-2H-1-benzopyran-2-one

CH₃ Synthesis



-Obtained by Fries rearrangement of 7-butyryloxy-3-chloro-6-ethyl-4-methylcoumarin with aluminium chloride for 1.5 h at 155° [2827].

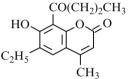
6-Ethyl-7-hydroxy-4-methyl-8-(1-oxobutyl)-2H-1-benzopyran-2-one

[109473-68-9]

 $C_{16}H_{18}O_4$

mol. wt. 274.32

H₃ Syntheses



-Obtained by Pechmann condensation of 1-(2,6-dihydroxy-3-ethylphenyl)-1-butanone with ethyl acetoacetate in presence of 80 % sulfuric acid [2811]. -Also obtained by Fries rearrangement of 7-butyryloxy-6-ethyl-4-methylcoumarin (m.p. 125°) [2811].

yellow needles [2811]; m.p. 110° [2811].

Oxime	$C_{16}H_{19}N$	O_4	mol. wt. 289.33
colourless	needles [2811]; m.p. 167	7° (d) [2811].	
Acetate	[110055-35-1]	$C_{18}H_{20}O_5$	mol. wt. 316.35

colourless needles [2811]; m.p. 114° [2811].

5-Ethyl-6-hydroxy-3-methyl-7-(1-oxobutyl)-2-benzofurancarboxylic acid

Synthesis

[109441-87-4]

 $\begin{array}{c} \text{CO}(\text{CH}_2)_2\text{CH}_3\\ \text{HO} \qquad & \text{O} \qquad & \text{CO}_2\text{H}\\ \text{C}_2\text{H}_5 \qquad & \text{CH}_3 \end{array}$

-Obtained by Fries rearrangement of 5-ethyl-6-butyryloxy-3-methylcoumarilic acid with aluminium chloride for 1 h at 150–160° [2827].

m.p. 223° [2827].

5,7-Dihydroxy-6-(1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one

¹H NMR [762], IR [762], UV [762].

Dimethyl ether	[98498-67-0]	$C_{18}H_{22}O_5$	mol. wt. 318.37
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-Preparation from a mixture of 6-butyryl-5,7-dihydroxy-4-propylcoumarin, dimethyl sulfate and potassium carbonate in refluxing acetone for 3 h (91 %) [763]. -Also refer to: [766].

white needles [763]; m.p. 66–68° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

Diacetate [98498-64-7] C₂₀H₂₂O₇ mol. wt. 374.39

-Obtained by reaction of acetic anhydride with 5,7-dihydroxy-6-(1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one in the presence of pyridine for 2 days at 20° (71 %) [763].

C16H18O5

-Also refer to: [766].

white needles [763]; m.p. 125–127° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

5,7-Dihydroxy-8-(1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one

[98192-68-8]

OH C₃H₇

HO

CO(CH₂)₂CH₃ Syntheses -Obtained by reaction of ethyl 3-oxohexanoate with phlorobutyrophenone in the presence of acetic acid containing 5 % (v/v) sulfuric acid (25–28 %) [762]. -Also refer to: [763].

mol. wt. 290.32

¹H NMR [762], IR [762], UV [762].

[111249-72-0] C18H22O5 **Dimethyl ether** mol. wt. 318.37

-Preparation from a mixture of 8-butyryl-5,7-dihydroxy-4-propylcoumarin, dimethyl sulfate and potassium carbonate in refluxing acetone for 3 h (95 %) [763].

white plates [763]; m.p. 93–95° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

5,7-Dihydroxy-4-(1-hydroxypropyl)-6-(1-oxobutyl)-2H-1-benzopyran-2-one

 $[111249-82-2] C_{16}H_{18}O_{6}$ HO O O Synt CH₃(CH₂)₂CO O - O t OH CHOHCH₂CH₃ xyc mat

Syntheses -Obtained by treatment of 5-acetoxy-4-(1-acetoxy-propyl)-6-butyryl-7-methoxycoumarin with boron tribromide in methylene chloride at -78° under nitrogen for 2 h (70 %) [763].

-Also obtained by treatment of 4-(1-acetoxypropyl)-6-butyryl-5,7-dihydroxy-coumarin with 10 % aqueous potassium hydroxide at 0° for 1.5 h [763].

yellow needles [763]; m.p. 226–228° [763]; ¹H NMR [763], IR [763], UV [763], MS [763]; TLC [763].

Synthesis

5,7-Dihydroxy-4-(1-hydroxypropyl)-8-(1-oxobutyl)-2H-1-benzopyran-2-one

[111249-85-5]	$C_{16}H_{18}O_{6}$	mol. wt. 306.32

$CO(CH_2)_2CH_3$
H0, , 0, 0
ĬŢŢŢ
$\langle \langle \rangle \rangle$
OH CHOHCH ₂ CH ₃

OH

-Obtained by adding lithium tert-butyl sulfide in HMPA to a solution of 4-(1-acetoxypropyl)-8-butyryl-7-hydroxy-5-methoxycoumarin in HMPA and the mixture wad heated at 75° for 2 h under nitrogen (26 %) [763].

white solid [763]; m.p. 180–183° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

1-(8-Hydroxy-4-propyl-3-quinolinyl)-1-butanone

	$C_{16}H_{19}NO_2$	mol. wt. 257.33
	Synthesis -Refer to: [170].	
	Methyl ether [189568-62-5]	
CO(CH ₂) ₂ CH ₃	$C_{17}H_{21}NO_2$	mol. wt. 271.36
I ₇	-Refer to: [170].	

1-(7-hydroxy-8-methyl-2,2-dimethyl-2H-1-benzopyran-6-yl)-1-butanone 6-Butyryl-7-hydroxy-2,2,8-trimethyl-2*H*-1-benzopyran

 $CH_{3}(CH_{2})_{2}CO$ CH_{3} CH_{3

mol. wt. 306.32

1-(7-hydroxy-8-methoxy-2,2-dimethyl-2H-1-benzopyran-6-yl)-1-butanone

6-Butyryl-7-hydroxy-8-methoxy-2,2-dimethyl-2H-1-benzopyran

$$C_{16}H_{20}O_4$$
 mol. wt. 27

76.33

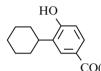
OCH₃ CH₃ HO CH₂ CH₃(CH₂)₂CC

Synthesis -Obtained by treatment of 6-butyryl-7-hydroxy-8-methoxy-2.2-dimethyl-3.4-dihydro-2H-1-benzopyran with DDQ in refluxing benzene for 48 h (70 %) [58].

m.p. 79–80° [58]; ¹H NMR [58].

1-(3-Cyclohexyl-4-hydroxyphenyl)-1-butanone

C₁₆H₂₂O₂ mol. wt. 246.35

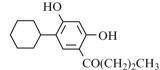


Synthesis -Refer to: [2704] (Japanese patent). m.p. 88° [2704].

USE: As colour developer [2704]. $CO(CH_2)_2CH_3$

1-(5-Cyclohexyl-2,4-dihydroxyphenyl)-1-butanone

C₁₆H₂₂O₃ mol. wt. 262.35



Synthesis -Obtained (XVIII) by reaction of butyric acid with 4-cyclohexylresorcinol in the presence of boron trifluoride etherate at 105-108° for 15 min, the BF₂-chelate hydrolysis of the (VIII) obtained [2382].

m.p. 124–125° [2382]; IR [2382], UV [2382].

BF ₂ -chelate (VIII)	$C_{16}H_{21}BF_2O_3$	mol. wt. 310.15 (54 %)	[2382].
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m.p. 113–114° [2382]; IR [2382].

1-(3,4-Dihydro-7-hydroxy-2,2,8-trimethyl-2H-1-benzopyran-6-yl)-1-butanone 6-Butyryl-7-hydroxy-2,2,8-trimethyl-3,4-dihydro-2H-1-benzopyran

> C₁₆H₂₂O₃ mol. wt. 262.35

Syntheses

CH₃ CH₃ HO CH₃ $CH_3(CH_2)_2CC$

[93970-94-6]

-Obtained by reaction of 2,4-dihydroxy-3-methyl-butyrophenone with 2-methyl-1,3-butadiene in petrol in the presence of polyphosphoric acid at 30-35° for 18 h (85 %) [58].

-Also refer to: [59].

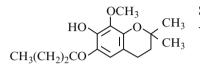
m.p. 85–86° [58]; ¹H NMR [58].

1-(3,4-Dihydro-7-hydroxy-8-methoxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-1-butanone

C₁₆H₂₂O₄

6-Butyryl-7-hydroxy-8-methoxy-2,2-dimethyl-3,4-dihydro-2H-1-benzopyran

[93970-95-7]



Syntheses

CH₃ -Obtained by treatment of 6-butyryl-CH₃
7,8-dihydroxy-2,2-dimethyl-3,4-dihydro-2*H*-1-benzopyran with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 2.5 h (90 %) [58].

-Also refer to: [59].

m.p. 66–67° [58]; ¹H NMR [58].

1-[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]-1-butanone (*HP57.4*)

[933786-84-6]

 $C_{16}H_{22}O_4$

mol. wt. 278.35

mol. wt. 278.35

 $(CH_3)_2C = CHCH_2 + CO(CH_2)_2CH_3 + From Helichrysum paronychioides (Asteraceae, tribe Inuleae) [2201]. yellow gum [2201];$

¹H NMR [2201], ¹³C NMR [2201], IR [2201], UV [2201], MS [2201]; TLC [2201].

BIOLOGICAL ACTIVITY: Antioxidant [2201].

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

OH O-β-D-Glc CO(CH₂)₂CH₃

Synthesis -Obtained by treatment of 4-acetyloxy-2-tetraacetyl-β-Dglucosyloxylbutyrophenone with sodium methoxide in boiling methanol for 3 min [3243].

Monohydrate

 $C_{16}H_{22}O_8, H_2O$ mol. wt. 360.40

 $(\alpha)_{D}^{30} = -73.2^{\circ}$ (water) [3243].

mol. wt. 360.40

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

 $\begin{array}{ccc} CO(CH_2)_2CH_3 & Synthesis \\ OH & -Obtained by treatment of resbutyrophenone tetra$ $acetyl-\beta-D-glucoside with 0.2 M sodium methoxide in metha$ $nol (70-75 %) [3243]. \\ O-\beta-D-Glc & m.p. 187-188^{\circ} [3243]. \end{array}$

Monohydrate

m.p. 134–135° [3243]; (α)_D²¹ = -38.8° (dimethylformamide) [3243].

C₁₆H₂₂O₈, H₂O

1-[2-(β-D-Glucopyranosyloxy)-4,6-dihydroxyphenyl]-1-butanone

[1067245-70-8]	$C_{16}H_{22}O_9$	mol. wt. 358.35
OH CO(CH ₂) ₂ CH ₃	Isolation from natural sources -From <i>Aster subulatus</i> Michx.	[1709].
HO O-β-D-Glc	USE: Antioxidant [1709].	

1-[4-(3-Bromopropoxy)-2-hydroxy-3-propylphenyl]-1-butanone

[194792-32-0] $C_{16}H_{23}BrO_3$ mol. wt. 343.26 OH Synthesis $C_{3}H_7$ CO(CH₂)₂CH₃ -Refer to: [307]. ¹H NMR [21].

1-[2-(N-Morpholinoethoxy)-4-hydroxyphenyl]-1-butanone

 $\begin{array}{cccc} C_{16}H_{23}NO_4 & mol. wt. 293.36 \\ OH & Synthesis \\ -Refer to: [787]. \\ Methyl ether (Hydrochloride) & [21092-65-9] \\ C_{17}H_{25}NO_4, HCl & mol. wt. 343.85 \\ CO(CH_2)_2CH_3 & C_{17}H_{25}NO_4, HCl & mol. wt. 343.85 \\ \end{array}$

-Obtained by adding a solution of 2-hydroxy-4-methoxybutyrophenone and sodium ethoxide in ethanol to the N-morpholinoethyl chloride. Then, the mixture was refluxed for 3 h [787].

m.p. 152-153° [787].

BIOLOGICAL ACTIVITY: Local anaesthetic [787]; Antispasmodic [787]; Depression [787].

LD₅₀ 180 [787].

1-[4-(N-Morpholinoethoxy)-2-hydroxyphenyl]-1-butanone

$$C_{16}H_{23}NO_4 \qquad \text{mol. wt. 293.36}$$

$$OH \qquad \qquad OH \qquad Synthesis \\ -Refer to: [787]. \\ Hydrochloride \qquad [20800-12-8] \\ C_{16}H_{23}NO_4, HCl \qquad \text{mol. wt. 329.82}$$

-Obtained by adding a solution of resbutyrophenone and sodium ethoxide in ethanol to the N-morpholinoethyl chloride. Then, the mixture was refluxed for 3 h [787].

m.p. 180-182° [787].

BIOLOGICAL ACTIVITY: Local anaesthetic [787]; Antispasmodic [787]; Depression [787].

LD₅₀ 700 [787].

1-[2-Hydroxy-3,5-bis(1-methylethyl)phenyl]-1-butanone

 $\begin{array}{c} C_{16}H_{24}O_2 \\ OH & Synthesis \\ (CH_3)_2CH & CO(CH_2)_2CH_3 & -Refer to: [2940]. \\ & & ESR [2940]. \end{array}$

1-(2-Hydroxy-3,4-dipropylphenyl)-butanone

[936642-87-4] $C_{16}H_{24}O_2$ mol. wt. 248.37 OH Synthesis $CH_3(CH_2)_2$ CO(CH_2)_2CH_3 -Obtained by reaction of 2,3-dipropylcyclobutenone with propyl vinyl ketone catalyzed by [RhCl(C_2H_4)_2]_2-P(cyclo-C_6H_{11})_3 in toluene at 130° for 12 h under argon atmosphere (65 %) [1731].

1-[4-(N,N-Diethylaminoethoxy)-2-hydroxyphenyl]-1-butanone

C₁₆H₂₅NO₃

mol. wt. 279.38

mol. wt. 248.37

OH CO(CH₂)₂CH₃

-Obtained by adding a solution of resbutyrophenone and sodium

Synthesis

resbutyrophenone and sodium ethoxide in ethanol to the N, N-diethylaminoethyl chloride. Then, the mixture was refluxed for 3 h [787].

Hydrochloride

C₁₆H₂₅NO₃, HCl

mol. wt. 315.84

m.p. 155–157° [787].

BIOLOGICAL ACTIVITY: Local anaesthetic [787]; Antispasmodic [787]; Convulsions [787]; Transient decrease of arterial blood pressure [787]; Inhibition of formaldehyde paw edema [787]; Protection against CaCl₂ ventricular fibrillation [787].

LD₅₀ 300 [787].

1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1-butanone

[230976-82-6] C₁₇H₁₈O₃ mol. wt. 270.33 OH Syntheses

OH Co(CH₂)₂CH₃

-Obtained by reaction of benzyl chloride with resbutyrophenone,

*in the presence of potassium hydroxide in methanol, first at r.t. overnight, then at reflux for 5 h [2181];

*in the presence of potassium carbonate in refluxing acetone for 8 h (50 %) [2181]. -Also refer to: [2411].

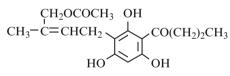
m.p. 85° [2181].

1-[3-[4-(Acetyloxy)-3-methyl-2-butenyl]-2,4,6-trihydroxyphenyl]-1-butanone (E)

[122585-61-9]

 $C_{17}H_{22}O_6$

mol. wt. 322.36



Isolation from natural sources

CO(CH₂)₂CH₃ -From *Helichrysum asperum* (Thunb.) Hilliard et Burtt. var. *albidulum* OH (DC) Hilliard [1488].

¹H NMR [1488], IR [1488], MS [1488].

1-[4-[[4-(Acetyloxy)-3-methyl-2-butenyl]oxy]-2,6-dihydroxyphenyl]-1-butanone (*E*)

[122585-51-7]

 $C_{17}H_{22}O_6$

mol. wt. 322.36

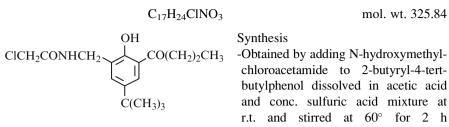
 CH_{3} $CH_{2}OCO(CH_{2})_{2}CH_{3}$ $CH_{2}OCOCH_{3}OH$ $CH_{2}OCOCH_{3}OH$

Isolation from natural sources

-From *Helichrysum asperum* (Thunb.) Hilliard et Burtt. var. *albidulum* (DC) Hilliard [1488].

¹H NMR [1488], IR [1488], MS [1488].

1-[3-(N-Chloroacetylaminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-butanone



¹H NMR [1475]; TLC [1475].

1-[2,4-Dihydroxy-3,6-dimethoxy-5-(3-methyl-2-butenyl)phenyl]-1-butanone

(82 %) [1475].

Diacetate [70219-90-8] C₂₁H₂₈O₇ mol. wt. 392.45 (14).

colourless oil [400]; ¹H NMR [400], IR [400].

1-[2-(N-Piperidinoethoxy)-4-hydroxyphenyl]-1-butanone

11 110

	$C_{17}H_{25}NO_3$	mol. wt. 291.39
OH OCH ₂ CH ₂ -N CO(CH ₂) ₂ CH ₃	Synthesis -Refer to: [787]. Methyl ether $C_{18}H_{27}NO_3$ -Refer to: [787].	mol. wt. 305.42

Hydrochloride of the methyl ether C₁₈H₂₇NO₃, HCl mol. wt. 341.88

-Obtained by adding a solution of 2-hydroxy-4-methoxybutyrophenone and sodium ethoxide in ethanol to the N-piperidinoethyl chloride. Then, the mixture was refluxed for 3 h [787].

m.p. 178° [787].

BIOLOGICAL ACTIVITY: Local anaesthetic [787]; Antispasmodic [787]; Convulsions [787]; Inhibition of formaldehyde paw edema [787].

LD₅₀ 130 [787].

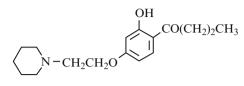
mol. wt. 325.84

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1-[4-(N-Piperidinoethoxy)-2-hydroxyphenyl]-1-butanone

C17H25NO3

mol. wt. 291.39



Synthesis -Obtained by adding a solution of resbutyrophenone and sodium ethoxide in ethanol to the N-piperidinoethyl chloride. Then. the mixture refluxed was for 3 h [787].

Hydrochloride

C₁₇H₂₅NO₃, HCl

mol. wt. 327.85

m.p. 145–147° [787].

BIOLOGICAL ACTIVITY: Local anaesthetic [787]; Antispasmodic [787]; Convulsions [787]; Transient decrease of arterial blood pressure [787]; Protection against CaCl₂ ventricular fibrillation [787].

LD₅₀ 200 [787].

1-[4-(Heptyloxy)-2-hydroxyphenyl]-1-butanone

 $\begin{array}{cccc} [22198-48-7] & C_{17}H_{26}O_3 & \mbox{mol. wt. } 278.39 \\ OH & \\ CO(CH_2)_2CH_3 & -Obtained by reaction of butanoyl chloride with resorcinol diheptyl ether in the presence of aluminium chloride at 80° for 2 h (60 %) [3469]. \end{array}$

-Also obtained by reaction of butanoyl chloride with m-diheptyloxybenzene in the presence of aluminium chloride in dichloroethane at 0° . Then, the mixture was stirred for 1 h at 10° , and then for 6 h at $20-25^{\circ}$. The temperature then raised to 80° and stirring was continued at that temperature for 5 h (60 %) [1194]. -Also refer to: [1861, 1862].

m.p. 41.5–42° [1194, 3469]; UV [1194, 3469].

USE: UV absorber [1194]; Light-stabilizer of polymeric materials [1194]; Substituted benzoimidazole compounds as transcription factor-modulating compounds useful as antiinfectives [1862].

BIOLOGICAL ACTIVITY: Antibacterial [1861].

9-Hydroxy-10-butyrylanthracene



Synthesis

-Obtained by Fries rearrangement of 9-butyryloxyanthracene with various metal halides in benzene under reflux, but it is rapidly transformed into 10-butyrylanthrone [3052].

4-[1-(Acetyloxy)propyl]-5,7-dihydroxy-6-(1-oxobutyl)-2H-1-benzopyran-2-one

[111249-78-6] C₁₈H₂₀O₇ mol. wt. 348.35 Syntheses .0 HO -To a mixture of 4-(1-acetoxypropyl)-CH₃(CH₂)₂CO

6-butyryl-5-hydroxy-7-methoxycoumarin and the 7-hydroxy-5-methoxy isomer and triethylamine in methylene chloride at 0° under nitrogen was added trimethylsilyl chloride.

The mixture was stirred for 30 min and the solvents were then evaporated under reduced pressure.

The residue was taken up in methylene chloride and the solution cooled to -78° under nitrogen.

Boron tribromide was added and the mixture stirred and allowed to warm to 0° . After stirring for 1 h the mixture was poured into dilute hydrochloric acid-ice (29 %) [763].

-Also obtained of 4-(1-acetoxypropyl)-6-butyrylby treatment 5,7-dimethoxycoumarin,

*in benzene with magnesium iodide-diethyl ether and refluxing for 2 h under nitrogen (3 %) [763];

*with boron tribromide (30 %) [766].

yellow needles [763]; m.p. 212–214° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

OH CH-CH₂CH₃

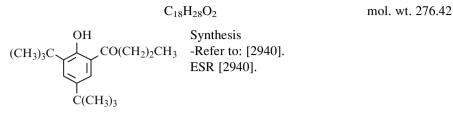
OCOCH₃

C₂₀H₂₄O₇ mol. wt. 376.41 **Dimethyl ether** [111249-75-3] [98498-69-2]

-Preparation by reaction of tetramethylammonium acetate with 4-(1-bromopropyl)-6-butyryl-5,7-dimethoxycoumarin in acetone for 2–3 days at 20° (82 %) [763, 766].

white plates [763]; m.p. 120–122° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

1-[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]-1-butanone



1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-butanone

[14035-35-9] $C_{18}H_{28}O_2$ mol. wt. 276.42 OH Syntheses (CH₃)₃C C(CH₃)₃ - Preparation by reaction of butyryl chloride with 2,6-di-tert-butylphenol in the presence of aluminium chloride, CO(CH₂)₂CH₃ *at -10° for 1–13 min (90 %) [2506]; *at 20° for 20 min, followed by hydrolysis of the keto ester obtained (**3c**) (87 %, m.p. 49.5–50.5°) [2971];

*in 1,1,2-trichloroethane at -10 to -20° [951].

-Preparation by reaction of butyryl chloride with 2,6-di-tert-butylphenol in the presence of titanium tetrachloride [1468].

-Also obtained by reaction of butyric anhydride with 2,6-di-tert-butylphenol in the presence of 5 drops 70 % perchloric acid, first at r.t. for 1 h and left overnight (90 %) [2136].

-Also refer to: [655, 2139].

m.p. 91–93° [2139], 88–90° [2506], 88–89° [2136], 85.5–86.5° (**2c**) [2971]; ¹H NMR [2136, 2971], IR [2136, 2971]; electrochemical characteristics [2136].

USE: Stabilize plastics, oils, and fats against heat, light, and oxidation [951].

BIOLOGICAL ACTIVITY: Inflammation inhibitor [2139].

1-(4-Butyloxy-3-butyl-2-hydroxyphenyl)-1-butanone

$C_{18}H_{28}O_3$					mol. wt. 292.42
$C_{4}H_{9}$	Synthesis -Obtained butyropher b.p. ₈ 200–2	none	[1587].	of	2,4-dihydroxy-

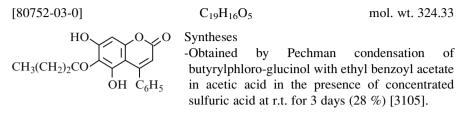
2,4-Dinitrophenylhydrazone C₂₄H₃₂N₄O₆

 $_{24}H_{32}N_4O_6$ mol. wt. 472.54

m.p. 140.5° [1587].

5,7-Dihydroxy-6-(1-oxobutyl)-4-phenyl-2H-1-benzopyran-2-one

5-Butyryl-5,7-dihydroxy-4-phenylcoumarin

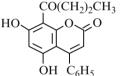


-Also obtained by reaction of butyryl chloride with 5,7-dihydroxy-4-phenyl-2*H*-1-benzopyran-2-one in the presence of aluminium chloride in carbon disulfide, then in refluxing nitromethane [2146].

pale yellow needles [3105]; m.p. 228–229° [3105]; ¹H NMR [3105], IR [3105].

5,7-Dihydroxy-8-(1-oxobutyl)-4-phenyl-2H-1-benzopyran-2-one

C₁₉H₁₆O₅ mol. wt. 324.33



Synthesis

-Obtained by reaction of butyryl chloride with 5,7-dihydroxy-4-phenyl-2*H*-1-benzopyran-2-one in the presence of aluminium chloride in carbon disulfide, then in refluxing nitromethane [2146].

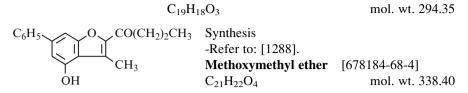
1-[6-[2-(Aminophenyl)thio]-8-hydroxy-3-quinolinyl]-1-butanone

$C_{19}H_{18}N_2O_2$	$_2$ S	mol. wt. 338.43
OH NH2 S CO(CH2)2CH3	Synthesis -Refer to: [973]. Methyl ether $C_{20}H_{20}N_2O_2S$	mol. wt. 352.46

Hydrochloride of the $\ [209479-38-9]$ $C_{20}H_{20}N_2O_2S,$ HCl mol. wt. 388.92 methyl ether

-Refer to: [973].

1-(4-Hydroxy-3-methyl-6-phenyl-2-benzofuranyl)-1-butanone



-Refer to: [1288].

4-[1-(Acetyloxy)propyl]-5-hydroxy-7-methoxy-6-(1-oxobutyl)-2*H*-1-benzopyran-2-one

[111249-77-5]

C₁₉H₂₂O₇ mol. wt. 362.38

Syntheses

-Obtained by treatment of 4-(1-acetoxypropyl)-6-butyryl-5,7-dimethoxy-

coumarin in benzene with magnesium iodide-diethyl ether and refluxing for 2 h under nitrogen (90 %) [763].

-Also obtained by adding boron tribromide to 4-(1-acetoxypropyl)-6-butyryl-5,7-dimethoxy-coumarin in methylene chloride at -78° under nitrogen. The solution was kept at -78° for 2 h then allowed to warm to 0° [766], (21 %) [763].

yellow solid [763]; m.p. 168–170° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

Acetate [111249-8	1-1] C ₂₁ H	$H_{24}O_8$ mol.	wt. 404.42
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-Preparation by treatment of 4-(1-acetoxypropyl)-6-butyryl-5-hydroxy-7-methoxycoumarin with acetic anhydride in the presence of pyridine for 2 days (72 %) [763].

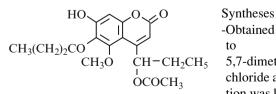
white needles [763]; m.p. 132–134° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

4-[1-(Acetyloxy)propyl]-7-hydroxy-5-methoxy-6-(1-oxobutyl)-2*H*-1-benzopyran-2-one

[111249-80-0]

 $C_{19}H_{22}O_7$

mol. wt. 362.38



-Obtained by adding boron tribromide to 4-(1-acetoxypropyl)-6-butyryl-5,7-dimethoxy-coumarin in methylene chloride at -78° under nitrogen. The solution was kept at -78° for 2 h then allowed to warm to 0° [766], (42 %) [763].

white needles [763]; m.p. 129–131° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

4-[1-(Acetyloxy)propyl]-7-hydroxy-5-methoxy-8-(1-oxobutyl)-2*H*-1-benzopyran-2-one

[111249-84-	4]	$C_{19}H_{22}O_7$		mol. wt. 362.38
HO CH ₃ O	CH ₂) ₂ CH ₃ O O CH-CH ₂ CH ₃ OCOCH ₃	of 4-(1-acetoxy) coumarin in methyl	propyl)-8-b ene chlorid xture was	bromide to a solution utyryl-5,7-dimethoxy- e at -78° under nitro- allowed to warm to
white needles [763]; m.p. 136–138° [763]; ¹ H NMR [763], IR [763], UV [763], MS [763].				
Methyl ethe	e r [111	249-76-4] C ₂	20H24O7	mol. wt. 376.41
Obtained by	was action of to	tram athe lamma anium	agatata wi	th 1 (1 here a near 1)

-Obtained by reaction of tetramethylammonium acetate with 4-(1-bromopropyl)-8-butyryl-5,7-dimethoxycoumarin in acetone for 2–3 days at 20° (85 %) [763].

white solid [763]; m.p. 130–132° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

1-(2-Hydroxy-5-nonylphenyl)-1-butanone

 $\begin{array}{cccc} C_{19}H_{30}O_2 & \text{mol. wt. 290.45} \\ OH & Synthesis \\ \hline CO(CH_2)_2CH_3 & -Refer to: [1869]. \\ \hline Oxime & [758691-87-1] \\ C_{19}H_{31}NO_2 & \text{mol. wt. 305.46} \\ \end{array}$

USE: Extraction agent for copper [1869].

1-[4-(Nonyloxy)-2-hydroxyphenyl]-1-butanone

 $[22198-49-8] C_{19}H_{30}O_3 mol. wt. 306.45$ OH CO(CH₂)₂CH₃ Obtained by reaction of butanoyl chloride with resorcinol dinonyl ether in the presence of aluminium chloride at 80° for 2 h (60 %) [3469].

-Also obtained by reaction of butanoyl chloride with m-dinonyloxybenzene in the presence of aluminium chloride in dichloroethane at 0° . Then, the mixture was stirred for 1 h at 10° , and then for 6 h at $20-25^{\circ}$. The temperature was then raised to 80° and stirring was continued at that temperature for 5 h (60 %) [1194].

m.p. 38–38.5° [1194, 3469]; UV [1194, 3469].

USE: UV absorber [1194], Light-stabilizer of polymeric materials [1194].

1-[8-Hydroxy-4-[(2-methylphenyl)amino]-3-quinolinyl]-1-butanone

[125500-46-1]	$C_{20}H_{20}N_2O_2$	mol. wt. 320.39
$\bigcup_{\substack{i=1\\NH-C_6H_4-CH_3(2)}}^{OH}$	Syntheses -Refer to: [170, 296, 1841, 1904]. m.p. 114–115° [1841]. BIOLOGICAL ACTIVITY: Refer to	p: [1841].

Methyl ether [115607-61-9] C₂₁H₂₂N₂O₂ mol. wt. 334.42

m.p. 112–114° [1420], 102° [1904]; ¹H NMR [1420].

-Refer to: [296, 370, 876, 1841].

BIOLOGICAL ACTIVITY: Antimicrobial agent [876]; Combinations of 5-HT₄ agonist or antagonist or 5-HT₃ antagonist and co-agent for treatment of gastrointestinal and abdominal visceral disorders [370].

N-(3-Butanoyl-2-hydroxy-5-methylphenyl)-1-phenylmethyltetrazole-5-carboxamide

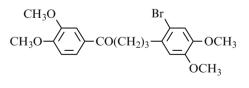
[70978-14-2]	$C_{20}H_{2}$	$_{21}N_5O_3$	mol. wt. 379.42
OH C ₃ H ₇ CO / NH-CO-	CH ₂ C ₆ H ₅ N-N ⟨ N-N	Synthesis -Refer to: [1017 (78 %)]. m.p. 166–169° [1017].	

4-(2-Bromo-4,5-dimethoxyphenyl)-1-(3,4-dimethoxyphenyl)-1-butanone

[111585-36-5]

 $C_{20}H_{23}BrO_5$

mol. wt. 423.30



Syntheses

-Obtained by reaction of 4-(2-bromo-4,5-dimethoxyphenyl)butyric chloride with veratrole in the presence of aluminium chloride in nitrobenzene for 24 h at r.t. (86 %) [1284].

-Also obtained by treatment of its semicarbazone with a saturated aqueous solution of oxalic acid at 100–110° for 2 h (91 %) [1284].

colourless prisms [1284]; m.p. 82-84° [1284].

Semicarbazone

[50874-48-1]

C21H26BrN3O5

C20H28O4

mol. wt. 480.36

mol. wt. 332.44

pale yellow needles [1284]; m.p. 172–173° [1284].

1-[2,4,6-Trihydroxy-3,5-bis(3-methyl-2-butenyl)phenyl]-1-butanone

OH $(CH_3)_2C = CHCH_2$ HO $CH_2CH = C(CH_3)_2$

Syntheses CO(CH₂)₂CH₃ -Obtained by reaction of resbutyrophenone with 1-bromo-3-methyl-2-buten (3 mol) in the presence of the weakly basic resin DeAcidite H-IP (OH⁻ form) in boiling benzene (12.6 %) [708].

N.B.: The yield was similar at this obtained by using 2-methyl-3-buten-2-ol but the isolation was simpler [708].

-Also obtained by reaction of phlorobutyrophenone with 1-chloro-3-methyl-2-butene (2 molar parts), magnesium oxide (0.5 equiv.) and potassium iodide (1%) [3309].

-Also refer to: [707].

oil [708].

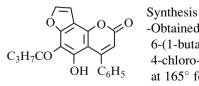
1-[2-Hydroxy-4-methoxy-6-[[tris(1-methylethyl)silyl]oxy]phenyl]-1-butanone

[326854-41-5] C20H34O4Si mol. wt. 366.57 Syntheses -Refer to: [1464, 3070]. $CO(CH_2)_2CH_3$

5-Hydroxy-6-(1-butanoyl)-4-phenyl-2H-furo[2',3':5,6] benzo[1,2-b]pyran-2-one (Furanoracemosone)

C21H16O5

mol. wt. 348.36



-Obtained by heating a mixture of 5,7-dihydroxy-6-(1-butanoyl)-4-phenyl-2*H*-1-benzopyran-2-one and 4-chloro-1,3-dioxolan-2-one first at 150° for 4 h, then at 165° for 30 min (10 %) [2146].

Isolation from natural sources

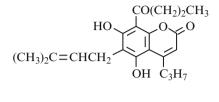
-From the leaves of Mesua racemosa (Clusiaceae) [2146].

white amorphous solid [2146]; ¹H NMR [2146], ¹³C NMR [2146], IR [2146], UV [2146], MS [2146],

5,7-Dihydroxy-6-(3-methyl-2-butenyl)-8-(1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one

(Normammein, Mammea B/BC)

[5085-54-1]



C21H26O5 mol. wt. 358.43

Syntheses -Obtained by reaction of prenyl bromide with 5,7-dihydroxy-8-(1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one in the presence of 10 % aqueous potassium hvdroxide at 0° (30 - 45)%) [765], (32 %) [762].

Isolation from natural sources

-From kernels of the fruit of Mammea americana L. and Mammea africana (Guttiferae) [765, 1006].

-From the seeds of Mammea americana L. [753, 758, 759, 1082].

-From the bark of Mammea africana G. Don (Guttiferae) [553].

needles [753]; white needles [762]; m.p. 132–134° [762], 132–133° [753], 130–132° [1006]; ¹H NMR [753, 762], IR [753, 762], UV [753, 762], MS [753, 762]; GC-MS [1082].

USE: Insecticide [753, 758, 759, 765].

BIOLOGICAL ACTIVITY: Antitumor [1006].

5,7-Dihydroxy-8-(3-methyl-2-butenyl)-6-(1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one

(Mammea B/AC)

[38537-84-7]

C21H26O5

Syntheses

mol. wt. 358.43

 $CH_2CH = C(CH_3)_2$ HO ~O CH₃(CH₂)₂CO OH C₃H₇

-Obtained by reaction of prenyl bromide with 5,7-dihydroxy-6-(1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one in the presence of 10 % aqueous potassium hydroxide at 0° (30–45 %) [765], (25 %) [762].

-Also obtained by isomerization of 5,7-dihydroxy-6-(3-methyl-2-butenyl)-8-(1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one (mammea B/BC) by treatment with methanolic 5 % potassium hydroxide at 20° overnight [757].

Isolation from natural sources

-From kernels of the fruit of *Mammea americana* L. [757] and *Mammea africana* (Guttiferae) [765].

yellow needles [757, 762]; m.p. 127–128.5° [757], 127–128° [762]; ¹H NMR [757, 762], IR [757, 762], UV [757, 762], MS [757, 762]; TLC [757].

USE: Insecticide [757, 765].

1-[7-Hydroxy-2-(3,4,5-trimethoxy)-2H-1-benzopyran-3-yl]-1-butanone

 $\begin{array}{cccc} C_{22}H_{24}O_6 & \text{mol. wt. 384.43} \\ & & & \\ OCH_3 & Synthesis \\ & & & \\ OCH_3 & -Refer to: [634]. \\ & & & \\ Methyl \ ether & [364039-57-6] \\ & & \\ OCH_3 & C_{23}H_{26}O_6 & \text{mol. wt. 398.46} \\ & & \\ CO(CH_2)_2CH_3 & -Refer \ to: [633, 634]. \end{array}$

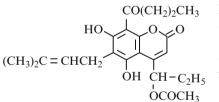
BIOLOGICAL ACTIVITY: As TNF-α-inhibitor [634].

4-[1-(Acetyloxy)propyl]-5,7-dihydroxy-6-(3-methyl-2-butenyl)-8-(1-oxobutyl)-2H-1-benzopyran-2-one

(Mammea E/BC)

 $C_{23}H_{28}O_7$ mol. v

mol. wt. 416.47



Isolation from natural sources -From *mammea americana* L. (Guttifferae) (Clusiaceae) [756, 758, 3381]. -Also refer to: [762]. m.p. 50–53° [756]; ¹H NMR [756, 758, 3381],

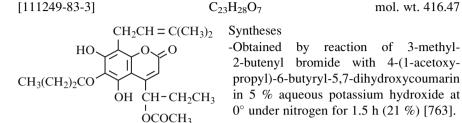
¹³C NMR [3381], IR [756], UV [756, 3381], MS [756]; (α)_D²⁵ = -28° (methanol) [3381].

USE: Insecticide [758].

BIOLOGICAL ACTIVITY: Apoptosis induction of human colon cancer SW-480 cells [3381]; Cytotoxicity [3381].

4-[1-(Acetyloxy)propyl]-5,7-dihydroxy-8-(3-methyl-2-butenyl)-6-(1-oxobutyl)-2H-1-benzopyran-2-one

(Mammea E/AC)



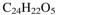
obtained by prenylation of 4-[1-(acetyloxy)propyl]-5,7-dihydroxy--Also 6-(1-oxobutyl)-2H-1-benzopyran-2-one using prenyl bromide in the presence of 2 equiv. of aqueous 5 % potassium hydroxide at 0° (21 %) [766].

yellow needles [763]; m.p. 139–141° [763]; ¹H NMR [763], IR [763], UV [763], MS [763].

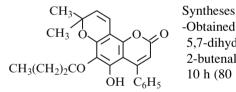
5-Hydroxy-8,8-dimethyl-6-(1-oxobutyl)-4-phenyl-2H,8H-benzo[1,2-b:5,6-b'] dipyran-2-one

(Mammea A/AC) 6-Butyryl-5-hydroxy-4-phenylseselin

[42290-21-1]



mol. wt. 390.44



-Obtained by treatment of 5-butyryl-5,7-dihydroxy-4-phenylcoumarin with 3-methyl-2-butenal in pyridine first at r.t., then at 110° for 10 h (80 %) [3105].

-Also obtained by oxidative cyclization of 5,7-dihydroxy-8-(3-methyl-2-butenyl)-6-(1-oxobutyl)-4-phenyl-2H-1-benzopyran-2-one with DDQ in benzene at r.t. overnight (76 %) [3105].

Isolation from natural sources

-From Ochrocarpus siamensis (Guttiferae) [3105].

-From flowers of Kayea assamica [3315].

-From seeds (Fruit and Spice Park) of Mammea americana L. (Clusiaceae) [3381].

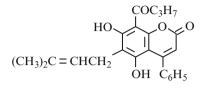
yellow needles [3105]; m.p. 138–139° [3105]; ¹H NMR [3105], IR [3105], UV [3105], MS [3105].

BIOLOGICAL ACTIVITY: Pancreatic cancer PANC-1 cells of human [3315]; Human colon cancer HCT-116 cells [3381]; Human colon cancer HT-29 cells [3381]; Human colon cancer SW-480 cells [3381]; Cytotoxicity [3381].

mol. wt. 416.47

5,7-dihydroxy-6-(3-methyl-2-butenyl)-8-(1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one

(Mammea A/BC)



Synthesis -Obtained by reaction of 3-methyl-2-butenyl bromide with 5,7-dihydroxy-8-(1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one in the presence of 10 % KOH at 0° for 1.5 h (15 %) [2146].

Isolation from natural sources

-From the leaves of Mesua racemosa (Clusiaceae) [2146].

m.p. 123–124° [2146]; ¹H NMR [2146], ¹³C NMR [2146], IR [2146], UV [2146], MS [2146].

5,7-dihydroxy-8-(3-methyl-2-butenyl)-6-(1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one

(Mammea A/AC)

C₂₄H₂₄O₅ mol. wt. 392.45

mol. wt. 392.45

$$\begin{array}{c} CH_2CH = C(CH_3)_2 \\ HO \\ C_3H_7CO \\ OH \\ C_6H_5 \end{array} \qquad \begin{array}{c} Syntheses \\ -Obtained \\ 5,7-dihydroxy-4-phenylcoumarin with 2-methyl- \\ 3-buten-2-ol in the presence of boron trifluoride \\ etherate in dioxan at 50° for 90 min \\ (10 \%) [3105]. \\ -Also refer to: [762]. \end{array}$$

Isolation from natural sources

-From flowers of Kayea assamica [2222].

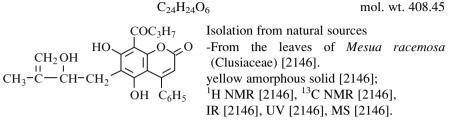
-From Ochrocarpus siamensis (Guttiferae) [3105].

pale yellow needles [3105]; m.p. 116–117° [3105]; ¹H NMR [3105], IR [3105], UV [3105], MS [3105].

BIOLOGICAL ACTIVITY: Cytotoxicity [2222].

5,7-dihydroxy-6-(2-hydroxy-3-methyl-3-butenyl)-8-(1-oxobutyl)-4-phenyl-2H-1-benzopyran-2-one

(Isoracemosol)



1-[2-(Tetraacetyl-β-D-glucopyranosyloxy)-4-hydroxyphenyl]-1-butanone (Resbutyrophenon-4-tetraacetyl- β -D-glucoside)

$$C_{24}H_{30}O_{12}$$
 mol. wt. 510.50

OH CO(CH₂)₂CH₃

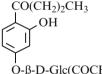
Svnthesis -Refer to: [3243]. Acetate C₂₆H₃₂O₁₃ mol. wt. 552.50 Acetate $C_{26}H_{32}O_{13}$ mol. wt. 5O-β-D-Glc(COCH_3)_4-Obtained by reaction of α-acetobromoglucose

 $(\alpha$ -ABG) with 4-acetoxyresbutyrophenone in the presence of silver oxide in quinoline for 30 min (16 %) [3243].

m.p. 182–184° [3243]; $(\alpha)_D^{21} = -46.2^\circ$ (water) [3243].

1-[4-(Tetraacetyl-β-D-glucopyranosyloxy)-2-hydroxyphenyl]-1-butanone (*Resbutyrophenon-4-tetraacetyl-\beta-D-glucoside*)

$$C_{24}H_{30}O_{12}$$
 mol. wt. 510.50



Synthesis -Obtained by reaction of α -acetobromoglucose (α -ABG) with resbutyrophenone in the presence of silver oxide in quinoline for 2 h [3243].

O-β-D-Glc(COCH₃)₄

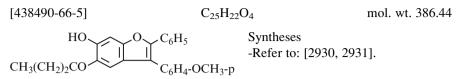
m.p. 125–126° [3243]; $(\alpha)_{D}^{20} = -21.9^{\circ}$ (chloroform) [3243].

1-(6-Hydroxy-3-(4-methylphenyl)-2-phenyl-5-benzofuranyl)-1-butanone

[438490-69-8] C25H22O3 $C_{6}H_{5}$ Synthesis -Refer to: [2930]. Synthesis CH₃(CH₂)₂CC

mol. wt. 408.45

1-(6-Hydroxy-3-(4-methoxyphenyl)-2-phenyl-5-benzofuranyl)-1-butanone



2 Aromatic Hydroxyketones Derived from Various Alkyl-1-Butanoic Acids

2.1 From 2-Methyl-1-Butanoic Acid

2.1.1 Unsubstituted Hydroxyketones

1-(2-Hydroxyphenyl)-2-methyl-1-butanone

[344408-25-9]	$C_{11}H_{14}O_2$	mol. wt. 178.23
OH CO-CH-CH ₂ CH ₃	Syntheses -Obtained by photo-Fries phenyl 2-methylbutanoate -Also refer to: [369 (2 %),	

IR [1002], UV [1002].

Methyl ether	[1196852-29-5]	$C_{12}H_{16}O_2$	mol. wt. 192.26

-Refer to: [1084].

1-(3-Hydroxyphenyl)-2-methyl-1-butanone

[195393-39-6]	$C_{11}H_{14}O_2$	mol. wt. 178.23
OH CO-CH-CH ₂ CH ₃	Synthesis -Also obtained (10) by react pentanoyl-1,3-cyclohexadien plex (8) with triethylamine r.t. for 1 h (53 %) [246].	e tricarbonyl iron com-

Acetate [195393-36-3]	$C_{13}H_{16}O_3$	mol. wt. 220.27
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-Obtained in the same reaction that the phenol above (14 %) [246].

1-(4-Hydroxyphenyl)-2-methyl-1-butanone

(S)-4-(1-oxo-2-methylbutyl)phenol

N.B.: Metabolite of T-018 in the urine of female rats [1511].

Methyl ether	[90269-46-8]	$C_{12}H_{16}O_2$	mol. wt. 192.26
wieury i eurei	[90209-40-0]	$C_{12} I_{16} O_2$	11101. wt. 192.20

-Obtained by treatment of anisole with 2-methylbutyric anhydride or 2-methylbutyryl chloride in the presence of sulfated zirconia [867].

-Also obtained by reaction of 2-methylbutanoyl chloride with anisole in the presence of aluminium chloride in carbon disulfide first at 0° for 15 min, then at r.t. for 1 h (54 %) [79].

-Also obtained from 4-methoxybenzaldehyde (52 %) [3024]. -Also refer to: [179, 1136, 1736, 1781, 2510].

b.p._{0.5} 99–100° [79], b.p._{0.4} 105° [1736], b.p.₁₂ 145–150° [1781], b.p.₁₀ 152° [2510], b.p.₁ 152–153° [1136]; (α)_D = + 25.3° [2510]; $n_D^{17.5} = 1.534$ [2510]; ¹H NMR [79, 3024], IR [79], UV [1736].

2,4-Dinitrophenylhydrazone of the methyl ether

[14248-30-7]	$C_{18}H_{20}N_4O_5$	mol. wt. 372.38
m.p. 97–100° [1781], 101.5–102.2° [2368].	

Methyl ether (2*S*) [27763-55-9] C₁₂H₁₆O₂ mol. wt. 192.26

-Obtained by reaction of 2-methylbutanoyl chloride with anisole (70 %) [179]. -Also refer to: [850, 867, 2069].

b.p._{0.3} 155° [179], b.p.₁₅₋₂₀ 160° [850]; $(\alpha)_D = +3.2^\circ$ neat (no solvent) [850], $(\alpha)_D = +25^\circ$ (dioxane) [179]; $n_D^{35} = 1.518$ [850]; Circular dichroism spectra [1736].

Methyl ether (R)	[77942-74-6]	$C_{12}H_{16}O_2$	mol. wt. 192.26
-Refer to: [1107].			

Phenyl ether	[90269-45-7]	$C_{17}H_{18}O_2$	mol. wt. 254.33
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-Obtained by reaction of 2-methylbutanoyl chloride with diphenyl oxide in the presence of aluminium chloride in carbon disulfide first at 0° for 15 min, then at r.t. for 1 h (66 %) [79].

b.p._{0.18} 129–131° [79]; ¹H NMR [79], IR [79].

1-(2,3-Dihydroxyphenyl)-2-methyl-1-butanone

	$C_{11}H_{14}O_3$	mol. wt. 194.23
ОН НО СО-СН-С ₂ Н ₅	Synthesis -Refer to: [2747].	
CH ₃	Dimethyl ether [15121-99-0] $C_{13}H_{18}O_3$	mol. wt. 222.28

-Obtained by oxidation of 1-(2,3-dimethoxyphenyl)-1-(2-methylpropyl) carbinol with sodium dichromate in dilute sulfuric acid (75–80 %) [2747].

pale yellow viscous oil [2747]; b.p._{0.2} 90–92° [2747].

2,4-Dinitrophenylhydrazone of the methyl ether

[15116-04-8]	$C_{19}H_{22}N_4O_6$	mol. wt. 402.41
m.p. 224.5–225.5° [2747].		

1-(2,4-Dihydroxyphenyl)-2-methyl-1-butanone

[15116-15-1] [123020-85-9] (2S)	$C_{11}H_{14}O_3$	mol. wt. 194.23
HO HCO-CH-CH ₂ CH ₃	Syntheses -Obtained by Friedel-Craft resorcinol dimethyl ether 2-methylbutyryl chloride in th minium chloride in ethylene d	with (S)-(+)- e presence of alu-

-Also obtained by reaction of sec-valeric acid with resorcinol in the presence of zinc chloride (55–75 %) [2747], for 2 h at 150° (55 %) [2829].

-Also obtained by enzymatic enantioselective deacetylation of its 2,4-diacetyl ester [2829].

-Also refer to: [1719, 3025].

yellow viscous oil [2747]; oil [2829]; b.p.₁ 143–148° [2747]; ¹H NMR [2829], ¹³C NMR [2829], IR [2829], UV [2829], MS [2829]; TLC [2829].

Diacetates

C₁₅H₁₈O₅

mol. wt. 278.30

-Refer to: [2829].

*racemic [406174-67-2].

-Obtained by reaction of acetic anhydride with 1-(2,4-dihydroxyphenyl)-2-methyl-1-butanone in the presence of catalytic amount of N,N-dimethylaminopyridine at 22–25° (90 %) [2829].

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oil [2829];
<sup>1</sup>H NMR [2829], <sup>13</sup>C NMR [2829], IR [2829], UV [2829],
MS [2829]; TLC [2829].
```

*levogyre (-) [406174-79-6].

-Obtained by chemical acetylation of monoacetate [2829].

 $(\alpha)_{\rm D}^{25} = -23.9^{\circ}$ (chloroform) [2829].

*dextrogyre (+)

[406174-75-2].

-Obtained by treatment of racemic ketone with PPL pre-incubated in THF at $40-42^{\circ}$ for 12 h in the presence of butanol (81 %) [2829].

 $(\alpha)_{\rm D}^{25} = +32.5^{\circ}$ (chloroform) [2829].

1-(3,4-Dihydroxyphenyl)-2-methyl-1-butanone

BIOLOGICAL ACTIVITY: Central nervous system depressant [2127]; Antimelanoma and skin depigmentation by, *in vitro* method for screening [612].

Dimethyl ether [67049-70-1] C₁₃H₁₈O₃ mol. wt. 222.28

-Obtained by reaction of 2-methylbutyryl chloride with veratrole in the presence of aluminium chloride in refluxing benzene for 30 min (66 %) [2127].

-Also obtained by metallation of veratric acid with s-BuLi/TMEDA (2.2 equiv.) at -30° [66], (22 %) [617].

colourless liquid [2127]; b.p._{0.025} 101° [2127]; $n_D^{25} = 1.5409$ [2127].

BIOLOGICAL ACTIVITY: Central nervous system depressant [2127].

1-(3,5-Dihydroxyphenyl)-2-methyl-1-butanone

$$\begin{array}{ccc} C_{11}H_{14}O_3 & \text{mol. wt. 194.23} \\ OH & Synthesis \\ & -Refer \text{ to: [31].} \\ HO & CO-CH-CH_2CH_3 & C_{13}H_{18}O_3 & \text{mol. wt. 222.28} \end{array}$$

-Preparation from 3,5-dimethoxybenzamide (76 %) [31].

b.p._{0.5} 124–128° [31]; $n_D^{20} = 1.5266$ [31].

2-Methyl-1-(2,4,6-trihydroxyphenyl)-1-butanone

(Multifidol)

[39652-80-7] [125074-06-8]

Syntheses

C11H14O4

	OH	
	, co·	-CH-CH ₂ C
ĺ		L CH ₃
HO	S∽OH	C113

'H₃ -Obtained by reaction of 2-methylbutanoic acid with phloroglucinol in the presence of boron trifluoride etherate [3019].

-Also obtained by adding phloroglucinol to a solution of phosphorous oxychloride plus aluminium chloride and stirred under nitrogen. 2-Methyl-butanoic acid was added and the reaction stirred under nitrogen at 0° for 8 h, then at 6° for 40 h (40–54 %) [3201].

-Also obtained by acid hydrolysis of 2-(2-methylbutyryl)phloroglucinol 1-*O*-(6"-*O*-β-D-apiofuranosyl)-β-D-glucopyranoside [3455].

-Also refer to: [762, 763, 765, 766, 1373, 1401, 1731, 1866, 2621, 3202, 3310].

Isolation from the natural sources

-From the latex of Jatropha multifada (Euphorbiaceae) [337, 1737].

m.p. 116–118° [1737], 62–64° [1401], 62–63° [1866], 61–64° [2621], 61–63° [1373].

N.B.: One of the reported melting point is obviously wrong.

¹H NMR [762, 1737, 3019, 3455], ¹³C NMR [1737, 3019],

IR [762, 1737, 3019], UV [1737, 3019], MS [1737].

BIOLOGICAL ACTIVITY: Antiprotozoal and antimicrobial [337]; Antimicrobial for *Staphylococcus aureus* [3372]; Antagonist both thromboxane A₂ and Leukotriene D₄ [3019]; Antifungal [3202]; Cytotoxicity [3202].

N.B.: Three CAS Registry Numbers attributed at this ketone:

[39652-80-7], [125074-06-8] and [98498-56-7] (racemic).

mol. wt. 210.23

(S)-(+) isomer [111556-27-5] $C_{11}H_{14}O_4$ mol. wt. 210.23

Synthesis

-Obtained by reaction of S-(*) 2-methylbutanoyl chloride with phloroglucinol in the presence,

*of boron trifluoride etherate at 40° for 2 h (30.5 %) [2434];

*of aluminium chloride in nitrobenzene/carbon disulfide mixture (84 %) [286]. -Also refer to: [1115, 1737, 3455].

Isolation from the natural sources

-From the latex collected from the leaf-stalks of *Jatropha multidisa* (Euphorbiaceae) [1737].

m.p. 116–118° [1737], 76–78° [2434]. **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [286, 1737, 3455], ¹³C NMR [1737], IR [1737], UV [1737, 2434], MS [1737]. (α)¹⁸_D = + 26.1 (chloroform) [2434], (α)²⁵_D = + 17.23 (ethanol) [286]; circular dichroism [1737];

2-\beta-D-Glucoside C₁₇H₂₄O₉ mol. wt. 372.37

Isolation from the natural sources

-From the latex of Jatropha multifada (Euphorbiaceae) [1737].

m.p. 139–140° [1737]; ¹H NMR [1737], ¹³C NMR [1737], IR [1737], UV [1737], MS [1737].

Trimethyl ether (S) [124598-12-5] C₁₄H₂₀O₄ mol. wt. 252.31

-Preparation by reaction of S-2-methylbutanoyl chloride with phloroglucinol trimethyl ether in the presence of aluminium chloride (18 %) [2434].

¹H NMR [2434], UV [2434]; (α)_D²¹ = +4.54° (acetone) [2434].

Triacetate (S) [124598-16-9] $C_{17}H_{20}O_7$ mol. wt. 336.34

-Obtained by reaction of acetic anhydride with S-2-methyl-1-(2,4,6-trihydroxyphenyl)-1-butanone in the presence of pyridine (58 %) [2434].

¹H NMR [2434]; (α)²¹_D = + 9.41° (acetone) [2434].

2-Methyl-1-(2,3,4,6-tetrahydroxyphenyl)-1-butanone

[209122-44-1] $C_{11}H_{14}O_5$ mol. wt. 226.23

$$HO \xrightarrow{HO} CO \xrightarrow{CH_3} CO \xrightarrow{I} CO \xrightarrow{I} CH \xrightarrow{CH_2} CO \xrightarrow{I} CH \xrightarrow{$$

Synthesis -Obtained by reaction of 2-methylbutanoic acid with 1,2,3,5-tetrahydroxybenzene (benzenetetrol) in the presence of boron trifluoride etherate and powdered molecular sieves 4 Å at 80° for 1 h under argon (77.5 %) [2721].

pale brown oil [2721]; ¹H NMR [2721], IR [2721], MS [2721].

1-(2-Hydroxyphenyl)-2,2-dimethyl-1-butanone

Synthesis

[106141-14-4]

 $C_{12}H_{16}O_2$

mol. wt. 192.26

 $CO-C-C_2H_5$ -Obtained by treatment of o-bromophenyl 2,2-dimethyl-butyrate with 1.1 equiv. sec-butyllithium (0.25 M in THF/ether/hexane) at -95° and stirred for 30 min. After an additional 30 min at -78° , the mixture was hydrolyzed with saturated NH₄Cl (85 %) [2076].

¹H NMR [2076], IR [2076], UV [2076], MS [2076].

2.1.2 Substituted Hydroxyketones

1-(2,3-Dichloro-4-hydroxyphenyl)-2-methylene-1-butanone

 $C_{11}H_{10}Cl_2O_2$ [4115-00-8] mol. wt. 245.10 Syntheses OH -Refer to: [1517, 2054, 2055, 2058–2061]. .C1 m.p. 84-85° [2054, 2055, 2058-2061]. C-CH₂CH₃ CH₂

[59043-82-2] C₁₂H₁₂Cl₂O₂ mol. wt. 259.13 Methyl ether

-Obtained by adding dropwise acetic anhydride to a suspension of the 4-methoxybutyrophenone in N,N,N',N'-tetramethylmethanediamine. The reaction temperature was maintained at 90° (86 %) [833]. -Also refer to: [742 (94 %), 1517, 3333].

liquid [742];

m.p. 46–48° [833, 3333]; ¹H NMR [742].

2-Hydroxyethyl ether [327023-36-9] C₁₃H₁₄Cl₂O₃ mol. wt. 289.16

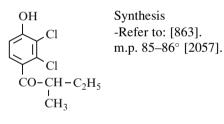
-Refer to: [863].

1-(2 Chloro-4-hydroxyphenyl)-2-methylene-1-butanone

1-(2,3-Dichloro-4-hydroxyphenyl)-2-methyl-1-butanone

C₁₁H₁₂Cl₂O₂ mo

mol. wt. 247.12

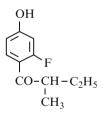


1-(2-Fluoro-4-hydroxyphenyl)-2-methyl-1-butanone (2S)

[505084-77-5]

 $C_{11}H_{13}FO_2$

mol. wt. 196.22



Synthesis -Refer to: [3025].

USE: In preparation of polymeric ferroelectric liquid crystals [3025].

1-(4,6-Dihydroxy-1,3-benzodioxol-5-yl)-2-methyl-1-butanone

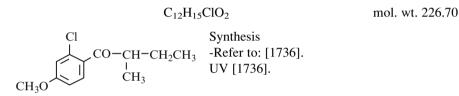
$$\begin{array}{c} C_{12}H_{14}O_5 \\ HO \\ CH_3 \\ CH_2 \\ -CH \\ -CH$$

Isolation from natural sources

-From Helichrysum chrysargyrum [411].

colourless oil [411]; ¹H NMR [411], IR [411], MS [411].

1-(2-Chloro-4-methoxyphenyl)-2-methyl-1-butanone



1-(4-Hvdroxy-2-methylphenyl)-2-methyl-1-butanone (2S)

[505084-75-3]	$C_{12}H_{16}O_2$	mol. wt. 192.26
OH CH	Synthesis -Refer to: [3025]. ¹ H NMR [1352], IR [1352].	

 $\begin{array}{ccc} & & & \\ & & & \\ CO-CH-C_2H_5 & & \\ & & \\ & & \\ CH_3 & \end{array} \quad USE: \quad In \quad preprint for all or preprint for all of the set of th$ USE: In preparation of polymeric ferroelectric liquid

1-(4-Hydroxy-3-methylphenyl)-2-methyl-1-butanone

	$C_{12}H_{16}O_2$	mol. wt. 192.26
OH →CH ₃	Synthesis -Refer to: [218].	
	Methyl ether [927911-91-9] C ₁₃ H ₁₈ O ₂	mol. wt. 206.28
CO-CH-CH ₂ CH ₃		

-Obtained by reaction of 2-methylbutanoic acid with 2-methylanisole in the presence of HSiMe₂Cl and InCl₃ in 1,2-dichloroethane, first 1 h at r.t., then heated at 80° for 4 h (81 %) [218].

¹H NMR [218], ¹³C NMR [218], IR [218], MS [218].

1-(2-Hydroxy-3-methoxyphenyl)-2-methyl-1-butanone

$$[15116-07-1] C_{12}H_{16}O_3 mol. wt. 208.26$$

(50-70 %) [2747].

oil [2747]; b.p._{0.4} 89–94° [2747].

2-Methyl-1-(2,4,6-trihydroxy-3-methylphenyl)-1-butanone (S)

C12H16O4 [124598-11-4] mol. wt. 224.26 OII

Synthesis
-Obtained by reaction of S-2-methylbutanoyl chlo- ride with 2-methylphloroglucinol in the presence of
ride with 2-methylphloroglucinol in the presence of
aluminium chloride (68.3 %) [2434].

m.p. 132–135° [2434]; UV [2434]; $(\alpha)_{D}^{18} = +31^{\circ}$ (acetone) [2434].

[124598-14-7] C₁₅H₂₂O₄ **Trimethyl ether** (S) mol. wt. 266.34

-Obtained bv reaction of S-2-methyl-1-butanoyl chloride with 2-methylphloroglucinol trimethyl ether in the presence of aluminium chloride (21 %) [2434].

¹H NMR [2434], UV [2434]; $(\alpha)_{D}^{21} = +10.6^{\circ}$ (acetone) [2434].

[124598-18-1] C₁₈H₂₂O₇ mol. wt. 350.37 **Triacetate** (S)

-Obtained by reaction of acetic anhydride with S-2-methyl-1-(2,4,6-trihydroxy-3-methylphenyl)-1-butanone in the presence of pyridine (77 %) [2434].

¹H NMR [2434]: $(\alpha)_{\rm D}^{21} = +21.9^{\circ}$ (acetone) [2434].

1-[2-(Acetyloxy)-4-hydroxyphenyl]-2-methyl-1-butanone (-)

[406174-71-8] C13H16O4 mol. wt. 236.27 OCOCH₃ CH_3 CO-CH-C₂H₅ Synthesis -Obtained by enzymatic enantioselective deacetylation of diester in the presence of PPL (72 %) [2829]. oil [2829];

¹H NMR [2829], ¹³C NMR [2829], IR [2829], UV [2829], MS [2829]; TLC [2829]; $(\alpha)_D^{25} = -41.4^{\circ}$ (chloroform) [2829].

1-(2-Hydroxy-5-methylphenyl)-2,2-dimethyl-1-butanone

OH

[106141-15-5] C13H18O2 mol. wt. 206.28 Synthesis CH3

 $^{1}_{C-C_{2}H_{5}}$ -Obtained by treatment of 2-bromo-5-methylphenyl 2,2-dimethylbutyrate with 1.1 equiv. sec-butyllithium (0.25 M in THF/ether/hexane) at -95° and stirred for ĊH₃ 30 min. After an additional 30 min at -78° , the mixture was hydrolyzed with saturated NH₄Cl (76 %) [2076].

¹H NMR [2076], IR [2076], UV [2076], MS [2076].

1-(2,4-Dihydroxy-6-methoxy-3-methylphenyl)-2-methyl-1-butanone

[918896-70-5] $C_{13}H_{18}O_{4}$ mol. wt. 238.28 Isolation from natural sources CH₃ OH $CO-CH-CH_2CH_3$ -From the aerial parts of *Hypericum beanii* (Guttiferae) [2496]. CH₃ pale yellow oil [2496]; HO

¹H NMR [2496], ¹³C NMR [2496], IR [2496], UV [2496], MS [2496].

BIOLOGICAL ACTIVITY: Antistaphylococcal [2496].

1-(2,6-Dihydroxy-4-methoxy-3-methylphenyl)-2-methyl-1-butanone (*Pulverulentone B*)

[89647-61-0]	$C_{13}H_{18}O_4$	mol. wt. 238.28
$CH_{3} \xrightarrow{OH} CO^{-}CH_{2}CH_{2}CH_{3}$	Isolation from natural sou -From <i>Eucalyptus pulveru</i> -From <i>Eucalyptus p</i> (Myrtaceae) [1106].	

MS [420]; HPLC [420]; GC/MS [420].

1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)-2-methyl-1-butanone (Pseudoaspidinol 2-MeB)

[55382-32-6]	$C_{13}H_{18}O_4$	mol. wt. 238.28
HO CH_3 $CO-CH-CH_2CH_3$ HO CH_3 CH_3 CH_3	Syntheses -Obtained by reductive alkaline *of kosotoxin (II) and protok female flowers of <i>Hagenia ab</i> Gmelin [1916] (IVc); *of "kosin" (IV), from <i>Flos kos</i> treatment with 15 % potassium presence of zinc powder on a w	xosin (III) from <i>pyssinica</i> (Bruce) so "Siegfried" by hydroxide in the

[1915] (VIIIc).

m.p. 59–61° [1915]; ¹H NMR [2744].

- **N.B.**: "Kosin" (IV), others names: Methylene-bis-pseudo-aspidinol; pseudo-aspidin.
 - m.p. 148–150° [1915];
 ¹H NMR [1915], ¹³C NMR [1912], IR [1915], UV [1915], MS [1915]; GLC [2531].

(S)-(+) [124598-08-9].

-Obtained by reaction of S-2-methylbutanoyl chloride (4 mol) with methyl 2,6-dihydroxy-4-methoxy-3-methylbenzoate (4 mol) in the presence of aluminium chloride (12 mol) in carbon disulfide for 1.5 h (7 %) [2434].

m.p. 39–40° [2434]; ¹H NMR [2434], UV [2434]; $(\alpha)_D^{23} = +18.6^\circ$ (acetone) [2434].

Diacetate (S)-(+) [124598-17-0]
$$C_{17}H_{22}O_6$$
 mol. wt. 322.36

-Obtained by reaction of acetic anhydride with S-1-(4,6-dihydroxy-2-methoxy-3-methylphenyl)-2-methyl-1-butanone in the presence of pyridine (84 %) [2434].

¹H NMR [2434]; $(\alpha)_{\rm D}^{21} = +10.3^{\circ}$ (acetone) [2434].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-methyl-1-butanone (S)

 $[124598-07-8] C_{13}H_{18}O_4 mol. wt. 238.28$ $OH CH_3 CO-CH-C_2H_5 OCH_3 O$

¹H NMR [2434], UV [2434]; $(\alpha)_D^{18} = +16.2^{\circ}$ (chloroform) [2434].

2-Methyl-1-(2,4,6-trihydroxy-3,5-dimethylphenyl)-1-butanone

[87035-85-6] [65792-31-6] $CH_3 \rightarrow CO^{-}CH^{-}CH_2CH_3$ $HO \rightarrow CH_3$ $HO \rightarrow CH_3$ $CH_3 \rightarrow CO^{-}CH^{-}CH_2CH_3$ $HO \rightarrow CH_3$ $CH_3 \rightarrow CO^{-}CH^{-}CH_2CH_3$ $CH_3 \rightarrow CO^{-}CH^{-}CH_3CH_3$ $CH_3 \rightarrow CO^{-}CH^{-}CH_3CH_3$ $CH_3 \rightarrow CO^{-}CH^{-}CH_3$

m.p. 105-106° [2858].

Trimethyl ether	[245063-60-9]	$C_{16}H_{24}O_{4}$	mol. wt. 280.36
(Isotorquatone)			

Isolation from natural sources

-From the leaf oils of the genus Xanthostemon (Myrtaceae) in Australia [462].

-From essential oil of Eucalyptus apodophylla (Myrtaceae) [2046].

-From volatile leaf oils of some southwestern and southern Australian species of the genus *Eucalyptus* [353–356, 359, 366].

-Compound of leaf essential oils of *Eucalyptus chartaboma* (chemotype II) (3.1 %) and *Eucalyptus miniata* (1.3 %) (Myrtaceae) [1453].

¹H NMR [1453, 2046], ¹³C NMR [1453, 2046], MS [1453, 2046]; GC-MS [462].

1-(4,6-Dihydroxy-2,3-dimethoxyphenyl)-2-methyl-1-butanone

 $[73694-18-5] \qquad C_{13}H_{18}O_5 \qquad \text{mol. wt. } 254.28$ $Iorred CH_3 \qquad Isolation from natural sources -From Helichrysum nanum (Compositae) (15) [401].$ $HO \qquad OCH_3 \qquad Interpret Helichrysum nanum (Compositae) (15) [401].$

Synthesis

-Obtained by heating a mixture of acetic anhydride, 1-(4,6-dihydroxy-2,3-dimethoxyphenyl)-2-methyl-1-butanone and 4-pyrrolidinopyridine for 1 h at 70° (7 %) [401].

oil [401]; ¹H NMR [401], MS [401]; TLC [401].

1-(2-Amino-5-methoxy-4-methylphenyl)-2-methyl-1-butanone

[959137-60-1] $C_{13}H_{19}NO_2$ mol. wt. 221.30 NH₂ CH_3 CH_3 Synthesis -Refer to: [2027]. CH₃ OCH_3

1-(2,6-Dihydroxy-4-methoxy-3,5-dimethylphenyl)-2-methyl-1-butanone

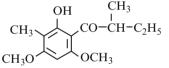
[138690-39-8]		$C_{14}H_{20}O_4$	mol. wt. 252.31
CH ₃ CH ₃ O CH ₃ O CH ₃ O CH ₃ O	CH ₃ -CH-C ₂ H ₅	Isolation from natural sources -From <i>Hagenia Abyssinica</i> (F -From <i>Eucalyptus robusta</i> [637, 1106]. -Also refer to: [727, 2533].	

pale yellow [2744]; m.p. 47–49° [2744]; ¹H NMR [637, 2744], ¹³C NMR [637, 1106, 2744], IR [637, 2744], UV [637], MS [637, 2744]; $(\alpha)_{\rm D} = +17^{\circ}$ (chloroform) [2744]; TLC [2744].

BIOLOGICAL ACTIVITY: Refer to: [637].

1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-2-methyl-1-butanone (S)

[124598-13-6] C14H20O4 mol. wt. 252.31



 $\begin{array}{c} \begin{array}{c} CH_{3} \\ \hline \\ CO-CH-C_{2}H_{5} \end{array} \\ \begin{array}{c} CO-CH-C_{2}H_{5} \\ OCH_{3} \end{array} \\ \begin{array}{c} Synthesis \\ -Obtained by reaction of S-2-methyl-1-butanoyl \\ chloride with 3,5-dimethoxy-2-methylphenol in \\ the presence of aluminium chloride (8 %) [2434]. \end{array}$

¹H NMR [2434], UV [2434]; $(\alpha)_{D}^{21} = +21.2^{\circ}$ (acetone) [2434].

[124598-19-2] Acetate C16H22O5 mol. wt. 294.35

-Obtained by reaction of acetic anhydride with S-1-(2-hydroxy-4,6-dimethoxy-3-methylphenyl)-2-methyl-1-butanone in the presence of pyridine (91 %) [2434].

¹H NMR [2434], UV [2434]; $(\alpha)_{\rm D}^{21} = +14.4^{\circ}$ (acetone) [2434].

1-(6-Hydroxy-2,4-dimethoxy-3-methylphenyl)-2-methyl-1-butanone

(Homoisobaeckeol)

[808751-13-5]

C14H20O4 mol. wt. 252.31

OH CH_3 $CO-CH-C_2H_5$ $CO-CH_3$ CH_3 Isolation from natural sources -Compound of leaf essential oils of *Eucalyptus chartaboma* (chemotype I) (27.5 %), *Eucalyptus chartaboma* (chemotype II) (8 %) and *Eucalyptus miniata* (6.3 %) (Myrtaceae) [1453]

¹H NMR [1453], ¹³C NMR [1453], MS [1453].

Methyl ether	[808749-86-2]	$C_{15}H_{22}O_4$	mol. wt. 266.34
(Homobaeckeol met	hyl ether)		

Isolation from natural sources

-Compound of leaf essential oils of Eucalyptus chartaboma (chemotype II) (4.7 %) and Eucalyptus miniata (7.3 %) (Myrtaceae) [1453].

¹H NMR [1453], ¹³C NMR [1453], MS [1453].

1-[2,4-Bis(acetyloxy)-6-hydroxyphenyl]-2-methyl-1-butanone (S)

 $C_{15}H_{18}O_{6}$ [124598-15-8] mol. wt. 294.30 Synthesis

CH₃ OH

¹H NMR [2434]; $(\alpha)_D^{21} = +7.94^{\circ}$ (acetone) [2434].

 $CO-CH-C_2H_5$ -Obtained by reaction of acetic anhydride with S-2-methyl-1-(2,4,6-trihydroxyphenyl)-1-butanone in the presence of pyridine (54 %) [2434].

1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-2-methyl-1-butanone

C₁₅H₂₂O₂ mol. wt. 234.34 [80356-13-4] Syntheses OH -Obtained by photo-Fries rearrangement of thymyl 2-methylbutanoate in methanol for 6 h at 25° under nitrogen (16.5%) (7) $(CH_3)_2CH_3$ CH₂ $CO-CH-CH_2CH_3$ -Also refer to: [2421]. m.p. 126° [2421]; ĊH₃ ¹H NMR [2421], IR [2421].

1-(2-Hydroxy-4,6-dimethoxy-3,5-dimethylphenyl)-2-methyl-1-butanone (Chartabomone)

[808751-11-3]

$C_{15}H_{22}O_4$	mol. wt. 266.34
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CH₃ OH OCH₃ CH₃O

Isolation from natural sources $-CO-CH-C_2H_5$ -Compound of leaf essential oils of *Eucalyptus* chartaboma (chemotype II) (0.5 %) and Eucalyptus miniata (0.3 %) (Myrtaceae) [1453].

¹H NMR [1453], ¹³C NMR [1453], MS [1453].

CH₃COO

¹H NMR [2434];

1-(4-Hydroxy-2,6-dimethoxy-3,5-dimethylphenyl)-2-methyl-1-butanone

[87035-88-9]	$C_{15}H_{22}O_4$	mol. wt. 266.34
$CH_{3} \rightarrow CH_{3} CH_{3} CH_{3} CH_{3} CH_{3} CH_{3} CO-CH-CH_{2}CH_{3} CO-CH-CH_{2}CH_{3} CH_{3} CO-CH-CH_{2}CH_{3} CH_{3} CH_{$	Syntheses -Obtained by deprotection of 1-[4 hylsilyl)-oxy-2,6-dimethoxy-3,5- 2-methyl-1-butanone with tetra-r fluoride in THF for 1 h (82 %) [4 -Also refer to: [2512].	-dimethylphenyl]- n-butylammonium
m.p. 58.5–59° [475]; ¹ H NMP [2512] ¹³ C NMP	2 [2512] IR [2512] UV [475]	

¹H NMR [2512], ¹³C NMR [2512], IR [2512], UV [475], MS [2512].

USE: Asym. synthesis of wasabidienones B_1 an B via SIBX-mediated hydroxylative phenol dearomatization [2512].

1-(4-Hydroxy-2,6-dimethoxy-3,5-dimethylphenyl)-2-methyl-1-butanone (2R)

[1080023-65-9]
$$C_{15}H_{22}O_4$$
 mol. wt. 266.34
OH Synthesis
CH₃ CH₃ -Refer to: [2512].
CH₃O OCH₃ MS [2512], ¹³C NMR [2512], IR [2512],
CO-CH-CH₂CH₃ (α)²⁰_D = -4.1° [2512].

USE: Asym. synthesis of wasabidienones B_1 an B via SIBX-mediated hydroxylative phenol dearomatization [2512].

1-(4-Hydroxy-2,6-dimethoxy-3,5-dimethylphenyl)-2-methyl-1-butanone (2S)

[1080023-64-8] $C_{15}H_{22}O_4$ mol. wt. 266.34 OH Synthesis -Refer to: [2512]. CH₃O OCH₃ [2512]; CO-CH-CH₂CH₃ (α)²⁰_D = +5° [2512]. I (α)²⁰_D = +5° [2512].

USE: Asym. synthesis of wasabidienones B_1 an B via SIBX-mediated hydroxylative phenol dearomatization [2512].

1-[2,3-Dihydro-4,6-dihydroxy-2-(1-methylethenyl)-5-benzofuranyl]-2-methyl-1-butanone

[103771-75-1]	$C_{16}H_{20}O_4$	mol. wt. 276.33
CH ₃ HO C ₂ H ₅ -CH-CO	 H₂ Isolation from natural -From <i>Helichrysum</i> <i>Helichrysum</i> (Compositae) [1487]. 	cephaloideum and mixtum

1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-2-methyl-1-butanone

Synthesis

CH₃

CH₂

[39652-88-5]

-Obtained by heating of 2-methyl-1-[2,4,6-tri-hydroxy-3-(3-methyl-2-butenyl) phenyl]-1-butanone in benzene with sulfuric acid in the presence of acetic anhydride [398].

mol. wt. 278.35

Isolation from natural sources

CH₃

CH₃CH₂-CH-CO

-From the aerial parts of *Helichrysum platypterum* DC (Compositae) [1487]. -From *Helichrysum gymnnoconum* (Compositae) [398].

¹H NMR [398], IR [398], MS [398].

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όн

1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl)-2-methyl-1-butanone

C16H22O4

CO-		C ₂ H ₅ CH ₃ CH ₃
T	•	
ÓН		
		CO-CH-

[39652-87-4]

Syntheses -Obtained by heating of 2-methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-butanone in benzene with sulfuric acid in the presence of acetic anhydride [398]. -Also refer to: [708].

Isolation from natural sources

-From *Helichrysum platypterum* (Compositae) [1487]. -From *Helichrysum gymnnoconum* (Compositae) [398].

¹H NMR [398, 1487], IR [398], MS [398, 1487].

1-[2,6-Dihydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-2-methyl-1-butanone

[70219-82-8]
$$C_{16}H_{22}O_4$$
 mol. wt. 278.35
OH CH_3 Isolation from natural sources
-From *Helichrysum crispum*
(Compositae) (**30**) [401].
-From *Helichrysum squarrosus*
DC (Compositae) (**6**) [400].

-From *Helichrysum asperum* (Thunb.) Hilliard et Burtt. var. *albidulum* (DC) Hilliard [1488].

colourless oil [400]; ¹H NMR [400], IR [400], MS [400].

2-Methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-butanone (*HP34.12*)

C₁₆H₂₂O₄

[71539-60-1]

 $(CH_3)_2C = CHCH_2$ OH CH_3 $CO - CH - CH_2CH_3$

HC

mol. wt. 278.35

Isolation from natural sources -From *Helichrysum infaustum* (Compositae) [401].

-From *Helichrysum paronychioides* (Asteraceae, tribe Inuleae) [2201].

-From Helichrysum platypterum DC (Compositae) [404, 1487].

-From Helichrysum gymmnoconum (Compositae) [398].

OH

-From *Helichrysum asperum* (Thunb.) Hilliard et Burtt. var. *albidulum* (DC) Hilliard [1488].

-From Helichrysum indicum (L.) Grieson (86/248, near Clanwilliam) [1488].

-From Helichrysum moeserianum Thell. (86/249, near Clanwilliam) [1488].

-From Helichrysum odoratissimum (Compositae) [1238].

colourless oil [398]; ¹H NMR [398, 2201], ¹³C NMR [2201], IR [398, 2201], UV [2201], MS [2201]; TLC [2201].

BIOLOGICAL ACTIVITY: Antioxidant [2201].

Triacetate [71539-61-2] C₂₂H₂₈O₇ mol. wt. 404.46

-Obtained by heating 2-methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl) phenyl]-1-butanone with acetic anhydride and sodium acetate [398].

colourless oil [398]; ¹H NMR [398], IR [398], MS [398].

Trimethyl ether [1175530-97-8] $C_{19}H_{28}O_4$ mol. wt. 320.43

¹H NMR [3334], ¹³C NMR [3334], IR [398], MS [3334].

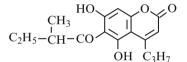
1-[2,6-Dihydroxy-4-[(4-hydroxy-3-methyl-2-butenyl)
oxy]phenyl]-2-methyl-1-butanone $({\cal E})$

 $[122585-54-0] C_{16}H_{22}O_5 mol. wt. 294.35$ $OH CH_3 - CO-CH-CH_2CH_3 - CO-CH-CH_2CH_3 - From Helichrysum asperum (Thunb.) Hilliard et Burtt. var. albidulum (DC) Hilliard [1488].$

¹H NMR [1488], IR [1488], MS [1488].

5,7-Dihydroxy-6-(2-methyl-1-oxobutyl)-4-propyl-2*H***-1-benzopyran-2-one** (*MAB 2*)

 $[5022-22-0] C_{17}H_{20}O_5 mol. wt. 304.34$



Syntheses -Obtained by condensation of 2-(2-methylbutyryl)-phloroglucinol with ethyl 4-bromo-3-oxohexanoate [763].

-Also obtained by reaction of ethyl 3-oxohexanoate with 1-(2,4,6-trihydroxy-phenyl)-2-methyl-1-butanone in the presence of concentrated sulfuric acid in acetic acid at r.t. for 3 days [753].

-Also refer to: [553, 762].

crystals [753]; m.p. 209–211° [762], 207–208° [753]; ¹H NMR [553, 753, 762], IR [753, 762], UV [553, 753, 762], MS [753].

Syntheses

5,7-Dihydroxy-8-(2-methyl-1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one

$$\begin{array}{c} CH_3\\ CO-CH-C_2H_5\\ HO \\ HO \\ OH \\ C_3H_7\end{array}$$

[5022-23-1]

 $C_{17}H_{20}O_5$

mol. wt. 304.34

-Obtained by condensation of 2-(2-methylbutyryl)phloroglucinol with ethyl 4-bromo-3-oxohexanoate (3 %) [763].

-Also obtained by reaction of ethyl 3-oxohexanoate with 1-(2,4,6-trihydroxyphenyl)-2-methyl-1-butanone in the presence of concentrated sulfuric acid in acetic acid at r.t. for 3 days (34 %) [753].

-Also refer to: [553, 762].

crystals [753]; m.p. 248–250° for racemic material [286], 235–236° [753]; ¹H NMR [286, 753], IR [753], UV [553, 753], MS [753].

(S)-(-) isomer

-Obtained by adding ethyl 3-oxohexanoate followed by concentrated sulfuric acid to (S)-(+)-(2-methylbutyryl)phloroglucinol in glacial acetic acid. The mixture was allowed to stand for 4 days (27 %) [286].

m.p. 254–256° [286]; ¹H NMR [286]; $(\alpha)_D^{25} = -5.4^\circ$ (ethanol) [286].

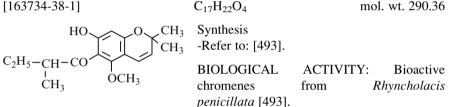
1-(7-Hydroxy-5-methoxy-2,2-dimethyl-2H-1-benzopyran-6-yl)-2-methyl-1-butanone

(Rhynchonin B)(-)

OH

[71539-62-3]

[163734-38-1]

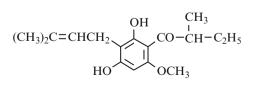


1-(3,4-Dihydro-5-hydroxy-7-methoxy-2,2-dimethyl-2H-1-benzopyran-8-yl)-2-methyl-1-butanone

 $C_{17}H_{24}O_{4}$ mol. wt. 292.37 Isolation from natural sources CH₂ -From Helichrysum platypterum (Compositae) [1487]. $O-CH-C_2H_5$ ¹H NMR [1487], MS [1487]. CH₃

1-[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-methyl-1-butanone

C17H24O4



¹H NMR [398], IR [398], MS [398].

mol. wt. 292.37

Isolation from natural sources -From the aerial parts of Helichrysum cephaloideum (Compositae) [1487]. -From Helichrysum gymmnoconum (Compositae) [398].

Diacetate [71539-64-5] $C_{21}H_{28}O_{6}$ mol. wt. 376.45

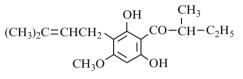
-Obtained by reaction of acetic anhydride with 1-[2,4-dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-methyl-1-butanone in the presence of sodium acetate for 12 h at 70° [398].

¹H NMR [398], IR [398], MS [398],

1-[2,6-Dihydroxy-4-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-methyl-1-butanone

 $C_{17}H_{24}O_4$

[103771-69-3]



Isolation from natural sources -From the aerial parts of Helichrysum platypterum (Compositae) [1487].

¹H NMR [1487], MS [1487].

1-(3,4-Dihydro-5-hydroxy-7-methoxy-2,2-dimethyl-2H-1-benzopyran-6-yl)-2-methyl-1-butanone

 $C_{17}H_{24}O_{4}$ [71539-70-3] mol. wt. 292.37

$$\begin{array}{c} CH_{3}O\\CH_{3}\\C_{2}H_{5}-CH-CO\\OH\end{array} \xrightarrow{O}CH_{3}\\CH_{$$

Synthesis -Obtained by heating of 1-[2,4-dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-methyl-1-butanone in benzene with sulfuric acid in the presence of acetic anhydride [398].

Isolation from natural sources

-From Helichrysum platypterum (Compositae) [1487]. -From Helichrysum gymmnoconum (Compositae) [398].

¹H NMR [398, 1487], IR [398, 1487], MS [398, 1487].

1-[2-(β-D-Glucopyranosyloxy)-4,6-dihydroxyphenyl]-2-methyl-1-butanone

[124960-73-2]	$C_{17}H_{24}O_9$	mol. wt. 372.37
OH CH ₃ CO-CH- HO O-β-D-glu	Gaertn. (syn. <i>Phys</i>	gs of <i>Emblica officinalis</i> <i>llanthus emblica</i>) com- <i>nla</i> (Indian goose berry)

mol. wt. 292.37

-From hops [277].

-From the latex of Jatropha multifada [337, 1737].

-From the leaves and branches of *Phyllanthus emblica* (Euphorbiaceae) [3455].

m.p. 139–140° [1737]; ¹H NMR [1737, 3455], ¹³C NMR [1737, 3455], IR [1737], UV [1737], MS [1737].

BIOLOGICAL ACTIVITY: Antiinflammatory agent [277]; Antiprotozoal and antimicrobial [337].

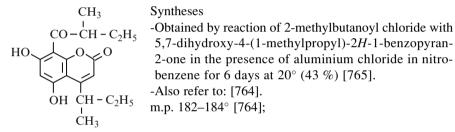
5,7-Dihydroxy-8-(2-methyl-1-oxobutyl)-4-(1-methylpropyl)-2H-1-benzopyran-2-one

[98192-74-6]

C18H22O5

mol. wt. 318.37

mol. wt. 314.38



¹H NMR [764], IR [764], UV [764], MS [764].

1-[4-[[(4-Acetyloxy)-3-methyl-2-butenyl]oxy]-2,6-dihydroxyphenyl]-2-methyl-**1-butanone** (E)

benzene for 6 days at 20° (43 %) [765].

[122585-55-1] mol. wt. 336.38 $C_{18}H_{24}O_6$ CH₃ Isolation from natural sources OH $CO - CH - CH_2CH_3$ -From Helichrysum asperum (Thunb.) Hilliard et Burtt. var. albidulum (DC) Hilliard [1488]. $CH_3 - C = CHCH_2O$ OH ¹H NMR [1488]. CH₂OCOCH₃

1-[2,6-Dihydroxy-3,5-dimethyl-4-(phenyloxy)phenyl]-2-methyl-1-butanone

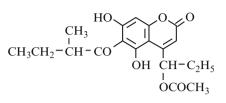
 $C_{10}H_{22}O_4$

$$\begin{array}{c|cccccc} OH & Synthesis \\ CH_3 & CO - CH - CH_2CH_3 \\ C_6H_5O & OH \\ CH_3 & CH_3 \\ CH_3 & CH_3$$

USE: Asym. synthesis of wasabidienones B_1 an B via SIBX-mediated hydroxylative phenol dearomatization [2512].

4-[1-(Acetyloxy)propyl]-5,7-dihydroxy-6-(2-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one

C₁₉H₂₂O₇ mol. wt. 362.38



Synthesis

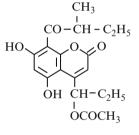
-Obtained by condensation of ethyl 3-oxo-4-acetoxyhexanoate with (2-methylbutyroyl)-phloroglucinol in the presence of 5 % sulfuric acid in acetic acid (traces) [766].

4-[1-(Acetyloxy)propyl]-5,7-dihydroxy-8-(2-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one

[98498-77-2]

C₁₉H₂₂O₇ mol. wt. 362.38

Syntheses



-Obtained by condensation of ethyl 3-oxo-4-acetoxyhexanoate with (2-methylbutyroyl)phloroglucinol in the presence of 5 % sulfuric acid in acetic acid (traces) [766].

-Also obtained by reaction of 2-methylbutyryl chloride with 4-[1-(acetyloxy)propyl]-5,7-dihydroxy-2*H*-1-benzopyran-2-one in the presence of aluminium chloride in carbon disulfide/nitrobenzene for 4 days at 20° (41 %) [766].

-Also refer to: [764].

m.p. 182–184° [764]; ¹H NMR [764], IR [764], UV [764], MS [764].

Stereoisomer (1'RS,2"S)

 $C_{19}H_{22}O_7$

mol. wt. 362.38

[98498-79-4] [98498-80-7] [98574-78-8] [98574-81-3].

-Refer to: [286].

 $(\alpha)_{\rm D}^{25} = +16.83^{\circ}$ (chloroform) [286].

5,7-Dihydroxy-6-(2-methyl-1-oxobutyl)-4-pentyl-2H-1-benzopyran-2-one

Syntheses

$$[98192-60-0] C_{19}H_{24}O_5 mol. wt. 332.40$$

 $\begin{array}{c} CH_3 \\ C_2H_5 - CH - CO \\ OH \\ C_5H_{11} \end{array} \xrightarrow{O} O$

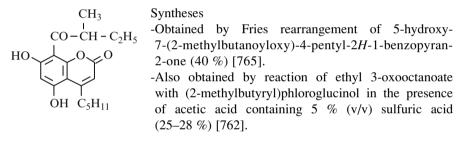
-Obtained by Fries rearrangement of 5-hydroxy-7-(2-methylbutanoyloxy)-4-pentyl-2*H*-1-benzopyran-2-one [765].

-Also obtained by reaction of ethyl 3-oxooctanoate with (2-methylbutyryl) phloroglucinol in the presence of acetic acid containing 5 % (v/v) sulfuric acid (38-42 %) [762].

m.p. 196–197° [762]; ¹H NMR [762], IR [762], UV [762].

5,7-Dihydroxy-8-(2-methyl-1-oxobutyl)-4-pentyl-2H-1-benzopyran-2-one

[36478-56-5] (*Mammea C/OB*) C₁₉H₂₄O₅ mol. wt. 332.40 [111821-07-9] (*Unusual mammea C/O*)



Isolation from natural sources

-From the bark of *Mammea africana* G. Don (Guttiferrae) [553, 751]. -Also refer to: [827].

needles [553]; m.p. 218–220° [553], 218° [762, 2591]; ¹H NMR [553, 751, 2591], IR [553, 762, 2591], UV [553, 762, 2591], MS [553, 751, 2591].

3,4-Dihydro-5,7-dihydroxy-8-(2-methyl-1-oxobutyl)-4-pentyl-2*H*-1-benzopyran-2-one

(Dihydromammea C/OB)

USE: Insecticide [751].

1-[4-(β-D-Glucopyranosyloxy)-2,6-dihydroxy-3,5-dimethylphenyl]-2-methyl-1-butanone

[241133-23-3] $C_{19}H_{29}O_9$ mol. wt. 401.43 OH Isolation from natural sources CH_3 CO-CH-C₂H₅ -From *Hypericum japonicum* [3342]. β -D-Glc-O OH CH₃

5,7-Dihydroxy-6-(2-methyl-1-oxobutyl)-4-phenyl-2H-1-benzopyran-2-one

 $C_{20}H_{18}O_5$

[98192-58-6]

-Preparation as a mixture (70 %) from (2-methylbutyryl)-phloroglucinol and ethyl benzoylacetate [762] in acetic acid/concentrated sulfuric acid for 7 days at 20° [754].

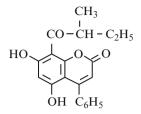
mol. wt. 338.36

-Also refer to: [765, 2380, 2381, 2556].

yellow needles [762]; yellow crystals [754]; m.p. 207–209° [762], 201–202° [754]; ¹H NMR [754, 762], IR [754, 762], UV [754, 762], MS [754, 762].

5,7-Dihydroxy-8-(2-methyl-1-oxobutyl)-4-phenyl-2H-1-benzopyran-2-one

 $[98192-65-5] C_{20}H_{18}O_5 mol. wt. 338.36$



Syntheses -Preparation as a mixture (70 %) from (2-methylbutyryl)phloroglucinol and ethyl benzoylacetate [762] in acetic acid/concentrated sulfuric acid for 7 days at 20° [754]. -Also refer to: [765, 2380, 2381].

white plates [762]; m.p. 222–224° [762], 210–211° [754]; ¹H NMR [754, 762], IR [754, 762], UV [754, 762], MS [754, 762].

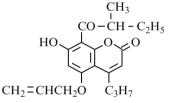
7-Hydroxy-8-(2-methyl-1-oxobutyl)-5-(2-propenyloxy)-4-propyl-2*H*-1-benzopyran-2-one

Synthesis

[111761-38-7]

 $C_{20}H_{24}O_5$

mol. wt. 344.41



-Obtained by treatment of 5,7-dihydroxy-8-(2-methyl-butyryl)-4-propylcoumarin with allyl bromide in the presence of potassium carbonate and potassium iodide in refluxing acetone overnight [762].

white needles [762]; m.p. 95–96° [762];

¹H NMR [762], IR [762], UV [762], MS [762].

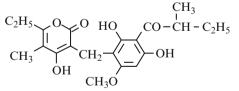
3-[[2,4-Dihydroxy-6-methoxy-3-(2-methyl-1-oxobutyl)phenyl]methyl]-6-ethyl-4-hydroxy-5-methyl-2*H*-pyran-2-one

(*Methyl-nor-auricepyrone*)

[103766-18-3]

 $C_{21}H_{26}O_7$

mol. wt. 390.43



Isolation from natural sources				
-From Helichrysum cephaloideum				
[1487].				
yellow oil [1487];				
¹ H NMR [1487], IR [1487],				
MS [1487].				

1-[3,4-Dihydro-5,7-dihydroxy-2-methyl-2-(4-methyl-3-pentenyl)-2H-1-benzopyran-8-yl]-2-methyl-1-butanone

[658702-63-7] (+)	$C_{21}H_{30}O_4$	mol. wt. 346.47
$HO \xrightarrow{CH_3}_{I} CO - CH - C_2H_5$ $HO \xrightarrow{CH_3}_{CH_2CH_2CH=C(CH_3)} CH_2CH = C(CH_3)$	Isolation from natural s -From the aerial part <i>amblycalyx</i> (Guttiferae yellow oil [3316]; $(\alpha)_D^{22} = +14^\circ$ (methanol	s of <i>Hypericum</i> e) [3316].

¹H NMR [3316], ¹³C NMR [3316], UV [3316], MS [3316].

BIOLOGICAL ACTIVITY: Antibacterial [3316]; Cytotoxicity against KB cancer cells [3316].

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

[71539-67-8]

CH₃

C₂₁H₃₀O₄

mol. wt. 346.47

CH₃ Isolation from OH $CO - CH - C_2H_5$ natural sources -From Helia Helichr-CH₃ $CH_3-C=CHCH_2CH_2-C=CHCH_2O$ vsum gymmnoconum (Compositae) [398].

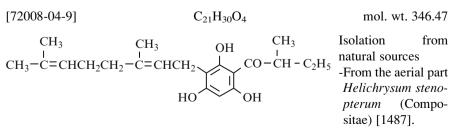
¹H NMR [398], IR [398], MS [398],

Diacetate (E)	[71539-68-9]	$C_{25}H_{34}O_{6}$	mol. wt. 430.54
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-Obtained by reaction of acetic anhydride with 1-[3-(3,7-dimethyl-2,6-octadienyl)-2,4,6-trihydroxyphenyl]-2-methyl-1-butanone in the presence of sodium acetate for 12 h at 70° [398].

¹H NMR [398], IR [398], MS [398].

1-[3-(3,7-Dimethyl-2,6-octadienyl)-2,4,6-trihydroxyphenyl]-2-methyl-**1-butanone** (*E*)



-From Helichrysum natalitium DC (Compositae) [404].

-From the aerial parts of Achyrocline alata (HBK) DC (Compositae) [406].

-From Helichrysum krookii Moeser (Compositae) [412].

-From Helichrysum monticola [1488].

-From Esenbeckia nesiotica Stand. (Rutaceae) [2625].

colourless oil [404]; ¹H NMR [404, 2625], ¹³C NMR [2625], IR [404], MS [404].

Triacetate [72008-09-4] C₂₇H₃₆O₇ mol. wt. 472.58

-Obtained by reaction of acetic anhydride with 1-[3-(3,7-dimethyl-2,6-octadienyl)-2,4,6-trihydroxyphenyl]-2-methyl-1-butanone in chloroform in the presence of pyrrolidinopyridine at 70° for 1 h [404]. -Also refer to: [2625].

colourless oil [404]; ¹H NMR [404, 2625], IR [404], MS [404].

1-[2,4,6-Trihydroxy-3,5-bis(3-methyl-2-butenyl)phenyl]-2-methyl-1-butanone (*Deoxyadhumulone*)

[4374-92-9] C ₂₁	H ₃₀ O ₄ mol. wt. 346.47	
$(CH_3)_2C = CHCH_2 \qquad \qquad$	2-methyl-3-buten-2-ol with 2,4,6-trihydroxy-phenyl sec-	
-Also obtained by reaction of Z-methyl-1-(2,4,6-trihydroxyphenyl)-1-butanone with 1-bromo-3-methyl-2-buten (3 mol) in the presence of the weakly basic resin		

with 1-bromo-3-methyl-2-buten (3 mol) in the presence of the weakly basic resin DeAcidite H-IP (OH-form) in boiling benzene (14 %) [708]. **N.B.**: The yield was similar at this obtained by using 2-methyl-3-buten-2-ol but the isolation was simpler [708].

-Also obtained by reaction of 2-methyl-1-(2,4,6-trihydroxyphenyl)-1-butanone with 1-chloro-3-methyl-2-butene (2 molar parts), magnesium oxide (0.5 equiv.) and potassium iodide [3310].

-Also refer to: [907, 1397].

oil [702]; m.p. 82° [708]; UV [702].

Tribenzoate [39652-89-6] C₄₂H₄₂O₇ mol. wt. 658.79

-Refer to: [704, 1397].

m.p. 127° [704], 126–127° [1397].

2-Methyl-1-[3-(3,7-dimethyloctyl)-2,4,6-trihydroxyphenyl]-1-butanone

 $\begin{array}{cccc} C_{21}H_{34}O_4 & \text{mol. wt. 350.50} \\ OH & CH_3 & Synthesis \\ + & CO-CH-C_2H_5 & -Refer to: [404]. \\ HO & OH & CH_3 & CH_3 & C_{27}H_{40}O_7 & \text{mol. wt. 476.28} \\ + & CH_2CH_2-CH-CH_2CH_2CH_2-CH-CH_3 & C_{27}H_{40}O_7 & \text{mol. wt. 476.28} \end{array}$

-Obtained by hydrogenation of 1-[2,4,6-tris(acetoxy)-3-(3,7-dimethyl-2,6-octadienyl)phenyl]-2-methyl-1-butanone in ethyl ether in the presence of 5 % palladium/barium sulfate.

colourless oil [404]; ¹H NMR [404], IR [404], MS [404].

5,7-Dihydroxy-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one

(Neomammein, Mammea B/BB)

[5022-20-8] racemic [37975-64-7]

Syntheses

C22H28O5

-Obtained by reaction of prenyl bromide with 5,7-dihydroxy-8-(2-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one in the presence of 10 % aqueous potassium hydroxide at 0° (30–45 %) [765], (29 %) [762].

mol. wt. 372.46

-Also obtained by treatment of its dipotassium salt with trifluoroethanol [765].

Isolation from natural sources

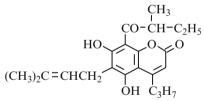
-From kernels of the fruit of *Mammea americana* L. and *Mammea africana* (Guttiferae) [765, 1006].

-From only seed of Mammea africana (Guttiferae) [751].

-From the seed of Mammea americana L. (Guttiferae) [753, 758, 759].

-From the bark of Mammea africana G. Don (Guttiferae) [553, 1082].

pale yellow needles [751]; white needles [762]; m.p. 122–123° [1006], 122° [553, 753], 121–122° [762], 117–118° [755], 72–74° [751];



¹H NMR [553, 751, 753, 762], ¹³C NMR [751], IR [553, 751, 753, 755, 762], UV [553, 751, 753, 755, 762, 1150], MS [553, 751, 753, 755, 762]; GC-MS [1082].

USE: Insecticide [753, 758, 759, 765].

BIOLOGICAL ACTIVITY: Antitumor [1006].

(S) isomer

[98244-57-6]

-(S) - (-) – isomer was obtained by prenylation of 5,7-dihydroxy-8-(2-methylbutanoyl)-4-propylcoumarin. Natural mammea B/BB has the (S) – configuration [765].

m.p. $121-122^{\circ}$ [765]; ¹H NMR [2591], ¹³C NMR [2591], IR [2591]; $(\alpha)_{\rm D} = -2.78^{\circ}$ [765].

5,7-Dihydroxy-8-(3-methyl-2-butenyl)-6-(2-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one

(Mammea B/A)

[30390-12-6] C₂₂H₂₈O₅ mol. wt. 372.46

$$\begin{array}{c} CH_2CH=C(CH_3)_2\\ HO & O\\ CH_3 & O\\ C_2H_5-CH-CO & O\\ OH & C_3H_7 \end{array}$$

Syntheses -Obtained by reaction of prenyl bromide with 5,7-dihydroxy-6-(2-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one in the presence of 10 % aqueous potassium hydroxide at 0° (30–45 %) [765], (22 %) [762].

-Also obtained by treatment of its dipotassium salt with trifluoroethanol [765]. -Also obtained by isomerization of 5,7-dihydroxy-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one (mammea B/BB) by treatment with methanolic 5 % potassium hydroxide at 20° overnight [757]. -Also obtained by treatment of 5,7-dihydroxy-6-(2-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one with 2-methyl-3-buten-2-ol in the presence of boron trifluoride-ether complex [757] in dioxane solution at r.t. for 24 h (2 %) [1081].

Isolation from natural sources

-From kernels of the fruit of *Mammea americana* L. [757] and *Mammea africana* (Guttiferae) [765].

-From the bark of Mammea africana G. Don (Guttiferae) [1082].

yellow needles [757, 762];

m.p. 118–120° [1081], 115–116° [552, 553], 114° [760], 98–100° [757], 97–100° [762];

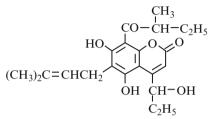
¹H NMR [553, 756, 757, 760, 762], IR [553, 756, 757, 760, 762], UV [552, 553, 756, 757, 760, 762], MS [552, 757, 762]; GC-MS [1082]; TLC [757].

USE: Insecticide [757, 765].

5,7-Dihydroxy-4-[1-(hydroxy)propyl]-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one

(Assamene)

C₂₂H₂₈O₆ mol. wt. 388.46



Isolation from natural sources -From *Kayea assamica* (Guttiferae) [428]. yellow plates [428]; m.p. 135° [428]; ¹H NMR [428], ¹³C NMR [428], IR [428], UV [428], MS [428].

BIOLOGICAL ACTIVITY: Inhibition of cell growth [2264].

5,7-Dihydroxy-4-(1-hydroxypropyl)-8-(3-methyl-2-butenyl)-6-(2-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one

IR [756, 758], UV [756, 758], MS [756, 758].

Triacetate

[38534-70-2]

C₂₈H₃₄O₉ mol.

mol. wt. 514.57

-Obtained by treatment of the titled compound with pyridine-acetic anhydride mixture [758].

IR [756, 758], UV [756, 758], MS [756, 758].

5,7-Dihydroxy-8-(2-hydroxy-3-methylbut-3-enyl)-6-(2-methyl-1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one

(Disparpropylinol B)

C22H28O6 mol. wt. 388.46 Isolation from natural sources -From the fruits and the stem bark of $CH = C = CH_2$ Calophyllum dispar (Clusiaceae) [1178]. CH₂ -Also refer to: [2544]. .0、 HO vellow crystals [1178]; m.p. 111–112° [1178]; ¹H NMR [1178], ¹³C NMR [1178], IR [1178], UV [1178], MS [1178];

 $(\alpha)_{\rm D}^{25} = 0^{\circ}$ (chloroform) [1178].

HO

OH

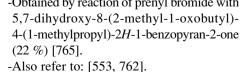
 $CH-C_2H_5$ CH₃

(CH₃)₂C=CHCH₂

BIOLOGICAL ACTIVITY: Human erythroleukemic K562/R7 MDR cells [2544]; Cytotoxicity [1178].

1-[2-[(6-O-D-Apio-β-D-furanosyl-β-D-glucopyranosyl)oxy]-4.6-dihydroxyphenyl]-2-methyl-1-butanone (2S)

[467437-62-3] Isolation from natural sources CH₃ CO-CH-C₂H₅ -From the leaves and branches of *Phyllanthus emblica* (Euphorbiaceae) [3455]. HO $OR = (6-O-D-apio-\beta-D-furanosyl-\beta-D-glucopyranosyl)oxy$ yellow amorphous powder [3455]; ¹H NMR [3455], ¹³C NMR [3455], UV [3455], circular dichroism [3455], MS [3455]; $(\alpha)_{\rm D}^{18} = -60.3^{\circ}$ (methanol) [3455]. 5,7-Dihydroxy-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxobutyl)-4-(1-methylpropyl)-2H-1-benzopyran-2-one (Ferruol A, Mammea D/BB) [92844-55-4] (*R*,*R*) mol. wt. 386.49 [98244-56-6] (R,S) C23H30O5 [16117-33-2] Syntheses -Obtained by reaction of prenyl bromide with $CH - C_2H_5$



C₂H₅-CH-CO

C22H33O13 mol. wt. 505.50 Isolation from natural sources

-From the trunk bark of Mesuua ferrea L. [753, 765, 1150].

m.p. 126.5° [1150], 126–127° [753]; ¹H NMR [753, 764, 765, 1150], IR [753, 764, 1150], UV [753, 764, 1150], MS [753, 764, 1150].

4-[1-(Acetyloxy)propyl]-5,7-dihydroxy-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one

(1^{'''}-Acetoxy-mammea E/BB)

[26477-65-6] C₂₄H₃₀O₇ [111321-12-1] [188817-91-6]



-Obtained by reaction of prenyl bromide with 4-[1-(acetyloxy)propyl]-5,7-dihydroxy-8-(2-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one in the presence of aqueous potassium hydroxide at 0° (40 %) [766].

-Also obtained by acetylation of *Assamene* with acetic anhydride in the presence of pyridine at r.t. overnight (80 %) [428].

-Also refer to: [373, 762, 764, 765, 912, 3381].

Isolation from natural sources

-From Mammea americana [756, 758, 759, 766, 912].

-From the Bark of Mammea africana G. Don (Guttiferae) [553, 759].

-From Mammea siamensis seeds [1828].

-From twigs of Mammea suriga [2554].

semi-solid [2554]; m.p. 114–116° [764], 50–53° [756], 50–51° [758]; **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [428, 756, 758, 759, 764, 912, 1828, 2554], ¹³C NMR [1828, 2554], IR [428, 756, 758, 759, 764], UV [756, 758, 759, 764], MS [428, 756, 758, 759, 764, 2554]; TLC [2554]; HPLC [912, 2554]; (α)_D²⁵ = -28° (ethanol) [428];

BIOLOGICAL ACTIVITY: Insecticidal [553, 758, 759, 766]; Inhibitor of IKBα kinase [2554]; Toxicity [759].

-Also refer to: [912].

 $(CH_3)_2C = CHCH_2$ $(CH_3)_$

mol. wt. 430.50

Isomers of Mammea E/BB $C_{24}H_{30}O_7$ mol. wt. 430.50

(N° 1) Isolation from seeds (Fruit and Spice Park) Mammea americana L (Clusiaceae) [3381].

¹³C NMR [3381]; $(\alpha)_D^{25} = -62^\circ$ (methanol) [3381].

BIOLOGICAL ACTIVITY: Cytotoxicity [3381].

(N°2) [1263908-96-8]; Isolation from stem bark of *Mammea americana* [912].

BIOLOGICAL ACTIVITY: Refer to: [912].

Diacetate $C_{28}H_{34}O_9$ mol. wt. 514.57

-Obtained by treatment of the titled ketone with pyridine-acetic anhydride at 100° temperature overnight [758].

m.p. 20–22° [758]; IR [758], UV [758], MS [758].

Dimethyl ether [26477-66-7] $C_{26}H_{34}O_7$ mol. wt. 458.55

-Refer to: [758, 759].

m.p. 88–90° [758, 759]; IR [758], UV [758], MS [758].

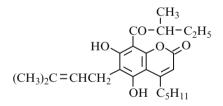
5,7-Dihydroxy-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxobutyl)-4-pentyl-2*H*-1-benzopyran-2-one

(Mammea C/BB)

[5085-55-2]

 $C_{24}H_{32}O_5$

mol. wt. 400.52



Synthesis -Obtained by reaction of prenyl bromide with 5,7-dihydroxy-8-(2-methyl-1-oxobutyl)-4-pentyl-2*H*-1-benzopyran-2-one in the presence of 10 % aqueous potassium hydroxide at 0° (19 %) [762].

Isolation from natural sources

-From kernels of the fruit of *Mammea americana* L. and *Mammea africana* [765] (Guttiferae).

-From only seed of Mammea africana (Guttiferae) [751].

-From the seeds of Mammea americana L. [753, 758, 759].

-From Mammea africana G. Don (Guttiferae) [1082].

needles [751, 753]; m.p. 100–101° [753], 85–86° [762], 81–83° [751]; ¹H NMR [751, 753, 762], IR [751, 753, 762], UV [751, 753, 762, 1150], MS [751, 753, 762]; GC-MS [1082].

USE: Insecticide [751, 753, 758, 759, 765].

5,7-Dihydroxy-8-(3-methyl-2-butenyl)-6-(2-methyl-1-oxobutyl)-4-pentyl-2*H*-1-benzopyran-2-one

 $(Mammea\ C/AB)$

$$[98192-70-2] C_{24}H_{32}O_5 \qquad \text{mol. wt. 400.52}$$

$$\begin{array}{c} CH_2CH=C(CH_3)_2 \\ CH_3 \\ C_2H_5-CH-CO \\ OH \\ C_5H_{11} \end{array} \qquad \begin{array}{c} CH_2CH=C(CH_3)_2 \\ OH \\ C_5H_{11} \\ OH \\ C_5H_{11} \end{array} \qquad \begin{array}{c} Synthesis \\ -Obtained \\ S,7-dihydroxy-6-(2-methyl-1-benzopyran-2-one in the presence of 10 \% aqueous \end{array}$$

potassium hydroxide at 0° (19 %) [762].

Isolation from natural sources

-From kernels of the fruit of *Mammea americana* L. and *Mammea africana* (Guttiferae) [765].

yellow crystals [762]; m.p. 78–80° [762]; ¹H NMR [762], IR [762], UV [762], MS [762].

USE: Insecticide [765].

5-Hydroxy-8-(1-hydroxy-1-methylethyl)-6-(2-methyl-1-oxobutyl)-4-phenyl-2*H*-furo[2',3':5,6]benzo[1,2-b]pyran-2-one (*Ochrocarpin A*)

 $C_{25}H_{24}O_{6}$ mol. wt. 420.46 $HO CH_{3}$ CH_{3} CH_{3} $C_{2}H_{5}-CH-CO$ $C_{1}H_{3}$ $C_{2}H_{5}-CH-CO$ $C_{1}H_{5}$ $C_{1}H_{3}$ $C_{6}H_{5}$ $C_{2}H_{5}-CH-CO$ $C_{1}H_{5}$ $C_{1}H_{5}$

BIOLOGICAL ACTIVITY: Against ovarian cancer cells [616, 1884]; Cytotoxicity [616].

mol. wt. 406.48

5,7-dihydroxy-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one

C25H26O5

(Mammea A/BB)

[6916-62-7] [870084-45-0]

Synthesis -Obtained by reaction of 3-methyl-2-butenyl bromide with 5,7-dihydroxy-8-(2-methylbutyryl)-4-phenylcoumarin in the presence of 10 % potassium hydroxide at 0° under nitrogen over 1.5 h (21 %) [762].

Isolation from natural sources

-From kernels of the fruit of *Mammea americana* L. [754, 758, 759] and *Mammea africana* (Guttiferae) [765].

-From the leaves of Mesua ferrea [2554].

white needles [762]; colourless needles [754]; white solid [2554]; m.p. 124–125° [754, 2554], 111–112° [762]; ¹H NMR [754, 762, 2554], ¹³C NMR [2554], IR [754, 762], UV [754, 762], MS [754, 762, 2554]; TLC [2554]; HPLC [2554].

USE: Insecticide [758, 759, 765].

BIOLOGICAL ACTIVITY: Inhibitor of IKBα kinase [2554].

5,7-dihydroxy-8-(3-methyl-2-butenyl)-6-(2-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one

(Mammea A/AB, MAB 1)

[7058-70-0] C25H26O5 mol. wt. 406.48 $CH_2CH = C(CH_3)_2$ Synthesis -Obtained by reaction of 3-methyl-2-butenyl bromide with 5,7-dihydroxy-6-(2-methylbutyryl)-4-phenylcoumarin in C₂H₅-CH-CO C₆H₅ the presence of 10 % potassium hydroxide at 0° under nitrogen over 1.5 h (21 %) [762].

Isolation from natural sources

-From kernels of the fruit of *Mammea americana* L. [754, 758, 759] and *Mammea africana* (Guttiferae) [765].

-From the bark of *Mammea africana* G. Don (Guttiferae) [553, 1082]. -From the fruits of *Mammea suriga* [2554].

yellow needles [754]; yellow crystalline solid [2554]; m.p. 109–110° [762], 107–108° [754], 106–107° [2554], 105–106° [553]; ¹H NMR [553, 754, 762, 2554], ¹³C NMR [2554], IR [553, 754, 762], UV [553, 754, 762], MS [553, 754, 762, 2554]; GC-MS [1082]; TLC [2554]; HPLC [2554].

USE: Insecticide [758, 759].

BIOLOGICAL ACTIVITY: Inhibitor of IKBα kinase [2554].

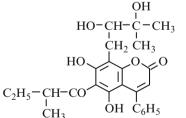
9,10-Dihydro-5,9-dihydroxy-8,8-dimethyl-6-(2-methyl-1-oxobutyl)-4-phenyl-2H,8H-benzo[1,2-b:5,6-b']dipyran-2-one

(Mammea A/AB cyclo E)

5,7-Dihydroxy-8-(2,3-dihydroxy-3-methylbutyl)-6-(2-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one

(Dispardiol B)

C₂₅H₂₈O₇ mol. wt. 440.49



Isolation from natural sources -From the fruits and the stem bark of *Calophyllum dispar* (Clusiaceae) [1178]. ¹H NMR [1178], ¹³C NMR [1178], IR [1178], UV [1178], MS [1178]; $(\alpha)_D^{25} = 0^\circ$ (chloroform) [1178]. 3-[[2,3-Dihydro-4,6-dihydroxy-2-(1-methylethenyl)-5-(2-methyl-1-oxobutyl)-7-benzo-furanyl]methyl]-6-ethyl-4-hydroxy-5-methyl-2H-pyran-2-one

[103834-39-5]

CH₂ CH₃

HO

-OH

CO-CH-C₂H₅

ĊH₃

C25H30O7

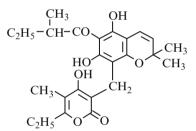
mol. wt. 442.51

Isolation from natural sources -From Helichrysum cephaloudeum and Helichrysum mixtum (Compositae) [1487].

3-[[5,7-Dihydroxy-2,2-dimethyl-6-(2-methyl-1-oxobutyl)-2H-1-benzopyran-8-yl]methyl]-6-ethyl-4-hydroxy-5-methyl-2*H*-pyran-2-one

[103771-76-2]

C25H30O7 mol. wt. 442.51



Isolation from	n natural sources	
-From	Helichrysum	mixtum
(Compositae	e) [1487].	
yellow oil [1	487];	
¹ H NMR [14	87], IR [1487],	
MS [1487].		

1-[4,6-(Diacetyloxy)-2-hydroxy-3-(3,7-dimethyl-2,6-octanediene)phenyl]-2-Methyl-1-butanone

[144785-81-9] C25H34O6 mol. wt. 430.54 Isolation from $\begin{array}{c} CH_3 & CH_3 \\ CH_3 - C = CHCH_2CH_2 - C = CHCH_2. \end{array}$ CH₃ OH CO-CH-C2H5 natural sources -Refer to: [2625]. CH₃COC OCOCH₃

¹H NMR [2625].

 C_2H_5

CH₃

5-Hydroxy-8-(1-methoxy-1-methylethyl)-6-(2-methyl-1-oxobutyl)-4-phenyl-2*H*-furo[2',3':5,6]benzo[1,2-b]pyran-2-one (*Ochrocarpin D*)

 $C_{26}H_{26}O_{6}$ mol. wt. 434.49 $C_{13}O_{CH_{3}}CH_{3}$ Isolation from natural sources -From the bark of *Ochrocarpos punctatus* H. Perrier (Clusiaceae = Guttiferae) [616]. viscous liquid [616]; ¹H NMR [616], ¹³C NMR [616], IR [616], UV [616]; (α)²⁰_D = -0.48° (chloroform) [616].

BIOLOGICAL ACTIVITY: Against ovarian cancer cells [616, 1884]; Cytotoxicity [616].

1-[6-[(3-Acetyl-2,4,6-trihydroxy-5-methylphenyl)methyl]-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-2-methyl-1-butanone

(Mallotophilippen B)

[500109-65-9]

$C_{26}H_{30}O_8$

mol. wt. 470.52

Isolation from natural sources -From *mallotus philippinensis* pericarp (Euphorbiaceae) [793, 1697]. yellow powder [793];

¹H NMR [793], ¹³C NMR [793], IR [793], UV [793], MS [793]; $(\alpha)_D^{23} = 0^\circ$ (methanol) [793].

BIOLOGICAL ACTIVITY: Antiallergic agent [793]; Antiallergic agent for the treatment of asthma, atopic dermatitis, allergic rhinitis, and hay fever [1697].

3-[[2,4-Dihydroxy-6-methoxy-5-(3-methyl-2-butenyl)-3-(2-methyl-1-oxobutyl) phenyl]-methyl]-6-ethyl-4-hydroxy-5-methyl-2H-pyran-2-one

[103771-71-7]	C ₂₆ H ₃₄ O ₇	mol. wt. 458.55
C ₂ H ₅ O O CH ₃ CH	$HO \qquad CO-CH-C_2H_5$ $H_2 \qquad -OH$ $CH_3O \qquad CH_2CH=C(CH_3)_2$	Isolation from natural sources -From <i>Helichrysum cephaloideum</i> (Compositae) [405, 1487]. -From <i>Helichrysum auriceps</i> (Compositae) [405].

¹H NMR [405], IR [405], MS [405].

Triacetate

 $C_{32}H_{40}O_{10}$

mol. wt. 584.66

-Obtained by reaction of acetic anhydride with the title ketone in chloroform in the presence of 4-pyrrolidinopyridine [405].

colourless oil [405]; ¹H NMR [405], MS [405].

1-[(2*R***,3***S***)-3,4-Dihydro-5,7-dihydroxy-2-methyl-3-(3-methyl-2-butenyl)-2-(4-methyl-3-pentenyl)-2***H***-1-benzopyran-8-yl]-2-methyl-1-butanone (***Hypercalyxone B***)**

[658702-61-5] (-)	$C_{26}H_{38}O_4$	mol. wt. 414.58
$HO \xrightarrow[OH]{} CH_{3}$ $HO \xrightarrow[O]{} CH_{2}CH_{2}CH_{2}CH=C(CH_{3})_{2}$ $HO \xrightarrow[OH]{} CH_{2}CH=C(CH_{3})_{2}$	Isolation from natural s -From the aerial part <i>amblycalyx</i> (Guttiferat 4 ₃₎₂ yellow oil [3316];	ts of Hypericum
¹ H NMR [3316], ¹³ C NMR [331	6], UV [3316], MS [3316];	

 $(\alpha)_{\rm D}^{22} = -1^{\circ}$ (methanol) [3316].

BIOLOGICAL ACTIVITY: Antibacterial [3316]; Cytotoxicity against KB cancer cells [3316].

5,7-Dihydroxy-3,6-bis(3-methyl-2-butenyl)-8-(2-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one

[111761-40-1]	$C_{27}H_{36}O_5$	mol. wt. 440.58
HO、 (CH ₃) ₂ C=CHCH ₂	CH_{3} $CO-CH-C_{2}H_{5}$ O $CH_{2}CH=C(CH_{3})_{2}$ $OH C_{3}H_{7}$	Syntheses -Obtained by reaction of prenyl bromide with 5,7-dihydroxy- 8-(2-methyl-1-oxobutyl)-4-pro- pyl-2 <i>H</i> -1-benzopyran-2-one in the presence of 10 % aqueous potassium hydroxide at 0° (1 %) [762, 765].

¹H NMR [762], IR [762], UV [762], MS [762].

6-(3,7-Dimethyl-2,6-octadienyl)-5,7-dihydroxy-8-(2-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one

(Surangin A) (E)

 $[98244-54-3] C_{27}H_{36}O_{5}$ $CH_{3} CH_{3} CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{3}CH$

mol. wt. 440.58

Syntheses -Obtained by reaction of geranyl chloride with 5,7-dihydroxy-8-(2-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one in the presence of 10 % potassium hydroxide for 24 h at 40–50° (7 %) [762, 765].

Isolation from natural sources

-From *Mammea longifolia* (Wight) Planch and Triana (Syn.) *Ochrocarpus longifolius* (Wt.) Benth and Hook. f. ex T. Anders. (Guttiferae) [765, 1548].

colourless needles [1548]; white solid [762]; m.p. 83–85° [1548], 81–82° [762]; ¹H NMR [762, 1548], IR [762, 1548], UV [762, 1548], MS [762, 1548]; $(\alpha)_D^{26} = -1.6^\circ$ (chloroform) [1548].

BIOLOGICAL ACTIVITY: High in vitro antibacterial [1548].

5-[(3,7-Dimethyl-2,6-octadienyl)oxy]-7-hydroxy-8-(2-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one

[98193-78-3]

 $C_{27}H_{36}O_5$

 $\begin{array}{c} CH_{3}\\ CO^{-}CH^{-}C_{2}H_{5}\\ HO \\ CH_{3} \\ CH_{3} \\ CH_{3} \\ CH_{2} \\ CH_{3} \\ CH_{$

Syntheses -Obtained by reaction of geranyl chloride with 5,7-dihydroxy-8-(2-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one in the presence of 10 % potassium hydroxide for 24 h at 40–50° (6 %) [762, 765].

mol. wt. 440.58

white needles [762]; m.p. 54–55° [762]; ¹H NMR [762], IR [762], UV [762], MS [762].

3-[[2,4-Dihydroxy-6-methoxy-5-(3-methyl-2-butenyl)-3-(2-methyl-1-oxobutyl) phenyl]-methyl]-6-ethyl-4-methoxy-5-methyl-2*H*-pyran-2-one

C27H36O7

$$\begin{array}{c} C_{2}H_{5} \\ C_{2}H_{5} \\ CH_{3} \\ CH_{3}O \end{array} \xrightarrow{C} CH_{2} \\ CH_{3}O \\ CH_{3}O \\ CH_{3}O \\ CH_{2}O \\ CH_{2}CH = C(CH_{3})_{2} \end{array}$$

Synthesis

mol. wt. 472.58

-Obtained by reaction of diazomethane with 3-[[2,4-dihydroxy-6-methoxy-5-(3-methyl-2-butenyl)-3-(2-methyl-1-oxobutyl)-phenyl] methyl]-6-ethyl-4-hydroxy-5-methyl-2*H*-pyran-2-one in ethyl ether at r.t. for 1 h [405].

colourless oil [405]; ¹H NMR [405], ¹³C NMR [405], IR [405], MS [405].

Diacetate $C_{31}H_{40}O_9$ mol. wt. 556.65

-Obtained by reaction of acetic anhydride with the title ketone in chloroform in the presence of 4-pyrrolidinopyridine for 3 h [405].

colourless oil [405]; ¹H NMR [405], ¹³C NMR [405], IR [405], MS [405].

CH₂

CO-CH-C₂H₅

CH₃

CH₃

1-[5,7-Dihydroxy-2,2-dimethyl-6-[[2,4,6-trihydroxy-3-methyl-5-(2-methyl-1-oxopropyl) phenyl]methyl]-*2H***-1-benzopyran-8-yl]-2-methyl-1-butanone** (*Mallotophilippen A*)

[500109-64-8]

HO

COCH(CH₃)₂

OHHO.

 $C_{28}H_{34}O_8$

mol. wt. 498.57

Isolation from natural sources -From *mallotus philippinensis* pericarp (Euphorbiaceae) [793, 1697]. yellow powder [793]; ¹H NMR [793],

CH₃ CH₂ CH₂ OH ¹⁴ NMR [13 C NMR [793], IR [793], UV [793], MS [793]; (α)²³₂ = 0° (methanol) [793].

 $(\omega)_{\rm D} = 0$ (incluation) [755].

BIOLOGICAL ACTIVITY: Antiallergic agent [793]; Antiallergic agent for the treatment of asthma, atopic dermatitis, allergic rhinitis, and hay fever [1697].

4-[1-(Acetyloxy)propyl]-6-(3,7-dimethyl-2,6-octadienyl)-5,7-dihydroxy-8-(2-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one

(Surangin B)

[28319-38-2] [98574-77-7] R,R [98574-79-9] R-R,S [98574-80-2] S-R,R [98575-56-5] R,S $C_{29}H_{38}O_7$

mol. wt. 498.62

 $CH_{3} CH_{3} CH_{3} CH_{3} CH_{2} CH_{2}$

sources -From Mammea longifolia (Wight) Planch and Triana (Syn.) Ochrocarpus longifolius (Wt.) Benth and Hook. f. ex T. Anders. (Guttiferae) [758, 759, 766, 1548].

Isolation from natural

-Also refer to: [373, 762, 1150].

m.p. 98–100° [1548]; ¹H NMR [1548], IR [1548], UV [1548], MS [1548]; $(\alpha)_D^{24} = -30^{\circ}$ [1548].

BIOLOGICAL ACTIVITY: Insecticidal [758, 759, 766]; High *in vitro* antibacterial [758, 759, 1548]; Toxic to mosquito larvae [759].

Dimethyl ether $C_{31}H_{42}O_7$ mol. wt. 526.27

-Obtained by reaction of dimethyl sulfate with *Surangin B* in the presence of potassium carbonate in refluxing acetone overnight (71 %) [1548].

gummy solid [1548]; IR [1548], UV [1548], MS [1548].

ĊH₃

3"-Mergtlachyroclinopyrone

$$C_{30}H_{40}O_{7}$$
 mol. wt. 512.64

$$CH_{3}$$

$$CH_{2}$$

$$CH_{2}$$

$$CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{2}CH_{3}$$

$$CH_{3}$$

$$CH_{3}$$

$$CH_{3}$$

$$CH_{3}$$

$$CH_{3}$$

$$CH_{3}$$

$$Isolation from natural sources Isolated as its tetraacetate [406].$$

Tetraacetate

 $C_{38}H_{48}O_{11}$

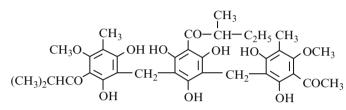
mol. wt. 680.79

colourless gum [406]; ¹H NMR [406], MS [406].

1-[3-[(3-Acetyl-2,6-dihydroxy-4-methoxy-5-methylphenyl)methyl]-5-[[2,6-dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl)phenyl] methyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-butanone (Agrimol D)

C35H42O12

[55576-64-2]



mol. wt. 654.71

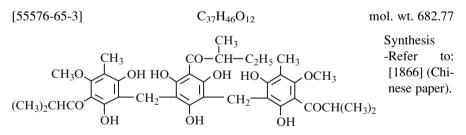
Synthesis -Refer to: [1866] (Chinese paper).

Isolation from natural sources

-From the whole plant *Agrimol pilosa*, ledeb [2860] (Chinese paper). -From the Chinese herb medicine *Agrimonia pilosa* [632] (Chinese paper).

m.p. 147–149° [2860]; ¹H NMR [632], IR [632], UV [632], MS [632].

1-[3,5-Bis[[2,6-dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl)phenyl]methyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-butanone (Agrimol A)



Isolation from natural sources

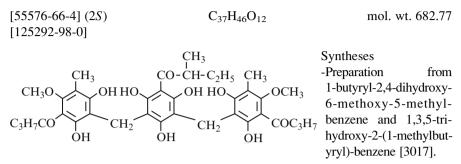
-From the whole plant *Agrimol pilosa*, ledeb [2860] (Chinese paper). -From the Chinese herb medicine *Agrimonia pilosa* [632] (Chinese paper).

m.p. 176–178° [2860]; ¹H NMR [632], IR [632], UV [632], MS [632].

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1-[3,5-Bis[[2,6-dihydroxy-4-methoxy-3-methyl-5-(1-oxobutyl)phenyl]methyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-butanone

(Agrimol B)



-Also refer to: [1866] (Chinese paper).

Isolation from natural sources

-From the Chinese herb medicine Agrimonia pilosa [632, 2860] (Chinese papers).

m.p. 167–169° [2860], 166–168° [1866];

¹H NMR [632], IR [632, 1866], UV [632], MS [632].

BIOLOGICAL ACTIVITY: Inhibited melanin formation in mouse melanoma cells [3017].

2.2 From 2-Ethyl-1-Butanoic Acid

2.2.1 Unsubstituted Hydroxyketones

2-Ethyl-1-(4-hydroxyphenyl)-1-butanone

[100256-47-1]	$C_{12}H_{16}O_2$	mol. wt. 192.26
OH CO-CH-CH ₂ CH ₃	Syntheses -Obtained by treatment of p-(2'-ethylb *with boiling pyridinium chloride [50 *with hydrobromic acid in acetic acid -Also obtained by reaction of diethylac in the presence of boron trifluoride (8	8], (68 %) [2087]; [508]. etic acid with phenol

-Also refer to: [509].

colourless and microcrystalline substance [2087]; b.p.₁ 175° [2087], b.p.₁₀ 204–205° [508, 509]; m.p. 70° [2087]; UV [2087]; $n_D^{23} = 1.5585$ [509].

Methyl ether [84836-32-8] C₁₃H₁₈O₂ mol. wt. 206.28

-Preparation of aryl ketones *via* modified Friedel-Crafts acylation catalyzed by rare metal triflates that generates reduced amounts of toxic by-products [3258].

-Also obtained by Friedel-Crafts reaction of anisole with diethylacetic acid (82 %) [2583].

-Also obtained by reaction of diethylacetyl chloride with anisole in the presence of aluminium chloride in carbon disulfide (70 %) [2087].

-Also obtained by methylation of 2-ethyl-1-(4-hydroxyphenyl)-1-butanone (74 %) [2087].

-Also obtained by ruthenium-catalyzed cross-coupling diethylacetaldehyde with 4-methoxy-phenylboronic acid (84 %) [1864].

-Also refer to: [508, 509, 1606, 3178].

colourless oil [2583]; Pleasantly smelling pale yellow oil [2087]; b.p.₁₁ 154° [2583], b.p.₁₂ 156° [508, 509], b.p.₁₄ 168–170° [3178], b.p.₁₆ 172° [2087]; ¹H NMR [1864, 2583], ¹³C NMR [1864], IR [1864, 2583], UV [2087], MS [1864]; $n_D^{24} = 1.528$ [2087].

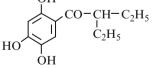
2-Ethyl-1-(2,3,4-trihydroxyphenyl)-1-butanone

 $\begin{array}{c} C_{12}H_{16}O_4 & \text{mol. wt. } 224.26 \\ OH & \\ HO & CO-CH-CH_2CH_3 \\ HO & CH_2CH_3 \end{array} \qquad \begin{array}{c} Syntheses \\ -Preparation \ by \ reaction \ of \ diethylacetic \ acid \\ with \ pyrogallol \ in \ the \ presence \ of \ boron \\ trifluoride \ in \ ethyl \ ether \ at \ 0^{\circ} \ (71 \ \%) \ [540, 2822]. \end{array}$

m.p. 111° [540, 2822], UV [540].

2-Ethyl-1-(2,4,5-trihydroxyphenyl)-1-butanone

[100257-71-4] C₁₂H₁₆O₄ mol. wt. 224.26 OH Synthesis



CH- C_2H_5 -Preparation by reaction of diethylacetic acid chloride with 1,3,4-trihydroxybenzene in the presence of aluminium chloride in nitrobenzene [292]. m.p. 123-124° [292].

USE: Antioxidant for fats, oils and paraffin waxes [292].

2.2.2 Substituted Hydroxyketones

1-(2,3-Dichloro-4-hydroxyphenyl)-2-ethyl-1-butanone

[1539-14-6]	$C_{12}H_{14}Cl_2O_2$	mol. wt. 261.15
-------------	-----------------------	-----------------

OH 	Cl
Ť CO-	-CH-CH ₂ CH
	ĊH ₂ CH ₃

Syntheses

-Obtained by reaction of diethylacetyl chloride with 2,3-dichloroanisole in the presence of aluminium chloride in carbon disulfide at r.t. for 22 h and at 55° for 90 min.
H₃ Then, after carbon disulfide elimination, addition of heptane and to heat on a water bath for 3 h (44 %) [2929].
-Also obtained by treatment of its methyl ether with aluminium chloride (2 equiv.) in refluxing heptane [2060].

-Also refer to: [2047, 2050, 2054–2056, 2058, 2059, 2061, 2767 (44 %), 2768, 3294].

b.p._{0.5} 140–142° [2054, 2767, 2929]; m.p. 85–86° [2047, 2054–2056, 2058–2061, 2767, 2768, 2929, 3294], 84–86° [2050].

Methyl ether $C_{13}H_{16}Cl_2O_2$ mol. wt. 275.17

-Preparation by reaction of 2-ethylbutyryl chloride with 2,3-dichloroanisole [2768] (m.p. 32–33°) in petroleum ether at r.t. [2060].

1-(2-Chloro-4-hydroxyphenyl)-2-ethyl-1-butanone

[4804-56-2]	$C_{12}H_{15}ClO_2$	mol. wt. 226.70
OH Cl CO-CH-CH ₂ CH ₃ CH ₂ CH ₃	Syntheses -To diethylacetyl chloride, 3-chl- disulfide was added in small port chloride; the mixture stirred 1 h at pentane and aluminium chloride 80° [2056]. -Also refer to: [2047, 2057, 2767].	ions at 25° , aluminium t r.t. and 45 min at 55° ;

b.p._{0.3} 148–181° [2047, 2057, 2767].

5-(2-Ethylbutyryl)-2-hydroxybenzoic acid

5-Diethylacetylsalicylic acid

1-(2-Chloro-4-hydroxy-3-methylphenyl)-2-ethyl-1-butanone

[92019-26-6] C13H17ClO2 mol. wt. 240.73

OH CH₃ $CO-CH-C_2H_5$

OH

Syntheses -To diethylacetyl chloride, 3-chloro-2-methylanisole and carbon disulfide was added in small portions at 25°, aluminium chloride; the mixture stirred 1 h at r.t. and 45 min at 55°; pentane and aluminium chloride added, heated 3 h at 80° [2056]. -Also refer to: [2046].

m.p. 87-89° [2046, 2056].

1-(3-Chloro-4-hydroxy-2-methylphenyl)-2-ethyl-1-butanone

[4798-10-1] C13H17ClO2 mol. wt. 240.73 Synthesis OH -Refer to: [2767 (44 %)]. m.p. 87–89° [2767]. CH₃ $CO-CH-C_2H_5$

2-Hydroxy-5-(2-ethylbutyryl)benzamide

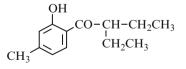
C₁₃H₁₇NO₃ mol. wt. 235.28 Synthesis CONH₂ -Obtained by Fries rearrangement of salicylamide diethylacetate in the presence of aluminium chloride in nitrobenzene for 3 h at 20° [1158].

2-Ethyl-1-(2-hydroxy-4-methylphenyl)-1-butanone

-CH-CH₂CH₃ m.p. 231° [1158].

$$C_{13}H_{18}O_2$$

mol. wt. 206.28



CO-CH-CH₂CH₃ CH₂CH₃ -Obtained by reaction of [Ru₂(p-cymene)₂Cl₂]₂ with bis(acetoxy)iodobenzenze in trifluoroacetic acid and trifluoroacetic aphysicid in a sealed tube (13 %) [2830].

¹H NMR [2830], ¹³C NMR [2830]; MS [2830].

1-(2,6-Dihydroxy-4-methylphenyl)-2-ethyl-1-butanone

$$\begin{array}{c} C_{13}H_{18}O_3 \\ OH \\ CH_3 \\ CH_3 \\ OH \\ CH_3 \\ OH \\ CH_2CH_3 \\ OH \\ CH_2CH_3 \\ OH \\ CH_2CH_3 \\ OH \\ CH_3 \\ CH_3 \\ OH \\ CH_3 \\ OH \\ CH_2CH_3 \\ OH \\ CH_3 \\ OH \\ CH_3 \\ OH \\ CH_3 \\ OH \\ CH_2CH_3 \\ OH \\ CH_3 \\ CH_3 \\ OH \\ CH_3 \\$$

¹H NMR [2830], ¹³C NMR [2830]; MS [2830].

2.3 From 3-Methyl-1-Butanoic Acid

2.3.1 Unsubstituted Hydroxyketones

1-(2-Hydroxyphenyl)-3-methyl-1-butanone

 $[19019-21-7] C_{11}H_{14}O_2 mol. wt. 178.23$

Syntheses -Obtained by Fries rearrangement of phenyl isovalerate in the presence of aluminium chloride, *in nitrobenzene for 18 h at 50° (24 %) [776];

*without solvent for 4 h at $130-140^{\circ}$ [186].

-Also obtained by reaction of isovaleric acid with phenol in the presence of aluminium chloride,

*in petroleum ether, first at r.t. for 12-14 h, then on a water bath for 3-5 h (25 %) [3478];

*in benzene [3477].

-Also obtained by treatment of 1-(2-hydroxyphenyl)-3-methyl-1-butanol with manganese dioxide in methylene chloride for 7 h at r.t. (33 %) [77].

-Also obtained from 2,2-dimethyl-4-chromanone, also named 2,2-dimethyl-4-oxo-4*H*-1-benzopyran (13 %) [118].

oil [118], colourless oil [77]; b.p._{0.1} 76–77° [776], b.p.₂₀ 138–140° [186], b.p. 248–250° [3477, 3478]; ¹H NMR [77, 118], ¹³C NMR [77], IR [77, 118], MS [77]; $n_D^{20} = 1.5295$ [118].

BIOLOGICAL ACTIVITY: Antifeedant against storage pests and aphids [1076].

Semicarbazone

C₁₂H₁₇N₃O₂ mo

mol. wt. 235.29

m.p. 195° [812], 173° [3477].

p-Nitrophenylhydrazon	e ($C_{17}H_{19}N_{2}$	3O3	mol. wt. 313.36
m.p. 121–122° [186].				
Methyl ether [8	57803-59-9]	C	$_{2}H_{16}O_{2}$	mol. wt. 192.26
-Refer to: [186, 1084].				
b.p. ₁₂ 142–144° [186] ¹ H NMR [1231], ¹³ C	; NMR [1231], IR	. [1231],	MS [1231].	
Acetate	C ₁₃ H ₁₆ O	3		mol. wt. 220.27
b.p. ₂₀ 164–166° [186]				
2,4-Dinitrophenylhydra	zone [93650-	62-5]	$C_{17}H_{18}N_4O_5$	mol. wt. 358.35
m.p. 187–189° [423],	181° [1523].			
1-(3-Hydroxyphenyl)-3-	methyl-1-butar	one		
	$C_{11}H_{14}O_2$			mol. wt. 178.23
OH COCH ₂ CH(CH ₃) ₂	with m-aceto	xybenzo	yl chloride (70	hylbutyl)cadmium) %), then saponi- 0 %) [2586].
b.p. ₂ 136–138° [2586]	; m.p. 83° [25	86].		
Acetate b.p. _{2.5} 76–77° [2586].	C ₁₃ H ₁₆ O	3		mol. wt. 220.27
Methyl ether [1 -Refer to: [624].	183770-52-6]		₁₂ H ₁₆ O ₂	mol. wt. 192.26
¹ H NMR [624, 1231],	¹³ C NMR [624	, 1231], 1	IR [624, 1231]	, MS [624, 1231].
2,4-Dinitrophenylhydra m.p. 171° [2586].	zone	C ₁₇ H ₁₈	N ₄ O ₅	mol. wt. 358.35

1-(4-Hydroxyphenyl)-3-methyl-1-butanone

[34887-83-7] C11H14O2 mol. wt. 178.23



Syntheses -Preparation by Fries rearrangement of phenyl isovalerate in the presence of aluminium chloride, *in nitrobenzene for 18 h at 50° (68 %) [776]; $COCH_2CH(CH_3)_2$ *without solvent at 130–140° for 4 h [186].

-Also obtained by Fries rearrangement of phenylisovalerate in the presence of BF_3 -H₂O in a closed pressure tube at 80° for 1 h (87 %) [2514].

-Also obtained by reaction of isovaleric acid with phenol,

*in the presence of boron trifluoride for 2 h at 70° (52 %) [1685];

*in the presence of aluminium chloride in petroleum ether, first at r.t. for 12–14 h, then on a water bath for 3-5 h (38 %) [3478].

-Also obtained by dealkylation of its ethyl ether with aluminium chloride in carbon disulfide for 8 h at $60-70^{\circ}$ [191].

- -Also obtained by treatment of its methyl ether with pyridinium chloride at 190° [627].
- -Also obtained by reaction of 4-iodophenol with iso-Bu₃In in the presence of $(PPPh_3)_4$ in THF at 66° under atmospheric pressure of CO gas (61 %) [1851]. -Also refer to: [884, 1414, 3477 (63 %)].

b.p._{0.1} 145–147° [812], b.p.₃ 201–202° [1685]; m.p. 97–98° [191], 96–97° [3477, 3478], 95.5–96.5° [186], 95–96° [776, 1685];

¹H NMR [881, 2514], ¹³C NMR [2514], MS [2514].

[82938-20-3] $C_{12}H_{16}O_{2}$ Methyl ether mol. wt. 192.26

-Obtained by reaction of isovaleric acid/isovaleric anhydride mixture with anisole in the presence of perchloric acid (73 %) [1781].

- -Also obtained by reaction of isovaleric acid with anisole in the presence of PPA at 80° (76–100 %) [627].
- -Also obtained by reaction of isovaleric chloride with anisole in the presence of aluminium chloride,

*in methylene chloride at 0° (76–100 %) [627];

- *in 1,2-dichloroethane first at 0° , then at r.t. for 8–15 h (84 %) [2942].
- -Also obtained by direct acylation of 4-bromoanisole with isovaleraldehyde by palladium catalysis (75 %) [2668].
- -Also obtained by reaction of 1,3-dimethyl-1-phenyl-3-buten-1-ol with 4-methoxybenzaldehyde in the presence of Ru/CeO_2 as catalyst in mesitylene at 170° for 24 h under argon atmosphere (93 %) [2101].
- -Synthesis and *Rhizopus oryzae* mediated enantioselective hydrolysis of α -acetoxy derivative [846].
- -Also obtained from 4-methoxybenzaldehyde (77 %) [3024].

mol. wt. 282.34

-Also refer to: [698 (55 %), 1114].

pale yellow oil [2942]; b.p.₁₂ 145–159° (bath temp.) [1781]. b.p.₁₂ 158.5–159.5° [3178], b.p.₂₅ 167° [251]; ¹H NMR [698, 2942, 3024], ¹³C NMR [698, 2942], IR [698], MS [698, 2942].

Ethyl ether [92035-99-9]
$$C_{13}H_{18}O_2$$
 mol. wt. 206.28

-Obtained by reaction of isovaleryl chloride with phenetole in the presence of, *aluminium chloride in carbon disulfide at $60-70^{\circ}$ for 8 h [191];

*of zinc chloride for 4 h at 70° (68 %) [1780].

-Also obtained by reaction of isovaleric acid with phenetole in the presence of aluminium chloride, in petroleum ether, first at r.t. for 12–14 h, then on a water bath for 3–5 h (82 %) [3477, 3478].

-Also refer to: [964].

b.p.₁₈ 133–137° [3477, 3478], b.p.₂₀ 136–138° [1780]; $n_D^{15} = 1.5332$ [3478], $n_D^{20} = 1.5338$ [1780].

Semicarbazone of the ethyl ether	$C_{14}H_{21}N_3O_2$	mol. wt. 263.34
m.p. 191–192° [3477].		
Oxime of the ethyl ether	$C_{13}H_{19}NO_2$	mol. wt. 221.30

C₁₈H₁₈O₃

Benzoate

m.p. 75–76° [3477, 3478].

1-(2,3-Dihydroxyphenyl)-3-methyl-1-butanone

[104216-24-2]	$C_{11}H_{14}O_3$	mol. wt. 194.23

	OH
HO	COCH ₂ CH(CH ₃) ₂
	L I
	\sim

Syntheses ² -Obtained by treatment of guaiacol isovalerate with aluminium chloride in carbon disulfide at 90° for 50 min, then at 135–140° for 2 h after solvent elimination [2075].

-Also obtained by treatment of its dimethyl ether below with hydriodic acid (d = 1.71) in refluxing acetic acid for 6 h (88 %) [198].

m.p. 93–95° [2075], 48° [198]; **N.B.**: One of the reported melting point is obviously wrong. IR [198].

Dimethyl ether [15121-98-9] C₁₃H₁₈O₃ mol. wt. 222.28

-Preparation by action of isobutylmagnesium iodide with 2,3-dimethoxybenzaldehyde (73 %) [198].

-Also obtained by treatment of 1-(2,3-dimethoxyphenyl)-3-methyl-1-butanol with potassium dichromate in dilute sulfuric acid (59 %) [198].

-Obtained by oxidation of 1-(2,3-dimethoxyphenyl)-1-(3-methylpropyl) carbinol with sodium dichromate in dilute sulfuric acid (75–80 %) [2747].

pale yellow oil [198, 2747]; b.p._{0.2} 103–105° [2747], b.p._{0.5} 118–120° [198], b.p.₃ 130° [198]; ¹H NMR [1231], ¹³C NMR [1231], IR [198, 1231], MS [1231].

2,4-Dinitrophenylhydrazone of the dimethyl ether

[15116-03-7] C₁₉H₂₂N₄O₆ mol. wt. 402.41 m.p. 225–226° [2747].

1-(2,4-Dihydroxyphenyl)-3-methyl-1-butanone

[15116-14-0]	$C_{11}H_{14}O_3$	mol. wt. 194.23
HO	resorcinol,	of isovaleric acid with on trifluoride for 2 h at 70°

*in the presence of zinc chloride at 150° [2517], (70–78 %) [2747], (68–78 %) [2501], for 2 h at 125–136° [893].

-Also obtained by reaction of iso-amyl chloride with resorcinol at 85–90° for 20– 30 min (85.3 %) [731].

-Also refer to: [773, 1300, 2685].

yellow viscous oil [2747]; b.p.₁ 145–152° [2747], b.p._{6–7} 183–185° [893]; m.p. 108–110° [893, 2245], 73° [2312]; ¹H NMR [829].

BIOLOGICAL ACTIVITY: Antitumor [1300]; For the prevention and treatment of bone and cartilage diseases [2685].

USE: Spectrophotometric determination of uranium [3213].

Oxime	[159457-03-1]	$C_{11}H_{15}NO_3$	mol. wt. 209.25
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-For preparation of transition metal complexes [1506].

m.p. 203–205° [3375], 205–207° [2267].

Diacetate [251463-54-4] C₁₅H₁₈O₅ mol. wt. 278.30

-Obtained by reaction of acetic anhydride with the title ketone in the presence of pyridine (>80 %) [2517].

oil [2517]; ¹H NMR [2517], ¹³C NMR [2517], IR [2517], UV [2517], MS [2517].

Dimethyl ether [54874-25-8] C₁₃H₁₈O₃ mol. wt. 222.28

-Obtained by reaction of dimethyl sulfate with 2,4-dihydroxyisovalerophenone in the presence of potassium carbonate in refluxing acetone for 4 h [1788].

-Also obtained by hydrogenation of 2',4'-dimethoxydehydroisovalerophenone (b.p._{3–4} 152–153°) in the presence of 10 % Pd/C in ethanol in an hydrogen atmosphere (96 %) [1613].

-Also obtained by reaction of isovaleric acid with 1,3-dimethoxyphenyl in the presence of a mixture of phosphorous pentoxide and orthophosphoric acid at 90° for 1 h (73 %) [1613].

b.p.₂ 120–125° [1613]; ¹H NMR [1613], IR [1613].

2,4-Dinitrophenylhydrazone [94711-66-7] C₁₇H₁₈N₄O₆ mol. wt. 374.35

m.p. 234° [2245].

1-(2,5-Dihydroxyphenyl)-3-methyl-1-butanone

(2-Isovaleroylhydroquinone)

[124557-52-4]	$C_{11}H_{14}O_3$	mol. wt. 194.23
OH	Syntheses	
COCH ₂ CH(CH ₃) ₂	-Obtained by reaction of isova	aleric acid with hydroqui-
	none in the presence of boror	h trifluoride for 2 h at 125°
\mathbf{i}	(71 %) [2312].	
ÓН	-Also obtained by reaction of	f isovaleryl chloride with

-Also obtained by reaction of isovaleryl chloride with hydroquinone in the presence of aluminium chloride in nitrobenzene for 3 h in a water bath [1442].

-Also obtained by Fries rearrangement of hydroquinone diisovalerate in the presence of hydroquinone and aluminium chloride at 150–160° for 1 h (77 %) [770]. -Also obtained by Fries rearrangement of hydroquinone diisovalerate in the presence of aluminium chloride at 150–155° (oil bath) and maintained at that temperature till no more hydrogen chloride was evolved (1 h) (50 %) [613].

-Also obtained by hydrogenation of 1-(2,5-dihydroxyphenyl)-3-methyl-2-buten-1-one in the presence of 10 % Pd/C in ethanol at r.t. [2540].

-Also obtained by Fries rearrangement of 4-methoxyphenyl isovalerate with aluminium chloride (5 part)/sodium chloride (2 part) mixture at 180–200° (30 %) [1796].

-Also refer to: [1629, 1703, 1794, 2882].

yellow plates [613]; m.p. 115° [1703], 114–115° [1442], 111° [2312], 110° [770, 1796, 2540], 106–108° [613]; ¹H NMR [613], IR [613, 2540], UV [613, 2540, 2882].

USE: As antioxidant for vitamin A [1629].

Dimethyl ether	[124557-51-3]	$C_{13}H_{18}O_{3}$	mol. wt. 222.28
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-Obtained by treatment of 2-hydroxy-5-methoxy isovalerophenone with dimethyl sulfate in the presence of 10 % a queous sodium hydroxide in refluxing acetone for 20 min [770].

-Also obtained by Friedel-Crafts reaction of isovaleryl chloride with hydroquinone dimethyl ether [2540].

colourless oil [770]; b.p.₁ 124–126° [770].

1-(2,6-Dihydroxyphenyl)-3-methyl-1-butanone

[13936-90-8]	$C_{11}H_{14}$	D_3	mol. wt. 194.23
OH COCH ₂ CH(CH ₃)	-	8]. 52], 67–68° [2631].	
Dimethyl ether	[52856-20-9]	$C_{13}H_{18}O_{3}$	mol. wt. 222.28

-Obtained by condensation of 2,6-dimethoxyphenyllithium with ethyl isovalerate (42 %) [1858].

b.p._{0.5} 116° [1858].

2,4-Dinitrophenylhydrazone of the dimethyl ether

[52856-31-2]	$C_{19}H_{22}N_4O_6$	mol. wt. 402.41

m.p. 147.2–148° [1858].

1-(3,4-Dihydroxyphenyl)-3-methyl-1-butanone

(L-158,870) [2904]

[67239-25-2]	$C_{11}H_{14}O_3$	mol. wt. 194.23
OH OH	Syntheses -Obtained by Fries rearrangement diisovalerate with aluminium chloride,	of pyrocatechol
COCH ₂ CH(CH ₃) ₂	 *in nitrobenzene for 1 h at 80° (40 %) [2 *in the presence of pyrocatechol for (69 %) [2075]. 	

-Also refer to: [885, 899, 2127, 2904].

b.p.₄ 200–210° [2075]; m.p. 108° [2646], 106.5–107.5° [2075].

BIOLOGICAL ACTIVITY: Central nervous system depressant [2127]; Agonist specific for the modified β -₂AR [2904]; Antimelanoma and skin depigmentation, *in vitro* method for screening [612].

Dimethyl ether [132858-47-0] C₁₃H₁₈O₃ mol. wt. 222.28

-Also obtained by reaction of isovaleric acid with veratrole in the presence of polyphosphoric acid for 2.5 h at 60° (91.6 %) [1364].

pale yellow oil [1364]; b.p._{0.37} 134.5° [1364]; ¹H NMR [1231], ¹³C NMR [1231], IR [1231], MS [1231].

Oxime of the dimethyl ether $C_{13}H_{19}NO_3$ mol. wt. 237.30

oil [1364].

colourless short thick prisms [1364]; m.p. 183.4–185.7° [1364].

1-(3,5-Dihydroxyphenyl)-3-methyl-1-butanone

[104216-80-0]	$C_{11}H_{14}O_3$	mol. wt. 194.23
HO COCH ₂ CH(CH ₃) ₂	sodium hydroxide a	of its diacetate with 5 % t reflux for 4–5 h
Monohydrate	$C_{11}H_{14}O_3, H_2O$	mol. wt. 212.24
m.p. 44° [1406].	[101502.65.1] C. H.	N.O. mol. wt. 274.25

2,4-Dinitrophenylhydrazone [101593-65-1] $C_{17}H_{18}N_4O_6$ mol. wt. 374.35 m.p. 204° (d) [1406].

Diacetate [108715-26-0] C₁₅H₁₈O₅ mol. wt. 278.30

-Preparation by reaction of diisobutylcadmium with 3,5-diacetoxybenzoyl chloride in refluxing benzene for 1 h (62 %) [1406].

b.p._{0.5} 178–182° [1406].

2,4-Dinitrophenylhydrazone of the diacetate

[102661-34-7]	$C_{21}H_{22}N_4O_8$		mol. wt. 458.43
m.p. 160° [1406].			
Dimethyl ether	C ₁₃	H ₁₈ O ₃	mol. wt. 222.28
-Obtained by reaction 3,5-dimethoxybenzonitrile (17		isobutylmagnesium 2].	bromide with
m.p. 143–145° [1212].			
Semicarbazone of the dimeth	yl ether	$C_{14}H_{21}N_{3}O_{3}$	mol. wt. 279.34
m.p. 195–196° [1212].			
3-Methyl-1-[2,3,4-trihydroxy]	phenyl]-1	l-butanone	
[757408-19-8]	$C_{11}H$	$H_{14}O_4$	mol. wt. 210.23
HO HO HO	gallol i *of bor (72 %)	es ed by reaction of isoval n the presence, on trifluoride in ethyl [538, 540]; c chloride (Nencki reac	ether at 0° for 1 h
-Also refer to: [1054, 1260, 334	49].		
b.p. _{0.01} 138–140° [811];			

m.p. 109° [538, 540], 106–110° [811]; UV [540].

3-Methyl-1-[2,4,5-trihydroxyphenyl]-1-butanone

[79744-63-1]	$C_{11}H_{14}O_{2}$	1	mol. wt. 210.23
HO OH COCH ₂ C		508]. 508], ¹³ C NMR [1508].
Trimethyl ether	[98230_17_2]	CuHarOu	mol wt 25231

Trimethyl ether[98230-17-2] $C_{14}H_{20}O_4$ mol. wt. 252.31m.p. 76.5° [1406];UV [1406].

2,4-Dinitrophenylhydrazone of the trimethyl ether

[102458-53-7]	$C_{20}H_{24}N_4O_7$	mol. wt. 432.43
m.p. 132° [1406].		

3-Methyl-1-(2,4,6-trihydroxyphenyl)-1-butanone

[26103-97-9] $C_{11}H_{14}O_4$ mol. wt. 210.23 OH Syntheses -Preparation by reaction of isovaleryl chloride with phloroglucinol in the presence of aluminium chloride [(88.5 %) 491, (60 %) 621, 2534, 3415],

*in nitrobenzene [334, (60–70 %) 421, (45 %) 898, (30–38 %) 1665, (25 %) 2615, (47 %) 2618, 2646];

*in a methylene chloride/nitromethane mixture [2580];

*in a carbon disulfide/nitrobenzene mixture [(35–38 %) 1066, (46 %) 2113, (47 %) 2620];

*without solvent at 50° (70 %) [335].

-Also obtained by reaction of isovaleronitrile with phloroglucinol (Hoesch reaction) [1608, 1665, (23 %) 2926].

-Also obtained by reaction of isovaleric acid with phloroglucinol in the presence of boron trifluoride etherate [3019, 3020] at 100° for 2 h (27 %) [338].

-Also obtained by acid hydrolysis of its 2- β -D-glucopyranoside in methanol in the presence of 1 N HCl at reflux for 4 h [209].

-Also obtained by acid hydrolysis of its 2,4-di-β-D-glucopyranoside [1089].

-Also obtained by adding phloroglucinol to a solution of phosphorous oxychloride plus aluminium chloride and stirred under nitrogen. 3-Methylbutanoic acid was added and the reaction stirred under nitrogen at 0° for 8 h, then at 6° for 40 h (40–54 %) [3201].

-Also obtained from valerophenone synthase (VPS) which uses isovaleryl-CoA to synthetize phloroisovalerophenone [3482].

-Also refer to: [5, 337, 542, 763, 1254, 1292, 1374, 1983, 2405, 2610, 2614, 2616, 2685, 2686, 2771, 3033, 3120, 3202, 3310, 3481].

Isolation from natural sources

-From hop plant, *Humulos lupulus* (Cannabinaceae) [1066, 2389]. -From hop bittering [2846].

yellow crystals [1066]; b.p._{0.01} 180–190° [2926]; m.p. 176–178° [1608], 146° [2113], 145° [421, 1374, 1665, 2615, 2618, 2620, 2646, 2771], 144–145° [1066, 2534, 2926], 143–145° [209], 142° [898], 141–143° [541]; ¹H NMR [209, 421, 541, 898, 1066, 2534, 3019], ¹³C NMR [3019], IR [421, 898, 3019], UV [898, 1066, 3019], MS [421, 1066, 2531, 2534, 2846]; GLC [2531]; TLC [1066]; HPLC [1066, 3481]. USE: As bone metabolism improving agent [1042]; Antagonist both thromboxane A_2 and Leukotriene D_4 [3019]; Effects on transpiration and stomatal closure [3408]; In lupulone preparation [3120].

BIOLOGICAL ACTIVITY: Antimicrobial activity against *Bacillus subtilis* [3020]; Antimicrobial activity of, for *Staphylococcus aureus* [3372]; Germination inhibition [421]; Inhibitory effects of treatment with this acylphenol on TPA Induced EBV-EA Activation [3033]; Antifungal [2113]; As bone resorption inhibitor for treatment of bone diseases [2686]; For the prevention and treatment of bone and cartilage diseases [2685].

Monohydrate	$C_{11}H_{14}O_4, H_2O$	mol. wt. 228.25

m.p. 95° [1608].

Trimethyl ether	[101032-04-6]	$C_{14}H_{20}O_4$	mol. wt. 252.31
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-Obtained by reaction of isovaleryl chloride with phloroglucinol trimethyl ether in the presence of aluminium chloride [1818] (88 %), in methylene chloride, *at 0° for 3.5 h (81 %) [3071, 3072];

*at -10 to -5° over 2 h, then at 0° for 30 min (88 %) [1819].

oil [1819]; ¹H NMR [1819].

2,4-Dinitrophenylhydrazone $C_{17}H_{18}N_4O_7$ mol. wt. 390.35

m.p. 196° [1665].

3-Methyl-1-(3,4,5-trihydroxyphenyl)-1-butanone

[216300-88-8]	$C_{11}H_{14}O_4$	mol. wt. 210.23
HO HO COCH ₂ CH(CH ₃) ₂	Synthesis -Refer to: [2684]. BIOLOGICAL ACTIVITY: Fo treatment of bone and cartilage d	-
Trimethyl ether	$C_{14}H_{20}O_4$	mol. wt. 252.31

-Obtained by reaction of 3,4,5-trimethoxybenzonitrile with isobutylmagnesium bromide in toluene at 40° for 5 h (42 %) [1407] or at reflux for 3 h (26 %) [1212].

b.p.₁ 147–150° [1212], b.p.₆ 164–166° [1407]; m.p. 37–39° [1212, 1407].

Semicarbazone of the trimethyl ether	$C_{15}H_{23}N_3O_4$	mol. wt. 309.37

m.p. 205° [1212].

1-(3,4-Dihydroxyphenyl)-3-hydroxy-3-methyl-1-butanone

	$C_{11}H_{14}O_4$	mol. wt. 210.23
ŌН	Synthesis	
ОН	-Obtained by thermal Fries rearranger	ment of pyrocatechol
	di-3-methyl-2-butenoate (13 %) [150	05].
Ý	pale yellow needles [1505];	
$COCH_2 - C - (CH_3)_2$	m.p. 132–133° [1505];	
Ь́Н	¹ H NMR [1505], IR [1505], UV [150	5].

1-(2,3,4,6-Tetrahydroxyphenyl)-3-methyl-1-butanone

[100059-66-3]	$C_{11}H_{14}O_5$			mol. wt. 226.23	
OH	Syntheses				
COCH ₂ CH(CH ₃) ₂		2		demethylation	
	•	•	•	isovalerophenone	
НО ОН				chlorobenzene at	
OH	80–85° for 5 h	(83 %)	[2610].		

-Also obtained by reaction of isovaleryl chloride with 1,2,3,5-tetrahydroxybenzene in the presence of aluminium chloride in nitrobenzene first at 0° , then at r.t. for 3 days (38 %) [2307].

m.p. 178–179° [2610], 167–168° [2307]; sublimation _{0.2} 130–140° [2610]; ¹H NMR [2307], UV [2307], MS [2307, 2683].

N.B.: Stability [2610, 2616].

1-(3,4-Dihydroxyphenyl)-2-ethyl-3-methyl-1-butanone

[67114-29-8]	$C_{13}H_{18}O_3$	mol. wt. 222.28
OH OH CO-CH-CH(CH ₃) ₂	Synthesis -Obtained by treatment of its pyridinium chloride under nitroge (30 %) [2127]. white crystals [2127]; b.p. _{0.025} 13 m.p. 100–101.5° [2127].	en at 200–220° for 1 h

BIOLOGICAL ACTIVITY: Central nervous system depressant [2127].

Dimethyl ether [92730-14-8] C₁₅H₂₂O₃ mol. wt. 250.34

-Obtained by reaction of 2-ethyl-3-methylbutyryl chloride with veratrole in the presence of aluminium chloride in refluxing benzene for 30 min (39 %) [2127].

colourless liquid [2127]; b.p._{0.025} 117° [2127]; $n_D^{25} = 1.5329$ [2127].

BIOLOGICAL ACTIVITY: Central nervous system depressant [2127].

2.3.2 Substituted Hydroxyketones

1-(2,3-Dichloro-4-hydroxyphenyl)-3-methyl-1-butanone

m.p. 112-114° [2048, 2766], 110-112° [2047, 2056, 2057, 2767].

Methyl ether [53107-50-9] C₁₂H₁₄Cl₂O₂ mol. wt. 261.15

-Preparation by reaction of isovaleryl chloride with 2,3-dichloroanisole in the presence of aluminium chloride (72 %) [3333]. -Also refer to: [732, 734, 736, 737, 2052].

m.p. 54–55° [3333], 49–54° [732, 734, 736, 737, 2052].

1-(3,5-Dichloro-2,4-dihydroxyphenyl)-3-methyl-1-butanone

C	$L_{11}H_{12}Cl_2O_3$	mol. wt. 263.21
CI COCH ₂ CH(CH ₃	Synthesis) ₂ -Refer to: [1052].	
но	BIOLOGICAL ACTIVITY	Y: Bactericide [1052].

1-(4-Fluoro-2-hydroxy-5-nitrophenyl)-3-methyl-1-butanone

[120292-07-1] $C_{11}H_{12}FNO_4$ mol. wt. 241.22 OH Synthesis F COCH₂CH(CH₃)₂ -Refer to: [958].

1-(3-Bromo-2,4-dihydroxyphenyl)-3-methyl-1-butanone

 $\begin{array}{cccc} [664376-91-4] & C_{11}H_{13}BrO_3 & \text{mol. wt. 273.13} \\ & OH & Syntheses \\ Br & COCH_2CH(CH_3)_2 & -Refer to: [2485, 2486]. \\ & HO \end{array}$

1-(5-Chloro-2-hydroxyphenyl)-3-methyl-1-butanone

	$C_{11}H_{13}ClO_2$	mol. wt. 212.68
OH	Synthesis	
COCH ₂ CH(CH ₃) ₂	-Obtained by Fries rearra	angement of 4-chlorophenyl
	isovalerate with a	luminium chloride in
γ	tetrachloroethane at 150-	160° for 2 h [3170].
Cl	b.p. _{0.20-0.25} 98-98.5° [317	0]; UV [3170].

1-(5-Chloro-2,4-dihydroxyphenyl)-3-methyl-1-butanone

[216300-97-9]	$C_{11}H_{13}ClO_3$	mol. wt. 228.68		
OH COCH ₂ CH(CH ₃) ₂	Synthesis -Refer to: [2684].			
HO CI	BIOLOGICAL ACTIVITY: For and treatment of bone diseases [2684].	r the prevention and cartilage		

1-(3-Fluoro-4-hydroxyphenyl)-3-methyl-1-butanone

$C_{11}H_{13}FO_2$		mol. wt. 196.22	
F COCH ₂	chlori		ement with aluminium de [922].
Methyl ether	[4374-24-7]	$C_{12}H_{15}FO_2$	mol. wt. 210.25

b.p._{0.09} 90° [671].

1-(2,4-Dihydroxy-3-iodophenyl)-3-methyl-1-butanone

[1204737-61-0]	$C_{11}H_{13}IO_3$	mol. wt. 320.13
OH I COCH ₂ CH(CH ₃) ₂ HO	Synthesis -Obtained by action of iodina ethanol at 20° (93 %) [1672] MS [1672].	

3-Methyl-1-(2,4,6-trihydroxy-3-nitrophenyl)-1-butanone

[119691-94-0]	$C_{11}H_{13}NO_6$	mol. wt. 255.23
NO ₂ HO OH COCH ₂ CH(CH ₃) ₂ OH	Synthesis -Obtained by adding hexane, of concentrated sulfuric acc nitric acid at 0° to 2,4,6-trihydroxyisovaleropheno trated sulfuric acid below 0° (7)	a solution of in concen-
1 1 1 11 11 12 12 14 14	04 060 [2414]	

bright yellow needles [3414]; m.p. 94–96° [3414]; ¹H NMR [3414], IR [3414], MS [3414].

BIOLOGICAL ACTIVITY: Germination inhibition [3414];

1-(3-Amino-5-bromo-2,4-dihydroxyphenyl)-3-methyl-1-butanone

[909255-16-9]	$C_{11}H_{14}BrNO_3$	mol. wt. 288.14
OH NH ₂ HO Br	Synthesis -Obtained by reduction of the 3-nitro derivative by zinc an methanol at 20° (73 %) [3350 m.p. 119–121° [3350]; ¹ H NI ¹³ C NMR [3350].	nd acetic acid in].

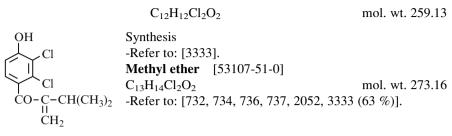
1-(2-Amino-5-hydroxyphenyl)-3-methyl-1-butanone

[404918-99-6]	$C_{11}H_{15}NO_2$	mol. wt. 193.25
NH ₂ COCH ₂ CH(CH ₃) ₂	Synthesis -Refer to: [3331].	
OH	USE: Preparation of indazoles to that of a thyroid hormone duction thereof, and their use i	and method for the pro-

1-(3-Amino-2,4-dihydroxyphenyl)-3-methyl-1-butanone

[909255-15-8]	$C_{11}H_{15}NO_3$	mol. wt. 209.25
NH ₂ CO-CH ₂ -CH(CH ₃)	Synthesis -Obtained by reduction the 3-nitro derivative by in methanol at 20° (75 m.p. 117–119° [3350]; ¹³ C NMR [3350].	y zinc and acetic acid %) [3350].

1-(2,3-Dichloro-4-hydroxyphenyl)-3-methyl-2-methylene-1-butanone



m.p. 56–58° [3333], 46–51° [732, 734, 736, 737, 2052].

2-Hydroxy-5-(3-methylbutyryl)benzoic acid

	C ₁₂ H ₁₄	$_{4}O_{4}$		mol. wt. 2	22.24
$\bigcup_{COCH_2CH(CH_3)_2}^{OH}$	Synthesis -Obtained 5-isovaleryls m.p. 178° [11	•	alkaline ide [1158].	hydrolysis	of

1-(2-Chloro-6-hydroxy-4-methoxyphenyl)-3-methyl-1-butanone

[112954-17-3]	$C_{12}H_{15}ClO_3$	mol. wt. 242.70
CH ₃ O ^{OH} COCH ₂ CH(CH ₃) ₂ CH ₃ O ^{Cl}	Syntheses -Obtained by Frieder isovaleryl chloride 5-methoxyphenol in the chloride [1695, 1696].	with 3-chloro-

1-(5-Chloro-2-hydroxy-4-methoxyphenyl)-3-methyl-1-butanone

[136741-47-4]	$C_{12}H_{15}ClO_3$		n	nol. wt. 242	.70
OH	Synthesis		с с	.1. 1.1.1.	. 1
COCH ₂ CH(CH ₃) ₂	with 4-chloro	•		•	
CH ₃ O	presence o		•	•	in
Ċl	1,2-dichloroe	thane (9	2 %) [2718	3].	

1,2-dichloroethane (92 %) [2718]. m.p. 81–82° [2718].

2-Hydroxy-5-(3-methylbutyryl)benzamide

$$\begin{array}{ccc} C_{12}H_{15}NO_3 & \text{mol. wt. } 221.26 \\ OH & Synthesis \\ \hline & CONH_2 & -Obtained by Fries rearrangement of salicylamide isovalerate in the presence of aluminium chloride in nitrobenzene for 3 h at 20° [1158]. \\ \hline & COCH_2CH(CH_3)_2 & \text{m.p. } 182^\circ [1158]. \end{array}$$

1-(2-Hydroxy-3-methylphenyl)-3-methyl-1-butanone

[105337-84-6]	$C_{12}H_{16}O_2$		mol. wt. 192.26
CH ₃ COCH ₂ CH(CH ₃) ₂	Syntheses -Obtained by isovalerate wit at 160–180° (2 -Also refer to: [-
b.p. ₄ 127° [2390], b.p. ₈ 143° [2390].	137° [2390], b.	.p. _{11.5} 140–146°	[1644], b.p. ₁₁
Phenylhydrazone m.p. 114–115° [2390].	$C_{18}H_{22}N_2O$		mol. wt. 282.39
2,4-Dinitrophenylhydrazone	[109252-17-7]	$C_{18}H_{20}N_4O_5$	mol. wt. 372.38

m.p. 191-192° [2390].

1-(2-Hydroxy-4-methylphenyl)-3-methyl-1-butanone

[105337-38-0]	$C_{12}H_{16}O_2$	mol. wt. 192.26
CH ₃ OH COCH ₂ CH(CH ₃) ₂	-	of isovaleric acid with ce of boron trifluoride for 5].

-Also obtained by Fries rearrangement of m-cresyl isovalerate in the presence of aluminium chloride,

*without solvent for 30 min at 120–148° (73 %) [1644] or at 140–150° [906];

*first in refluxing carbon disulfide for 2 h, then at $145-150^{\circ}$ for 4 h after solvent elimination, (95 %) [3063].

-Also obtained from 2,2,7-trimethyl-4-chromanone, also named 2,2,7-trimethyl-4-oxo-4*H*-1-benzopyran (4 %) [118, 3063].

b.p._{1.5} 95–97° [906], b.p._{11.5} 137–142° [1644], b.p.₂₁ 151–152° [3063], b.p.₁₈ 152–156° [1685]; ¹H NMR [118, 3063], IR [118, 3063]; $n_D^{20} = 1.5295$ [118]. $\label{eq:2.39} {\mbox{Phenylhydrazone}} \ \ \, [101784-96-7] \ \ \, C_{18}H_{22}N_2O \ \ \, \mbox{mol. wt. } 282.39$

m.p. 107–110° [1644].

Methyl ether	[71898-88-9]	$C_{13}H_{18}O_2$	mol. wt. 206.28
wieuryr euler	[/1090-00-9]	$C_{13}\Pi_{18}O_2$	11101. wt. 200.

-Obtained by treatment of 2-hydroxy-4-methylisovalerophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 10 h (96 %) [3063].

b.p.₁₀ 146° [3063]; ¹H NMR [3063], IR [3063].

1-(2-Hydroxy-5-methylphenyl)-3-methyl-1-butanone

 $\begin{array}{c} [55813-81-5] \\ OH \\ COCH_2CH(CH_3)_2 \\ CH_3 \end{array} \begin{array}{c} C_{12}H_{16}O_2 \\ Syntheses \\ -Obtained by Fries rearrangement of p-cresyl isovalerate in the presence of aluminium chloride for 45 min at 130–160° (67 %) [1644]. \\ -Also obtained by treatment of its methyl ether with boiling pyridinium chloride [515, 2089]. \end{array}$

-Also obtained by reaction of aluminium chloride with its methyl ether in boiling carbon disulfide [185].

b.p._{0.1} 85° [640], b.p.₁₂ 138–140° [1644], b.p.₂₁ 151° [185]; ¹H NMR [640], IR [640], UV [640]; $n_D^{18.4} = 1.5685$ [185].

2,4-Dinitrophenylhydrazone	$C_{18}H_{20}N_4O_5$	mol. wt. 372.38
m.p. 174–175.5° [1644].		
4-Nitrophenylhydrazone	$C_{18}H_{21}N_3O_3$	mol. wt. 327.38

Methyl ether	$C_{13}H_{18}O_2$	mol. wt. 206.28
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-Preparation by reaction of isovaleryl chloride with 4-methylanisole in the presence of aluminium chloride in carbon disulfide for 24 h at r.t. [515, (50 %) 2089].

pale yellow oil; b.p.₁₇ 157–158° [515, 2089]; $n_D^{20} = 1.5200$ [515, 2089].

1-(4-Hydroxy-2-methylphenyl)-3-methyl-1-butanone

C₁₂H₁₆O₂ mol. wt. 192.26

OH	Synthesis
\checkmark	-Obtained by reaction of aluminium chloride with
	4-hydroxy-2-methyl-5-isopropylisovalerophenone in chlo-
CH ₃	robenzene, first 20 h at r.t., then for 4 h at 50° (53 %) [1523].
COCH ₂ CH(CH ₃) ₂	b.p. _{0.0008} 115–120° [1523]; m.p. 51° [1523].

1-(4-Hydroxy-3-methylphenyl)-3-methyl-1-butanone

[105337-39-1]	$C_{12}H_{16}O_2$	mol. wt. 192.26
OH CH3 COCH2CH(CH3)2	Syntheses -Obtained by treatment of p-isovalerocar ium chloride in chlorobenzene, first at 50° for 4 h (71 %) [1522, 1523]. -Also obtained by Fries rearrangement of in the presence of aluminium chlorid 30 min (18 %) [1644].	r.t. for 20 h, then at fo-cresyl isovalerate
b.p. 10 5 184–188°	[1644], b.p., 188–190° [2390], b.p., 219	-220° [2390]:

b.p._{10.5} 184–188° [1644], b.p.₂ 188–190° [2390], b.p.₁₈ 219–220° [2390]; m.p. 83° [1522, 1523], 76–77° [2390]; IR [2777].

Methyl ether [1096879-14-9] C₁₃H₁₈O₂ mol. wt. 206.29

¹H NMR [1231], ¹³C NMR [1231], IR [1231], MS [1231].

2,4-Dinitrophenylhydrazone [109248-77-3] C₁₈H₂₀N₄O₅ mol. wt. 372.38 m.p. 200–201° [2390].

1-(2,4-Dihydroxy-3-methylphenyl)-3-methyl-1-butanone

[664376-65-2]	$C_{12}H_{16}O_3$		mol. wt	. 208.26
CH ₃ HO	3-(bromome 4-mercaptop carbonate in	(by-product) (thyl)-benzoate, pyridine in the pr acetone at 45 y LiAlH ₄ in	°, then the	product

-Also obtained by reaction of isovaleroyl chloride with 2-methylresorcinol in the presence of aluminium chloride in methylene chloride first at 0° , then at r.t. for 16 h (57 %) [2486].

-Also refer to: [773].

white solid [2486].

1-(2,4-Dihydroxy-6-methylphenyl)-3-methyl-1-butanone

[198879-07-1]	$C_{12}H_{16}O_3$	mol. wt. 208.26
OH COCH ₂ CH(CH ₃) ₂	Syntheses -Refer to: [2684, 2685].	
HO CH ₃	BIOLOGICAL ACTIVITY: For and treatment of bone and ca [2684, 2685].	-

1-(2,6-Dihydroxy-4-methylphenyl)-3-methyl-1-butanone

[216300-92-4]	$C_{12}H_{16}O_3$	mol. wt. 208.26
OH COCH ₂ CH(CH ₃) ₂	Synthesis -Refer to: [2684].	
сн3 ОН	BIOLOGICAL ACTIVITY: For and treatment of bone diseases [2684].	-

1-(2-Hydroxy-3-methoxyphenyl)-3-methyl-1-butanone

[15116-06-0]	$C_{12}H_{16}O_3$	mol. wt. 208.26
CH ₃ O CH ₃ O COCH ₂ CH(CH ₃) ₂	Syntheses -Obtained by partial demen ether with aluminium (50–60 %) [2747]. -Also refer to: [1681].	thylation of its methyl chloride in toluene

oil [2747]; b.p._{0.2} 97–101° [2747].

1-(2-Hydroxy-4-methoxyphenyl)-3-methyl-1-butanone

$[193687-88-6]$ $C_{12}H_{16}O_3$	mol. wt. 208.26
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CH₃O

COCH₂CH(CH₃)₂ -Obtained by reaction of isovaleryl chloride with resorgined dimethed in the of aluminium chloride in refluxing carbon disulfide (60 %) [1788].

-Preparation by partial methylation of 2,4-dihydroxyisovalerophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 4-6 h (85-90 %) [2501].

-Also obtained from 2,2-dimethyl-7-methoxy-4-chromanone, also named 2,2-dimethyl-7-methoxy-4-oxo-4H-1-benzopyran (31 %) [118].

-Also obtained by treatment of 2-(*t*-butyldimethylsilyloxy)-2'-isopropyl-4-methoxyacetophenone (m.p. 110–112°) with $(n-Bu)_4NF$ in THF at 0° for 45 min (82 %) [1467].

oil [118]; b.p.₄ 165° [1788]; m.p. 125–127° [1467]; ¹H NMR [118, 1467], ¹³C NMR [1467], IR [118, 1467], MS [1467]; $n_D^{20} = 1.5475$ [118].

Phenylhydrazone

C₁₈H₂₂N₂O₂ mol. wt. 298.38

mol. wt. 265.31

m.p. 85° [1788].

1-(2-Hydroxy-5-methoxyphenyl)-3-methyl-1-butanone

[876511-19-2]	$C_{12}H_{16}O_3$	mol. wt. 208.26
OH COCH ₂ CH(CH ₃) ₂ OCH ₃	Syntheses -Obtained by reaction of isova dimethyl ether in the presend [770] in carbon disulfide [16 -Also obtained by hydroge 5-methoxyphenyl)-3-methyl- presence of 10 % Pd/C in eth	ce of aluminium chloride 1]. nation of 1-(2-hydroxy- 2-buten-1-one in the
-Also refer to: [458]		
oil [2540]; b.p. ₁₅ 155	–163° [161], b.p. ₁₈ 177–178° [458].
Na salt	C ₁₂ H ₁₅ O ₃ Na	mol. wt. 230.24
-Refer to: [770].		
yellow needles [770].		

Semicarbazone

m.p. 171° [770, 2540].

1-(3-Hydroxy-4-methoxyphenyl)-3-methyl-1-butanone

[99783-85-4]	$C_{12}H_{16}O_3$	mol. wt. 208.26
CH ₃ O COCH ₂ CH(CH ₃) ₂	refluxing with 30 % s	3-methylbutanoate by sodium hydroxide for 2 h
	1	

 $C_{13}H_{19}N_3O_3$

m.p. 51–53° [2779], 45–48° [144]; ¹H NMR [144].

1-(2,4-Dihydroxy-6-methoxyphenyl)-3-methyl-1-butanone

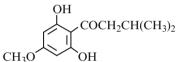
mol. wt. 224.26 $C_{12}H_{16}O_{4}$ **Synthesis** $COCH_2CH(CH_3)_2$ -Refer to: [2356]. m.p. 132° [2356]; UV [2356].

1-(2,6-Dihydroxy-3-methoxyphenyl)-3-methyl-1-butanone

C12H16O4 mol. wt. 224.26 Synthesis .COCH₂CH(CH₃)₂ -Refer to: [829]. ¹H NMR [829].

1-(2,6-Dihydroxy-4-methoxyphenyl)-3-methyl-1-butanone

[91555-33-8] C12H16O4 mol. wt. 224.26



Syntheses $COCH_2CH(CH_3)_2$ -Refer to: [829, 2356]. Isolation from natural sources -Refer to: [2707].

-Gradient-enhanced nuclear Overhauser effect spectroscopy (GOESY) in structure elucidation of plant secondary metabolites [2707].

m.p. 96–97° [2356]: ¹H NMR [829], UV [2356].

3-Methyl-1-(2,4,6-trihydroxyphenyl)-1-butanone

[49583-27-9]	$C_{12}H_{16}O_4$	mol. wt. 224.26
OH CH ₃ COCH ₂ CH(CH ₃) ₂ HO OH	Syntheses -Obtained by reaction of isoval 2,4,6-trihydroxytoluene [3176] -Also obtained by treatment 3-methyl-4,6-bis(1-methylethoxy 1-butanone with titanium tetrau lene chloride at r.t. for 48 h (8)	according to [641]. of 1-[2-hydroxy-)phenyl]-3-methyl- chloride in methy-

-Also refer to: [361, 769, 1917, 2435, 3405, 3437 (23 %)].

red crystals [642]; m.p. 160–161° [1917], 154–155° [642], 148° [3437]; ¹H NMR [642, 3437], ¹³C NMR [642], IR [642, 3437], UV [642], MS [642, 1917, 3437]; GLC [2531].

BIOLOGICAL ACTIVITY: As photosynthetic electron transport (PET) [3405].

CH₃O

Trimethyl ether [149053-77-0] C₁₅H₂₂O₄ mol. wt. 266.34

-Refer to: [2435].

Isolation from natural sources

-From volatile leaf oils of some south-western and southern Australian species of the genus Eucalyptus: Subgenus Symphyomyrtus, section Bisectaria, series Macrocarpae [361].

¹H NMR [2435]; GC/MS [361].

1-[2-(Acetyloxy)-4-hydroxyphenyl]-3-methyl-1-butanone

[251463-59-9]

 $C_{13}H_{16}O_4$

mol. wt. 236.27

OH OCOCH₃ COCH₂CH(CH₃)₂

Synthesis -Obtained by selective deacetylation of 2,4-diacetoxyphenyl isobutyl ketone mediated by porcine pancreatic lipase (PPL) in THF at 42–45° for 48 h in the presence of n-butanol (45 %) [2517].

oil [2517]; ¹H NMR [2517], ¹³C NMR [2517], IR [2517], UV [2517], MS [2517].

Synthesis

5-Hydroxy-6-(2-methyl-1-oxobutyl)-1,4-benzodioxane

C₁₃H₁₆O₄ mol. wt. 236.27

(CH₃)₂CHCH₂CO OH

-Obtained by Fries rearrangement of 5-isovaleryloxy-1,4-benzodioxane in the presence of aluminium chloride in nitrobenzene at 20° (62 %) [801].

b.p._{0.1} 162–164° [801]; m.p. 59–60° [801]; UV [801].

1-[2-(Acetyloxy)-4,6-dihydroxyphenyl]-3-methyl-1-butanone

[62545-33-9]	$C_{13}H_{16}O_5$		mol. wt. 252	2.27
HO HO OCOCH ₃	Synthesis -Obtained phloroisobut			of

1-[4-(Acetyloxy)-2,6-dihydroxyphenyl]-3-methyl-1-butanone

2,6-Dihydroxy-3-isovaleryl-4-methoxybenzoic acid

$$\begin{array}{cccc} C_{13}H_{16}O_6 & \text{mol. wt. } 268.27 \\ OH & Synthesis \\ (CH_3)_2CHCH_2CO & CO_2H & -Refer to: [493]. \\ CH_3O & OH & C_{14}H_{18}O_6 & \text{mol. wt. } 282.29 \end{array}$$

-Obtained by reaction of isovaleryl chloride with methyl 2,6-dihydroxy-4-methoxybenzoate in the presence of aluminium chloride in nitrobenzene at 0° , then 3 days at r.t. (80 %) [493].

m.p. 102–104° [493]; IR [493].

1-(2-Chloro-4-hydroxyphenyl)-2-ethyl-3-methyl-1-butanone

$$[5590-61-4] C_{13}H_{17}ClO_2 mol. wt. 240.73$$

$$C_1 C_2H_5 Synthesis -Obtained by condensation of 2-ethyl-3-methylbutyryl chloride on chloro-3-anisole [2767]. m.p. 75-77° [2047, 2057, 2767].$$

1-(3-Chloro-4-hydroxyphenyl)-2-ethyl-3-methyl-1-butanone

 $Cl \qquad CO-CH-CH(CH_3)_2$

Synthesis -To 2-isopropylbutyryl chloride, 2-chloroanisole and carbon disulfide was added in small portions at 25°, aluminium chloride; the mixture stirred 1 h at r.t. and 45 min at 55°; pentane and aluminium chloride added, heated 3 h at 80° [2056].

m.p. 75–76° [2056].

1-(5-Ethyl-2-hydroxyphenyl)-3-methyl-1-butanone

 $\begin{array}{cccc} [77346-69-1] & C_{13}H_{18}O_2 & \mbox{mol. wt. } 206.28 \\ OH & Syntheses \\ -Preparation by Fries rearrangement of 4-ethylphenyl \\ 3-methylbutanoate in the presence of aluminium chloride for 2 h at 140° (94 %) [403]. \\ C_2H_5 & -Also refer to: [2220]. \\ b.p._{0.3} 96-98° [403]; \ ^1H NMR [403], IR [403]. \end{array}$

Isovalerate [77346-70-4] C₁₈H₂₆O₃ mol. wt. 290.40

-Obtained by reaction of isovaleryl chloride with 5-ethyl-2-hydroxyisovalerophenone in the presence of pyridine in chloroform at r.t. (96 %) [403].

b.p.₃ 180–185° [403]; ¹H NMR [403], IR [403].

Methyl ether [72247-02-0] C₁₄H₂₀O₂ mol. wt. 220.31

-Obtained by reaction of isovaleric acid with p-ethylanisole in the presence of PPA at 80° for 2 h (87 %) [2220, 2701].

b.p.₄ 126–127° [2220]; ¹H NMR [2220], IR [2220], UV [2220].

1-(2,4-Dihydroxy-3,5-dimethylphenyl)-3-methyl-1-butanone

CH₂

HO

 CH_3

 $\begin{array}{c} C_{13}H_{18}O_3 & \mbox{mol. wt. } 222.28 \\ \mbox{COCH}_2CH(CH_3)_2 & \begin{subarray}{c} Synthesis \\ -Refer to: [829]. \\ \end{subarray}^1 H NMR [829]. \end{array}$

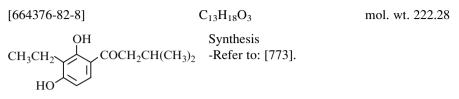
1-(2,5-Dihydroxy-3,4-dimethylphenyl)-3-methyl-1-butanone

mol. wt. 222.28

 $\begin{array}{c|c} OH & Synthesis \\ CH_3 & \bigcirc & COCH_2CH(CH_3)_2 \\ CH_2 & & m.p. \ 138^{\circ} \ [2538]; \\ \end{array} UV \ [2538]. \end{array}$

C₁₃H₁₈O₃

1-(3-Ethyl-2,4-dihydroxyphenyl)-3-methyl-1-butanone



1-[5-(1-Hydroxyethyl)-2-hydroxyphenyl]-3-methyl-1-butanone

	$C_{13}H_{18}O_3$		mol. wt. 222.28
OH COCH ₂ CH(C	572 E	natural sources Brachyclados	megalanthus
$(\alpha)_{\rm D}^{24} = -4^{\circ}$ (chlo	s [3444]; m.p. 150° proform) [3444]; IR [3444], MS [3444		
2-Methyl ether	[51995-88-1]	$C_{14}H_{20}O_3$	mol. wt. 236.31

-Obtained by heating its isovalerianate with methanolic 2 N potassium hydroxide for 1 h at 65° (90 %) [399].

Isolation from natural sources

-From Verbesina luetzelburgii (Compositae) [407].

colourless oil [399]; ¹H NMR [399], IR [399].

Isovalerianate of the 2-methyl ether $C_{14}H_{20}O_3$ mol. wt. 236.31

-Refer to: [399].

1-(2,6-Dihydroxy-4-methoxy-3-methylphenyl)-3-methyl-1-butanone (*Aspidinol C*)

[1257216-80-0]	$C_{13}H_{18}O_4$	mol. wt. 238.28
CH ₃ OH CH ₃ OCOCH ₂ CH(CH ₃) ₂ OH	Synthesis -Refer to: [3268]. m.p. 155–158° [3268]; NMR [3268], IR [3268],	

1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)-3-methyl-1-butanone

[55382-31-5]	$C_{13}H_{18}O_4$	mol. wt. 238.28
HO COCH ₂ CH(CH ₃) ₂ HO CH ₃	female flowers of <i>Hag</i> Gmelin [1916] (IVb); *of "kosin" (IV), from treatment with 15 % po	Alkaline cleavage, d protokosin (III) from <i>genia abyssinica</i> (Bruce) <i>Flos koso</i> "Siegfried" by potassium hydroxide in the er on a water bath for 24 h
m.p. 59–61° [1915].		

N.B.: "Kosin" (IV), others names: Methylene-bis-pseudo-aspidinol; pseudoaspidin. [1911].

m.p. 148–150° [1915]; ¹H NMR [1915], IR [1915], UV [1915], MS [1915].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-3-methyl-1-butanone

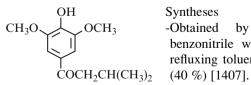
[68754-16-5]	$C_{13}H_{18}O_4$	mol. wt. 238.28
CH ₃ O ^{OH} COCH ₂ CH(CH ₃) ₂ OCH ₃ O ^{OCH} 3	Syntheses -Preparation by reaction of with 3,5-dimethoxyphenol is boron trifluoride etherate (98 %) [643].	in the presence of

-Also refer to: [176, 177, 3085, 3086].

m.p. 50–50.5° [1005], 48° [2356], 32° [643]; ¹H NMR [643], ¹³C NMR [643], IR [643, 1005], UV [643, 1005, 2356], MS [643].

1-(4-Hydroxy-3,5-dimethoxyphenyl)-3-methyl-1-butanone

[873380-96-2] $C_{13}H_{18}O_4$ mol. wt. 238.28



Syntheses OCH₃ -Obtained by reaction of 3,4,5-trimethoxy-benzonitrile with isobutyImagnesium bromide in refluxing toluene for 3 h (25 %) [1212] or for 5 h refluxing toluene for 3 h (25 %) [1212] or for 5 h

-Also obtained by treatment of its methyl ether with concentrated sulfuric acid at 35–40° for 20 h (85 %) [1407].

m.p. 94° [1212], 93–93.5° [1407].

mol. wt. 253.30

mol. wt. 295.34

Oxime

m.p. 110° [1212].

Semicarbazone

m.p. 162.5° [1212].

Benzoate $C_{20}H_{22}O_5$ mol. wt. 342.39

C14H21N3O4

C13H19NO4

-Obtained by reaction of benzoyl chloride with the title ketone in the presence of pyridine in boiling benzene for 1 h (70 %) [1212].

m.p. 111° [1212].

3-Methyl-1-(2,4,6-trihydroxy-3,5-dimethylphenyl)-1-butanone

(Kunzeaphlogin D aglycone)

C₁₃H₁₈O₄ mol. wt. 238.28

Syntheses

ŎН	Syntheses
CH ₃ COCH ₂ CH(CH ₃) ₂	-Obtained by
	kosotoxin (II
но Он	flowers of H
ĊH ₃	[1916] (XIX

Obtained by reductive alkaline cleavage of kosotoxin (**II**) and protokosin (**III**) from female flowers of *Hagenia abyssinica* (Bruce) Gmelin [1916] (**XIXb**).

-Also obtained by reaction of isovaleric acid with 2,4-dimethylphloroglucinol in the presence of boron trifluoride in carbon tetrachloride during 3 h on the steam bath (51 %) [440].

-Also obtained from *Kunzeaphlogin* D, isolated from the leaves of *Kunzea ambigua* (SM) Bruce (Myrtaceae) [1612].

-Also refer to: [1838].

brown oil [1612]; ¹H NMR [1612, 1838].

BIOLOGICAL ACTIVITY: Refer to: [1838].

Trimethyl ether [3567-96-2] $C_{16}H_{24}O_4$ mol. wt. 280.36 (*Torquatone*)

Synthesis

-Obtained by treatment of triphenol above with excess dimethyl sulfate in the presence of potassium carbonate in acetone solution [440].

Isolation from natural sources

-From volatile leaf oils of,

*Eucalyptus incrassata Labill. [367] (1.56 %), [1106];

*Eucalyptus angulosa Schau [367] (1.37 %), [1106];

*Eucalyptus ceratocorys (Blakely) L. A. S. Johnson & K. D. Hill [367, 1106];

*Eucalyptus erythrandra Blakely & Steedm. [367, 1106];

*Eucalyptus flocktoniae Maiden [440, 1106]; *Eucalyptus tetraptera Turcz. [367, 1106]; *Eucalyptus stoatei Gardner [367, 1106]; *Eucalyptus brachycalyx Blakely [360]; Maiden [1106]; *Eucalyptus rugosa R.Br. ex Blakely [360, 1106]; *Eucalyptus griffithsii Maiden [360, 1106]; *Eucalyptus concinna Maiden & Blakely [360, 1106]; *Eucalyptus corrugata Luehm. [360, 1106]; *Eucalyptus torquata Luehm. [360 (40.91 %), 439, 1106, 2037]; *Eucalyptus pimpiniana Maiden [360, 1106]; *Eucalyptus caesia Benth. [439, 1106]; *Eucalyptus calygona Turcz. [461 (3.6 %), 1106]; *Eucalyptus celastroides Turcz. ssp. celastroides [461 (4.2 %), 1106]; *Eucalvptus clelandii (Maiden) Maiden [461 (7.2 %), 1106]: *Eucalyptus salubris var. glauca Maiden [461 (10.2 %), 1106]; *Eucalyptus stricklandii Maiden [461 (32.9 %), 1106]; *Eucalyptus woodwardii Maiden [461 (6.2 %), 1106]; *Eucalyptus macrocarpa subsp. macrocarpa Hopper [361 (6.68 %)]; *Eucalyptus carnabyi Blakely & Steedm. ex. Blakely [361 (1.96 %)]; *Eucalyptus rhodantha Blakely & Steedm. [361 (0.84 %)]; *Eucalyptus pachyphylla F. Muell. [361 (0.98 %)]; *Eucalyptus oxymitra Blakely [361 (0.75 %)]; *Eucalyptus spathulata Hook ssp. grandiflora Benth [440, 1106]; *Eucalyptus chartaboma (chemotype II) (3 %) and Eucalyptus miniata (1.6 %) (Myrtaceae) [1453];

- *Eucalyptus torquata folage [439, 1838];
- *Eucalyptus leaf essential oils in relation to taxonomy [924].
- -From essential oil of Eucalyptus apodophylla (Myrtaceae) [2046].
- -From genus Angophora taxa (Myrtaceae) [923],
- *on volatile leaf oils of 8 south-western and southern Australian species of the genus *Eucalyptus* [357, 358, 365, 925].
- -Also refer to: [362-364, 1107].
 - colourless crystalline solid [439]; cubes [1107]; m.p. 40–41° [439], 38–39° [440]; ¹H NMR [1107, 1453, 2046], ¹³C NMR [1107, 1453, 2046], IR [439, 440, 461, 2046], UV [439], MS [361, 461, 1453, 2046]; X-ray crystal data [1107]; GC [357, 358, 361, 461, 925]; GC-MS [357, 358].
- **N.B.**: The formula mentioned in the paper [439] (page 444), 3-methyl-1-(2,4,6-trimethoxy-3,5-dimethylphenyl)-**2**-butanone was erroneous.

BIOLOGICAL ACTIVITY: Antiherbivore Chemistry of Eucalyptus-Cues and Deterrents for Marsupial Folivores [2138]; Inhibition of drug metabolism enzymes [1154].

-Also refer to: [1838].

2,4-Dinitrophenylhydrazone of the trimethyl ether

[111979-31-8] C22H28N4O7 mol. wt. 460.49

m.p. 204–205° [440], 202° [439].

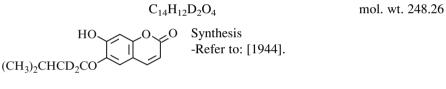
1-(2,4-Dihydroxy-3,6-dimethoxyphenyl)-3-methyl-1-butanone

(3,6-Dimethoxy-resisovalerophenone)

C13H18O5 [106276-13-5] mol. wt. 254.28 Synthesis OCH₃ COCH₂CH(CH₃)₂ -Obtained by reaction of isovaleryl chloride with 2,5-dimethoxyresorcinol (XI) (88 %, m.p. HO OH $86-88^{\circ}$) in the presence of aluminium chloride ÓCH₃ in a carbon disulfide/nitrobenzene mixture [2610].

m.p. 93.5° [2610]; sublimation 0.1 85° [2610].

7-Hydroxy-6-(2,2-dideuterio-3-methyl-1-oxobutyl)-2H-1-benzopyran-2-one (Dideuterogeijerin)



Methyl ether [38409-24-4] $C_{15}H_{14}D_2O_4$ mol. wt. 262.27

-Refer to: [1944].

MS [1944].

4-Hydroxy-3-(3-methyl 1-oxobutyl)-2H-1-benzopyran-2-one

[4139-75-7]	$C_{14}H_{14}O_4$	mol. wt. 246.26
	Syntheses -Obtained by reaction	of isovalervl chloride with

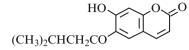
Obtained by reaction of isovaleryl chloride with COCH₂CH(CH₃)₂ 4-hydroxycoumarin in pyridine containing one drop of piperidine for 3 h on a water bath (33 %) [3174].

-Also refer to: [525, 3144].

m.p. 75-76° [3174].

7-Hydroxy-6-(3-methyl-1-oxobutyl)-2H-1-benzopyran-2-one

 $[109966\text{-}52\text{-}1] \qquad \qquad C_{14}H_{14}O_4 \qquad \qquad \text{mol. wt. } 246.26$



Syntheses -Obtained by Fries rearrangement of 7-iso-valeroxycoumarin with aluminium chloride in nitrobenzene first at $120-130^{\circ}$ overnight. The next day, the mixture was heated at $160-170^{\circ}$ for 1 h [2823].

-Also refer to: [886].

m.p. 168-170° and 146-148° (dimorphic forms) [2823].

Methyl ether	[450-16-8]	$C_{15}H_{16}O_4$	mol. wt. 260.29
(Geijerin)			

-Obtained by dehydrogenation of 3,4-dihydro-7-methoxy-6-(3-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one in the presence of Pd/C in refluxing a diphenyl oxide/1-dodecene (5 equiv.) mixture (66 %) [529].

-Also obtained by cyclization of methyl 3-[2-hydroxy-4-methoxy-5-(3-methyl-1-oxobutyl)-phenyl]propanoate in the presence of Pd/C in refluxing diphenyl oxide (66 %) [529].

-Also obtained by treatment of 6-isovaleryl-7-hydroxycoumarin with methyl iodide in the presence of potassium carbonate in refluxing acetone for 6 h (89.9 %) [2823].

-Also refer to: [530, 886, 1814, 1944].

Isolation from natural sources

-From the bark of Geijera salicifolia Schott (Rutaceae) [529, (0.6 %) 1814].

flat colourless prisms [1814]; colourless needles [2823]; m.p. 121–123° [2823], 121° [1814]; ¹H NMR [529, 530], IR [530], UV [530], MS [530, 1944].

2,4-Dinitrophenylhydrazone of the methyl ether

[16850-97-8]	$C_{21}H_{20}N_4O_7$	mol. wt. 440.41

m.p. 187° [2823], 181° [1814].

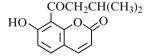
7-Hydroxy-8-(3-methyl-1-oxobutyl)-2H-1-benzopyran-2-one

Synthesis

[109966-03-2]

 $C_{14}H_{14}O_4$

mol. wt. 246.26



-Obtained by Fries rearrangement of 7-isovaleroxycoumarin with aluminium chloride in nitrobenzene for 1 h at 150° (18 %) [2823].

m.p. 103–105° [2823].

Methyl ether [109068-12-4] $C_{15}H_{16}O_{4}$ mol. wt. 260.29 (Isogeijerin)

-Obtained by treatment of 8-isovaleryl-7-hydroxycoumarin with methyl iodide in the presence of potassium carbonate in refluxing acetone for 6 h [2823].

m.p. 94–96° [2823].

2,4-Dinitrophenylhydrazone $C_{21}H_{20}N_4O_7$ mol. wt. 440.41 of the methyl ether

m.p. 194–195° [2823].

1-(2,4-Dihydroxy-3-quinolinyl)-3-methyl-1-butanone

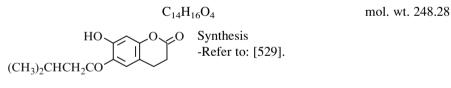
юH OH

Synthesis -Obtained by reaction of isovaleryl chloride -Obtained by reaction of isovaleryl chloride COCH₂CH(CH₃)₂ with 2,4-dihydroxyquinoline in the presence of aluminium chloride in nitrobenzene on the steam bath for 3 h (26 %) [3123].

m.p. 195–198° [3123]; UV [3123].

BIOLOGICAL ACTIVITY: Antibacterial properties (Staphylococcus aureus and Escherichia coli) [3123].

3,4-Dihydro-7-hydroxy-6-(3-methyl-1-oxobutyl)-2H-1-benzopyran-2-one



Methyl ether [117285-87-7] $C_{15}H_{18}O_{4}$ mol. wt. 262.31

-Obtained by cyclization of methyl 3-[2-hydroxy-4-methoxy-5-(3-methyl-1-oxobutyl)phenyl]-propanoate in refluxing diphenyl oxide (90 %) [529]. -Also refer to: [530, 1814].

m.p. 86.5-87.5° [530], 84° [1814]; ¹H NMR [530], IR [530], UV [530], MS [530].

2,4-Dihydroxy-5-isovalerylcinnamic acid

$$\begin{array}{ccc} & C_{14}H_{16}O_5 & \text{mol. wt. } 264.28 \\ & OH & Synthesis \\ & CH=CHCO_2H & -Refer to: [2823]. \\ & Dimethyl \ ether & [101430-14-2] \\ & C_{16}H_{20}O_5 & \text{mol. wt. } 292.33 \\ & Methylgeijerinic \ acid \end{array}$$

-Obtained by heating 6-isovaleryl-7-methoxycoumarin with dimethyl sulfate in aqueous alkali (20 per cent) on a steam bath for 4 h [2823]. -Also refer to: [1814].

colourless needles [2823]; m.p. 158° [1814], 157–158° [2823].

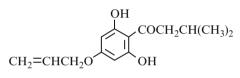
1-[2,6-Dihydroxy-4-(2-propen-1-yloxy)phenyl]-3-methyl-1-butanone

[918814-68-3]

 $C_{14}H_{18}O_4$

mol. wt. 250.29

mol. wt. 254.76



COCH₂CH(CH₃)₂ -Obtained by Friedel-Crafts acylation of phloroglucinol monoallyl ether with isopentanoyl chloride in the presence of titanium tetrachloride (20 %) [337].

light brown viscous oil [337]; ¹H NMR [337], IR [337], MS [337].

BIOLOGICAL ACTIVITY: Antiprotozoal and antimicrobial [337].

1-(3-Chloro-4-hydroxyphenyl)-3-methyl-2-(1-methylethyl)-1-butanone

	$C_{14}H$	$_{19}CIO_2$
	CH(CH ₃) ₂ -CH-CH(CH ₃) ₂	Synthesis -To diisop and carbo tions at 2
HO		tions at

_

-To diisopropylacetyl chloride, 2-chloroanisole and carbon disulfide was added in small portions at 25° , aluminium chloride; the mixture stirred 1 h at r.t. and 45 min at 55° ; pentane and aluminium chloride added, heated 3 h at 80° [2056].

m.p. 123–124.5° [2056].

3-Methyl-1-(2,4,6-trihydroxy-3-nitro-5-propylphenyl)-1-butanone

[119692-00-1]	$C_{14}H_{19}NO_6$	mol. wt. 297.31

Synthesis $COCH_2CH(CH_3)_2$ -Obtained by adding a mixture of fuming NO₂ nitric acid and acetic acid to the solution of 3-methyl-1-(2,4,6-tri-hydroxy-3-propylphenyl)-HO 1-butanone in acetic acid at 60° for 30 min (30-40 %) [3414].

m.p. 53–55° [3414]; ¹H NMR [3414], IR [3414], MS [3414].

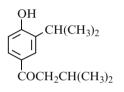
BIOLOGICAL ACTIVITY: Germination inhibition [3414]; PET inhibition [3414].

1-[4-Hydroxy-3-(1-methylethyl)phenyl]-3-methyl-1-butanone

[95185-73-2]

 $C_{14}H_{20}O_2$

mol. wt. 220.31



Synthesis -Obtained by Fries rearrangement of 2-isopropylphenyl isovalerate with aluminium chloride in nitrobenzene, first 2 h at 30°, then at r.t. for 24 h [2704].

1-(4-Hydroxy-2-methylphenyl)-2-ethyl-3-methyl-1-butanone

$$C_{14}H_{20}O_2$$
 mol. wt. 220.31

 $\begin{array}{c} C_2H_5 & \text{Symmesus} \\ I & \text{CH-} CH(CH_3)_2 \\ \text{m.p. } 123-124.5^{\circ} \ [2057]. \end{array}$

Synthesis

1-(2,4-Dihydroxy-3-propylphenyl)-3-methyl-1-butanone

[664376-79-8]

C14H20O3

mol. wt. 236.31



COCH₂CH(CH₃)₂ -Obtained by reaction of isovaleroyl chloride with 2-propylresorcinol in the presence of aluminium chloride in methylene chloride first at 0°, then at r.t. for 16 h [2486].

-Also refer to: [773].

1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-3-methyl-1-butanone

(Homobaeckeol, Pallidusol)

[60831-55-2] $C_{14}H_{20}O_4$ mol. wt. 252.31 Synthesis OH $COCH_2CH(CH_3)_2$ -Obtained treatment of 2,4,6-trime-CH₂ by thoxytoluene with isovaleric acid and boron CH₂O OCH₂ trifluoride, after hydrolysis of 1-oxa-3-oxonia-2-boratanaphthalene, which on hydrolysis gave homobaeckeol (61 %) [2743].

Isolation from natural sources

-From the leaves of Mallotus palliduss (Euphorbiaceae) [2988].

pale yellow [2743]; m.p. 109° [2743], 101–102° [2988]; ¹H NMR [2743, 2988], ¹³C NMR [2988], IR [2988], UV [2743, 2988], MS [2988].

Na salt C14H19O4Na mol. wt. 274.29

-Refer to: [2988].

1-(6-Hydroxy-2,4-dimethoxy-3-methylphenyl)-3-methyl-1-butanone (Homoisobaeckeel)

(ITOMOISODUeckeoi)		
[126211 12 0]		

[126211-12-9]	$C_{14}H_{20}O_4$	mol. wt. 252.31
CH ₃ O ^{OH} CH ₃ O ^{OCH} ₂ CH(CH ₃) ₂ CH ₃ O ^{OCH} ₃	Isolation from natural sources -From the essential oil of <i>Thry</i> (Myrtaceae) [803]. -From <i>Eucalyptus</i> species (My	L

1-[3-(3-Hydroxypropoxy)-4-hydroxyphenyl]-3-methyl-1-butanone

	$C_{14}H_{20}O_4$	mol. wt. 252.31
OH OCH ₂ CH ₂ CH ₂ OH COCH ₂ CH(CH ₃) ₂	Synthesis -Refer to: [2779]. 4-methyl ether [919995-26-9] $C_{15}H_{22}O_4$ -Refer to: [2779].	mol. wt. 266.34

[919995-27-0] Dimethyl ether $C_{16}H_{24}O_4$ mol. wt. 280.36 -Refer to: [1085, 2206, 2779].

¹H NMR [1231, 2206], ¹³C NMR [1231], IR [1231], MS [1085, 1231].

5,7-Dihydroxy-6-(3-methyl-1-oxobutyl)-4-methyl-2H-1-benzopyran-2-one

$$\begin{array}{c} C_{15}H_{16}O_5 & \text{mol. wt. 276.29} \\ HO & O & O \\ (CH_3)_2CHCH_2CO & O & \text{Syntheses} \\ OH & CH_3 & -Preparation & from & 2,4,6-trihydroxy & isovalerophenone (54 \%) [1884]. \\ -Preparation & [650] & according & to & the \\ method [1884]. \end{array}$$

canary-yellow solid [1884]; m.p. 272–274° [1884]; ¹H NMR [1884], ¹³C NMR [1884], IR [1884], MS [1884]; TLC [1884].

BIOLOGICAL ACTIVITY: Inhibition of growth (Escherichia coli, Staphylococcus aureus, Klebsiella pneumoniae, Pseudomonas aeruginosa, Salmonella typhimurium) [650].

1-[5-(Acetoxyethyl)-2-hydroxyphenyl]-3-methyl-1-butanone

	$C_{15}H_{20}O_4$	mol. wt. 264.32
OH COCH ₂ CH(CH ₃) ₂	Synthesis -Refer to: [2701]. Methyl ether [77311-66-1]	
CH-CH ₃ OCOCH ₃	C ₁₆ H ₂₂ O ₄ -Obtained by treatment of 5-ethyl-2- phenone with BTAP (benzyltrieth manganate) in acetic acid at (8.6 %) [2701].	yl-ammonium per-

b.p.₁ 153–155° [2701]; ¹H NMR [2701], IR [2701].

1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-3-methyl-1-butanone

[80356-12-3]	$C_{15}H_{22}O_2$	mol. wt. 234.34
(CH ₃) ₂ CH CH ₃ COCH ₂ CH(CH ₃) ₂	Syntheses -Obtained by treatment of its boiling pyridinium chloride ((20 %) [2660]. -Also obtained by photo-Fries thymyl isovalerate in metha under nitrogen (28 %) (2b) [2	$(205-215^{\circ})$ for 2 h is rearrangement of nol for 6 h at 25°

-Also obtained by Fries rearrangement of thymyl isovalerate with aluminium chloride in nitrobenzene for 12 h at 30° (92 %) [2647]. -Also refer to: [2648].

b.p.₁₃ 198° [2660], b.p.₁₃ 202° [2647]; m.p. 119° [2421], 108° [2647, 2648], 106° [2660]; ¹H NMR [2421], IR [2421].

Methyl ether (VI) $C_{16}H_{24}O_2$ mol. wt. 248.37

-Obtained by reaction of isovaleryl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (71 %) [2660].

b.p.₁₆ 179–180° [2660]; $n_D^{20.5} = 1.523$ [2660].

1-[4-Hydroxy-5-methyl-2-(1-methylethyl)phenyl]-3-methyl-1-butanone

b.p.₁₃ 201° [2647]; m.p. 108° [2647], 86° [1522].

1-(4-Hydroxy-2-methylphenyl)-3-methyl-2-(1-methylethyl)-1-butanone

1-(4-Hydroxy-3-methylphenyl)-3-methyl-2-(1-methylethyl)-1-butanone

 $C_{15}H_{22}O_{2} mtext{mol. wt. 234.34}$ $CH(CH_{3})_{2} Synthesis -Refer to: [2767]. mtext{m.p. 123-124.5° [2767].}$

1-[3,5-Dihydroxy-4-(2-methylpropyl)phenyl]-3-methyl-1-butanone

$$C_{15}H_{22}O_3 \qquad \text{mol. wt. } 250.34$$

$$HO \qquad OH \qquad -\text{Refer to: } [1407].$$

$$Dimethyl \ ether \\ C_{17}H_{26}O_3 \qquad \text{mol. wt. } 278.39$$

-Obtained by reaction of 3,4,5-trimethoxybenzonitrile with a greater excess of isobutylmagnesium bromide in refluxing toluene for 5 h (28 %) [1407].

b.p.5 163-167° [1407].

CH₃

HO

(CH₃)₂CH CH₃

Semicarbazone of the dimethyl ether

m.p. 183-184° [1407].

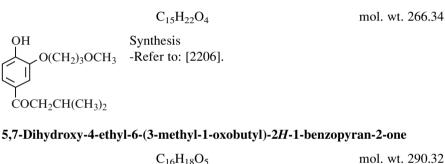
1-(2-Hydroxy-4,6-dimethoxy-3,5-dimethylphenyl)-3-methyl-1-butanone (*Miniatone*)

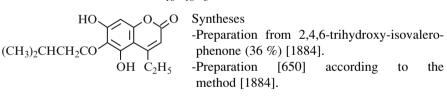
$$[808751-12-4] C_{15}H_{22}O_4 mol. wt. 266.34$$

$$CH_3 - COCH_2CH(CH_3)_2 FCH_3 OCH_3 OCH_$$

¹H NMR [1453], ¹³C NMR [1453], MS [1453].

1-[4-Hydroxy-3-(3-methoxypropoxy)phenyl]-3-methyl-1-butanone

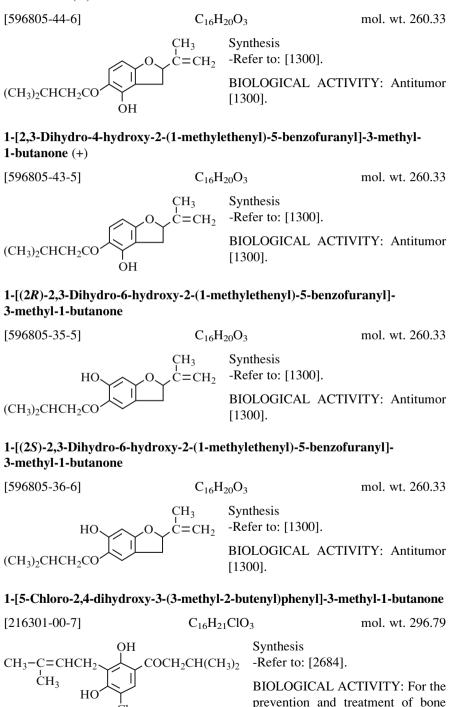




canary-yellow solid [1884]; m.p. 229–232° [1884]; ¹H NMR [1884], ¹³C NMR [1884], IR [1884], MS [1884]; TLC [1884].

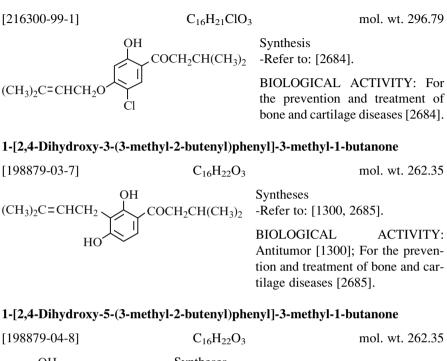
BIOLOGICAL ACTIVITY: Inhibition of growth (Escherichia coli, Staphylococcus aureus, Klebsiella pneumoniae, Pseudomonas aeruginosa, Salmonella typhimurium) [650].

1-[2,3-Dihydro-4-hydroxy-2-(1-methylethenyl)-5-benzofuranyl]-3-methyl-1-butanone (-)



and cartilage diseases [2684].

1-[5-Chloro-2-hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-3-methyl-1-butanone



Syntheses OH COCH₂CH(CH₃)₂ -Refer to: [1300, 2685]. BIOLOGICAL ACTIVITY: Antitumor [1300]. CH₂CH=C(CH₃)₂

-For the prevention and treatment of bone and cartilage diseases [2685].

1-[2-Hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-3-methyl-1-butanone

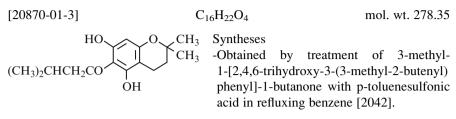
[198879-02-6] $C_{16}H_{22}O_3$ $COCH_2CH(CH_3)_2$ -Refer to: [2685].

mol. wt. 262.35

Synthesis

BIOLOGICAL ACTIVITY: For the prevention and treatment of bone and cartilage diseases [2685].

1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-3-methyl-1-butanone



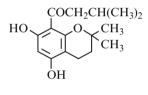
-Also obtained by reaction of isovaleronitrile with 5,7-dihydroxy-2,2-dimethylchroman (Hoesch reaction) [2481]. -Also refer to: [702, 1611].

Pale yellow needles [2042]; Almost colourless needles [2481]; m.p. 144–145° [2481], 142° [702, 2614], 141–142° [2042]; ¹H NMR [2042], IR [2042], UV [702].

1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl)-3-methyl-1-butanone

[20869-99-2]

C₁₆H₂₂O₄ mol. wt. 278.35



Syntheses -Obtained by treatment of 3-methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-butanone with p-toluenesulfonic acid in refluxing benzene (40 %) [2042].

-Also obtained by reaction of isovaleronitrile with 5,7-dihydroxy-2,2-dimethylchroman (Hoesch reaction) (70 %) [2481].

-Also refer to: [702, 708, 1611].

pale yellow prisms [2042]; pale greenish-yellow plates [2481]; m.p. 138° [2481], 136° [702], 135–136° [2042], ¹H NMR [2042], IR [2042], UV [702].

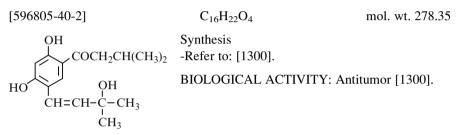
1-[2,4-Dihydroxy-3-(2-hydroxy-3-methyl-3-butenyl)phenyl]-3-methyl-1-butanone

[596805-37-7]	$C_{16}H_{22}O_4$	mol. wt. 278.35
СН ₃ ОН сн ₂ =С—СН-	CH ₂ COCH ₂ CH(CH ₃) ₂ HO	Synthesis -Refer to: [1300]. BIOLOGICAL Antitumor [1300].

1-[2,4-Dihydroxy-5-(2-hydroxy-3-methyl-3-butenyl)phenyl]-3-methyl-1-butanone

[596805-39-9] C16H22O4 mol. wt. 278.35 **Synthesis** OH $COCH_2CH(CH_3)_2$ -Refer to: [1300]. **BIOLOGICAL ACTIVITY: Antitumor** [1300]. OH CH₃ HC CH₂-CH-C=CH₂

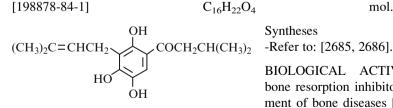
1-[2,4-Dihydroxy-5-(3-hydroxy-3-methyl-1-butenyl)phenyl]-3-methyl-1-butanone



1-[3,5-Dihydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-3-methyl-1-butanone

[216300-90-2] C₁₆H₂₂O₄ mol. wt. 278.35 Synthesis (CH₃)₂C=CHCH₂O -Refer to: [2684]. BIOLOGICAL ACTIVITY: For OCH₂CH(CH₃)₂ the prevention and treatment of bone and cartilage diseases [2684].

3-Methyl-1-[2,4,5-trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-butanone



mol. wt. 278.35

Syntheses

BIOLOGICAL ACTIVITY: As bone resorption inhibitor for treatment of bone diseases [2686]; For the prevention and treatment of bone and cartilage diseases [2685].

3-Methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-butanone

[54614-64-1] $C_{16}H_{22}O_4$ (CH₃)₂C=CHCH₂ OH COCH₂CH(CH₃)₂ HO OH mol. wt. 278.35

-Obtained by reaction of prenyl bromide with phloroisovalerophenone,

Syntheses

*in the presence of sodium methoxide in an ethyl ether/ methanol solution [1254], (9 %) [2616];

- *in the presence of potassium hydroxide in water at 0° for 22 h under argon (13 %) [1066];
- *in the presence of 10 % aqueous potassium hydroxide containing crushed ice (21 %) [2042].
- -Also prepared by adding at 5° a solution of sodium methoxide in methanol, followed by isopentenyl bromide in ethyl ether to a solution of 3-methyl-1-(2,4,6-trihydroxyphenyl)-1-butanone in benzene and ethyl ether; then the mixture was stirred at r.t. for 6 h (17 %) [2113].
- -Also obtained from isoprenyl pyrophosphate and 6-isovaleryl-3,5-diketocapronyl-CoA [909].
- -Also refer to: [542, 2609, 2614, 2685, 2686, 3481].

Isolation from natural sources

-From hop plant, Humulos lupulus (Cannabinaceae) [1066].

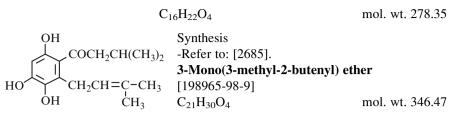
yellow crystals [1066]; pale yellow prisms [2042]; m.p. 140° [2113], 139–140° [2609], 138.5–140° [2616], 138.5–139.5° [1066], 138–139° [2042], 134–141° [541]; ¹H NMR [541, 1066, 2042], IR [2042], UV [1066], MS [1066]; TLC [1066]; HPLC [1066, 3481].

BIOLOGICAL ACTIVITY: Antifungal [2113]; As bone resorption inhibitor for treatment of bone diseases [2686]; For the prevention and treatment of bone and cartilage diseases [2685].

3-Methyl-1-[3,4,5-trihydroxy-2-(3-methyl-2-butenyl)phenyl]-1-butanone

[216300-91-3]	$C_{16}H_{22}O_4$	mol. wt. 278.35
ОН НО ОН	Synthesis -Refer to: [2684].	
$CH_2CH=C(CH_3)_2$ $COCH_2CH(CH_3)_2$	BIOLOGICAL ACTIVITY: Fo and treatment of bone and c [2684].	

3-Methyl-1-[3,4,6-trihydroxy-2-(3-methyl-2-butenyl)phenyl]-1-butanone



¹H NMR [2683].

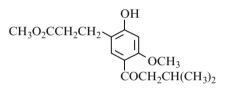
BIOLOGICAL ACTIVITY: For the prevention and treatment of bone and cartilage diseases [2685].

Methyl 3-(2-hydroxy-5-isovaleroyl-4-methoxyphenyl)propanoate

[117285-75-3]

 $C_{16}H_{22}O_5$

mol. wt. 294.35



OH Syntheses -Preparation by Fries rearrangement of methyl 3-(2-isovaleroyloxy-4-methoxyphenyl)-propanoate with aluminium chloride (5 equiv.) in nitromethane at r.t. (65 %) [529].

-Also refer to: [530].

m.p. 117–119° [530]; ¹H NMR [530], IR [530], UV [530], MS [530].

1-[2,6-Dihydroxy-4-[(3-methylbutoxyl)]phenyl]-3-methyl-1-butanone

[918814-70-7]

 $C_{16}H_{24}O_{4}$

Synthesis

mol. wt. 280.36

(CH₃)₂CHCH₂CH₂OH

COCH₂CH(CH₃)₂ -Obtained by Friedel-Crafts acylation of phloroglucinol monoisopentyl ether with isopentanoyl chloride in the presence of titanium tetrachloride (35 %) [337].

off white solid [337]; m.p. 168–172° [337]; ¹H NMR [337], IR [337], MS [337].

BIOLOGICAL ACTIVITY: Antiprotozoal and antimicrobial [337].

3-Methyl-1-(2,4,6-trihydroxy-3-pentylphenyl)-1-butanone

[74478-03-8]	$C_{16}H_{24}O_4$	mol. wt. 280.36
OH C ₅ H ₁₁ HO OH OH	Synthesis -Obtained by adding a solution chloride in nitrobenzene to 2,4,6-trihydroxy-pentylbenzen chloride in carbon disulfide at the mixture for 6 h at 30–35°	a suspension, of e and aluminium t r.t., then stirring

m.p. 155° [2113].

BIOLOGICAL ACTIVITY: Antifungal [2113].

1-[2,3,4-Trihydroxy-5-(3-methylbutyl)phenyl]-3-methyl-1-butanone

C	₆ H ₂₄ O ₄	mol. wt. 280.36
HO HO HO CH ₂ CH ₂ CH(CH ₃) ₂ CH ₂ CH ₂ CH(CH ₃) ₂	Syntheses -Obtained by reaction 4-iso-amylpyrogallol in ride for 3 h at 130–140° -Also refer to: [1054]. b.p. _{0.01} 148–160° [811];	the presence of zinc chlo- [811].

Tribenzoate

C₃₇H₃₆O₇

mol. wt. 592.69

-Obtained treatment of the title ketone with benzoyl chloride in the presence of pyridine [811].

-Also refer to: [1054].

m.p. 170° [811].

3-Methyl-1-[2,4,6-trihydroxy-3-(3-methylbutyl)phenyl]-1-butanone

2',4',6'-Trihydroxy-3'-isopentylisovalerophenone (**XIII**)

[26104-01-8]	$C_{16}H_{24}O_4$	mol. wt. 280.36
(CH ₃) ₂ CHCH ₂ CH	OH H ₂ O O O H	Syntheses -Obtained by reaction of isovaleryl chloride with 2-isopentylphloroglucinol in the presence of aluminium chloride in nitrobenzene (52 %) (II) [2610], at 0° for 3 days (41 %) (XIII) [898].

-Preparation by hydrogenation of 3-prenylphloroisovalerophenone (**XV**) with hydrogen in acetic acid in the presence of Pd (**XVI**) (81 %) [2616].

-Preparation by hydrogenation of 2,4,6-trihydroxy-3-(3-methyl-2-butenyl) isovalerophenone,

*in methanol over 5 % Pd/C (91 %) [542];

*in the presence of PtO_2 in methanol under a hydrogen atmosphere at r.t. for 1 h (86 %) [2113].

-Also obtained by hydrogenation of 3,5-dihydroxy-2-isovaleryl-6,6-bis(3-methyl-2-butenyl)-cyclohexa-2,4-dien-1-ol in methanol over 5 % Pd/C (42 %) [542].

-Also obtained by condensation of isobutyl cyanide with isoamylphloroglucinol (Hoesch reaction) (5 %) [811].

m.p. 173–175° [898], 172–174° [542], 171° [2113], 169–170° [2616], 167–169° [2610], 163–165° [541], 103° [811]; **N.B.**: One of the reported melting point is obviously wrong.

¹H NMR [541, 898], IR [898], UV [898].

BIOLOGICAL ACTIVITY: Antifungal [2113].

3-Methyl-1-[2,3,4,6-tetrahydroxy-5-(3-methylbutyl)phenyl]-1-butanone

C₁₆H₂₄O₅

(Humulo-hydrochinon) (IV), (Humuloquinol)

[107152-23-8] OH (CH₃)₂CHCH₂CH₂

Syntheses

 $(CH_3)_2CHCH_2CH_2CH_2CH_2CH(CH_3)_2 + Obtained (II) by treatment of humuloquinone (III) with sulfur dioxide in 80 % ethanol for 90 min (92 %) [2610].$

mol. wt. 296.36

-Also obtained by hydrogenation of Humulon (II) in the presence of palladium chloride in methanol [1255, 2613, 3329]. -Also refer to: [3304–3306].

Isolation from natural sources

-Refer to: [1254, 2613, 3330].

b.p._{0.2} 135–140° [2610]; m.p. 128–130° [2610], 126–128° [1254, 1255], 123–125° [3329, 3330], 118° [2613, 3304, 3305]; UV [1255].

Tetrabenzoate [103567-06-2] C₄₄H₄₀O₉ mol. wt. 712.80

-Obtained by reaction of benzoyl chloride with humuloquinol in the presence of pyridine [1255].

-Also refer to: [2610, 3329, 3330].

m.p. 172–172.5° [2610], 168° [3329, 3330], 167° [1255]; UV [1255].

1-[3-Amino-2,4,6-trihydroxy-5-(3-methylbutyl)phenyl]-3-methyl-1-butanone 5-Amino-3-isoamyl-phlorisovalerophenone

C ₁₆ H	H ₂₅ NO ₄	mol. wt. 295.38
NH_2 H_2 $COCH_2CH(CH_3)_2$	Synthesis -Refer to: [2610]. Hydrochloride	
HO CH ₂ CH ₂ CH(CH ₃) ₂	$C_{16}H_{25}NO_4$, HCl	mol. wt. 331.84

-Obtained by treatment of 5-phenylazo-3-isoamylphloroisovalerophenone with stannous chloride dihydrate in the presence of concentrated hydrochloric acid in boiling acetic acid for 10 min (77 %) [2610].

C44H41NO8

C51H45NO9

m.p. 125-130° [2610].

Tetrabenzoate

-Refer to: [2610].

Pentabenzoate

-Refer to: [2610].

1-(2-Hydroxy-4-phenoxyphenyl)-3-methyl-1-butanone

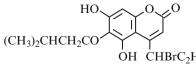
[307000-33-5]	$C_{17}H_{18}O_3$	mol. wt. 270.32
C ₆ H ₅ O ^H COCH ₂ CH(CH ₃) ₂	Syntheses -Refer to: [1018–1022, 1345].	

USE: Cosmetic compounds containing derives of hydroxydiphenyl ether for inhibiting body odours [1018–1022].

BIOLOGICAL ACTIVITY: Antimicrobial [1345].

4-(1-Bromopropyl)-5,7-dihydroxy-6-(3-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one

[98498-59-0]	racemic	$C_{17}H_{19}BrO_5$	mol. wt. 383.24
[111249-67-3]			



Syntheses

-Obtained by condensation of 2-(3-methylbutyryl)phloroglucinol with ethyl 4-bromo-3-oxohexanoate (small amount) [347].

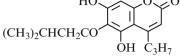
-Obtained from a Pechmann reaction (low yield) [766].

yellow-brown solid [347]; m.p. 205–206° (d) [347]; ¹H NMR [763], IR [763], UV [763], MS [763]. mol. wt. 711.80

mol. wt. 815.90

5,7-Dihydroxy-6-(3-methyl-1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one

[98192-61-1]
$$C_{17}H_{20}O_5$$
 mol. wt. 304.34
HO₅ O_5 O_5 Syntheses



-Obtained by reaction of ethyl 3-oxohexanoate with 1-(2,4,6-trihydroxyphenyl)-3-methyl-1-butanone in the presence of concentrated sulfuric acid in acetic acid at r.t. for 2 days (35 %) [753].

-Also refer to: [762].

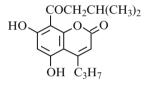
m.p. 228–229° [1005], 223–224° [753]; ¹H NMR [753], IR [753], UV [753], MS [753].

5,7-Dihydroxy-8-(3-methyl-1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one

[98192-67-7]

 $C_{17}H_{20}O_5$

mol. wt. 304.34



-Obtained by reaction of ethyl 3-oxohexanoate with 1-(2,4,6-trihydroxyphenyl)-3-methyl-1-butanone in the presence of concentrated sulfuric acid in acetic acid at r.t. for 2 days (4 %) [753]. -Also refer to: [553, 762].

m.p. 219° [753]; ¹H NMR [753], IR [753], UV [553, 753], MS [753].

Syntheses

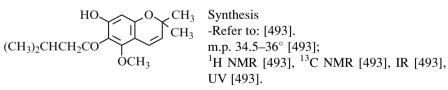
1-(7-Hydroxy-5-methoxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-3-methyl-1-butanone

(Rhynchonin A)

[163734-37-0]

 $C_{17}H_{22}O_4$

mol. wt. 290.36



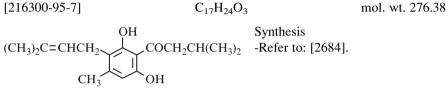
BIOLOGIC ACTIVITY: Bioactive chromenes from *Rhyncholacis penicillata* [493].

1-[2,4-Dihydroxy-6-methyl-3-(3-methyl-2-butenyl)phenyl]-3-methyl-1-butanone

[198878-99-8]
$$C_{17}H_{24}O_3$$
 mol. wt. 276.38
 $CH_3-C=CHCH_2$ OH Synthesis
 $CH_3-C=CHCH_2$ COCH₂CH(CH₃)₂ -Refer to: [2685].
HO CH₃

BIOLOGICAL ACTIVITY: As preventive and therapeutic agent for bone and cartilage diseases [2685].

1-[2,6-Dihydroxy-4-methyl-3-(3-methyl-2-butenyl)phenyl]-3-methyl-1-butanone



BIOLOGICAL ACTIVITY: Prevention and treatment of bone and cartilage diseases [2684].

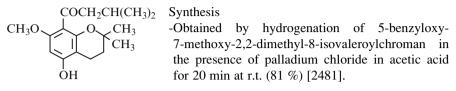
1-[4,6-Dihydroxy-2-methyl-3-(3-methyl-2-butenyl)phenyl]-3-methyl-1-butanone

[198879-00-4]	$C_{17}H_{24}O_3$	mol. wt. 276.38
OH COCH ₂ CH(CH ₃) ₂	Synthesis -Refer to: [2685].	
HO CH ₃ CH ₂ CH=C-CH ₃ CH ₃	BIOLOGICAL ACTIVITY: As therapeutic agent for bone diseases [2685].	•

1-[2-Hydroxy-6-methyl-4-[(3-methyl-2-butenyl)oxy]phenyl]-3-methyl-1-butanone

[198878-98-7]
$$C_{17}H_{24}O_3$$
 mol. wt. 276.38
OH
CH₃
CH₃
CH₃-C=CHCH₂O
CH₃
CH₃-C=CHCH₂O
CH₃
CH₃-C=CHCH₂O
CH₃

1-(3,4-Dihydro-5-hydroxy-7-methoxy-2,2-dimethyl-2H-1-benzopyran-8-yl)-3-methyl-1-butanone

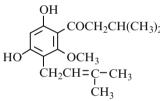


small needles [2481]; m.p. 142–143° [2481].

1-[4,6-Dihydroxy-2-methoxy-3-(3-methyl-2-butenyl)phenyl]-3-methyl-1-butanone

[162071-07-0]

C17H24O4 mol. wt. 292.37



Synthesis COCH₂CH(CH₃)₂ -Obtained by refluxing methyl 2,6-dihydroxy-3-isovaleryl-4-methoxy-5-(3-methyl-2-butenyl) benzoate with 5 % aqueous KOH for 4 h under nitrogen (75 %) [493].

chromenes

m.p. 80-82° [493]; IR [493].

Rhyncholacis

penicillata [493].

1-[2,6-Dihydroxy-3-methyl-4-[(3-methyl-2-butenyl)oxy]phenyl]-3-methyl-1-butanone

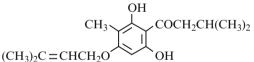
Bioactive

[140400-68-6]

BIOLOGICAL

C17H24O4

mol. wt. 292.37



ACTIVITY:

Isolation from natural sources $_{\rm COCH_2CH(CH_3)_2}$ -From the aerial parts of *Hyper*icum calycinum (Clusiacees, Guttuferes) [337, 836].

from

pale yellow needles [836]; m.p. 118–121° [836]; ¹H NMR [836], ¹³C NMR [836], IR [836], UV [836], MS [836]; TLC [836].

BIOLOGICAL ACTIVITY: Antiprotozoal and antimicrobial activities [337]; In vitro malarial activity [334, 337, 836]; Fungicide [337, 836].

Diacetate [140400-69-7] C₂₁H₂₈O₆ mol. wt. 376.45

-Obtained by reaction of acetic anhydride with the title ketone in the presence of pyridine at r.t. for 25 h [836].

colourless oil [836]; ¹H NMR [836], ¹³C NMR [836], MS [836]; TLC [836].

1-[2-(β-D-Glucopyranosyloxy)-4,6-dihydroxyphenyl]-3-methyl-1-butanone (Lysidiciside A, Lysidiside A)

[718608-83-4]	$C_{17}H_{24}O_9$	mol. wt. 372.37
OH COCH ₂ CH(CH ₃) ₂ HO O-β-Glc	Isolation from natural sources -From strawberry Fruit, <i>Fragaria</i> cv. Tochiotome [3154]. -From the whole plant of <i>Indig</i> (Leguminosae) [209].	

-From the roots of *Lysidice rhodostega* Hance (Fabaceae) [1089, 1091]. -In hop extracts [3396].

-From Lysidice brevicalyx [2536].

colourless gummy solid [209]; light yellow amorphous powder [1089]; m.p. 112–115° [1089]; ¹H NMR [209, 1089, 2536, 3154], ¹³C NMR [209, 1089, 3154], IR [209, 1089, 3154], UV [209, 1089, 2536, 3154], MS [209, 1089, 2536, 3154]; HPLC [2536]; (α)_D²⁶ = -55° (methanol) [3154]; (α)_D²⁵ = -63.4° (acetone) [1089].

BIOLOGICAL ACTIVITY: Lipoxygenase enzyme inhibitor [209]; Cytochrome P 450 inhibitor [3154]; Vasodilator [1089].

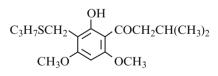
1-[2-Hydroxy-4,6-dimethoxy-3-[(propylthio)methyl]phenyl]-3-methyl-1-butanone

[179630-66-1]

 $C_{17}H_{26}O_4S$

Synthesis

mol. wt. 326.46



COCH2CH(CH3)2-Obtained in two steps: First, treatment
of 2-hydroxy-4,6-dimethoxy-3-(1-oxo-
3-methyl-butyl)benzaldehyde with
NaBH3CN in methanol at r.t. for 3 h.
Then, after evaporation, the residue was
treated with propane-1-thiol in methylene
chloride in the presence of zinc iodide for
16 h at r.t. (88 %) [643].

oil [643]; ¹H NMR [643], ¹³C NMR [643], IR [643], UV [643], MS [643].

1-[2-Hydroxy-5-(phenylmethoxy)phenyl]-3-methyl-1-butanone

	$C_{18}H_{20}O_3$	mol. wt. 284.36
$\bigcup_{OCH_2C_6H_5}^{OH} COCH_2CH(CH_3)_2$	Synthesis -Obtained from a mixture of 2,5- isovalerophenone, benzyl chlorid in refluxing ethanol for 8 h (58 %	le and sodium ethoxide

yellow rhombs. [770]; b.p.₂ 160–170° [770]; m.p. 60° [770].

2,6-Dihydroxy-3-isovaleryl-4-methoxy-5-(3-methyl-2-butenyl)benzoic acid

mol. wt. 336.38 $C_{18}H_{24}O_{6}$ $(CH_3)_2CHCH_2CO \qquad OH \qquad Synthesis$ $(CH_3)_2CHCH_2CO \qquad CO_2H \qquad -Refer to: [493].$ Methyl ester [162071-06-9] $CH_3O \qquad OH \qquad C_{19}H_{26}O_6 \qquad mol. v$ mol. wt. 350.41

-Obtained by treatment of methyl 2,6-dihydroxy-3-isovaleryl-4-methoxybenzoate with 2-methyl-3-buten-2-ol in the presence of boron trifluoride etherate in dioxane at r.t. for 48 h (50 %) [493].

oil [493]; IR [493].

1-[2-Hydroxy-6-methoxy-3-methyl-4-[(3-methyl-2-butenyl)oxy]phenyl]-3-methyl-1-butanone

[140400-70-0]

 $(CH_3)_2C = CHCH_2O$

 $C_{18}H_{26}O_4$

mol. wt. 306.40

OH CH_3 CH_4 $CH_2CH_2CH(CH_3)_2$ $COCH_2CH(CH_3)_2$ Obtained by treatment of 1-[2,6-dihydroxy-3-methyl- $<math>CHCH_2O$ OCH_3 4-[(3-methyl-2-butenyl)oxy]phe- $<math>TH_3$ TH_3 TH_3 THnyl]-3-methyl-1-butanone with diazomethane in ethyl ether at r.t. for 30 h [836].

colourless crystals [836]; m.p. 63–67° [836]; ¹H NMR [836], ¹³C NMR [836], MS [836]; TLC [836].

1-(4-Heptyl-2-hydroxyphenyl)-3-methyl-1-butanone

[101873-66-9]	$C_{18}H_{28}O_2$	mol. wt. 276.42
OH COCH ₂ CH(CH ₃) ₂	3-heptylphen	by Fries rearrangement of yl isovalerate with aluminium 10° for 15 min (66 %) [855].

-Also obtained by Friedel-Crafts acylation of 3-heptylanisole with isovaleryl chloride in the presence of aluminium chloride in carbon disulfide between -5 and -10° , then boiling for 5 h after solvent elimination (76 %) [551].

b.p._{1.7} 173–174° [551], b.p._{0.8–0.9} 177° [551], b.p._{1.5} 183° [551]; $n_{\rm D}^{20} = 1.5148$ [551].

p-Nitrophenvlhvdrazone [102812-21-5] C₂₄H₃₃N₃O₃ mol. wt. 411.54

m.p. 130-131° [551].

2,4-Dinitrophenylhydrazone [102897-71-2] C₂₄H₃₂N₄O₅ mol. wt. 456.54

m.p. 112–113° [551].

1-[2-Hydroxy-3-methyl-4,6-bis(1-methylethoxy)phenyl]-3-methyl-1-butanone

[181227-34-9] C18H28O4 mol. wt. 308.42 Syntheses

CH₃ (CH₃)₂CHO

OH Syntheses -Obtained by reaction of isovaleryl chloride with 2-methyl-3,5-diisopro-poxyphenol in the presence of titanium tetrachloride [641] in methylene chloride at 0° C under argon.

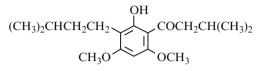
The mixture was allowed to stand at r.t. for 30 min (84.6 %) [642].

yellow oil [642]; ¹H NMR [642], ¹³C NMR [642], IR [642], UV [642], MS [642].

1-[2-Hydroxy-4,6-dimethoxy-3-(3-methylbutyl)phenyl]-3-methyl-1-butanone

3-Isoamyl-phlorisovalerophenon-4,6-dimethylether

[101874-06-0] C18H28O4 mol. wt. 308.42



Syntheses

 \bigcirc COCH₂CH(CH₃)₂ -Obtained by reaction of methyl iodide with 3-isoamylphloroisovalerophenone in the presence potassium carbonate of in refluxing acetone for 6 h (27 %) [2610].

-Also refer to: [778].

yellow prisms [2610]; b.p._{0.2} 100° [2610]; m.p. 86° [2610].

1-[2,4-Dihydroxy-3,6-dimethoxy-5-(3-methylbutyl)phenyl]-3-methyl-1-butanone

(Humulo-hydrochinon-dimethyläther) (XII)

OCH₃

CH₂CH₂CH(CH₃)₂

[101874-19-5]

CH₃O

HO

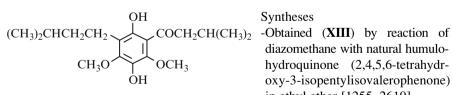
C18H28O5 mol. wt. 324.42

OH Synthesis $COCH_2CH(CH_3)_2$ -Obtained by reaction of isovaleryl chloride with 2.5-dimethane 4.1 the presence of aluminium chloride in a carbon disulfide/nitrobenzene mixture (55 %) (**XII**) [2610].

yellow oil [2610]; b.p._{0.2} 120–140° [2610]; UV [2610].

1-[2,5-Dihydroxy-4,6-dimethoxy-3-(3-methylbutyl)phenyl]-3-methyl-1-butanone

C18H28O5



Syntheses

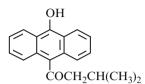
oxy-3-isopentylisovalerophenone) in ethyl ether [1255, 2610].

mol. wt. 324.42

b.p._{0.01} 100–115° [1255, 2610].

9-Hydroxy-10-isovalerylanthracene

C₁₉H₁₈O₂ mol. wt. 278.35



Synthesis -Obtained by Fries rearrangement of 9-isovaleryloxyanthracene with various metal halides in benzene under reflux, but it is rapidly transformed into 10-isovalerylanthrone [3052].

4-[1-(Acetyloxy)propyl]-5,7-dihydroxy-8-(3-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one

[98498-78-3] racemic $C_{19}H_{22}O_7$ mol. wt. 362.38 Syntheses $COCH_2CH(CH_3)_2$ -Obtained ,0 by reaction of isovaleroyl chloride HO with 4-[1-(acetyloxy)propyl]-5,7-dihydroxy-2H-1-benzopyran-2-one in the presence of aluminium OH CH-C₂H₅ chloride in carbon disulfide/nitrobenzene for 4 days at 20° (33 %) [766]. OCOCH₃ -Also refer to: [764].

m.p. 210–212° [764]; ¹H NMR [764], IR [764], UV [764], MS [764].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-3-methyl-1-butanone

[14102-16-0]	$C_{19}H_{30}O_2$	mol. wt. 290.45
(CH ₃) ₃ C COCH ₂ CH(CH ₃) ₃	Syntheses -Preparation by reaction of iso 2,6-di-tert-butylphenol in the *aluminium chloride at -10° [2506]; *titanium tetrachloride [1468]	presence of, for 1–13 min (88 %)

-Also refer to: [951].

m.p. 110-113° [2506], 110-111° [951].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-3-mercapto-3-methyl-1-butanone

[174635-35-9]	$C_{19}H_{30}O_2S$	mol. wt. 322.51
$(CH_3)_3C \xrightarrow{OH} C(CH_3)_3$ $SH \\ COCH_2 - C - CH_3$ CH_3	Synthesis -Refer to: [2482]. BIOLOGICAL ACTIVITY: As and analgesic agent [2482].	antiinflammatory

1-(2,5-Dihydroxy-4-heptyl-3-methylphenyl)-3-methyl-1-butanone

(Dihydroflavoglaucine)

C ₁₉ H	$I_{30}O_3$	mol. wt. 306.45
$CH_{3} \xrightarrow{OH} COCH_{2}CH(CH_{3})_{2}$ $C_{7}H_{15} \xrightarrow{OH} OH$	Syntheses -Obtained by hydrogena [2882] in the presence or at r.t. [2540]. -Also obtained by Auroglaucine* [2882].	U

*Flavoglaucine: 1-(2,5-dihydroxy-4-heptyl-3-methylphenyl)-3-methyl-2-buten-1-one *Auroglaucine: 1-[2,5-dihydroxy-3-methyl-4-(1,3,5-heptanetriene)phenyl]-3-methyl-2-buten-1-one

-Also refer to: [2541].

UV [2882].

1-(4-Heptyl-2-hydroxy-5-methoxyphenyl)-3-methyl-1-butanone

 $[102020-42-8] C_{19}H_{30}O_3 mol. wt. 306.45$

CH₃(CH₂)₆ CH₃(CH₂)₆ OCH₃ Synthesis

-Obtained by reaction of isovaleryl chloride with heptylhydroquinone dimethyl ether in the presence of aluminium chloride in carbon disulfide for 2 h (39 %) [2391].

b.p._{0.5} 191–192° [2391], b.p.₃₋₄ 199–204° [2391].

2,4-Dinitrophenylhydrazone

m.p. 134° [2391].

5,7-Dihydroxy-6-(3-methyl-1-oxobutyl)-4-phenyl-2H-1-benzopyran-2-one

[98192-57-5]

 $C_{20}H_{18}O_5$

C₂₅H₃₄N₄O₆

mol. wt. 338.36

mol. wt. 486.57

HO (CH₃)₂CHCH₂CO OH C₆H₅ Syntheses

-Preparation as a mixture (36 %) from (3-methyl-butyryl)phloroglucinol and ethyl benzoylacetate [762] in acetic acid/concentrated sulfuric acid set aside or 5 days [754].

-Also obtained by reaction of benzoyl acetic ester with phloroisovalerophenone in acetic acid in the presence of concentrated sulfuric acid at r.t. for 3 days (31 %) [236].

-Also obtained from *Ochrocarpin* B [1884]. -Also refer to: [650, 765, 1884 (12 %)].

yellow needles [762]; canary-yellow solid [1884]; long colourless needles [236]; m.p. 257–258° [762], 253–255° [1884], 244–245° [754], 242–244° [236]; ¹H NMR [754, 762, 1884], ¹³C NMR [1884], IR [754, 762, 1884], UV [754, 762], MS [754, 762, 1884]; TLC [236, 1884].

BIOLOGICAL ACTIVITY: Inhibition of growth (Escherichia coli, Staphylococcus aureus, Klebsiella pneumoniae, Pseudomonas aeruginosa, Salmonella typhimurium) [650]; Antioxidant [1884]; Cytotoxicity [1884].

5,7-Dihydroxy-8-(3-methyl-1-oxobutyl)-4-phenyl-2H-1-benzopyran-2-one (*Isodispar B*)

[98192-64-4] C₂₀H₁₈O₅

mol. wt. 338.36

COCH ₂ CH(CH ₃) ₂	Syntheses
$HO \downarrow 0 0$	-Preparatio
	(3-methyl
$\langle \langle \rangle \rangle$	benzoylad
он с ₆ н ₅	furic acid

-Preparation as a mixture (36 %) from (3-methylbutyryl)-phloroglucinol and ethyl benzoylacetate [762] in acetic acid/concentrated. sulfuric acid set aside for 5 days [754].

-Also refer to: [236, 278, 650, 765, 1178, 1906].

white crystals [762]; m.p. 200–202° [762], 196–197° [754]; ¹H NMR [754, 762, 1178], ¹³C NMR [1178], IR [754, 762, 1178], UV [754, 762, 1178], MS [754, 762, 1178].

Isolation from natural sources

-From the leaves of *Marila pluricostata* [1906]. -From the fruits and the stem bark of *Calophyllum dispar* (Clusiaceae) [1178].

BIOLOGICAL ACTIVITY: Inhibition of growth (Escherichia coli, Staphylococcus aureus, Klebsiella pneumoniae, Pseudomonas aeruginosa, Salmonella typhimurium) [650]; Antiviral, anti HIV [278]; Antioxidant [1884]; Cytotoxicity [278, 1178, 1906].

5,7-Dihydroxy-6-(3-methyl-1-oxobutyl)-4-(4-methylphenyl)-2*H*-1-benzopyran-2-one

$$\begin{array}{ccc} C_{21}H_{20}O_5 & \text{mol. wt. 352.39} \\ HO & O & O \\ (CH_3)_2CHCH_2CO & OH & C_6H_4\text{-}CH_3\text{-}p \\ OH & C_6H_4\text{-}CH_3\text{-}p \end{array} \begin{array}{c} \text{Syntheses} & -\text{Preparation from $2,4,6\text{-trihydroxy-isovalerophenone (24 \%) [1884].} \\ -\text{Preparation [650] according to the method [1884].} \end{array}$$

canary-yellow solid [1884]; m.p. 253–255° [1884]; ¹H NMR [1884], ¹³C NMR [1884], IR [1884], MS [1884]; TLC [1884].

BIOLOGICAL ACTIVITY: Inhibition of growth (Escherichia coli, Staphylococcus aureus, Klebsiella pneumoniae, Pseudomonas aeruginosa, Salmonella typhimurium) [650].

1-[3-Bromo-4-[4-(2,3-difluorophenoxy)butoxy]-2-hydroxyphenyl]-3-methyl-1-butanone

[811801-19-1]
$$C_{21}H_{23}BrF_2O_4$$
 mol. wt. 457.31
OH Synthesis
-Refer to: [2485].
¹H NMR [2485], MS [2485].

BIOLOGICAL ACTIVITY: Study of its activity as allosteric potentiator of metabotropic glutamate receptor 2 (mGlu2) [2485].

1-[3-Bromo-2-hydroxy-4-(4-phenoxybutoxy)phenyl]-3-methyl-1-butanone

[811801-17-9]

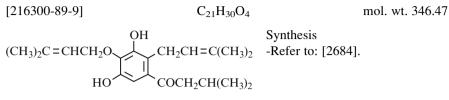
 $C_{21}H_{25}BrO_4$

mol. wt. 421.33

OH Br $C_{6}H_{5}O(CH_{2})_{4}O$ Synthesis -Refer to: [2485]. ¹H NMR [2485], MS [2485].

BIOLOGICAL ACTIVITY: Study of its activity as allosteric potentiator of metabotropic glutamate receptor 2 (mGlu2) [2485].

1-[3,5-Dihydroxy-2-(3-methyl-2-butenyl)-4-[(3-methyl-2-butenyl)oxy]phenyl]-3 methyl-1-butanone



BIOLOGICAL ACTIVITY: For the prevention and treatment of bone and cartilage diseases [2684].

3-Methyl-1-[2,4,5-trihydroxy-3,6-bis(3-methyl-2-butenyl)phenyl]-1-butanone

[198878-83-0] $C_{21}H_{30}O_4$ mol. wt. 346.47 (CH₃)₂C=CHCH₂ OH $COCH_2CH(CH_3)_2$ -Refer to: [2686]. HO $CH_2CH=C(CH_3)_2$ BIOLOGICAL ACTIVITY: As bone resorption inhibitor for treatment of bone diseases [2686].

3-Methyl-1-[2,4,6-trihydroxy-3,5-bis(3-methyl-2-butenyl)phenyl]-1-butanone

(3,5-Bis(Ÿ,Ÿ-dimethylallyl)phloroisovalerophenone) (4-Desoxyhumulone) (Deoxycohumulone)

-Also obtained from phloroisovalerophenone which, in 2 N KOH and chlorobenzene containing Aliquat 336 as phase-transfer catalyst and the mixture adjusted to pH 12 with 2 N HCl, was prenylated with prenyl bromide at 0° for 4 h (54.8 %) [491].

-Also obtained by reaction of prenyl bromide with 2,6-disodium salt of phloroisovalerophenone in benzene suspension at 0° under nitrogen [2615].

-Also obtained by thermal rearrangement of 2-isovaleryl-4,4-bis(3-methylbut-2-enyl)-cyclohexane-1,3,5-trione in a sealed vial at 170° for 4 h (10 %) [703].

-Also obtained by treatment of phloroisovalerophenone in dioxan with 3-hydroxy-3-methylbutene and boron trifluoride etherate [705] at 20° for 8 h (24 %) [704].

-Also obtained by reaction of phloroisovalerophenone with 1-bromo-3-methyl-2-buten (3 mol) in the presence of the weakly basic resin DeAcidite H-IP (OH⁻ form) in boiling benzene (12.1 %) [708]. **N.B.**: The yield was similar at this obtained by using 2-methyl-3-buten-2-ol but the isolation was simpler [708].

- -Also obtained by reaction of phloroisovalerophenone with 1-chloro-3-methyl-2butene (2 molar parts), magnesium oxide (0.5 equiv.) and potassium iodide (1%) [3309, 3310].
- -Also refer to: [542, 907, 908, 1254, 1292, 2532, 2580, 2685, 2686, 3481].

Isolation from natural source

-From the hop (0.2–0.5 %) [2608] and the hop resins [164].

oil [703]; m.p. 86° [708], 82–84° [541, 704], 81–83° [2609], 81–82° [2608]; ¹H NMR [541, 703], UV [703], MS [703]; HPLC [3481]; TLC [703].

BIOLOGICAL ACTIVITY: As bone resorption inhibitor for treatment of bone diseases [2686]; for the prevention and treatment of bone and cartilage diseases [2685].

[121426-03-7] $C_{42}H_{42}O_{7}$ Tribenzoate mol. wt. 658.79 m.p. 138–139° [1401, 2609], 127° [702].

3-Methyl-1-[2,4,6-trihydroxy-3-(3,7-dimethyl-2,6-octanediene)phenyl]-1-butanone

3-Geranyl-1-(3-methylbutanoyl)phloroglucinol

[144785-80-8] $C_{21}H_{30}O_4$

CH₃ ÇH₃ CH₃-C=CHCH₂CH₂-C=CHCH₂ COCH₂CH(CH₃)₂ HO

-From the leaves of *Hypericum styphelloides* (Clusiaceae) [1083].

-From the leaves of *Esenbeckia nesiotica* Stand. (Rutaceae) [2625].

-From Helichrysum species (monticola and anomalym) [1488].

-From the aerial parts of Helichrysum stenopterum [1487].

-From the aerial parts of Helichrysum platypterum [1487].

-From the aerial parts of Achyrocline alata [406].

-From Helichrysum infaustum [401].

-Also refer to: [1782, 3269].

¹H NMR [2625, 3269], ¹³C NMR [169, 2625, 3269], IR [3269], MS [2625, 3269].

BIOLOGICAL ACTIVITY: Antioxidant (human skin fibroblasts) [169].

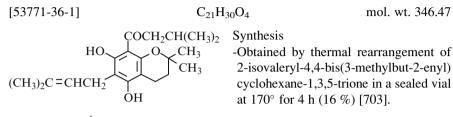
mol. wt. 346.47

Isolation from natural sources -From the aerial parts of Hypericum jovis [169].

Triacetate	[144785-84-2]	$C_{27}H_{36}O_{7}$	mol. wt. 472.58
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-Refer to: [2625]; ¹H NMR [2625].

3-Methyl-1-(3,4-dihydro-5,7-dihydroxy-2,2-dimethyl-6-(3-methyl-2-butenyl)-2H-1-benzopyran-8-yl)-1-butanone



oil [703]; ¹H NMR [703], UV [703], MS [703]; TLC [703]; GLC [703].

1-[2,6-Dihydroxy-4-methyl-3,5-bis(3-methyl-2-butenyl)phenyl]-3-methyl-1-butanone

[216300-96-8] C₂₁H₃₂O₃ mol. wt. 332.48 Synthesis $COCH_2CH(CH_3)_2$ -Refer to: [2684]. $(CH_3)_2C = CHCH_2.$ BIOLOGICAL ACTIVITY: For the prevention and treatment of $H_2CH = C(CH_3)_2$ bone and cartilage diseases [2684].

3-Methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)-5-(3-methylbutyl)phenyl]-1-butanone

(Prehumulone)

[93796-34-0]

 $C_{21}H_{32}O_4$

 $\begin{array}{c|c} COCH_2CH(CH_3)_2 & -Obtained \\ \hline \\ OH & of 2,4,0 \end{array}$ (CH₃)₂C=CHCH₂. H₂CH₂CH(CH₃)₂

mol. wt. 348.48

Syntheses by adding boron trifluoride etherate to a solution 2,4,6-trihydroxy-3-isopentylisovalerophenone and 2-methyl-3-buten-2-ol in dioxane and stirring the mixture at 20° for 7 h (22 %) [542].

-Also obtained by reaction of 4-bromo-2-methyl-2-butene with phlor-4-methylpentanophenone in the presence of potassium hydroxide in dilute methanol first at 0°, then at r.t. for 25 h (4 %) [2624].

oil [542]; yellow oil [2624];

¹H NMR [542], UV [542], MS [542].

mol. wt. 346.47

1-[2,4-Dihydroxy-3,5-bis(3-methylbutyl)phenyl]-3-methyl-1-butanone

mol. wt. 334.50

Obtained by perhydrogenation of (-) tetrahydrohumulone in methanol in the presence of 5 % PtO2 (Adam's catalyst) and 2 % Pt as chloroplatinic acid (35–40 %) [828].

-Also refer to: [829].

white crystals [828]; m.p. 70–70.5° [828]; ¹H NMR [828, 829], IR [828], UV [828].

3-Methyl-1-[2,4,6-trihydroxy-3,5-bis(3-methylbutyl)phenyl]-1-butanone (4-Deoxytetrahydrohumulone), (3,5-diisoamyl-phloroisovalerophenone) (**III**)

[22748-59-0]	$C_{21}H_{34}O_4$	mol. wt. 350.50
(CH ₃) ₂ CHCH ₂ CH ₂ HO		Syntheses -Obtained by hydrogenation of Lupulon (m.p. 93–94°) [557], *in the presence of palladium [811, 2615];

*in the presence of palladium chloride in methanol [557, 1254, 1255, 2613, 3330]. -Also refer to: [898, 1181, 2685, 2686, 3304, 3306, 3330, 3339].

¹H NMR [898], UV [557, 898, 3330].

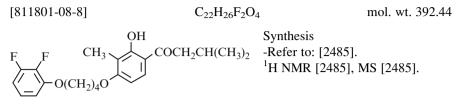
BIOLOGICAL ACTIVITY: As bone resorption inhibitor for treatment of bone diseases [2686]; For the prevention and treatment of bone and cartilage diseases [2685].

Tribenzoate [26104-08-5] C₄₂H₄₆O₇ mol. wt. 662.83

-Obtained by reaction of benzoyl chloride with the title ketone in the presence of pyridine for 3 days at r.t. under CO_2 atmosphere [813]. -Also refer to: [811, 898, 2609, 3330].

m.p. 167–168° [898], 165–166° [2609], 164–165° [3330], 164° [813], 163–164° [811]; UV [898].

1-[4-[4-(2,3-Difluorophenoxy)butoxy]-2-hydroxy-3-methylphenyl]-3-methyl-1-butanone



BIOLOGICAL ACTIVITY: Study of its activity as allosteric potentiator of metabotropic glutamate receptor 2 (mGlu2) [2485].

1-[4-[4-(2-Fluorophenoxy)butoxy]-2-hydroxy-3-methylphenyl]-3-methyl-1-butanone

 $[811801-06-6] C_{22}H_{27}FO_4 mol. wt. 374.45$ OH Synthesis -Refer to: [2485]. $I OH O(CH_2)_4O + I OO(CH_2)_4O + I O(CH_3)_2 OO(CH_3)_4O + I O(CH_3)_4O + I O$

BIOLOGICAL ACTIVITY: Study of its activity as allosteric potentiator of metabotropic glutamate receptor 2 (mGlu2) [2485].

3-Methyl-1-(2,4,6-trihydroxy-3-isopentyl-5-phenylazophenyl)-1-butanone

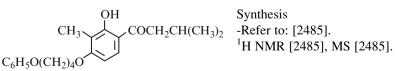
 $[115120-54-2] C_{22}H_{28}N_2O_4 mol. wt. 384.48$ $(CH_3)_2CHCH_2CH_2 \xrightarrow{OH} N=N-\swarrow N$ $HO \xrightarrow{OH} OH COCH_2CH(CH_3)_2$ Synthesis -Refer to: [2610]. m.p. 143° [2610].

1-[2-Hydroxy-3-methyl-4-(4-phenoxybutoxy)phenyl]-3-methyl-1-butanone

[811801-04-4]

C22H28O4

mol. wt. 356.46



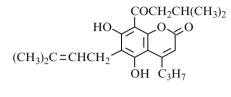
BIOLOGICAL ACTIVITY: Study of its activity as allosteric potentiator of metabotropic glutamate receptor 2 (mGlu2) [2485].

5,7-Dihydroxy-6-(3-methyl-2-butenyl)-8-(3-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one

C22H28O5

(Mammein) (Mammea B/BA)

[521-38-0]



mol. wt. 372.46

Syntheses -Obtained by reaction of prenyl bromide with 5,7-dihydroxy-8-(3-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one in the presence of 10 % aqueous potassium hydroxide at 0° (30–45 %) [765], (23 %) [762].

Isolation from natural sources

-From kernels of the fruit of *Mammea americana* L. and *Mammea africana* (Guttiferae) [765, 1006].

-From only seed of Mammea africana (Guttiferae) [751].

-From the seed of *Mammea americana* L. (Guttiferae) [753, 758, 759, 886, 887, 1001, 1003, 1004, 1007].

-From the bark of Mammea africana G. Don (Guttiferae) [553, 1082].

needles [751, 753]; white needles [762]; transparent prisms [887]; m.p. 128.5–129.5° [887], 128–129° [762], 128° [751], 127° [753, 754, 1006]; ¹H NMR [751, 753, 762], ¹³C NMR [751], IR [751, 753, 762], UV [751, 753, 762, 886, 887], MS [751, 753, 762]; GC-MS [1082].

USE: Insecticide [753, 758, 759, 765, 1001, 1003, 1004].

BIOLOGICAL ACTIVITY: Antitumor [1006].

Hydrochloride	$C_{22}H_{28}O_5$, HCl	mol. wt. 408.96
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-Obtained by treatment of mammein in ethyl ether with hydrogen chloride for 1.5 h (70 %) [887].

m.p. 142–144° [887]; UV [887].

Diacetate	[38789-16-1]	$C_{26}H_{32}O_7$	mol. wt. 456.54
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-Preparation by reaction of acetic anhydride with mammein in the presence of pyridine at r.t. overnight [887].

-Also refer to: [753, 756].

m.p. 118–120° [753, 756], 105–107.5° [887]; IR [753, 756, 887], UV [887]. **Dimethyl ether** [119042-58-9] C₂₄H₃₂O₅ mol. wt. 400.52

-Preparation by treatment of mammein with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 1.5 h (74 %) [887].

-Also obtained by reaction of diazomethane in ethyl ether with mammein in acetone at 0° , then at r.t. for 24 h [887].

m.p. 103–103.5° [887]; UV [887].

5,7-Dihydroxy-8-(3-methyl-2-butenyl)-6-(3-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one

C22H28O5

(Isomammein, Mammea B/AA)

[478-67-1]

 $CH_2CH=C(CH_3)_2$ Syntheses

 $(CH_3)_2CHCH_2CO$ OH C_3H_7

-Obtained by reaction of prenyl bromide with 5,7-dihydroxy-6-(3-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one in the presence of 10 % aqueous potassium hydroxide at 0° (30–45 %) [765], (22 %) [762].

-Also obtained by treatment of 5,7-dihydroxy-6-(3-methyl-2-butenyl)-8-(3-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one (*mammein*) with methanolic potassium hydroxide at r.t. overnight, followed by acidification [886], (61 %) [887].

-Also obtained by saponification of mammein diacetate with 1 N methanolic potassium hydroxide overnight at r.t. [887].

-Also obtained by isomerization of 5,7-dihydroxy-6-(3-methyl-2-butenyl)-8-(3-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one (mammea B/BA) by treatment with methanolic 5 % potassium hydroxide at 20° overnight (80 %) [757]. -Also obtained by treatment of 5,7-dihydroxy-6-(3-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one with 2-methyl-3-buten-2-ol in the presence of boron trifluoride-ether complex [757] in dioxane solution at r.t. for 24 h (2 %) [1081].

Isolation from natural sources

-From kernels of the fruit of *Mammea americana* L. [757, 1007] and *Mammea africana* (Guttiferae) [765].

-From only seed of Mammea africana (Guttiferae) (small amount) [751].

-From Mammea africana G. Don (Guttiferae) [1082].

yellow compound [886]; yellow needles [762]; m.p. 122.5–123° [1081], 119–121° [757], 119–120° [762, 887]; ¹H NMR [751, 757, 762], IR [757, 762, 887], UV [757, 762, 887], MS [751, 757, 762]; GC-MS [1082]; TLC [757].

USE: Insecticide [757, 765].

mol. wt. 372.46

Diacetate

 $C_{26}H_{32}O_7$

mol. wt. 456.54

-Obtained by treatment of isomammein with acetic anhydride in the presence of pyridine at r.t. overnight [887].

colourless crystals [887]; m.p. 98–99° [887]; UV [887].

Dimethyl ether [114002-07-2] C₂₄H₃₂O₅ mol. wt. 400.52

-Preparation by reaction of dimethyl sulfate with isomammein in the presence of potassium hydroxide in refluxing acetone for 25.5 h (57 %) [887].

colourless oil [887]; IR [887], UV [887].

5,7-Dihydroxy-4-(1-hydroxypropyl)-8-(3-methyl-2-butenyl)-6-(3-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one

[38534-76-8]	C ₂₂ H	$_{28}O_{6}$	mol. wt. 388.46
HO (CH ₃) ₂ CHCH ₂ CO	CH ₂ CH=C(CH ₃) ₂ O O OH CH-C ₂ H ₅ OH	5,7-dihydroxy-6 8-(3-methyl-1-ox yran-2-one wa deacylated w	hen treated with potassium hydroxide

-Also refer to: [756].

IR [756, 758], UV [756, 758], MS [756, 758].

Triacetate [38534-63-3] C₂₈H₃₄O₉ mol. wt. 514.57

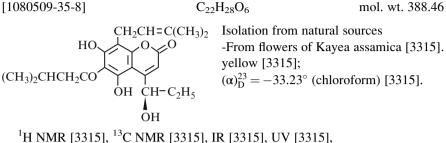
-Obtained by treatment of the titled compound with pyridine-acetic anhydride mixture [758].

-Also refer to: [756].

IR [756, 758], UV [756, 758], MS [758].

5,7-Dihydroxy-4-(1-hydroxypropyl)-8-(3-methyl-2-butenyl)-6-(3-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one (1S)

(Kayeassamin G)



MS [3315].

BIOLOGICAL ACTIVITY: Cytotoxicity [3315].

5,7-Dihydroxy-6-(3-methylbutyl)-8-(3-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one

(Dihydromammein)

[37975-69-2] C22H30O5 mol. wt. 374.48 $COCH_2CH(CH_3)_2$ Syntheses -Obtained by hydrogenation of HO -0 5,7-dihydroxy-6-(3-methyl-2-butenyl)-(CH₃)₂CHCH₂CH₂ 8-(3-methyl-1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one (mammein) [886], C_3H_7 (75 %) [887].

-Also refer to: [1005].

m.p. 133–134.5° [1005], 132–133° [887]; UV [886, 887].

Diacetate [120363-74-8] C₂₆H₃₄O₇ mol. wt. 458.55

-Obtained by hydrogenation of mammein diacetate in the presence of PtO_2 in methanol (90 %) [3014].

-Also obtained by acetylation of dihydromammein with acetic anhydride at r.t. (45 %) [1005].

m.p. 86–87° [887]; IR [887], UV [887].

Dimethyl ether [120363-73-7] $C_{24}H_{34}O_5$ mol. wt. 402.53

-Obtained by hydrogenation of mammein dimethyl ether in the presence of PtO_2 in methanol (76 %) [3014].

-Also obtained by methylation of dihydromammein with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 4 h (86 %) [887].

m.p. 89–90° [887]; UV [887].

5,7-Dihydroxy-8-(3-methylbutyl)-6-(3-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one

(Isodihydromammein) (Dihydroisomammein)

-Could also be obtained by isomerization of 5,7-dihydroxy-6-(3-methylbutyl)-8-(3-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one (dihydromammein) with potassium hydroxide in methanol at r.t. overnight [886], (81 %) [887].

m.p. 122–124° [887]; IR [887], UV [887].

Dimethyl ether [120363-75-9] C₂₄H₃₄O₅ mol. wt. 402.53

-Preparation by reaction of dimethyl sulfate with dihydroisomammein in the presence of potassium carbonate in refluxing acetone [887].

m.p. 82-84° [887]; IR [887], UV [886, 887].

1-[3,4-Dihydro-7-hydroxy-5-(phenylmethoxy)-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-3-methyl-1-butanone

[854465-73-9]	$C_{23}H_{28}O_4$	mol. wt. 368.47
HO HO COCH ₂ CH(CH ₃) ₂ OCH ₃ CH ₃ OCH ₂ C ₆ H ₅	Synthesis -Obtained by reaction of 5,7-dihydroxy-2,2-dimethyl- the presence of potassium ca for 2 h (53 %) [2481].	8-isovaleroylchroman in

prisms or plates [2481]; m.p. 92-93° [2481].

Methyl ether	[854465-70-6]	$C_{24}H_{30}O_4$	mol. wt. 382.50
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-Preparation by reaction of 5-benzyloxy-7-hydroxy-2,2-dimethyl-8-isovaleroylchroman with methyl iodide in the presence of potassium carbonate in boiling acetone during 60 h (92 %) [2481].

rectangular prisms [2481]; m.p. 90–91° [2481].

С

7-Hydroxy-6-(3-methyl-2-butenyl)-5-methoxy-8-(3-methyl-1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one

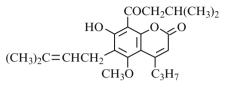
(Mammein monomethyl ether)

-Obtained by reaction of diazomethane

in ethyl ether with mammein in acetone

at 0°, then at r.t. for 24 h (37 %) [887].

Synthesis



pale yellow needles [887]; m.p. 80–82° [887]; IR [887], UV [887].

1-[4-[[6-O-(6-Deoxy-α-L-mannopyranosyl)-β-D-glucopyranosyl]oxy]-2,6-dihydroxy-phenyl]-3-methyl-1-butanone

(Lysidiside D)

[1000210-35-4]
$$C_{23}H_{34}O_{13}$$
 mol. wt. 518.52
OH $P = [6 O (6 \text{ Deoxy } \alpha \text{ L mannopyranosyl}) \beta D$

 $\mathbf{K} = [6 \text{-}O \text{-}(6 \text{-}Deoxy \text{-}\alpha \text{-}L \text{-}mannopyranosyl) \text{-}\beta \text{-}$ COCH₂CH(CH₃)₂ glucopyranosyl]

Isolation from natural sources

-From the roots of Lysidice rhodostegia Hance (family Fabaceae).

yellow powder [1090]; ¹H NMR [1090], ¹³C NMR [1090], IR [1090], m.p. 201–203° [1090]; UV [1090], MS [1090]; TLC [1090]; HPLC [1090]; $(\alpha)_{\rm D}^{25} = -8.5^{\circ}$ (acetone) [1090].

1-[2,4-Bis(β-D-glucopyranosyloxy)-6-hydroxyphenyl]-3-methyl-1-butanone (Lysidiciside B) (Lysidiside B)

[718608-84-5]
$$C_{23}H_{34}O_{14}$$
 mol. wt. 534.51
OH Isolation from natural sources
-From the roots of Lysidice rhodostega
(Fabaceae) [1089, 1091].

Yellow amorphous powder [1089]; m.p. 142–144° [1089]; ¹H NMR [1089], ¹³C NMR [1089], IR [1089], UV [1089], MS [1089]; $(\alpha)_{\rm D}^{25} = -86^{\circ}$ (acetone) [1089].

BIOLOGICAL ACTIVITY: Vasodilator [1089].

$1-[3-\beta-D-Glucopyranosyl-6-(\beta-D-glucopyranosyloxy)-2, 4-dihydroxyphenyl]-3-methyl-1-butanone$

[1000210-34-3]	C ₂₃ H ₃	4O ₁₄	mol. wt. 534.51
β-D-pyranosyl HO	COCH ₂ CH(CH ₃) ₂	Isolation from natural -From the roots of <i>Lyst</i> Hance (family Fabace yellow powder [1090]	<i>idice rhodostegia</i> eae).
m.p. $157-150^{\circ}$ [10 ¹ H NMR [1090], [1090]; HPL0 (α) _D ²⁵ = -25.2° (ac	¹³ C NMR [1090], II C [1090];	R [1090], UV [1090], N	MS [1090]; TLC

USE: Antioxidant [1090].

4-[1-(Acetyloxy)propyl]-5,7-dihydroxy-6-(3-methyl-2-butenyl)-8-(3-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one

(Mammea E/BA)

[98498-81-8] racemic [26477-64-5] [111321-13-2]

 $C_{24}H_{30}O_7$

mol. wt. 430.50

Syntheses

-Obtained by reaction of prenyl bromide with 4-[1-(acetyloxy)propyl]-5,7-dihydroxy-8-(3-methyl-1-oxobutyl)-2*H*-1-benzopyran-2-one in the presence of aqueous potassium hydroxide at 0° (40 %) [766].

-Also refer to: [373, 762].

Isolation from natural sources

-From *Mammea americana* [758, 766]. -From *Mammea africana* seeds [759].

m.p. 50–51° [758]; ¹H NMR [758, 759], IR [758, 759], UV [758, 759], MS [758, 759].

BIOLOGICAL ACTIVITY: Insecticidal [758, 759, 766]; Toxicity [759].

Diacetate [38534-84-8] C₂₈H₃₄O₉ mol. wt. 514.57

-Obtained by treatment of the titled ketone with pyridine-acetic anhydride at 100° temperature overnight [758].

m.p. 20–22° [756, 758]; IR [756, 758], UV [758], MS [756, 758].

Dimethyl ether [26481-09-4]
$$C_{26}H_{34}O_7$$
 mol. wt. 458.55

-Refer to: [756, 758, 759].

CH₃

CH3

m.p. 88–90° [756, 758, 759]; IR [756, 758], UV [756, 758], MS [756, 758].

5-Hydroxy-8,8-dimethyl-6-(3-methyl-1-oxobutyl)-4-phenyl-2*H*;8*H*-pyrano [2,3-f]chromen-2-one

C

(Mammea A/AA cyclo D) (Mammeigin)

[2289-11-4]

(CH₃)₂CHCH₂CO

Isolation from natural sources

-From mamey seed oil of *Mammea americana* L (Guttiferae) [1003].

-From flowers of Kayea assamica [3315].

-From the fruit of *Kilmeyera pumila* Pohl [901]. -From Mammea americana L (Clusiaceae) [1004], seeds (Fruit and Spice Park) [3381].

-From the leaves of Marila pluricostata [1906].

OH

-CO₂ extract of Mesua ferrea L. (Guttiferae) [236, 582, 3219].

 C_6H_5

-From Mammea harmandi (Pierre) Kosterm (Guttiferae), leaves and twigs [2589].

-From Kielmeyera elata [1156].

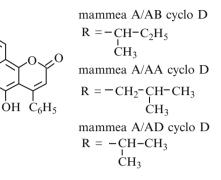
-From Kielmeyera pumila [2218].

-Also refer to: [553, 1006, 1081].

yellow crystalline substance [1003]; m.p. 150–151° [1081], 149–150° [582], 148–150° [2218], 146–147° [1006], 145–146° [553], 144–146° [236, 1003, 1004], 140–142° [2589]; ¹H NMR [553, 1003, 1004, 2218, 3219], ¹³C NMR [3219], IR [553, 1003, 1004, 2218], UV [236, 553, 1003, 1004, 2218], MS [553, 2218].

BIOLOGICAL ACTIVITY: Antiviral [278]; Cytotoxicity [278, 1906, 2589, 3315, 3381].

mixture (composition completely given)	Chemical Name	p. cent	
-mammea A/AB cyclo D	mammeigin	67 %	[3219]
-mammea A/AA cyclo D	mammea A/AA cyclo D	20 %	[3219]
-mammea A/AD cyclo D	mesuagin	13 %	[3219]



BIOLOGICAL ACTIVITY: Antibacterial [3219]; Antiprotozoal [3219].

5-Hydroxy-8-(1-hydroxy-1-methylethyl)-6-(3-methyl-1-oxobutyl)-4-phenyl-2H-furo[2',3':5,6]benzo[1,2-b]pyran-2-one

(Ochrocarpin B)

C ₂₅ H	H ₂₄ O ₆ mol. wt. 420.46
HO CH_3 CH_3 O	Isolation from natural sources -From the bark of <i>Ochrocarpos punctatus</i> H. Perrier (Clusiaceae = Guttiferae) [616, 1884]. viscous liquid [616]; ¹ H NMR [616], ¹³ C NMR [616], IR [616], UV [616]; (α) _D ²⁰ = 0.12° (chloroform) [616].

BIOLOGICAL ACTIVITY: Anticancer [1884]; Against ovarian cancer cells [616, 1884]; Cytotoxicity [616].

3-Methyl-1-[2,4,6-trihydroxy-3,5-bis(phenylmethyl)phenyl]-1-butanone

[198878-91-0] C	₂₅ H ₂₆ O ₄ mol. wt. 390.48
$C_6H_5CH_2$ HO $COCH_2CH(CH_3)_2$ HO $CH_2C_6H_5$	Syntheses -Refer to: [2685, 2686]. BIOLOGICAL ACTIVITY: As bone resorption inhibitor for treatment of bone diseases [2686]; For the prevention and treatment of bone and cartilage diseases [2685].

¹H NMR [2683].

CH₃

CH₃

R-CO

5,7-Dihydroxy-6-(3-methyl-2-butenyl)-8-(3-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one

(Mammea A/BA)

$$[5224-54-4] C_{25}H_{26}O_5 \qquad \text{mol. wt. 406.48}$$

$$COCH_2CH(CH_3)_2 Synthesis$$

$$COCH_2CH(CH_3)_2 Synthesis$$

$$-Obtained by reaction of 3-methyl-2-butenyl bromide with 5,7-dihydroxy-8-(3-methyl-butyryl)-4-phenylcoumarin in the presence of 10 % potassium hydroxide at 0° under nitrogen over 1.5 h (22 %) [762].$$

Isolation from natural sources

-From kernels of the fruit of *Mammea americana* L. [754, 758, 759, 1007] and *Mammea africana* (Guttiferae) [765].

-From the Bark of Mammea africana G. Don (Guttiferae) [553].

white needles [762]; colourless needles [754]; m.p. 125–126° [754], 123–125° [762], 122–124° [2591]; ¹H NMR [754, 762, 2591, 3219], ¹³C NMR [3219], IR [754, 762, 2591], UV [754, 762], MS [754, 762, 2591, 3219].

USE: Insecticide [758, 759, 765].

5,7-Dihydroxy-8-(3-methyl-2-butenyl)-6-(3-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one

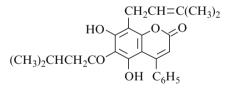
(Mammeisin) (Mammea A/AA)

[18483-64-2]

$$C_{25}H_{26}O_5$$

Syntheses

mol. wt. 406.48



-Obtained by reaction of 3-methyl-2-butenyl bromide with 5,7-dihydroxy-6-(3-methyl-butyryl)-4-phenylcoumarin in the presence of 10 % potassium hydroxide at 0° under nitrogen over 1.5 h (20 %) [762].

-Also obtained by adding 2-methyl-3-buten-2-ol to a solution of 4-phenyl-5,7-dihydroxy-6-isovalerylcoumarin in dioxane and boron trifluoride etherate for 90 min at 50° [236].

Isolation from natural sources

- -From kernels of the fruit of *Mammea americana* L. [754, 758, 759, 1004, 1007] and *Mammea africana* (Guttiferae) [765, 1006].
- -From the peelings of the fruit of Mammea americana L. (Guttiferae) [1003].

-From both bark and seed of Mammea africana (Guttiferae) [751].

- -From the Bark of Mammea africana G. Don (Guttiferae) [553].
- -From Mesua ferrea (Guttiferae) [236].
- -From the fruit peels (pericarpa of Kayea assamica) (Guttiferae) [428].

yellow needles [751, 754, 762]; yellow powdery solid [1007]; yellow substance [1001]; yellow crystalline solid [428]; m.p. 98–109° [1001, 1007], 98–102° [762], 98–100° [2547], 97° [428], 92–112° [236], 89–100° [751], 83–84° [754, 1007], 83° [553]; ¹H NMR [428, 553, 751, 754, 762, 1004, 3219], ¹³C NMR [751, 3219], IR [236, 428, 553, 751, 754, 762, 1007], UV [428, 553, 751, 754, 762, 1001, 1007, 2547], MS [428, 553, 751, 754, 762]; $(\alpha)_{D}^{31} = +22°$ (ethanol) [428].

USE: Insecticide [758, 759, 765, 1003, 1004].

BIOLOGICAL ACTIVITY: Antitumor [1006]; Toxicity [1007].

Diacetate	[27127-44-2]	$C_{29}H_{30}O_7$	mol. wt. 490.55
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-Obtained by reaction of acetic anhydride with 5,7-dihydroxy-8-(3-methyl-2-butenyl)-6-(3-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one in the presence of pyridine (80 %) [1007].

-Also refer to: [278, 1028, 1906, 2547].

m.p. 122–124° [1001, 1007], 118–120° [2547], 86° [754]; ¹H NMR [754, 1906], ¹³C NMR [1906], IR [754, 1007, 1906], UV [1007], MS [754].

BIOLOGICAL ACTIVITY: Antiviral, anti HIV [278]; Cytotoxicity [278, 1906].

Dimethyl ether	[103033-88-1]	$C_{27}H_{30}O_5$	mol. wt. 434.53
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-Obtained by reaction of dimethyl sulfate with 5,7-dihydroxy-8-(3-methyl-2-butenyl)-6-(3-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one in the presence of potassium carbonate in acetone (25 %) [1007].

-Also obtained by treatment of 5,7-dihydroxy-8-(3-methyl-2-butenyl)-6-(3-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one in ethyl ether with diazomethane [1007].

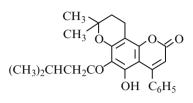
-Also refer to: [1001, 1177].

m.p. 87° [754], 86–89° [1001, 1007]; ¹H NMR [754], IR [754, 1007], UV [754, 1007], MS [754].

9,10-Dihydro-8,8-dimethyl-5-hydroxy-6-(3-methyl-1-oxobutyl)-4-phenyl-8*H*-pyrano-[2,3-*f*]chromen-2-one

(Dihydromammeigin)

[2289-30-7]



C₂₅H₂₆O₅ mol. wt. 406.48

Syntheses

-Obtained by hydrogenation of mammeigin in the presence of Pd-C in ethanol-THF at r.t. and atmosphere pressure [1003].

-Also obtained by treatment of mammeisin in acetic acid containing a trace of H₂SO₄ [1003].

-Also refer to: [760, 1004, 2547].

m.p. 164–165° [1003], 158–159° [2547]; IR [1004, 2547], UV [760, 1004, 2547].

Methyl ether

C₂₆H₂₈O₅

mol. wt. 420.50

-Refer to: [2547]; m.p. 132° [2547].

8,9-Dihydro-5-hydroxy-8-(1-hydroxy-1-methylethyl)-6-(3-methyl-1-oxobutyl)-4-phenyl-2*H*-furo[2',3':5,6]benzo[1,2-b]pyran-2-one

(Mammea A/AA cyclo F) (Cyclomammeisin)

OH C₆H₅

[30563-62-3] [188817-92-7] [111821-09-1] (racemic)

(CH₃)₂CHCH₂CO

HO CH₃

$$C_{25}H_{26}O_6$$
 mol. wt. 422.48

Isolation from natural sources

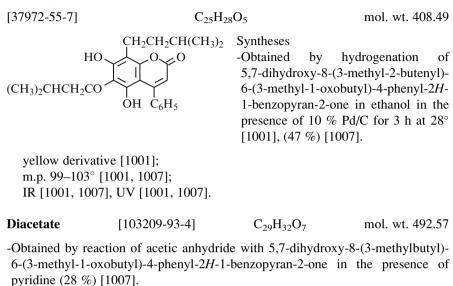
-From the fruit peels (pericarpa of *Kayea assamica*) (Guttiferae), compound **3** [428].

m.p. 131–132° [762], 115–117° [756], 105° [428];

¹H NMR [428, 756, 762],

IR [428, 756, 762], UV [428, 756], MS [428, 756, 762].

5,7-Dihydroxy-8-(3-methylbutyl)-6-(3-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one



m.p. 98–102° [1007]; IR [1007], UV [1007].

1-[4,6-(Diacetyloxy)-2-hydroxy-3-(3,7-dimethyl-2,6-octanediene)phenyl]-3-methyl-1-butanone

[144785-82-0]	C ₂₅	5H ₃₄ O ₆	mol. wt. 4	430.54
CH ₃ CH ₃ -C=CHCH ₂ CH ₂		OH COCH ₂ CH(CH ₃) ₂	Isolation natural sour -Refer to: [2	

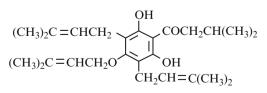
¹H NMR [2625].

1-[2,6-Dihydroxy-3,5-bis(3-methyl-2-butenyl)-4-(3-methyl-2-butenyloxy) phenyl]-3-methyl-1-butanone

(Lupulone)

$$C_{26}H_{38}O_4$$

mol. wt. 414.58



Syntheses -Obtained by reaction of prenyl bromide with 2,6-dihydroxy 3-prenyl-4-prenyloxyisovalerophenone in the presence of sodium ethoxide [1254].

-Also obtained by treatment of isovalerylphloroglucinol with 3-methyl-1-chloro-2-butene in sodium methoxide-methanol (24.5 %) [3120].

1-[2-Hydroxy-6-methyl-3,5-bis(3-methyl-2-butenyl)]-4-[(3-methyl-2-butenyloxy)phenyl]-3-methyl-1-butanone

[198879-01-5] C₂₇H₄₀O₃ mol. wt. 412.61 Synthesis CH_3 $CH_3-C=CHCH_2$ \sim COCH₂CH(CH₃)₂ -Refer to: [2685]. CH₃ CH₂CH=C-CH₃ BIOLOGICAL ACTIVITY: For CH₃-C=CHCH₂O the prevention and treatment CH₃ of bone and cartilage diseases [2685].

1-[3,4-Dihydro-7-hydroxy-5-(phenylmethoxy)-6-(phenylmethyl)-2,2-dimethyl-2H-1-benzopyran-8-yl]-3-methyl-1-butanone

$$C_{30}H_{34}O_4$$
 mol. wt. 458.60

COCH₂CH(CH₃)₂ Synthesis -Obtained by reaction of benzyl bromide with CH₃ HO CH₃ 5,7-dihydroxy-2,2-dimethyl-8-isovaleroylchroman in the presence of potassium carbonate in boiling acetone for 2 h [2481].

long, thin, pale greenish prisms [2481]; m.p. 117° [2481].

1-[3,5-Bis(3,7-dimethyl-2,6-octadienyl)-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone

[198878-75-0] $CH_{3}-C=CHCH_{2}CH_{2}-C=CHCH_{2}$ CH_{3} CH_{3} CH_{3} CH_{3} CH_{3} $CH_{2}CH=C-CH_{2}CH_{2}CH=C-CH_{3}$ CH_{3} CH_{3} C[198878-75-0] C31H46O4 mol. wt. 482.70 Syntheses -Refer to: [2685, 2686].

BIOLOGICAL ACTIVITY: As bone resorption inhibitor for treatment of bone diseases [2686]; As preventive and therapeutic agent for bone and cartilage diseases [2685].

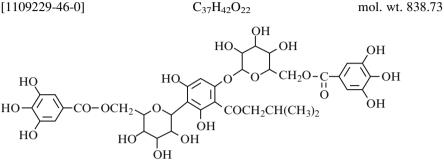
C₆H₅CH₂

249

1-[2,4-Dihydroxy-3-[6-O-(3,4,5-trihydroxybenzoyl)-β-D-glucopyranosyl]-6-[[6-O-(3,4,5-trihydroxybenzoyl)-β-D-glucopyranosyl]oxy]phenyl]-3-methyl-1-butanone

(Kunzeaphlogin B)

[1109229-46-0]



Isolation from natural sources

-From the leaves of Kunzen ambigua (Myrtaceae) [1612].

pale yellow amorphous powder [1612]; ¹H NMR [1612], ¹³C NMR [1612], UV [1612], MS [1612]; $(\alpha)_{\rm D}^{23} = -45^{\circ}$ (methanol) [1612].

From 3,3-Dimethyl-1-Butanoic Acid 2.4

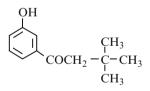
2.4.1 Unsubstituted Hydroxyketones

1-(3-Hydroxyphenyl)-3,3-dimethyl-1-butanone

[940307-82-4]

C₁₂H₁₆O₂

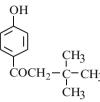
mol. wt. 192.26



Synthesis -Refer to: [1465].

1-(4-Hydroxyphenyl)-3,3-dimethyl-1-butanone

[14392-74-6] C12H16O2 mol. wt. 192.26



Syntheses -Obtained by Fries rearrangement of phenyl 3,3-dimethylbutyrate in the presence of BF_3 -H₂O in a closed pressure tube at 80° for 1 h (88 %) [2514].

 \dot{C} - CH₃ - Also obtained by treatment of its methyl ether with pyridinium chloride at 190° [627]. -Also refer to: [414, 415].

m.p. 102–105° [414], 105° [415]; ¹H NMR [2514], ¹³C NMR [2514], MS [2514].

Methyl ether	[85157-92-2]	$C_{13}H_{18}O_2$	mol. wt. 206.28
wiemyr emer	[05157-92-2]	$C_{13}I_{18}O_2$	mon. wt. 200.20

-Obtained by reaction of 3,3-dimethylbutanoic acid with anisole in the presence of PPA at 80° (76–100 %) [627].

-Also obtained by reaction of 3,3-dimethylbutanoyl chloride with anisole [549] in the presence of aluminium chloride in methylene chloride at 0° (76–100 %) [627]. -Also obtained from a mixture of palladium(II) chloride and 4-methoxy-β-tertbutylstyrene in aqueous N,N-dimethylformamide at 100° until completion shown by TLC [1096].

-Also refer to: [963, 1227, 1680].

b.p.₁ 130–132° [549]; ¹H NMR [549, 1096, 1680], ¹³C NMR [1680], MS [1227].

1-(2,4-Dihydroxyphenyl)-3,3-dimethyl-1-butanone

[1204737-56-3]	$C_{12}H_{16}O_3$	mol. wt. 208.26
HO COCH ₂ -C-CH ₃ COCH ₂ -C-CH ₃ CH ₃	Synthesis -Refer to: [1672]. ¹ H NMR [1672].	

1-(2,4,6-Trihydroxyphenyl)-3,3-dimethyl-1-butanone

mol. wt. 224.26 $C_{12}H_{16}O_4$

 $\begin{array}{c} CH_3 \\ COCH_2 - C - CH_3 \\ CH_3 \end{array}$ $\begin{array}{c} Synthesis \\ -Obtained by reaction of tert-butylacetyl chloride \\ with phloroglucinol in the presence of aluminium \\ \hline \end{array}$ chloride in nitrobenzene at 60° (70 %) [1884].

yellow solid [1884]; ¹H NMR [1884], ¹³C NMR [1884], MS [1884].

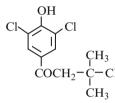
2.4.2 Substituted Hydroxyketones

1-(2,3-Dichloro-4-hydroxyphenyl)-3,3-dimethyl-1-butanone

 $[55507-71-6] C_{12}H_{14}Cl_2O_2 mol. wt. 261.15$ $Cl Cl COCH_2 - C-CH_3 CH_3 - Refer to: [739].$ $HO COCH_2 - C-CH_3 CH_3 - Refer to: [739].$

1-(3,5-Dichloro-4-hydroxyphenyl)-3,3-dimethyl-1-butanone

 $C_{12}H_{14}Cl_2O_2$ mol. wt. 261.15



CH₃

Synthesis -Obtained by treatment of 2,6-dichlorophenyl trimethylacetate, first in carbon disulfide, then at 135–145° for 2 h CH₃ after carbon disulfide elimination (28 %) [3078]. C-CH₃ m.p. 94–95.5° [3078].

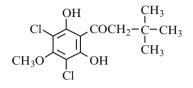
1-(2-Chloro-4-hydroxyphenyl)-3,3-dimethyl-1-butanone

1-(2,4-Dihydroxy-3-iodophenyl)-3,3-dimethyl-1-butanone

[1204737-68-7]	$C_{12}H_{15}IO_3$	mol. wt. 334.15
$HO \overset{OH}{\underset{HO}{\overset{OH}{\overset{CH_3}{\overset{H}{\overset{CH_3}{\overset{H}{\overset{CH_3}{\overset{H}{\overset{CH_3}{\overset{H}{\overset{H}{\overset{CH_3}{\overset{H}{\overset{H}{\overset{H}{\overset{H}{\overset{H}{\overset{H}{\overset{H}}{\overset{H}}{\overset{H}{}}{\overset{H}{}}{\overset{H}{\overset{H}{}}}{\overset{H}{\overset{H}{}}}{\overset{H}}{\overset{H}}{\overset{H}{}}}}{\overset{H}}{\overset{H}}}}}}}}$	Syntheses -Refer to: [1672]. ¹ H NMR [1672].	

1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-3,3-dimethyl-1-butanone (DIF-1)(3M)

[861889-83-0]
$$C_{13}H_{16}Cl_2O_4$$
 mol. wt. 307.17
OH CH₃ Syntheses



-Preparation by adding a solution of sulfuryl chloride (1.5 equiv.) in ethanol to a solution of 1-(2,6-dihydroxy-4-methoxyphenyl)-3.3-dimethyl-1-butanone in chloroform at r.t.: then, the solution was stirred for 1 h at r.t. [1129].

-Also refer to: [1772, 1773, 1777].

Yellow amorphous solid [1129]; MS [1129].

BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773]; Dictyostelium differentiation-inducing factor-1 derivs. promotion of glucose consumption in mammalian cells [1777]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

1-(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)-3,3-dimethyl-1-butanone (DIF-3)(3M)

 $\begin{array}{c|c} OH & CH_3 & Syntheses \\ Cl & COCH_2 - C - CH_3 & CH_3 & CH_3 \\ CH_3 & CH_3 & CH_3 & CH_3 \end{array}$

[861889-91-0]

C13H17ClO4 mol. wt. 272.73

tion of 1-(2,6-dihydroxy-4-methoxyphenyl)-3,3-dimethyl-1-butanone in chloroform at r.t., then, the solution was stirred for 1 h at r.t. [1129].

-Also refer to: [1772, 1777].

Yellow amorphous solid [1129]; MS [1129].

BIOLOGICAL ACTIVITY: Dictyostelium differentiation-inducing factor-1 derivs. promotion of glucose consumption in mammalian cells [1777]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cellinducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

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1-(4-Hydroxy-3-methylphenyl)-3,3-dimethyl-1-butanone

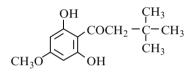
¹H NMR [218], ¹³C NMR [218], IR [218], MS [218].

1-(2,6-Dihydroxy-4-methoxyphenyl)-3,3-dimethyl-1-butanone

[861889-78-3]

 $C_{13}H_{18}O_4$

mol. wt. 238.28



OH CH_3 COCH₂ $-C-CH_3$ Synthesis $COCH_2 -C-CH_3$ -Preparation by reaction of 3,3-dimethylbutyryl CH₃ chloride with phloroglucinol monomethyl ether in the presence of aluminium chloride in methylene chloride at r.t. [1129].

BIOLOGICAL ACTIVITY: Structural requirements of Dictyostelium for their stalk-cell-inducing differentiation-inducing factors activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

1-(4-Ethyl-2,5-dihydroxyphenyl)-3,3-dimethyl-1-butanone

mol. wt. 236.31 $C_{14}H_{20}O_3$ OH CH_3 Synthesis $COCH_2 - C - CH_3$ -Refer to: [2441]. CH_3 CH_3 $C_{16}H_{24}O_3$ mol mol. wt. 264.37

Obtained by acylation of 2-ethyl-1,4-dimethoxybenzene with 3,3-dimethylbutanoic acid in the presence of trifluoroacetic anhydride [2441].

1-(2,4-Dihydroxy-3-propylphenyl)-3,3-dimethyl-1-butanone

[194855-72-6] $C_{15}H_{22}O_3$ mol. wt. 250.34 OH CH_3 Syntheses C_3H_7 $COCH_2$ C^-CH_3 CH_3 -Refer to: [18, 20, 21, 23, 307, 1534]. CH_3 1H NMR [21].

5,7-Dihydroxy-4-ethyl-6-(3,3-dimethyl-1-oxobutyl)-2H-1-benzopyran-2-one

$$\begin{array}{c} C_{17}H_{20}O_5 \\ HO \\ CH_3 \\ CH_5 \\$$

canary-yellow solid [1884]; m.p. 231–234° [1884]; ¹H NMR [1884], ¹³C NMR [1884], IR [1884], MS [1884]; TLC [1884].

BIOLOGICAL ACTIVITY: Inhibition of growth (Escherichia coli, Staphylococcus aureus. Klebsiella pneumoniae, Pseudomonas aeruginosa, Salmonella typhimurium) [650].

1-[2-(1,1-Dimethylethyl)-6-hydroxy-5-benzofuranyl]-3,3-dimethyl-1-butanone

 $CH_3 - C - CH_2CC$

CH₃ C-CH₃ C-CH₃ C-CH₃ CH₃ Synthesis -Obtained by treatment of 2,4-bis(tert-butylethynyl)-1,5-diacetoxybenzene with NaOH (6 equiv.) in THF/MeOH/H₂O at $^{\circ 0^{\circ}}$ (31 %) [1875].

mol. wt. 288.39

¹H NMR [1875], ¹³C NMR [1875], IR [1875].

1-[2-Hydroxy-4-(phenylmethoxy)-3-propylphenyl]-3,3-dimethyl-1-butanone

[194855-33-9] mol. wt. 340.46 C22H28O3 $C_{3}H_{7}$ $COCH_{2}$ C_{7} CH_{3} $COCH_{2}$ C_{7} $C-CH_{3}$ CH_{3} CH_{1} CH_{3} CH_{1} CH_{2} CH_{3} CH_{3} CH

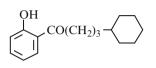
 $C_{18}H_{24}O_{3}$

2.5 From 4-Cyclohexyl-1-Butanoic Acid

2.5.1 Unsubstituted Hydroxyketones

4-Cyclohexyl-1-(2-hydroxyphenyl)-1-butanone

$$C_{16}H_{22}O_2$$
 mol. wt. 246.35



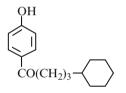
Synthesis -Obtained by Fries rearrangement of phenyl cyclohexylbutyrate with aluminium chloride at 140° [3116]. b.p._{1.5} 158° [2513]. b.p.₅ 220° [3116].

2,4-Dinitrophenylhydrazone [25804-67-5] C₂₂H₂₆N₄O₅ mol. wt. 426.47 m.p. 237° [2513], 125° [3116].

4-Cyclohexyl-1-(4-hydroxyphenyl)-1-butanone

[1760-65-2]

[25804-66-4]

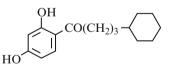


Synthesis -Obtained by Fries rearrangement of phenyl cyclohexylbutyrate with aluminium chloride at 110° (60 %) [3115]. m.p. 136–137° [2513], 137° [3115], 153–154° [2927]; IR [2927].

2,4-Dinitrophenylhydrazone [25804-69-7] C₂₂H₂₆N₄O₅ mol. wt. 426.47 m.p. 110° [3115], 127° [2513].

4-Cyclohexyl-1-(2,4-dihydroxyphenyl)-1-butanone

[709032-85-9] C₁₆H₂₂O₃



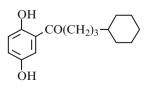
Synthesis -Obtained by reaction of 4-cyclohexylbutyric acid on resorcine in the presence of boron trifluoride [2036]. m.p. 98–107° [2036]

mol. wt. 262.35

4-Cyclohexyl-1-(2,5-dihydroxyphenyl)-1-butanone

[408310-63-4]

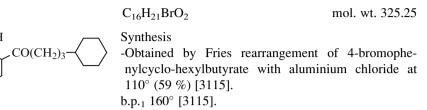
C₁₆H₂₂O₃ mol. wt. 262.35



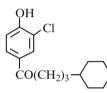
Synthesis -Obtained by reaction of 4-cyclohexylbutyric acid on hydroquinone in the presence of boron trifluoride [2036]. m.p. 109–110° [2036]

2.5.2 Substituted Hydroxyketones

4-Cyclohexyl-1-(5-bromo-2-hydroxyphenyl)-1-butanone



4-Cyclohexyl-1-(3-chloro-4-hydroxyphenyl)-1-butanone



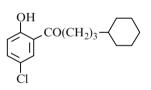
Synthesis								
Obtained	by	Fries	rear	rrangement	of	2-chlo	orop	henyl
cyclohexy	lbuty	rate	with	aluminium	ch	loride	at	110°
(50 %) [31	15].							
o.p. ₅ 230° [3115	5].						

2,4-Dinitrophenylhydrazone	C ₂₂ H ₂₅ ClN ₄ O ₅	mol. wt. 460.92
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m.p. 135° [3115].

4-Cyclohexyl-1-(5-chloro-2-hydroxyphenyl)-1-butanone

C ₁₆ H ₂₁ ClO ₂	mol. wt. 280.79
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Synthesis -Obtained by Fries rearrangement of 4-chlorophenyl cyclohexylbutyrate with aluminium chloride at 110° (52 %) [3115]. b.p.₁ 195° [3115].

4-Cyclohexyl-1-(2-hydroxy-4-methylphenyl)-1-butanone

[132180-62-2]	$C_{17}H_{24}O_2$	mol. wt. 260.38
OH CH ₃ CO(CH ₂) ₃	Synthesis -Refer to: [2036]. b.p. _{0.2} 160° [2036];	m.p. 29.5–30° [2036].

4-Cyclohexyl-1-(2-hydroxy-5-methylphenyl)-1-butanone

[25804-40-4] C17H24O2 mol. wt. 260.38 **Synthesis** CO(CH₂)₂ -Obtained by Fries rearrangement of 4-methylphenyl cyclohexylbutyrate with aluminium chloride at 110° (64 %) [3115]. ĊH3

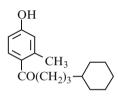
b.p.₃ 185° [2513], b.p.₃ 193° [3115].

[25804-41-5] C₂₃H₂₈N₄O₅ 2,4-Dinitrophenylhydrazone mol. wt. 440.50

m.p. 180° [3115], 174–175° [2513].

4-Cyclohexyl-1-(4-hydroxy-2-methylphenyl)-1-butanone

[132180-63-3]



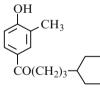
Synthesis -Obtained by Fries rearrangement of 3-methylphenyl cyclohexylbutyrate with aluminium chloride at 110° (55 %) [3115]. b.p.₂ 196–197° [3115].

2,4-Dinitrophenylhydrazone
$$C_{23}H_{28}N_4O_5$$
 mol. wt. 440.50

m.p. 160° [3115].

4-Cyclohexyl-1-(4-hydroxy-3-methylphenyl)-1-butanone

mol. wt. 260.38 [25804-38-0] C₁₇H₂₄O₂



Svnthesis -Obtained by Fries rearrangement of 2-methylphenyl cyclohexylbutyrate with aluminium chloride at 110° (45 %) [3115]. b.p.₁ 146° [2513]; m.p. 145° [3115].

mol. wt. 274.40

2,4-Dinitrophenylhydrazone [25804-39-1] C₂₃H₂₈N₄O₅ mol. wt. 440.50 m.p. 241° [2513], 201° [3115].

4-Cyclohexyl-1-(2-hydroxy-3,4-dimethylphenyl)-1-butanone

[855620-89-2] C18H26O2 Synthesis $CO(CH_2)_3$ -Refer to: [2036]. m.p. 38-40° [2036]. CH₃

4-Cyclohexyl-1-(2-hydroxy-4,6-dimethylphenyl)-1-butanone

[854866-87-8] C18H26O2 mol. wt. 274.40 Synthesis CO(CH₂)₃ -Refer to: [2036]. m.p. 45;2–46° [2036]. CH

4-Cyclohexyl-1-(4-hydroxy-2,3-dimethylphenyl)-1-butanone

C18H26O2 mol. wt. 274.40 CH₃ Synthesis -Refer to: [2036]. CO(CH₂)₃ CH₃ m.p. 77.7–78.4° [2036]. HO

4-Cyclohexyl-1-(2-ethyl-4-hydroxyphenyl)-1-butanone

mol. wt. 454.53

C2H5 CO(CH₂)₃

Synthesis -Obtained by Fries rearrangement of 3-ethylphenyl cyclohexylbutyrate with aluminium chloride at 110° (50 %) [3115]. b.p.7 220° [3115].

 $C_{24}H_{30}N_4O_5$

2,4-Dinitrophenylhydrazone

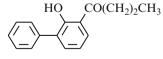
m.p. 155° [3115].

Aromatic Hydroxyketones Derived from Diphenyle 3

3.1 Unsubstituted Hydroxyketones

1,1'-(2-Hydroxy[1,1'-biphenyl]-3-yl)-1-butanone

$$C_{16}H_{16}O_2$$
 mol. wt. 240.30

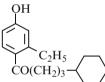


Syntheses -Obtained by Fries rearrangement of 2-(butyryloxy)-biphenyl with aluminium chloride at 160° for 30-45 min (15 %) [1257].

-Also refer to: [661].

b.p.3 5 185–190° [1257].





1,1'-(2-Hydroxy[1,1'-biphenyl]-5-yl)-1-butanone

[95185-62-9]	$C_{16}H_{16}O_2$	mol. wt. 240.30
HO CO(CH ₂) ₂ CH ₃	2-(butyryloxy)-bip at 160° for 30–45 -Also obtained by t	Fries rearrangement of ohenyl with aluminium chloride min (40 %) [1257]. reatment of its methyl ether with de for 1 h at reflux [504].

-Also refer to: [661, 2704].

shiny colourless leaflets [504]; m.p. 123° [504], 122° [2704], 116–117° [1257].

USE: As colour developer [2704].

Methyl ether	[854870-93-2]	$C_{17}H_{18}O_2$	mol. wt. 254.33
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-Preparation by reaction of butyryl chloride with 2-methoxydiphenyl in the presence of aluminium chloride in carbon disulfide for 4 h at r.t. (90 %) [504].

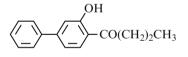
colourless leaflets [504]; b.p.₁₈ 243–245° [504]; m.p. 64° [504].

Semicarbazone of the methyl ether	$C_{18}H_{21}N_3O_2$	mol. wt. 311.38
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m.p. 186° [504].

1,1'-(3-Hydroxy[1,1'-biphenyl]-4-yl)-1-butanone

[131252-71-6] C ₁₆ H ₁₆	O ₂ mol. wt. 240.30
---	--------------------------------



-Preparation by reaction of butyryl chloride with CO(CH₂)₂CH₃ 3-methoxybiphenyl in the presence of aluminium chloride in refluxing methylene chloride (59 %) [443].

m.p. 80.5–81° [443].

1,1'-(3'-Hydroxy[1,1'-biphenyl]-4-yl)-1-butanone

C₁₆H₁₆O₂

mol. wt. 240.30

Synthesis -Refer to: [443]. -CO(CH₂)₂CH₃

Synthesis

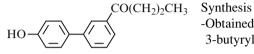
Methyl ether $C_{17}H_{18}O_2$ mol. wt. 254.33

-Obtained by reaction of butyryl chloride with 3-methoxybiphenyl in the presence of aluminium chloride in ethylene chloride at 35° for 45 min (64 %) [443].

irregular white plates [443]; m.p. 62–63° [443].

1,1'-(4'-Hydroxy[1,1'-biphenyl]-3-yl)-1-butanone

$C_{16}H_{16}O_2$ mol. wt. 240.30



-Obtained by diazotization of 4'-amino-3-butyryl-diphenyl [1879].

¹H NMR [1879], IR [1879], MS [1879].

Methyl ether

$$C_{17}H_{18}O_2$$
 mol. wt. 254.33

-Refer to: [1879].

¹H NMR [1879], IR [1879], MS [1879].

1,1'-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-1-butanone

[32339-35-8] $C_{16}H_{16}O_2$ mol. wt. 240.30 HO \leftarrow \leftarrow $CO(CH_2)_2CH_3$ Syntheses - Obtained by Eries rearrangement of

-Obtained by Fries rearrangement of 4-butyryl-oxybiphenyl with aluminium chloride in nitrobenzene, first at 20° for 12 h, then at 60° for 1 h [522].

-Also obtained by diazotization of 4'-amino-3-butyryldiphenyl [1879]. -Also refer to: [514].

prisms colourless [522]; m.p. 165° [514, 522]; ¹H NMR [1879], IR [1879], MS [1879].

Methyl ether	[32339-34-7]	$C_{17}H_{18}O_2$	mol. wt. 254.33
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-Refer to: [847, 1879].

¹H NMR [1879], IR [1879], MS [1879].

1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-2-methyl-1-butanone (S)

[120837-08-3]	C ₁₇	$H_{18}O_2$		mol. wt. 254.33
но-	CH-C ₂ H ₅ CH ₃		is o: [1752]. .5–98.5° [1457].	
Methyl ether (S)	[112231-64-8	8]	$C_{18}H_{20}O_2$	mol. wt. 268.36
Methyl ether	[132041-56-6	6]	$C_{18}H_{20}O_2$	mol. wt. 268.36
-Refer to: [1752].				
10-Undecenyl ether (S)	[175797	-78-1]	$C_{28}H_{38}O_2$	mol. wt. 406.61
-Refer to: [1752].				

1,1'-(3,5,2',4',6'-Pentahydroxy[1,1'-biphenyl]-4-yl)-3-methyl-1-butanone

[26103-99-1]	$C_{17}H_{18}$	₃ O ₆ mol. wt. 318.33
но-	OH COCH ₂ CH(CH ₃) ₂ OH	Synthesis -Obtained by reaction of isovaleryl chloride with phloroglucinol in the presence of aluminium chloride in nitrobenzene at 0° for 3 days [898].

Monohydrate

 $C_{17}H_{18}O_6, H_2O$

mol. wt. 336.34

-Refer to: [898] (X) (smaller amounts).

m.p. 256° (d) [898]; ¹H NMR [898], UV [898].

3.2 Substituted Hydroxyketones

1,1'-(3-Chloro-2-hydroxy[1,1'-biphenyl]-5-yl)-1-butanone

C	$C_{16}H_{15}ClO_2$		mol. wt. 274.75
HO CO(CH ₂) ₂ CH ₃	Synthesis -Refer to: [502 Methyl ether	-	mol. wt. 288.77

-Obtained by reaction of butyryl chloride with 3-chloro-2-methoxybiphenyl in the presence of aluminium chloride in carbon disulfide. After 12 h standing at r.t., the mixture was heated on a water bath (69 %) [502].

oil [502]; b.p.₁₅ 230° [502].

1-[4'-Fluoro-4-hydroxy-6-methoxy[1,1'-biphenyl]-3-yl]-1-butanone

[152609-12-6]	$C_{17}H_{17}FO_3$	mol. wt. 288.32
$F \longrightarrow CO(CH_2)_2CH_3$ $F \longrightarrow OH$ CH_3O	Syntheses -Refer to: [94, 233]. BIOLOGICAL ACTIVITY: antagonist for the treatment of Alzheimer' disease [94].	

1-[4-(3-Chloropropoxy)-4'-fluoro-6-hydroxy[1,1'-biphenyl]-3-yl]-1-butanone

$C_{19}H_{20}ClF$	O ₃	mol. wt. 350.82
$F \longrightarrow CO(CH_2)_2CH_3$ $F \longrightarrow OCH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2$	Synthesis -Refer to: [94]. Methyl ether C ₂₀ H ₂₂ ClFO ₃	[152609-14-8] mol. wt. 364.84

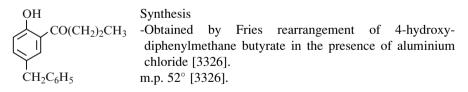
BIOLOGICAL ACTIVITY: As leukotriene antagonist for the treatment or prevention of Alzheimer' disease [94].

4 **Aromatic Hydroxyketones Derived** from Diphenylmethane

Unsubstituted Hydroxyketones 4.1

1-[2-Hydroxy-5-(phenylmethyl)phenyl]-1-butanone

C17H18O2 mol. wt. 254.33



1-[4-Hydroxy-3-(phenylmethyl)phenyl]-1-butanone

C₁₇H₁₈O₂ mol. wt. 254.33



Synthesis CH₂C₆H₅ -Obtained by Fries rearrangement of 2-hydroxydiphenylmethane butyrate with aluminium chloride in nitrobenzene, first at r.t. overnight, then between 50 and 60° for 4 h [3326]. m.p. 142° [3326].

Methyl ether

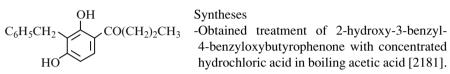
C₁₈H₂₀O₂

mol. wt. 268.36

-Obtained by reaction of dimethyl sulfate with 5-butyryl-2-hydroxydiphenylmethane in the presence of sodium hydroxide in dilute ethanol [3326].

m.p. 106° [3326].

1-[2,4-Dihydroxy-3-(phenylmethyl)phenyl]-1-butanone

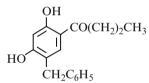


-Also obtained by reaction of 2-benzylresorcinol with butyronitrile (Hoesch reaction) (11 %) [2181].

Synthesis

m.p. 140–142° [2181].

1-[2,4-Dihydroxy-5-(phenylmethyl)phenyl]-1-butanone



Synthesis

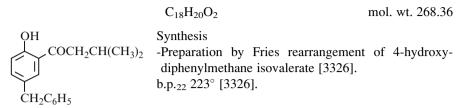
1-[2,4,6-Trihydroxy-3-(phenylmethyl)phenyl]-1-butanone

C17H18O4 mol. wt. 286.33

 $\begin{array}{c} \text{CO}(\text{CH}_2)_2\text{CH}_3 \\ \text{OH} \end{array} \qquad \begin{array}{c} \text{Oyntheod} \\ \text{-Obtained by adding cuprous chloride, a saturated aqueous solution of sodium carbonate and benzyl chloride to a solution of phlorobu$ tyrophenone in ethyl ether at r.t., then to keep the mixture for 12 h at this temperature [1026].

¹H NMR [1026], ¹³C NMR [1026], IR [1026].

1-[2-Hydroxy-5-(phenylmethyl)phenyl]-3-methyl-1-butanone

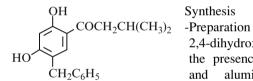


1-[4-Hydroxy-3-(phenylmethyl)phenyl]-3-methyl-1-butanone

$C_{18}H_{20}O_2$			mo	l. wt. 268.36		
$CH_2C_6H_5$	Synthesis -Preparation diphenylmet m.p. 121° [33	hane		rearrangement rate [3326].	of	2-hydroxy-

1-[2,4-Dihydroxy-5-(phenylmethyl)phenyl]-3-methyl-1-butanone

C₁₈H₂₀O₃ mol. wt. 284.36



Synthesis -Preparation by Fries rearrangement of 2,4-dihydroxy-diphenylmethane diisovalerate in the presence of 2,4-dihydroxydiphenylmethane and aluminium chloride in nitrobenzene (78 %) [3326].

m.p. 115° [3326].

4.2 Substituted Hydroxyketones

1-[2-Hydroxy-4-(phenylmethoxy)-3-(phenylmethyl)phenyl]-1-butanone

Synthesis

CO(CH₂)₂CH₃ -Obtained by reaction of benzyl chloride with resbutyrophenone in the presence of potassium hydroxide in methanol, first at r.t. overnight, then at reflux for 5 h (7 %) [2181].

 $C_6H_5CH_2$ $CO(CH_2)_2CH_3$ $C_6H_5CH_2O$

m.p. 108–109° [2181].

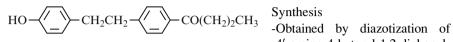
5 **Aromatic Hydroxyketones Derived** from 1,2-Diphenylethane

5.1 Unsubstituted Hydroxyketones

1-(4'-Hydroxy)-4-(1-oxobutyl)-1,2-diphenylethane

C₁₈H₂₀O₂

mol. wt. 268.36



4'-amino-4-butyryl-1,2-diphenylethane [1879].

¹H NMR [1879], IR [1879], MS [1879].

Methyl ether

C19H22O2

mol. wt. 282.38

-Refer to: [1879];

¹H NMR [1879], IR [1879], MS [1879].

1-[2-Hydroxy-5-(2-phenylethyl)phenyl]-1-butanone

	$C_{18}H_{20}O_2$		mol. wt. 268.36
OH CO(CH ₂) ₂ CH ₃	Synthesis -Obtained by	Fries rearrangement	of 4-hydroxy-
CH ₂ CH ₂ C ₆ H ₅	diphenylethane chlorobenzene [b.p. ₁₈ 240–242°	butyrate with alumini 3326].	ium chloride in

1-[2,4-Dihydroxy-5-(2-phenylethyl)phenyl]-1-butanone

	$C_{18}H_{20}O_3$	mol. wt. 284.36
OH CO(CH ₂) ₂ CH ₃	Synthesis -Obtained by action of alumin	ium chloride with a
HO CH ₂ CH ₂ C ₆ H ₅	2,4-dihydroxydiphenylethane 2,4-dihydroxydiphenyethane nitrobenzene [3326].	dibutyrate and mixture in
	m.p. 85–86° [3326].	

1-[2-Hydroxy-5-(2-phenylethyl)phenyl]-3-methyl-1-butanone

	$C_{19}H_{22}O_2$		1	mol. wt. 282.38
OH COCH ₂ CH(CH ₃) ₂ CH ₂ CH ₂ C ₆ H ₅	Synthesis -Preparation by diphenylethane is chloride in boilir b.p. ₁₉ 247° [3326	sovalerang chlor	ate in the presen	ce of aluminium

1-[2,4-Dihydroxy-5-(2-phenylethyl)phenyl]-3-methyl-1-butanone

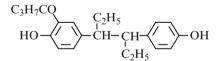
$$\begin{array}{c} C_{19}H_{22}O_{3} \\ \end{array} \qquad \mbox{mol. wt. } 298.38 \\ OH \\ COCH_{2}CH_{2}CH(CH_{3})_{2} \\ HO \\ CH_{2}CH_{2}C_{6}H_{5} \end{array} \qquad \begin{array}{c} Synthesis \\ -Preparation \ by \ Fries \ rearrangement \ of \\ 2,4-dihydroxy-diphenylethane \ diisovalerate \ in \\ the \ presence \ of \ 2,4-dihydroxydiphenylethane \\ and \ aluminium \ chloride \ in \ nitrobenzene \ at \ 50^{\circ} \\ for \ 3-4 \ h \ [3326]. \\ m.p. \ 102^{\circ} \ [3326]. \end{array}$$

5.2 Substituted Hydroxyketones

3-(4-Hydroxyphenyl)-4-[4-hydroxy-3-(1-oxobutyl)phenyl]hexane

(3-n-Butyrylhexestrol)

C₂₂H₂₈O₃ mol. wt. 340.46



-Obtained by treatment of its dimethyl ether by means of pyridinium chloride [510].

colourless needles; m.p. 134° [510].

Dimethyl ether

 $C_{24}H_{32}O_3$ mol. wt. 368.52

-Obtained by reaction of butyryl chloride with hexestrol dimethyl ether in the presence of aluminium chloride in nitrobenzene for 3 h at r.t. [510].

Synthesis

lustrous colourless leaflets [510]; m.p. 72° [510].

mol. wt. 298.38

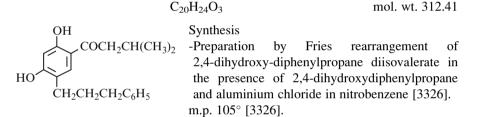
6 Aromatic Hydroxyketones Derived from 1,3-Diphenylpropane

1-[2,4-Dihydroxy-5-(3-phenylpropyl)phenyl]-1-butanone

C19H22O3

Synthesis OН -Obtained bv treatment of 2,4-dihydroxy-CO(CH₂)₂CH₃ а diphenylpropane (sym) dibutyrate and 2,4-dihydroxydiphenylpropane (sym) mixture with H aluminium chloride in nitrobenzene at 50° for $(CH_2)_3C_6H_5$ 3-4 h [3326]. m.p. 78° [3326].

1-[2,4-Dihydroxy-5-(3-phenylpropyl)phenyl]-3-methyl-1-butanone



7 Aromatic Hydroxyketones Derived from Various Halogenated 1-Butanoic Acid

7.1 From 2-Bromo-1-Butanoic Acid

2-Bromo-1-(3,5-dibromo-2-hydroxyphenyl)-1-butanone

[238074-77-6]	$C_{10}H_9Br_3O_2$	mol. wt. 400.89
Br Br	Syntheses -Obtained by reaction of 3,5-dibromo-2-hydroxybutyrop acetic acid (89 %) [375]. -Also refer to: [998].	
vellow crystals [375]:		

yellow crystals [375]; m.p. 135–136° [375]; ¹H NMR [375], IR [375].

2-Bromo-1-(3-bromo-2-hydroxyphenyl)-1-butanone

 $\begin{array}{cccc} [727687-90-3] & C_{10}H_{10}Br_2O_2 & \mbox{mol. wt. } 322.00 \\ OH & Synthesis \\ Br & COCHBrCH_2CH_3 & -Refer to: [375]. \end{array}$

2-Bromo-1-(3-bromo-4-hydroxyphenyl)-1-butanone

$$\begin{array}{cccc} C_{10}H_{10}Br_2O_2 & \text{mol. wt. 322.00} \\ & & \\ OH & & \\ Synthesis & \\ & & \\ Pr & & \\ Refer to: [441]. & \\ & & \\ Methyl \ ether \ (2S) \ \ [306972-94-1] & \\ & \\ C_{11}H_{12}Br_2O_2 & \\ & \\ COCHBrCH_2CH_3 & \\ \end{array}$$

-Preparation of nonracemic α -bromoketones by stereoselective bromination of aryl alkyl tartrate acetals followed by hydrolysis (50 %, 98 % ee) [441].

m.p. 82–83° [441]; ¹H NMR [441], ¹³C NMR [441], IR [441], MS [441].

2-Bromo-1-(3-hydroxyphenyl)-1-butanone

[90841-48-8]	$C_{10}H_{11}BrO_2$		mol. wt. 243.10
OH COCHBrCH ₂ COCHBrCH	Synthesis -Refer to: [3002] m.p. 59.1° [3002 CH ₃		
Methyl ether	[91335-45-4]	$C_{11}H_{13}BrO_2$	mol. wt. 257.13
m.p. 35° [3002].			
2-Bromo-1-(4-hydro	oxyphenyl)-1-butanon	e	
[53903-58-5]	$C_{10}H_{11}BrC$	D_2	mol. wt. 243.10
ОН	Syntheses -Refer to: [3002, 3471 m.p. 86.5–87.5° [3002	-	

COCHBrCH₂CH₃

Methyl ether [881-43-6] C₁₁H₁₃BrO₂ mol. wt. 257.13

-Obtained by treatment of 1-(4-methoxyphenyl)-1-butanone with bromine,

*in glacial acetic acid below 20°, the solution poured into ice water after disappearance of the bromine colour (88 %) [3062];

*in ethyl ether and glacial acetic acid at r.t. (80 %) [1114].

-Obtained by reaction of 2-bromobutanoyl chloride with anisole in the presence of aluminium chloride in 1,2-dichloroethane and the solution was cooled with an ice bath between 45 min and 90 min (43 %) [3308].

-Also refer to: [580, 1000, 1630, 2157, 2158, 2375, 2888, 2987, 3241, 3383, 3472].

colourless oil [3308]; m.p. 51° [3062], 50–52° [2157], 45–46° [1114]; ¹H NMR [1114, 2157, 3308], ¹³C NMR [2157], IR [3308], MS [1114, 3308].

Methyl ether (2S) [306972-91-8] C₁₁H₁₃BrO₂ mol. wt. 257.13

-Preparation of nonracemic α -bromoketones by stereoselective bromination of aryl alkyl tartrate acetals followed by hydrolysis (65 %, 92 % ee) [441].

m.p. 51–53° [441]; ¹H NMR [441], ¹³C NMR [441], IR [441], MS [441].

Benzyl ether [35081-43-3] C₁₇H₁₇BrO₂ mol. wt. 333.22

-Obtained by irradiation with UV lamp of solution of 4-benzyloxybutyrophenone and bromine in methylene chloride for 15 h between 15 and 18° (59 %) [556].

m.p. 60° [556].

2-Bromo-1-(2,4-dihydroxyphenyl)-1-butanone

$C_{10}H_{11}BrO_3$			mol. wt. 259.10	
HO	Synthesis -Obtained with reaction) m.p. 114–1	reso: [1700].	rcinol	f α-bromobutyronitrile (Houben-Hoesch

2-Bromo-1-(3,4-dihydroxyphenyl)-1-butanone

$$C_{10}H_{11}BrO_3$$
 mol. wt. 259.10



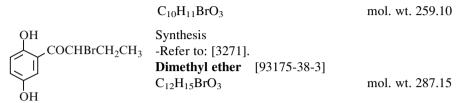
Synthesis -Obtained by reaction of α -bromobutyric acid with pyrocatechol in the presence of phosphorus oxychloride on a water bath [934].

COCHBrCH₂CH₃ m.p. 135° [934].

Dimethyl ether [23474-83-1] C₁₂H₁₅BrO₃ mol. wt. 287.15

m.p. 90–91.5° [713].

2-Bromo-1-(2,5-dihydroxyphenyl)-1-butanone



-Obtained by bromination of 2,5-dimethoxybutyrophenone (85 %) [3271].

2-Bromo-1-(2,4,5-trihydroxyphenyl)-1-butanone

$$\begin{array}{c} C_{10}H_{11}BrO_4 & \text{mol. wt. 275.10} \\ OH & Synthesis \\ + COCHBrCH_2CH_3 & -Refer to: [2696]. \\ HO & C_{13}H_{17}BrO_4 & \text{mol. wt. 317.18} \end{array}$$

-Obtained by reaction of bromine with 2,4,5-trimethoxybutyrophenone in acetic acid at $35-40^{\circ}$, then at 25° for 40 min [2695]. -Also refer to: [2696].

m.p. 65–65.5° [2695]; ¹H NMR [2695], IR [2695], MS [2695].

5-(2-Bromo-1-oxobutyl)-2-hydroxybenzoic acid

	$C_{11}H_{11}BrO_4$	mol. wt. 287.14
OH CO ₂ H	Synthesis -Refer to: [700]. Methyl ester [24085-13-0]	
Br CO-CH-C ₂ H ₅	$C_{12}H_{13}BrO_4$	mol. wt. 301.14

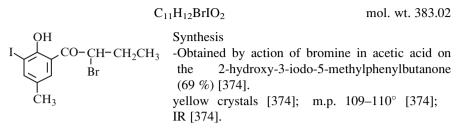
-Obtained by bromination of methyl 5-butyryl-2-hydroxybenzoate at r.t. in chloroform (96 %) [700].

-Also refer to: [434, 1932–1934].

m.p. 83° [700, 1932–1934].

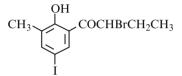
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2-Bromo-1-(2-hydroxy-3-iodo-5-methylphenyl)-1-butanone



2-Bromo-1-(2-hydroxy-5-iodo-3-methylphenyl)-1-butanone

[1186304-76-6] $C_{11}H_{12}BrIO_2$ mol. wt. 383.02



Synthesis .COCHBrCH₂CH₃ -Obtained by reaction of bromine 1-(2-hydroxy-5-iodo-3-methylphenyl) + bywith 1-(2-hydroxy-5-iodo-3-methylphenyl)-1-butanone in acetic acid (69 %) [374]. yellow crystals [374];

m.p. 109–110° [374]; ¹H NMR [374], ¹³C NMR [374], IR [374].

2-Bromo-1-(3-bromo-2-hydroxy-5-methylphenyl)-1-butanone

[396100-57-5] C11H12Br2O2 mol. wt. 336.02 **Synthesis** COCHBrCH₂CH₃ -Obtained by reaction of bromine with 3-bromo-2-hydroxy-5-methylbutyrophenone in refluxing acetic acid (70 %) [375]. CH₃ yellow crystals [375];

m.p. 117–118° [375]; ¹H NMR [375], IR [375].

2-Bromo-1-[4-hydroxy-3-(methylthio)phenyl]-1-butanone

9



Syntheses -Preparation by reaction of bromine with 4-hydroxy-3-(methylthio)butyrophenone in chloroform in the presence of calcium carbonate at 25° [2476].

COCHBrCH₂CH₃ -Also refer to: [2949].

2-Bromo-1-(3,5-dibromo-2-hydroxy-4,6-dimethylphenyl)-1-butanone

$$\begin{array}{c} C_{12}H_{13}Br_{3}O_{2} \\ \text{mol. wt. 428.95} \\ \\ OH \\ CH_{3} \\ Br \\ CH_{3} \\ Br \\ \end{array} \begin{array}{c} OH \\ CH_{3} \\ Br \\ CH_{3} \\ Br \end{array} \begin{array}{c} OH \\ CH_{3} \\ Br \\ CH_{3} \\ Br \\ CH_{3} \\ Br \end{array} \begin{array}{c} OH \\ Obtained \\ Obtaine$$

2-Bromo-1-(3-bromo-6-hydroxy-2,4-dimethylphenyl)-1-butanone

[861310-96-5]	$C_{12}H_{14}Br_2O_2$	mol. wt. 350.05
CH ₃ CH ₃ COCHBrCH ₂ CH ₃ Br CH ₃	Synthesis -Obtained by reaction of with 2-hydroxy-4,6-dimethy carbon disulfide [189]. m.p. 112.5–113.5° [189].	

2-Bromo-1-(2-hydroxy-4,6-dimethylphenyl)-1-butanone

C ₁₂ H	H ₁₅ BrO ₂	mol. wt. 271.15
CH ₃ COCHBrCH ₂ CH ₃	Synthesis -Refer to: [189]. Acetate $C_{14}H_{17}BrO_3$	mol. wt. 313.19

-Obtained by reaction of bromine with 1-(2-hydroxy-4,6-dimethylphenyl) acetate in carbon disulfide [189].

m.p. 36.5–38.5° [189].

2-Bromo-1-[4-hydroxy-3-(methylsulfonylmethyl)phenyl]butanone

OH	Synthesis		
CH ₂ SO ₂ CH ₃	-Refer to: [157.	3].	
	Benzyl ether	[56490-82-5]	
\mathbf{i}	$C_{19}H_{21}BrO_4S$		mol. wt. 425.34
COCHBrCH ₂ CH ₃			

-Obtained by refluxing a mixture of 1-[4-Hydroxy-3-(methylsulfonylmethyl)-phenyl]butanone, PHT,2-pyrrolidinone in THF for 2 h (83 %) [1573].

m.p. 93–96° [1573]; ¹H NMR [1573].

2-Bromo-1-[3,5-bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-butanone

[17055-14-0]	$C_{18}H_{27}BrO_2$	mol. wt. 355.32
$(CH_3)_3C$ $C(CH_3)_3$ $C(CH_3)_3$ $COCHBrCH_2CH_3$	Syntheses -Obtained by reaction of 2-brorr ride with 2,6-di-tert-butylpheno of titanium tetrachloride [1468].	l in the presence

-Also obtained by reaction of bromine with 4-hydroxy-3,5-di-tert-butylbutyrophenone in octane for 30 min at 70° (84 %) [3238, 3239].

-Also obtained from 4-hydroxy-3,5-di-tert-butylbutyrophenone by reaction with cupric bromide in ethyl acetate-chloroform (80 %) [2773].

m.p. 126–127° [2773], 120–122° [3238, 3239]; ¹H NMR [2773].

7.2 From 4-Bromo-1-Butanoic Acid

4-Bromo-1-(4-hydroxyphenyl)-1-butanone

 $\begin{array}{ccc} C_{10}H_{11}BrO_2 & \mbox{mol. wt. } 243.10 \\ & \\ OH & Synthesis & \\ -Refer to: [3354]. & \\ & \\ Methyl \ ether & [1023634-25-4] \\ & \\ C_{01}H_{13}BrO_2 & \mbox{mol. wt. } 257.13 \end{array}$

-Refer to: [565, 679, 1683, 3354].

上リ

¹H NMR [565, 3354], ¹³C NMR [565].

4-Bromo-1-(2,4-dihydroxyphenyl)-1-butanone

[105174-45-6]	$C_{10}H_{11}BrO_3$	mol. wt. 259.10
OH	Syntheses	
$CO(CH_2)_2CH_2Br$	-Obtained by reaction of	4-bromobutyric acid with

-Obtained by reaction of 4-bromobutyric acid with resorcinol in the presence of aluminium bromide in carbon disulfide (42 %) [1738, 1992].

-Also obtained by reaction of 4-bromobutyryl bromide with resorcinol in the presence of aluminium bromide in nitrobenzene (34 %) [1738]. -Also refer to: [3156].

colourless crystals [1738]; m.p. 79–80° [1738]; ¹H NMR [1738], IR [1738], UV [1738], MS [1738].

4-Bromo-1-(3,4-dihydroxyphenyl)-1-butanone

Dimethyl ether [105174-48-9] C₁₂H₁₅BrO₃ mol. wt. 287.15

-Obtained by reaction of 4-bromobutyryl bromide with veratrole in the presence of aluminium chloride in carbon disulfide for 30 min at r.t., then 15 min at 40° (59 %) [1738].

-Also obtained by reaction of 4-bromobutyryl chloride with veratrole in the presence of aluminium chloride in methylene chloride for 3 h at 0° (75 %) [3236]. -Also refer to: [492, 3237].

pale yellow crystals [1738]; white needles [3236]; m.p. 96–97° [1738, 3236]; ¹H NMR [1738, 3236], IR [1738], UV [1738], MS [1738].

4-Bromo-1-(2,3,4-trihydroxyphenyl)-1-butanone

[105174-47-8]	$C_{10}H_{11}BrO_4$	mol. wt. 275.10
HO HO HO	Synthesis -Obtained by reaction of 4-brom- with pyrogallol in the presence carbon disulfide at r.t. for 24 h (4	of aluminium in
colourlass readles [1729]	$m n = 101^{\circ} [1729]$	

colourless needles [1738]; m.p. 101° [1738]; ¹H NMR [1738], IR [1738], UV [1738], MS [1738].

7.3 From 2-Chloro-1-Butanoic Acid

2-Chloro-1-(2-hydroxyphenyl)-1-butanone

[103040-41-1]	$C_{10}H_{11}ClO_2$	mol. wt. 198.65
COCHCICH ₂ CH ₃		3-chloro-3-ethyl-2,4-chro- (53°) in a carbon tetrachlo- carbon dioxide evolution

-Also refer to: [1044].

b.p._{0.5} 110° [1043, 1044].

2-Chloro-1-(2-hydroxy-5-methylphenyl)-1-butanone

[871882-61-0]	$C_{11}H_{13}ClO_2$	mol. wt. 212.68
COCHCICH ₂ CH ₃	with p-cresol methyl ether i	of α -chlorobutyryl bromide in the presence of aluminium at r.t. for 5–6 h (80 %) [188]. .p. 61–62° [188].

7.4 From 3-Chloro-1-Butanoic Acid

3-Chloro-1-(2-hydroxyphenyl)-1-butanone

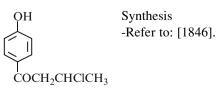
[129218-86-6]	$C_{10}H_{11}ClO_2$	mol. wt. 198.65
OH COCH ₂ CHCICH ₃	Synthesis -Obtained by treatment of aluminium chloride betwe (38 %) [95].	

¹H NMR [95], IR [95], MS [95].

3-Chloro-1-(4-hydroxyphenyl)-1-butanone

 $C_{10}H_{11}ClO_2$

mol. wt. 198.65



Methyl ether [654643-45-5] $C_{11}H_{13}CIO_2$ mol. wt. 212.68

-Refer to: [1846].

3-Chloro-1-(2-hydroxy-5-methylphenyl)-1-butanone

[871882-79-0]	$C_{11}H_{13}ClO_2$	mol. wt. 212.68
COCH ₂ CHCICH ₃ CH ₃	Syntheses -Preparation condensation of (oil; b.p. ₂₁ 51–53°) with 4-1 sence of aluminium chloride -Also refer to: [183].	methylanisole in the pre-

oil [193]; b.p. $_{20}$ 167–170° [183, 193]; m.p. 66–67° [183].

3-Chloro-1-[2-hydroxy-5-(1-methylethyl)phenyl]-1-butanone

	$C_{13}H_{17}ClO_2$	mol. wt. 240.73
OH ↓ COCH ₂ CHClCH ₃	Synthesis -Refer to: [1455].	
CH(CH ₃) ₂	Methyl ether [188405-38-1] $C_{14}H_{19}CIO_2$ -Refer to: [1455].	mol. wt. 254.76

7.5 From 4-Chloro-1-Butanoic Acid

1-(5-Bromo-2-hydroxyphenyl)-4-chloro-1-butanone

¹H NMR [125], IR [125], MS [125].

4-Chloro-1-(4-fluoro-2-hydroxyphenyl)-1-butanone

,			
[61053-78-9]	$C_{10}H_{10}C$	IFO ₂	mol. wt. 216.64
F CO(CH ₂) ₂ CH ₂ C	Syntheses ¹ -Refer to: [26 b.p. _{1.1} 128–12	534–2636, 2710, 2984]. 31° [2710, 2984].	
Acetate	$C_{12}H_{12}ClFC$	0	mol. wt. 258.68
O-Acetyloxime (1 <i>E</i>)	[313369-23-2]	C ₁₄ H ₁₅ ClFNO ₄	mol. wt. 315.73
-Refer to: [2634, 2635].			
Oxime [313544	-35-3] ($C_{10}H_{11}CIFNO_2$	mol. wt. 231.65
-Refer to: [2636].			
Refer to: [2050].			
Oxime (1 <i>E</i>) [854	85-53-6]	C ₁₀ H ₁₁ ClFNO ₂	mol. wt. 231.65
-Refer to: [2634, 2635].			
4-Chloro-1-(5-fluoro-2-h	ydroxyphenyl)-	1-butanone	
[79214-31-6]	$C_{10}H_{10}C$	IFO ₂	mol. wt. 216.64
OH CO(CH ₂) ₂ CH ₂ Cl	Synthesis -Refer to: [2623].	
4-Chloro-1-(2-hydroxyp	henvl)-1-butano	one	

4-Chloro-1-(2-hydroxyphenyl)-1-butanone

[313369-21-0]	С	$_{10}H_{11}ClO_2$	mol. wt. 198.65	
OH CO(CH ₂)	anisole i nitroethar	5		
¹ H NMR [24	.59], MS [2459].			
Oxime	[313544-36-4]	C ₁₀ H ₁₂ ClNO ₂	mol. wt. 213.66	
-Refer to: [2636].				
Oxime (1 <i>E</i>)	[313369-22-1]	$C_{10}H_{12}CINO_2$	mol. wt. 213.66	
-Refer to: [2634, 2635].				

Acetate		$C_{12}H_{13}ClO_3$	mol. wt. 240.68
O-Acetyloxime	[313369-24-3]	$C_{14}H_{16}ClNO_4$	mol. wt. 297.74
-Refer to: [2634, 2	635].		
Methyl ether	[40877-17-6]	$C_{11}H_{13}ClO_2$	mol. wt. 212.68

 $C_{11}H_{13}ClO_2$ -Refer to: [2459, 2775, 2776, 2815].

¹H NMR [2459], MS [2459], UV [2815].

4-Chloro-1-(3-hydroxyphenyl)-1-butanone

	$C_{10}H_{11}ClO_2$		mol. wt. 198.65
OH	Synthesis -Refer to: [328 Methyl ether	6]. [258882-48-3]	
CO(CH ₂) ₂ CH ₂ Cl		[230002 10 5]	mol. wt. 212.68

-Refer to: [2460 (61 %), 2782, 3286].

colourless oil [2460]; ¹H NMR [2460, 2782], MS [2460, 2782].

4-Chloro-1-(4-hydroxyphenyl)-1-butanone

[7150-55-2]

C10H11ClO2

mol. wt. 198.65



Syntheses -Obtained by reaction of 4-chlorobutyryl chloride with phenol in the presence of aluminium chloride in nitrobenzene at 40° for 3 h (50 %) [1581].

CO(CH₂)₂CH₂Cl

-Also obtained by Fries rearrangement of phenyl 4-chloro-butyrate in the presence of aluminium chloride in nitrobenzene for 18 h at r.t. (34 %) [3189].

-Also refer to: [111, 253, 299, 300, 711, 712, 968, 1492, 1498, 1580, 1868, 1886, 1949, 2319, 2385–2387, 2815, 2894, 2895, 3134, 3210].

m.p. 115–117° [1581], 114–115.2° [1492, 1498, 3189]; ¹H NMR [1581], UV [2815], MS [1581, 1886].

N.B.: Reported m.p. 113–116.5° in Aldrich Catalogue 1986–1987.

Acetate

C₁₂H₁₃ClO₃

mol. wt. 240.68

-Prepared by acetylation of 4'-hydroxy compound (50 %) [3189].

m.p. 30-31.5° [3189].

Methyl ether [

[40877-19-8] C₁

 $C_{11}H_{13}ClO_2$

mol. wt. 212.68

-Obtained by reaction of anisole with 4-chlorobutanoyl chloride,

*in the presence of aluminium chloride [2850], (71 %) [3189];

*in the presence of Si-Fe catalyst at 25° (48 %) [427].

-Also obtained by reaction of 4-methoxyphenylmagnesium bromide with 4-chlorobutyronitrile (32 %) [3044].

-Also obtained by cross coupling reaction of 4-chlorobutyryl chloride with $(4-CH_3OC_6H_5)_3Bi$ in the presence of Pd(o) as catalyst (66 %) [2565].

-Also refer to: [253, 456, 609, 668, 711, 712, 830, 1120, 1340, 1387, 1490–1497, 1710–1713, 1845, 1859, 1886, 2009, 2023, 2565, 2754, 2815, 2847, 2894, 2895, 3164, 3229, 3244, 3285–3287, 3389, 3390, 3422].

pale red liquid [3044]; b.p._{0.8-0.9} 152–154° [1845], b.p.₂ 159–163° [3189], b.p.₃₋₄ 162–165° [2754], b.p.₆ 175° [1496], b.p.₅ 175° [1490–1495, 1497]; m.p. 31–32° [2754], 31° [3164], 28.5–30° [3189]; ¹H NMR [1340, 2565, 3044, 3164], 3422], ¹³C NMR [2565, 3044, 3422], IR [2565, 3044, 3164], UV [2815], MS [2565].

USE: For preparation of acylaminothiazoles as inhibitors of 15-lipoxygenase [3285]; For preparation of imidazolyl inhibitors of 15-lipoxygenase [3287]; For preparation of piperidine derivatives as NMDA receptor antagonists [3389].

2,4-Dinitrophenylhydrazone of the methyl ether

 $[75349-76-7] C_{17}H_{17}ClN_4O_5 mol. wt. 392.80$

m.p. 153–154° [2754].

Ethyl ether	[75343-08-7]	$C_{12}H_{15}ClO_2$	mol. wt. 226.70
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-Obtained by reaction of phenetole with 4-chlorobutanoyl chloride in the presence of aluminium chloride (70 %) [3189].

-Also refer to: [40, 809, 1492, 2754, 2815].

m.p. 51–52° [2754], 50.4–51.8° [1491, 1492, 1497, 3189]; UV [2815].

2,4-Dinitrophenylhydrazone of the ethyl ether

[75343-28-1]	$\mathrm{C}_{18}\mathrm{H}_{19}\mathrm{ClN}_{4}\mathrm{O}_{5}$	mol. wt. 406.83
m.p. 151–153° [2754].		

Propyl ether [92019-28-8] $C_{13}H_{17}ClO_{2}$ mol. wt. 240.73

-Obtained by reaction of phenyl propyl ether with 4-chlorobutanoyl chloride in the presence of aluminium chloride (47 %) [3189]. -Also refer to: [1492, 1498].

b.p.₅ 183–185° [3189], b.p.₆ 183° [1492, 1498]; m.p. 29.5–32° [3189].

 $C_{14}H_{19}ClO_2$ **Butyl ether** [92317-86-7] mol. wt. 254.76

-Obtained by reaction of phenyl butyl ether with 4-chlorobutanoyl chloride in the presence of aluminium chloride (66 %) [3189]. -Also refer to: [1492, 1498].

m.p. 36.8-38° [1492, 1498, 3189].

Benzyl ether

Refer to: [1868].

Phenyl ether	[22620-38-8]	$C_{16}H_{15}ClO_2$	mol. wt. 274.75

C₁₇H₁₇ClO₂

-Refer to: [2009, 2905].

4-Chloro-1-(2,4-dihydroxyphenyl)-1-butanone

[105174-49-0]	$C_{10}H_{11}ClO_3$	mol. wt. 214.65
ОН	Synthesis	
CO(CH ₂) ₂ CH ₂ Cl	-Obtained by reaction of 4	-chlorobutyryl chloride
	with resorcinol in the prese	
HO	ride in carbon disulfid	e at r.t. for 24 h

mor in the presence ride in carbon disulfide at r.t. for 24 h (37 %) [1738].

yellow crystals [1738]; m.p. 92–93° [1738]; ¹H NMR [1738], IR [1738], UV [1738], MS [1738].

Dimethyl ether	[80269-97-2]	$C_{12}H_{15}ClO_3$	mol. wt. 242.70
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-Obtained by reaction of 4-chlorobutyryl chloride with resorcinol dimethyl ether in the presence of aluminium chloride in nitrobenzene at 0° , then at r.t. for 36 h (50 %) [3189].

-Also refer to: [2009, 2010].

mol. wt. 288.77

4-Chloro-1-(2,5-dihydroxyphenyl)-1-butanone

$$\begin{array}{ccc} C_{10}H_{11}ClO_3 & \text{mol. wt. 214.65} \\ OH & Synthesis \\ \hline CO(CH_2)_2CH_2Cl & -Refer to: [3189]. \\ \hline Dimethyl ether & [91767-62-3] \\ C_{12}H_{15}ClO_3 & \text{mol. wt. 242.70} \end{array}$$

-Obtained by reaction of 4-chlorobutyryl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride in nitrobenzene at 0°, then at r.t. for 36 h (42 %) [3189].

-Also refer to: [1491, 1494, 1497, 3209].

b.p.₄ 150–190° [1491, 1494, 1497, 3189]; m.p. 27–30° [1491, 1497, 3189].

4-Chloro-1-(3,4-dihydroxyphenyl)-1-butanone

[105133-00-4]

C10H11ClO3

mol. wt. 214.65



Syntheses

-Obtained by reaction of 4-chlorobutyryl chloride with pyrocatechol in the presence of aluminium chloride in carbon disulfide at r.t. for 30 min, then at 40° for 1 h (40 %) [1738]. CO(CH₂)₂CH₂Cl -Preparation [1948] using the general procedure [1738].

colourless crystals [1738]; m.p. 100–101° [1738]; ¹H NMR [1738], IR [1738], UV [1738], MS [1738].

Dimethyl ether	[57010-67-0]	$C_{12}H_{15}ClO_3$	mol. wt. 242.70
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-Obtained by reaction of 4-chlorobutyryl chloride with pyrocatechol dimethyl ether in the presence of aluminium chloride in nitrobenzene at 0° , then at r.t. for 36 h (57 %) [3189].

-Also refer to: [1492, 1498, 2560, 2815, 3164].

m.p. 92–93° [1492, 1498, 3189], 91–92° [2560], 90–92° [3164]; ¹H NMR [3164], IR [3164], UV [2815].

4-Chloro-1-(2,3,4-trihydroxyphenyl)-1-butanone

[105174-51-4]	$C_{10}H_{11}ClO_4$	mol. wt. 230.65
HO HO HO	Synthesis -Obtained by reaction of 4-chlo with pyrogallol in the presence ride in carbon disulfide at r.t. for	of aluminium chlo-

yellow crystals [1738]; m.p. 99–100° [1738]; ¹H NMR [1738], IR [1738], UV [1738], MS [1738]. **Trimethyl ether** [92019-50-6] C₁₃H₁₇ClO₄ mol. wt. 272.73

-Obtained by reaction of 4-chlorobutyryl chloride with pyrogallol trimethyl ether in the presence of aluminium chloride in nitrobenzene at 0° , then at r.t. for 36 h (12 %) [3189].

-Also refer to: [1494].

m.p. 83.8-85.4° [1494, 3189].

4-Chloro-1-(2,4,6-trihydroxyphenyl)-1-butanone

 $\begin{array}{cccc} C_{10}H_{11}ClO_4 & \text{mol. wt. } 230.65 \\ OH & Synthesis \\ + & CO(CH_2)_2CH_2Cl & -Refer to: [3282]. \\ HO & OH & C_{13}H_{17}ClO_4 & \text{mol. wt. } 272.73 \end{array}$

-Refer to: [1812, 2338, 3282].

m.p. 48° [1812]; HPLC [2338].

USE: Preparation of buflomedil by amination of the above trimethyl ether with pyrrolidine in cyclohexane in the presence of sodium iodide [3282].

4-Chloro-1-(3,4,5-trihydroxyphenyl)-1-butanone

	$C_{10}H_{11}ClO_4$		mol. wt. 230.65
ОН НО , , ОН	Synthesis -Refer to: [3188].		
	Trimethyl ether	[17766-59-5]	
CO(CH ₂) ₂ CH ₂ Cl	$C_{13}H_{17}ClO_4$		mol. wt. 272.73

-Obtained by heating of α -(3,4,5-trimethoxybenzoyl)- γ -butyrolactone with concentrated hydrochloric acid on a steam bath for 3 h. After cooling, fused zinc chloride was added and heating was continued for 2 h (85 %) [2465]. -Also refer to: [3188].

m.p. 80–82° [2465].

m.p. 139-140° [2465].

1-(2-Amino-4-hydroxyphenyl)-4-chloro-1-butanone

 $\begin{array}{cccc} [313545-13-0] & C_{10}H_{12}CINO_2 & mol. wt. 213.66 \\ OH & Synthesis \\ -Refer to: [2636]. \end{array}$

4-Chloro-1-(2-hydroxy-4-methylphenyl)-1-butanone

[113425-32-4]	$C_{11}H_{13}ClO_2$	mol. wt. 212.68
CO(CH ₂) ₂ CH ₂ Cl	Syntheses -Obtained by reaction of 4-ci with m-cresol in the presence ride in nitrobenzene at 40° for -Also refer to: [1580].	e of aluminium chlo-

m.p. 95–97° [1581]; ¹H NMR [1581], MS [1581].

4-Chloro-1-(2-hydroxy-5-methylphenyl)-1-butanone

-Also obtained by Fries rearrangement of 4-methylphenyl 4-chlorobutyrate with aluminium chloride in nitrobenzene at $60-70^{\circ}$ for 8 h (67 %) [2559]. -Also refer to: [1127].

b.p._{0.3} 135–136° [2559], b.p.₁ 149–152° [1127]; m.p. 42° [1127, 2311], 39–41° [2185], 36–38° [2559].

4-Chloro-1-(4-hydroxy-2-methylphenyl)-1-butanone

	$C_{11}H_{13}ClO_2$	mol. wt. 212.68
ОН	Syntheses -Refer to: [3082, 3244]. Methyl ether [838822-54-1]	
\downarrow CH ₃ CO(CH ₂) ₂ CH ₂ Cl	C ₁₂ H ₁₅ ClO ₂ -Refer to: [3082, 3244].	mol. wt. 226.70
¹ H NMR [3244]		

 \uparrow NH₂ CO(CH₂)₂CH₂Cl USE: Preparation of heterocycle-containing styrene derivatives as tyrosine kinase inhibitors for prevention/treatment of cancers, etc., [3082].

4-Chloro-1-(4-hydroxy-3-methylphenyl)-1-butanone

USE: Coordination complexes as catalyst in polycarbonate manuf. [1847]; Preparation of highly active and recyclable catalytic system for CO₂/Propylene oxide copolymn. for prepn. of polycarbonates with high mol. wt. [2675].

4-Chloro-1-(2-hydroxy-5-methoxyphenyl)-1-butanone

[173055-18-0]	$C_{11}H_{13}ClO_3$	mol. wt. 228.68
OH CO(CH ₂) ₂ CH ₂ Cl OCH ₃	Synthesis -Refer to: [2623].	

4-Chloro-1-(2-hydroxy-4-methoxyphenyl)-1-butanone

colourless needles [821]; m.p. 37–39° [821]; ¹H NMR [821], IR [821], UV [821], MS [821].

4-Chloro-1-(3-hydroxy-4-methoxyphenyl)-1-butanone

 $C_{11}H_{12}C_{10}$

CO(CH₂)₂CH₂Cl

DCH₃

mol. wt. 228.68

4-Chlorobutyrate

C15H18Cl2O4

mol. wt. 333.21

-Obtained by reaction of 4-chlorobutyric acid with guaiacol in the presence of phosphorous oxychloride for 3 h at 80° (51 %) [1738].

-Also obtained by reaction of 4-chlorobutyryl chloride with guaiacol in the presence of aluminium chloride in carbon disulfide for 1 h at 0° , then at r.t. for 5 h (43 %) [1738].

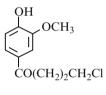
colourless crystals [1738]; m.p. 67° [1738]; ¹H NMR [1738], IR [1738], UV [1738], MS [1738],

N.B.: Or its 4-(4-chlorobutyryloxy)-3-methoxy isomer below; non precised.

4-Chloro-1-(4-hydroxy-3-methoxyphenyl)-1-butanone

Synthesis

 $C_{11}H_{13}ClO_3$ mol. wt. 228.68



-Refer to: [1738]. **4-Chlorobutyrate** $C_{15}H_{18}Cl_2O_4$ mol. wt. 333.21 -Obtained by reaction of 4-chlorobutyric acid with guaiacol in the presence of phosphorous oxychloride for 3 h at 80° (51 %) [1738].

-Also obtained by reaction of 4-chlorobutyryl chloride with guaiacol in the presence of aluminium chloride in carbon disulfide for 1 hat 0°, then at r.t. for 5 h (43 %) [1738].

colourless crystals [1738]; m.p. 67° [1738]; ¹H NMR [1738], IR [1738], UV [1738], MS [1738].

N.B.: Or its 3-(4-chlorobutyryloxy-)-4-methoxy isomer above; non precised.

4-Chloro-1-(4-hydroxy-3,5-dimethylphenyl)-1-butanone

[113425-31-3]	$C_{12}H_{15}ClO_2$	mol. wt. 226.70
CH ₃ CH ₃	Syntheses -Obtained by reaction of 4-chlor 2,6-dimethylphenol in the preser	

ride in nitrobenzene at 40° for 3 h (60 %) [1581]. CO(CH₂)₂CH₂Cl -Also refer to: [1580].

m.p. 83–85° [1581]; ¹H NMR [1581], MS [1581].

4-Chloro-1-(2-hydroxy-4,6-dimethoxyphenyl)-1-butanone

4-Chloro-1-(4-hydroxy-2,6-dimethoxyphenyl)-1-butanone

 $\begin{array}{c} [185835-74-9] & C_{12}H_{15}ClO_4 & \text{mol. wt. } 258.70 \\ \\ OH & Synthesis \\ -Refer to: [2338]. \\ HPLC [2338]. \\ \\ CH_{3}O & OCH_3 \\ COCH_2CH_2CH_2Cl \end{array}$

4-(4-Chlorobutyryl)-3-methylphenoxyacetic acid

[1148-02-3] $C_{13}H_{15}ClO_4$ mol. wt. 270.71 COUCH₂O CO(CH₂)₂CH₂Cl -Refer to: [2047, 2048, 2766, 2767].

m.p. 86.5-88° [2047, 2048, 2766, 2767].

4-Chloro-1-[4-hydroxy-3-(1-methylethyl)phenyl]-1-butanone

USE: Coordination complexes as catalyst in polycarbonate manuf. [1847]; Preparation of highly active and recyclable catalytic system for CO_2 /Propylene oxide copolymn. for prepn. of polycarbonates with high mol. wt. [2675].

4-Chloro-1-[3-(1,1-dimethylethyl)-4-hydroxyphenyl]-1-butanone

[1080021-90-4]	$C_{14}H_{19}ClO_2$	mol. wt. 254.76
OH C(CH ₃) ₃	Syntheses -Obtained by Friedel-Crafts acylation o using 4-chlorobutyryl chloride [2675]. -Also refer to: [1847].	f 2-tert-butylphenol
$CO(CH_2)_2CH_2Cl$	m.p. 121° [2675];	
1	12	

¹H NMR [2675], ¹³C NMR [2675], IR [2675].

USE: Coordination complexes as catalyst in polycarbonate manuf. [1847]; Preparation of highly active and recyclable catalytic system for $CO_2/Propylene$ oxide copolymn. for prepn. of polycarbonates with high mol. wt. [2675].

7.6 From Fluoro-1-Butanoic Acid

2,2,3,3,4,4,4-Heptafluoro-1-(2-hydroxyphenyl)-1-butanone

	$C_{10}H_5F_7O_2$	mol. wt. 290.14
OH CO(CF ₂) ₂ CF ₃	Synthesis -Refer to: [1848].	
	Methyl ether [217474-49-2] C ₁₁ H ₇ F ₇ O ₂	mol. wt. 304.16

-Obtained by refluxing a mixture of 2-(nonafluorobutyl)anisole in acetic acid with hydrobromic acid and aluminium oxide under nitrogen atmosphere during 16 days (43 %) [1848].

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oil [1848];
<sup>1</sup>H NMR [1848], <sup>19</sup>F NMR [1848], MS [1848]; GC [1848].
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2,2,3,3,4,4,4-Heptafluoro-1-(4-hydroxyphenyl)-1-butanone

[217474-50-5]	$C_{10}H_5F_7O_2$	mol. wt. 290.14
OH	Syntheses -Obtained by refluxing a mixture of 4-(nonaf- in acetic acid with hydrobromic acid and alun nitrogen atmosphere during 20 h (9 %) [1848	ninium oxide under
CO(CE) CE	Also refer to: $[2377]$	-

 $CO(CF_2)_2CF_3$ -Also refer to: [3377].

m.p. 62–64° [1848]; ¹H NMR [1848], ¹⁹F NMR [1848], MS [1848]; GC [1848]. **Methyl ether** [117482-22-1] C₁₁H₇F₇O₂ mol. wt. 304.16

-Preparation: An NMP solution containing $Pd(OAc)_2$, tributylphosphine, heptafluorobutyrate and 4-methoxyboronic acid in a 25 ml Schlenk tube was heated under argon at 80° for 4 h (87 %) [1577].

-Obtained by refluxing a mixture of 4-(nonafluorobutyl)anisole in acetic acid with hydrobromic acid and aluminium oxide under nitrogen atmosphere during 20 h (65 %) [1848].

-Also refer to: [1458].

colourless oil [1577]; oil [1848]; ¹H NMR [1577, 1848], ¹³C NMR [1577], ¹⁹F NMR [1577, 1848], MS [1577, 1848]; GC-MS [1577]; GC [1848].

O-[(Trifluoromethyl)sulfonyl]oxime of the methyl ether

[749924-46-7]	$C_{12}H_7F_{10}NO_4S$	mol. wt. 451.24
yellow crystals [3377];	¹ H NMR [3377], ¹⁹ F NMR [3377].	

USE: As latent acids for photoresist [3377].

Phenyl ether	$C_{16}H_{10}F_7O_2$	mol. wt. 367.24	[3377].
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O-[(Trifluoromethyl)sulfonyl]oxime of the phenyl ether

[749924-49-0]	$C_{17}H_9F_{10}NO_4S$	mol. wt. 513.31
10 100 [0077]		

m.p. 40–42° [3377]; ¹H NMR [3377], ¹⁹F NMR [3377].

USE: As latent acids for photoresist [3377].

2,2,3,3,4,4,4-Heptafluoro-1-(2,4-dihydroxyphenyl)-1-butanone

[65240-03-1]	$C_{10}H_5F_7O_3$	mol. wt. 306.14
HO HO HO HO	Syntheses -Refer to: [450, 2996]. m.p. 90° [450].	

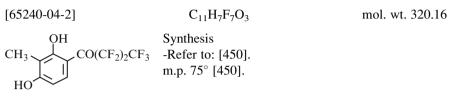
4,4,4-Trifluoro-1-(4-hydroxyphenyl)-1-butanone

	$C_{10}H_9F_3O_2$	mol. wt. 218.18
Н	Synthesis	
<	-Refer to: [698].	
	Methyl ether	
	$C_{11}H_{11}F_{3}O_{2}$	mol. wt. 232.20
$O(CH_2)_2 CF_3$		

-Obtained by reaction of 4-chloroanisole with 4,4,4-trifluorobutanal in the presence of Pd(dba)₂ (2 mol%), Pd phosphine (6 mol%), 4 Å MS and pyrrolidine in DMA at 140° for 4 h (54 %) [698].

¹H NMR [698], ¹³C NMR [698], IR [698], MS [698].

2,2,3,3,4,4,4-Heptafluoro-1-(2,4-dihydroxy-3-methylphenyl)-1-butanone



1-(2,4-Dihydroxy-3-propylphenyl)-4,4,4-trifluoro-1-butanone

[194981-87-8]	$C_{13}H_{15}F_{3}O_{3}$	mol. wt. 276.26
C ₃ H ₇ HO CO(CH ₂) ₂ CF ₃	Syntheses -Refer to: [20, 307]. ¹ H NMR [21].	

7.7 From 4-Iodo-1-Butanoic Acid

4-Iodo-1-(2-hydroxy-4-fluorophenyl)-1-butanone

	$C_{10}H_{10}FIO_2$	mol. wt. 308.09
F CO(CH ₂) ₂ CH ₂ I	Synthesis -Refer to: [3187]. m.p. 41.4° [3187].	

1-(4-Hydroxyphenyl)-4-iodo-1-butanone

$$C_{10}H_{11}IO_2$$
 mol. wt. 290.10



Syntheses -Obtained by treatment of 1-(4-hydroxyphenyl)-4-chloro-1-butanone with sodium iodide in methyl ethyl ketone at 90° [3134]. $CO(CH_2)_2CH_2I$ -Also refer to: [3212].

m.p. 96–97° [3134]; MS [3134].

Methyl ether [215667-87-1] C₁₁H₁₃IO₂ mol. wt. 304.13

-Obtained *via* the cerium ammonium nitrate mediated oxidative coupling of 2-(4-methoxy-phenyl)cyclobutanol and sodium iodide in 20 % H₂O/DME at 0° (67 %) [565].

-Preparation from 4-chloro-1-(4-methoxyphenyl)-1-butanone (75 %) [1120]. -Also refer to: [1122, 1928, 3081, 3212].

m.p. 43° [1120]; ¹H NMR [565, 1120], ¹³C NMR [565, 1120], IR [1120], MS [1120, 1928].

USE: Nickel-catalyzed cross-coupling of 4-iodo-1-(4-methoxyphenyl)-1-butanone with n-butyl-magnesium chloride in DMA at -35° for 30 min (68 %) [3212].

1-(3,4-Dihydroxyphenyl)-4-iodo-1-butanone

 $\begin{array}{cccc} [105174-61-6] & C_{10}H_{11}IO_3 & \text{mol. wt. } 306.10 \\ \\ OH & Synthesis \\ OH & -Obtained by reaction of sodium iodide with 4-chloro- 1-(3,4-dihydroxyphenyl)-1-butanone in boiling acetone for 24 h (56 %) [1738]. \\ CO(CH_2)_2CH_2I & m.p. \; 131^{\circ} \; [1738]; \end{array}$

¹H NMR [1738], IR [1738], UV [1738], MS [1738].

Dimethyl ether	[105174-55-8]	$C_{12}H_{15}IO_3$	mol. wt. 334.15
Dimentyl emer	[103174-33-8]	$C_{12} I_{15} I_{03}$	11101. wt. 554.1

-Obtained by reaction of sodium iodide with 4-chloro-1-(3,4-dimethoxyphenyl)-1-butanone in boiling acetone for 24 h (39 %) [1738].

m.p. 93° [1738]; ¹H NMR [1738], IR [1738], UV [1738], MS [1738].

4-Iodo-1-(2,3,4-trihydroxyphenyl)-1-butanone

[105174-58-1] $C_{10}H_{11}IO_4$ mol. wt. 322.10 OH Synthesis HO CO(CH₂)₂CH₂I -Obtained by reaction of sodium iodide with 4-bromo-1-(2,3,4-trihydroxyphenyl)-1-butanone in acetone at r.t. for 12 h (65 %) [1738]. m.p. 114° [1738];

¹H NMR [1738], IR [1738], UV [1738], MS [1738].

7.8 From Various Halogeno-1-Butanoic Acid

4-Bromo-3,3-difluoro-1-(4-hydroxyphenyl)-1-butanone

 $\begin{array}{cccc} C_{10}H_9BrF_2O_2 & \mbox{mol. wt. 279.08} \\ OH & Synthesis \\ -Refer to: [3354]. \\ \textbf{Methyl ether} & [1023272-24-3] \\ COCH_2-C-CH_2Br & C_{11}H_{11}BrF_2O_2 & \mbox{mol. wt. 293.11} \\ F & Obtained by treatment of 4-methoxyphenyl difluoro-cyclopropyl ketone with N-pentylpyridinium bromide at 70° for 10 h under nitrogen, \\ \end{array}$

*in trifluoroacetic acid (74 %) [3354];

*in trifluoromethanesulfonic acid (76 %) [3354].

¹H NMR [3354], ¹³C NMR [3354], ¹⁹F NMR [3354].

2,4-Dibromo-1-(2,6-dihydroxyphenyl)-1-butanone

$C_{10}H_{10}Br_2O_3$			mol. wt. 338.00
OH COCHBrCH ₂ CH ₂ Br	Synthesis -Refer to: [272]		
ОН	Dimethyl ether $C_{12}H_{14}Br_2O_3$	[753013-71-7]	mol. wt. 366.05

-Refer to: [272].

m.p. 99–100° [272]; MS [272].

USE: Preparation of hydroxycoumaranone derivs. as uPA receptor antagonists for treatment of cancers [272].

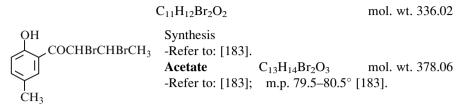
2-Bromo-1-(3-bromo-4-hydroxyphenyl)-3-methyl-1-butanone

	$C_{11}H_{12}Br_2O_2$		mol. wt. 336.02
OH Br	Synthesis -Refer to: [441].		
	Methyl ether (2S) $C_{12}H_{14}Br_2O_2$	[306972-95-2]	mol. wt. 350.05
$COCHBrCH(CH_3)_2$			

-Preparation of nonracemic α -bromoketones by stereoselective bromination of aryl alkyl tartrate acetals followed by hydrolysis (70 %, 71 % ee) [441].

pale brown oil [441]; ¹H NMR [441], IR [441], MS [441].

2,3-Dibromo-1-(2-hydroxy-5-methylphenyl)-1-butanone



2-Bromo-1-(4-hydroxyphenyl)-3-methyl-1-butanone

[412051-38-8]	$C_{11}H_{13}BrO_2$	mol. wt. 257.13
OH COCHBrCH(CH ₃) ₂	Syntheses -Refer to: [881–884, 1114]. ¹ H NMR [881].	

USE: For preparation of benzoxathiin derivatives as estrogen receptor modulators [882, 883].

Methyl ether	[35446-28-7]	$C_{12}H_{15}BrO_2$	mol. wt. 271.15
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-Obtained by treatment of 1-(4-methoxyphenyl)-4-methyl-1-pentanone with bromine in ethyl ether and glacial acetic acid at r.t. (93 %) [1114]. -Also refer to: [143, 896, 3231 (89 %)].

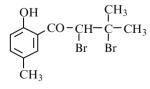
oil [1114]; m.p. 73° [3231]; ¹H NMR [896, 1114, 3231], IR [3231], MS [896, 1114].

2-Bromo-1-(2,3-dichloro-4-hydroxyphenyl)-2-ethyl-1-butanone

[4091-11-6]	$C_{12}H_{13}BrCl_2O_2$	mol. wt. 340.04
OH Cl CO-CCCH ₂ CH ₃ Br CH ₂ CH ₃	Syntheses -Obtained by reaction of bromine with 4-hydroxyphenyl)-2-ethyl-1-butanone in r.t. for 15 min [2060]. -Also refer to: [2054, 2058, 2059, 2061]. m.p. 122.5–123.5° [2054, 2058–2061].	

2,3-Dibromo-1-(2-hydroxy-5-methylphenyl)-3-methyl-1-butanone

 $C_{12}H_{14}Br_2O_2$ mol. wt. 350.05



CH₃ CH-C-CH₃ Br Br Sr Sr Synthesis Synthesis -Obtained by reaction of bromine with isobutenylp-cresyl-ketone (oil; b.p. 277–278°) in carbon disulfide [193]. m.p. 70–71° [193].

2-Bromo-1-(3-hydroxy-4-methoxyphenyl)-3-methyl-1-butanone

[99783-83-2]	C	$_{12}H_{15}BrO_3$			mol	l. wt. 287.15
CH ₃ O	COCHBrCH(CH ₃) ₂	4-methoxy mide in re	-isova fluxin	alerophenone	e with cetate	3-hydroxy- n cupric bro- e/chloroform

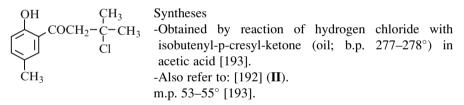
oil [144]; ¹H NMR [144].

2-Chloro-1-(2-hydroxy-5-methylphenyl)-3-methyl-1-butanone

[871886-71-4]	$C_{12}H_{15}ClO_2$	mol. wt. 226.70
$\bigcup_{CH_3}^{OH} \bigcup_{Cl}^{CH_3} \bigcup_{Cl}^{CH_3}$	Synthesis -Obtained by reaction of α -chloro with p-cresol methyl ether in the pro- chloride in carbon disulfide at r.t. [m.p. 75–76° [188].	esence of aluminium

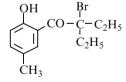
3-Chloro-1-(2-hydroxy-5-methylphenyl)-3-methyl-1-butanone

C12H15ClO2 mol. wt. 226.70



2-Bromo-2-ethyl-1-(2-hydroxy-5-methylphenyl)-1-butanone

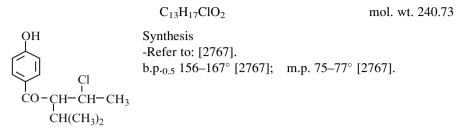
 $C_{13}H_{17}BrO_2$ mol. wt. 285.18



OH $CO - C - C_2H_5$ C_2H_5 Synthesis -Preparation by reaction of bromodiethylacetyl bromide with 4-methylanisole in the presence of aluminium chlo-ride in carbon disulfide on a water bath for 6 h (almost quantitative yield) [194].

b.p.₁₄ 175° [194]; $n_D^{20} = 1.565$ [194]; $d_{20} = 1.307$ [194].

3-Chloro-1-(4-hydroxyphenyl)-2-(1-methylethyl)-1-butanone



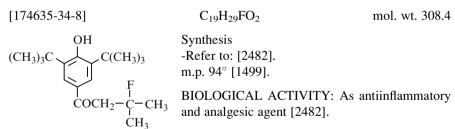
2-Bromo-1-[3,5-bis(1,1-dimethylethyl)-4-hydroxyphenyl]-3-methyl-1-butanone

[17055-16-2]	$C_{19}H_{29}BrO_2$	mol. wt. 369.34
(CH ₃) ₃ C C(CH ₃) ₃ C COCHBrCH(CH ₃) ₂	Syntheses -Obtained by reaction of 2-br chloride with 2,6-di-tert-butylp sence of titanium tetrachloride -Also obtained by reaction of 4-hydroxy-3,5-di-tert-butylisov octane for 30 min at 70° (79 %	bhenol in the pre- [1468]. of bromine with valerophenone in

-Also refer to: [3238].

m.p. 113-115° [3238, 3239].

1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-3-fluoro-3-methyl-1-butanone



$C_{19}H_{29}FO_2$

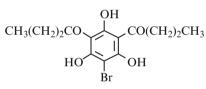
mol. wt. 308.4

Chapter 2 Butanones: Polyketones

1 Aromatic Hydroxyketones Derived from 1-Butanoic Acid

1,1'-(5-Bromo-2,4,6-trihydroxy-1,3-phenylene)bis-1-butanone

[2999-16-8] C₁₄H₁₇BrO₅ mol. wt. 345.19



 $CO(CH_2)_2CH_3$ Syntheses -Obtained by reaction of butyryl chloride OH with 2-bromophloroglucinol in the presence of boron trifluoride etherate (72–78 %) [3391].

-Also refer to: [457, 2911].

m.p. 146–148° [3391], 102–104° [457, 2911]. **N.B.**: One of the reported melting point is obviously wrong.

BIOLOGICAL ACTIVITY: Antischistosomal [3391].

1,1'-(4,6-Dihydroxy-5-nitro-1,3-phenylene)bis-1-butanone

[107522-52-1]	$\mathrm{C}_{14}\mathrm{H}_{17}\mathrm{NO}_{6}$	mol. wt. 2	295.29
NO ₂ HO CO(CH ₂) ₂ CH ₃	•	Fries rearrangement libutyrate (1 mol) with alum	
CO(CH ₂) ₂ CH ₃		: 100–110° for 3 h (33 %) [t 100–110° for 3 h (13 %) 13 %) [105].	

-Also obtained by reaction of butyric anhydride with 2-nitroresorcinol in the presence of aluminium chloride (3.3 mol) in nitrobenzene at 120–130° (56 %) [105].

m.p. 154° [105].

Diacetate	[107778-29-0]	$C_{18}H_{21}NO_8$	mol. wt. 379.37
-Refer to: [105];	m.p. 95° [105].		

1,1'-(2-Hydroxy-1,3-phenylene)bis-1-butanone

 $\begin{array}{cccc} [205067-94-3] & C_{14}H_{18}O_3 & \mbox{mol. wt. } 234.30 \\ & & CO(CH_2)_2CH_3 & Synthesis \\ & & -Refer to: [3450]. \\ & & N.B.: \ \mbox{Resonance-assisted} & \mbox{intramolecular hydrogen} \\ & & bonding [3450]. \end{array}$

1,1'-(2,4-Dihydroxy-1,3-phenylene)bis-1-butanone

[2999-22-6]	C ₁₄	$H_{18}O_4$	mol. wt. 250.29
	OH CO(CH ₂) ₂ CH ₃	Syntheses -Obtained by Fries rearran cinol dibutyrate with alu *at 130–135° for 4 h [265 *at 180–185° for 90 min	minium chloride, 51];
b.p. ₂₀ 190° [85	5, 2651]; m.p. 67°	[855].	

1,1'-(4,6-Dihydroxy-1,3-phenylene)bis-1-butanone

[2999-21-5]	$C_{14}H_{18}O_4$	mol. wt. 250.29
HO CO(CH ₂) ₂ CH ₃	dibutyrate with zinc c [2651].	earrangement of resorcinol hloride at 130° (40–50 %) 2,4-dihydroxyacetophenone

m.p. 64–65° [2651], 64° [385].

1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-butanone

phlorobutyrophenone in the presence of phosphorous pentoxide in methanesulfonic acid for 30 min at 70° under nitrogen atmosphere [1026]. -Also obtained by reaction of butyryl chloride,

*with phlorobutyrophenone in the presence of aluminium chloride in boiling carbon disulfide and nitrobenzene mixture for 2 h [1026];

*phloroglucinol in the presence of aluminium chloride in nitrobenzene for 3 days at r.t. (5–10 %) [421].

-Also obtained by reaction of butyric acid with phloroglucinol in the presence of boron trifluoride etherate [3019].

-Also refer to: [3033, 3391].

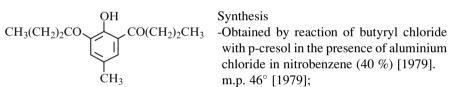
m.p. 180° [2646], 179–180° [1608, 1610, 2616, 2618], 135–137° [421], 126–128° [3391]; ¹H NMR [421, 3019], ¹³C NMR [1026, 3019], IR [421, 1026, 3019], UV [3019], MS [421].

BIOLOGICAL ACTIVITY: Antagonist both thromboxane A2 and Leukotriene D4 [3019]; Inhibitory effects of treatment with this acylphenol on TPA Induced EBV-EA Activation [3033].

1,1'-(2-Hydroxy-5-methyl-1,3-phenylene)bis-1-butanone

[88580-91-0]

C₁₅H₂₀O₃ mol. wt. 248.32



Synthesis

m.p. 46° [1979];

¹H NMR [1979], IR [1979], UV [1979].

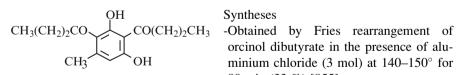
USE: For preparation of phenoxo-bridged macrocyclic dizinc (II) complex [2130].

1,1'-(2,6-Dihydroxy-4-methyl-1,3-phenylene)bis-1-butanone

[875854-88-9]

 $C_{15}H_{20}O_4$

mol. wt. 264.32



90 min (33 %) [855].

-Also obtained by treatment of 2,6-dihydroxy-4-methylbutyrophenone with butyric anhydride in the presence of aluminium chloride in nitrobenzene [854].

m.p. 67° [854, 855].

1,1'-[2,4,6-Trihydroxy-5-(2-propenyl)-1,3-phenylene]bis-1-butanone

2,4-Dibutyryl-6-(propen-2-yl)phloroglucinol (21) [1026]

 $C_{17}H_{22}O_5$

mol. wt. 306.36

 $\begin{array}{c} \text{OH} \\ \text{CH}_3(\text{CH}_2)_2\text{CO} \\ \text{HO} \\ \text{HO} \\ \text{CH}_2\text{CH} = \text{CH}_2 \end{array}$

Synthesis

IR [1026], MS [1026].

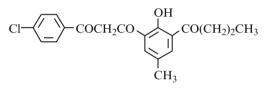
3-[2-(4-Chlorobenzoyl)acetyl]-2-hydroxy-5-methyl-1-butanone

 α -4-Chlorobenzoyl-2-hydroxy-5-methyl-3-butyrylacetophenone

[213622-31-2]

$C_{20}H_{19}ClO_4$

mol. wt. 358.82



Synthesis -Obtained by treatment of 1-[3-acetyl-2-(4-chlorobenzoyloxy)-5-methyl-phenyl]-1-butanone with powdered KOH in pyridine (Baker-Venkataraman rearrangement) (81 %) [344].

m.p. 126–127° [344]; ¹H NMR [344], IR [344].

1,1'-[Sulfonylbis(6-chloro-4-hydroxy-3,1-phenylene)]bis-1-butanone

[50444-95-6] CH₃(CH₂)₂CO $C_{20}H_{20}Cl_2O_5S$

mol. wt. 443.35

 $CO(CH_2)_2CH_3$ Synthesis

-Obtained by treatment of 4-chloro-2-hydroxybutyrophenone with thionyl chloride in the presence of aluminium chloride in carbon disulfide (52 %) [2430].

m.p. 227° [2430]; ¹H NMR [2430], IR [2430]. USE: Antifungal [2430].

3-(2-Benzoylacetyl)-2-hydroxy-5-methyl-1-butanone

 α -Benzoyl-2-hydroxy-5-methyl-3-butyrylacetophenone

 $[213622-30-1] C_{20}H_{20}O_4$ mol. wt. 324.38 OH COCH₂CO (-)CO(CH₂)₂CH₃ OH CH₃ OH CO(CH₂)₂CH₃ OB CO(CH₂)₂CH₃ CO(CH₂)CH₃ CO(CH₂)CH₃ CO(CH₂

m.p. 115–116° [344]; ¹H NMR [344], IR [344].

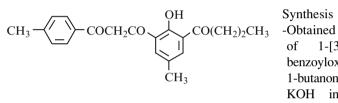
3-[2-(4-Methylbenzoyl)acetyl]-2-hydroxy-5-methyl-1-butanone

 α -4-Methylbenzoyl-2-hydroxy-5-methyl-3-butyrylacetophenone

[213622-32-3]

$C_{21}H_{22}O_4$

mol. wt. 338.40



-Obtained by treatment of 1-[3-acetyl-2-(4-methylbenzoyloxy)-5-methyl-phenyl]-1-butanone with powdered KOH in pyridine (Baker-Venkataraman rearrangement) (82 %) [344].

m.p. 134–135° [344]; ¹H NMR [344], IR [344].

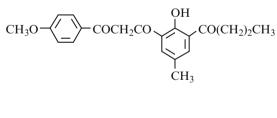
3-[2-(4-Methoxybenzoyl)acetyl]-2-hydroxy-5-methyl-1-butanone

 α -4-Methoxybenzoyl-2-hydroxy-5-methyl-3-butyrylacetophenone

[213622-33-4]

 $C_{21}H_{22}O_5$

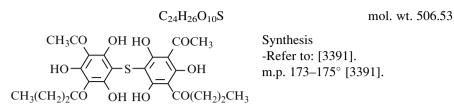
mol. wt. 354.40



Synthesis -Obtained by treatment of 1-[3-acetyl-2-(4-methoxybenzoyloxy)-5-methyl-phenyl]-1-butanone with powdered KOH in pyridine (Baker-Venkataraman rearrangement) (78 %) [344].

m.p. 118–119° [344]; ¹H NMR [344], IR [344].

1,1'-Thiobis[2,4,6-trihydroxy-3-(1-oxoethyl)-5,1-phenylene]bis-1-butanone



Aromatic Hydroxyketones Derived from 2-Methyl-2 **1-Butanoic Acid**

2,4,6-Trihydroxy-5-(2-methyl-1-oxobutyl)-1,3-benzenedicarboxaldehyde

C	$_{13}H_{14}O_{6}$	mol. wt. 266.25
$\begin{array}{c} OH & CH_3 \\ CHO & CO-CH-C_2H_5 \\ HO & OH \\ CHO \end{array}$	Isolation from natural sources -From the leaf essential oi <i>apodophylla</i> (Myrtaceae) [2046] MS [2046]; GC-MS [2046].	1 of <i>Eucalyptus</i> 5].

1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-2-methyl-1-butanone

[139409-36-2] C₁₆H₂₂O₅ mol. wt. 294.35 H Synuces $CO-CH-CH_2CH_3$ -Obtained CH_3 2-methyl-phloroglu CH₃CH₂-CH-CO CH₃ by reaction of 2-methyl-butanoic acid with phloroglucinol in the presence of boron trifluoride etherate [3019].

¹H NMR [3019], ¹³C NMR [3019], IR [3019], UV [3019],

BIOLOGICAL ACTIVITY: Antagonist both thromboxane A2 and Leukotriene D₄ [3019].

3 Aromatic Hydroxyketones Derived from 3-Methyl-1-Butanoic Acid

1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-3-methyl-1-butanone

2',4',6'-Trihydroxy-3'-isovalerylisovalerophenone

$$[2999-10-2] C_{16}H_{22}O_5 mol. wt. 294.35$$

$$(CH_3)_2CHCH_2CO + COCH_2CH(CH_3)_2 + ODE + ODE$$

(30 %) [621]. in nitrobenzene at 0° for 3 days (10 %) (**IX**) [898].

-Also obtained by reaction of isovaleric acid with phloroglucinol in the presence of boron trifluoride etherate [3019], (32 %) [2042], at 100° for 2 h (67 %) [338].

-Also obtained by reaction of isovaleric acid with phloroglucinol in the presence of titanium tetrachloride [3033].

-Also refer to: [337, 962, 1983, 2684, 2911].

cream coloured solid [338]; yellow prisms [2042]; m.p. 117–118° [2042], 114–115° [898], 113–114° [2911]; ¹H NMR [338, 898, 2042, 3019], ¹³C NMR [3019], IR [2042, 3019], UV [898, 3019], MS [338, 898].

BIOLOGICAL ACTIVITY: S-Euglobals: Biomimetic synthesis, antileishmanial, antimalarial, and antimicrobial activities [336]; Antagonist both thromboxane A_2 and Leukotriene D_4 [3019]; Effects on transpiration and stomatal closure [3408]; Inhibitory effects of treatment with this acylphenol on TPA Induced EBV-EA Activation [3033]; Industrial bactericidal and fungicidal agent, algicide and biofouling inhibitor [962]; For the prevention and treatment of bone and cartilage diseases [2684].

1,1'-(5-Ethyl-2-hydroxy-1,3-phenylene)bis-2-bromo-3-methyl-1-butanone

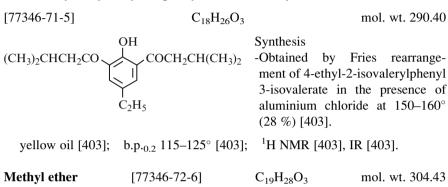
$C_{18}H_{24}Br_2O_3$	mol. wt. 448.19
(CH ₃) ₂ CHCHBrCO	Synthesis -Obtained by treatment of 1,1'-(5-ethyl-2-hydroxy- 1,3-phenylene)bis-3-methyl- 1-butanone with cupric bromide in ethyl acetate/chloroform for 2.5 h at r.t. under nitrogen (97 %) [403].
oil [403]; IR [403].	

Methyl ether [412340-39-7] C₁₉H₂₆Br₂O₃ mol. wt. 462.22

-Obtained by treatment of 4-ethyl-2,6-diisovalerylanisole in chloroform with a suspension of cupric bromide in ethyl acetate at reflux for 2.5 h (97 %) [403].

cognac coloured oil [403]; IR [403].

1,1'-(5-Ethyl-2-hydroxy-1,3-phenylene)bis-3-methyl-1-butanone



-Obtained by reaction of methyl iodide with the title diketone in the presence of potassium carbonate in acetone at r.t. for 20 h (95 %) [403].

-Preparation by treatment of 4-ethyl-2,6-diisovalerylphenol with methyl iodide in the presence of potassium carbonate in acetone at r.t. for 20 h (95 %) [403].

pale yellow oil [403]; b.p._{0.3} 110–115° [403]; ¹H NMR [403], IR [403].

1,1'-[2,4-Dihydroxy-6-(2-hydroxyethoxy)-1,3-phenylene]bis-3-methyl-1-butanone

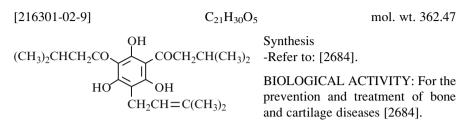
$[1103524-25-9] C_{18}H_{26}O_6 mol. wt. 338.4$
$(CH_3)_2CHCH_2CO \qquad \qquad OH \\ HO \qquad OCH_2CH_2CH(CH_3)_2 \\ HO \qquad OCH_2CH_2OH \qquad \qquad Synthesis \\ -Obtained by reaction of 2-bromoethanol with 2,4-diisoval erylphloroglucinol in the presence of potassium carbonate in aceton at r.t. for 10 h (50 %) [338].$

1,1'-[2,4-Dihydroxy-6-(2-propen-1-yloxy)-1,3-phenylene]bis-3-methyl-1-butanone

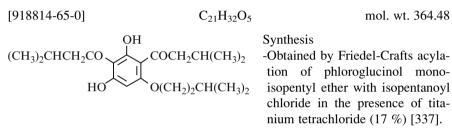
1 3-isovaleryliso-valerophenone in the presence of sodium methoxide in refluxing methanol for 2 h (60 %) [338].

yellow oil [338]; ¹H NMR [338], MS [338].

1,1'-[2,4,6-Trihydroxy-5-(3-methyl-2-butenyl)-1,3-phenylene]bis-3-methyl-1-butanone



1,1'-[2,4-Dihydroxy-6-(3-methylbutoxy)-1,3-phenylene]bis-3-methyl-1-butanone



yellow solid [337]; m.p. 65–67° [337]; ¹H NMR [337], ¹³C NMR [337], IR [337], UV [337], MS [337].

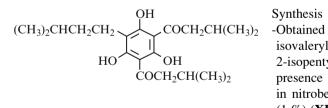
BIOLOGICAL ACTIVITY: Antiprotozoal and antimicrobial [337].

1,1'-[2,4,6-Trihydroxy-5-(3-methylbutyl)-1,3-phenylene]bis-3-methyl-1-butanone

[26104-02-9]

 $C_{21}H_{32}O_5$

mol. wt. 364.48



Synthesis -Obtained by reaction of isovaleryl chloride with 2-isopentylphloroglucinol in the presence of aluminium chloride in nitrobenzene at 0° for 3 days (1 %) (**XIV**) [898].

m.p. 109.5–111° [898]; ¹H NMR [898], UV [898], MS [898].

1,1'-(2,4-Dibutoxy-6-hydroxy-1,3-phenylene)bis-3-methyl-1-butanone

[1103524-20-4]	$C_{24}H_{38}O_5$	mol. wt. 406.56
$C_{4}H_{9}O \xrightarrow{OH} COCH_{2}CH(CH_{3})_{2}$ $C_{4}H_{9}O \xrightarrow{OC_{4}H_{9}} COCH_{2}CH(CH_{3})_{2}$	Synthesis -Obtained by reaction of n-bu 2,4-diisovaleroylphlorogluci sence of potassium carbona r.t. for 10 h (15 %) [338].	nol in the pre-

BIOLOGICAL ACTIVITY: A new class of GPR40 (FFAR1) agonists [338].

1,1'-(4,6-Dibutoxy-2-hydroxy-1,3-phenylene)bis-3-methyl-1-butanone

[1103524-19-1]

C₂₄H₃₈O₅

mol. wt. 406.56

 $(CH_3)_2CHCH_2CO$ C_4H_9O $COCH_2CH(CH_3)_2$ $COCH_3CH(CH_3)_2$ $COCH_3CH(CH_3)_2$ $COCH_3CH(CH_3)_2$ $COCH_3CH(CH_3)_2$ $COCH_3CH(CH_3)_2$ $COCH_3CH(CH_3)_2$

Synthesis of potassium carbonate in acetone at r.t. for 10 h (25 %) [338].

BIOLOGICAL ACTIVITY: A new class of GPR40 (FFAR1) agonists [338].

4 Aromatic Hydroxy-1,2-Butanediones

1-(4-Hydroxyphenyl)-1,2-butanedione

 $C_{10}H_{10}O_3$ mol. wt. 178.19 OH Synthesis -Refer to: [2165]. Methyl ether [10201-46-4] C₁₁H₁₂O₃ mol. wt. 192.21 COCOCH₂CH₃

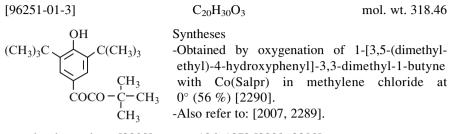
-Obtained by treatment of 1-(4-methoxyphenyl)-1-propyne with PdI₂ (2 mol%) in dimethyl sulfoxide at 140° for 1.5 h (58 %) [2165]. -Also refer to: [1114, 1405, 2986, 3217, 3230, 3232, 3307].

b.p._{0.2} 120–123° [3307], b.p.₁ 155° [1405], b.p.₂₅ 168–170° [3232]; ¹H NMR [3232], IR [3232]; $n_D^{20} = 1.5480$ [3307].

Dioxime of the methyl ether [10262-13-2] C₁₁H₁₄N₂O₃ mol. wt. 222.24

m.p. 207–208° [3307].

1-[3,5-(1,1-Dimethylethyl)-4-hydroxyphenyl]-3,3-dimethyl-1,2-butanedione



colourless prisms [2290]; m.p. 136–137° [2289, 2290]; ¹H NMR [2007, 2289, 2290], ¹³C NMR [2007], IR [2289, 2290], UV [2290].

5 Aromatic Hydroxy-1,3-Butanediones

5.1 Unsubstituted Aromatic Ring

1-(2-Hydroxyphenyl)-4,4,4-trifluoro-1,3-butanedione

 $\begin{array}{c} C_{10}H_7F_3O_3 & \text{mol. wt. } 232.16 \\ \hline OH & \\ COCH_2COCF_3 & -Obtained by treatment of o-hydroxyacetophenone \\ trifluoroacetate with base (pyridine/potassium hydroxide) \\ (Baker-Venkataraman rearrangement) [1564]. \end{array}$

Methyl ether	[15191-69-2]	$C_{11}H_9F_3O_3$	mol. wt. 246.19
	m.p. 36–38° [1086 ¹³ C NMR [2902], M		

2-Bromo-1-(4-hydroxyphenyl)-1,3-butanedione

	$C_{10}H_9BrO_3$	mol. wt. 257.08
OH COCHBrCOCH ₃	Synthesis -Refer to: [1094]. Methyl ether [91065-87-1] C ₁₁ H ₁₁ BrO ₃ -Obtained by treatment of the sodium of 1-(4-methoxyphenyl)-1,3-butanedione cooled carbon tetrachloride [1094].	11

-Also refer to: [2628].

Dirty white [1094]; m.p. 39° [1094].

2-Chloro-1-(2-hydroxyphenyl)-1,3-butanedione

 $C_{10}H_0ClO_3$ mol. wt. 212.63 OH **Synthesis** COCHClCOCH₃ -Refer to: [3400]. **Methyl ether** [1001024-95-8] C11H11ClO3 mol. wt. 226.66 -Obtained by reaction of N-chlorosuccinimide with 1-(2-hydroxyphenyl)-

1,3-butanedione in refluxing carbon tetrachloride for 4 h at $75-80^{\circ}$ (47 %) [3400].

yellow oil [3400]; ¹H NMR [3400], ¹³C NMR [3400], MS [3400]; GC-MS [3400].

1-(2-Hydroxyphenyl)-1,3-butanedione

[16636-62-7]

 $C_{10}H_{10}O_3$

Syntheses

mol. wt. 178.19

OH

COCH₂COCH₃ -Obtained by adding a solution of 2-hydroxyacetophenone in ethyl acetate to a suspension of pulverized sodium in ether [168, 947, (65 %) 3325].

-Also obtained by treatment of o-acetoxyacetophenone with metallic sodium in refluxing benzene for 3 h (poor yield) [3234].

-Also refer to: [383, 919, 1108, 1564, 3400].

colourless needles [3325]; pale yellow plates [3234]; m.p. 110° [1110], 102° [3234], 101° [234], 98° [1109], 90.5–91.5° [3325]; ¹H NMR [919, 2288, 2807, 3459], ¹³C NMR [3459], UV [3317], MS [2968].

Methyl ether [56290-53-0] $C_{11}H_{12}O_3$ mol. wt. 192.21

-Obtained by reaction of 2-methoxybenzoyl chloride with acetone in the presence of LDA (1.5 equiv.) in tetrahydrofuran (37 %) [3400].

-Also obtained from 2-methoxyacetophenone [2677].

-Also obtained from methyl 2-methoxybenzoate and acetone [383].

-Also refer to: [168, 942, 1011, 1207, 1413, 1856, 2313, 2526, 2628, 2935, 3259, 3459, 3463].

b.p.₂₀ 173–174° [2677]; m.p. 37° [2677], 36–37° [383], 36° [942]; ¹H NMR [3459], ¹³C NMR [3459].

Copper (II) salt

C22H22O6Cu

mol. wt. 445.96

m.p. 153° [2313].

Ethyl ether [60159-70-8] $C_{12}H_{14}O_3$ mol. wt. 206.24

-Obtained by reaction of 2-ethoxyacetophenone with ethyl acetate in the presence of sodium ethoxide [3083].

m.p. 58° [328], 57–58° [3083], 54–56° [101]; ¹H NMR [101], IR [101], UV [3083].

1-(3-Hydroxyphenyl)-1,3-butanedione

 $C_{10}H_{10}O_3$ OH Synthesis -Refer to: [3384]. Methyl ether [29681-99-0] COCH₂COCH₃ C₁₁H₁₂O₃ mol. wt. 192.21

-Obtained by reaction of 3-methoxyacetophenone with ethyl acetate in the presence of sodium ethoxide [3083].

obtained by Claisen condensation between -Also ethyl acetate and m-methoxyacetophenone in the presence of sodium amide [313].

-Also refer to: [34, 168, 314, 316, 1192, 1193, 1206, 1214, 2628, 2935, 3384].

b.p.₁ 115° [313], b.p. 280–283° [3083]; UV [313, 314, 316, 3083]; pK [313]; $n_D^{25} = 1.5930$ [313].

Cu salt

$$C_{22}H_{22}O_6Cu$$

-Refer to: [313]; m.p. 150° [313].

1-(4-Hydroxyphenyl)-1,3-butanedione

 $C_{10}H_{10}O_3$ [51944-08-2] mol. wt. 178.19 Syntheses OH -Obtained by treatment of p-acetoxyacetophenone in acetic anhydride with boron trifluoride-acetic acid complex for 2.5 h (78 %) [1538]. COCH₂COCH₃ -Also obtained by treatment of its acetate with sodium hydroxide in refluxing dilute methanol for 35 min [1538]. -Also refer to: [45, 294, 675, 774, 1292, 1433, 3384].

m.p. 114–116° [1433], 112–112.5° [1538], 112° [675]; ¹H NMR [1433], IR [1433], UV [1433].

mol. wt. 178.19

mol. wt. 445.96

Be salt	$C_{20}H_{18}O_6B_6$	2	mol. wt. 363.37	
-Refer to: [1538 (84 %)].				
m.p. 255° [1538].				
Cu salt	$C_{20}H_{18}O_6C$	u	mol. wt. 417.91	
-Refer to: [1538 (99 %)].				
m.p. 280–282° [1538].				
Mg salt	$C_{20}H_{18}O_6M$	g	mol. wt. 378.66	
-Refer to: [1538].				
m.p. >300° [1538].				
Zn salt	C ₂₀ H ₁₈ O ₆ Zi	1	mol. wt. 419.75	
-Refer to: [1538].				
m.p. >300° [1538].				
Acetate [9196	3-55-2]	$C_{12}H_{12}O_4$	mol. wt. 220.23	
-Obtained by action of	acetic anhydride		oylacetone in the	

-Obtained by action of acetic anhydride with p-hydroxybenzoylacetone in the presence of aqueous sodium hydroxide [1538].

m.p. 70–70.5° [1538], 70° [675].

Cu salt of the acetate	$C_{24}H_{22}O_8Cu$	mol. wt. 501.20
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m.p. 248° [1538].

	Methyl ether	[4023-80-7]	$C_{11}H_{12}O_3$	mol. wt. 192.21
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-Obtained by reaction of 4-methoxyacetophenone with ethyl acetate in the presence of sodium ethoxide [327, 3083].

-Also obtained by Claisen condensation between ethyl acetate and p-methoxyacetophenone in the presence of sodium amide [313]. -Obtained [2072] according to [2676].

-Also refer to: [45, 237, 294, 295, 314–316, 327, 330, 575, 612, 629, 630, 697, 744, 745, 774, 800, 819, 904, 960, 1015, 1031, 1072, 1094, 1141, 1190, 1191, 1207, 1214, 1220, 1241, 1275, 1292, 1350, 1433, 1920, 1952, 1959, 1976, 2230, 2502, 2525, 2853, 2872, 2897, 2898, 3257, 3359, 3360, 3384, 3446].

m.p. 57–58° [1433], 57° [612], 56.5° [2525], 55° [295],
54.5° [327, 1920], 54° [237, 1275, 1338, 1350, 2502], 53–54° [330], 53° [313],
52–54° [3083], 52–53° [1072, 1241], 51–53° [629], 48–51° [2230], 48–49° [2072];

¹H NMR [819, 1220, 1241, 1433, 1920, 2230], ¹³C NMR [745, 1220, 2230], IR [819, 1031, 1241, 1433, 1920, 2230], UV [313, 314, 316, 1031, 1433, 1920, 3083], MS [697, 1015, 1959, 2230].

C₁₁H₁₁O₃Na Na salt mol. wt. 215.20 -Refer to: [1094 (58 %)]. Cu salt C22H22O6Cu mol. wt. 445.96 -Refer to: [313, 1094 (60 %), 1920]. m.p. 229° [313], 220° [1920]. 1-(2,3-Dihydroxyphenyl)-1,3-butanedione mol. wt. 194.19 $C_{10}H_{10}O_4$ Synthesis $\begin{array}{c} \text{COCH}_2\text{COCH}_3 & \text{-Refer to: [48].} \\ & \text{Dimethyl ether} & [65547-52-6] \\ & \text{C} & \text{H} & \text{O} \end{array}$ HO. C12H14O4 mol. wt. 222.24

-Preparation from 2,3-dimethoxyacetophenone on Claisen condensation with ethyl acetate in the presence of pulverized sodium [48]. **N.B.**: Ratio of the keto and enol forms 1:3.

-Refer to: [48].

light yellow liquid [48]; ¹H NMR [48].

1-(2,4-Dihydroxyphenyl)-1,3-butanedione

	$C_{10}H_{10}O_4$		mol. wt. 194.19
OH COCH ₂ COCH ₃	Synthesis -Refer to: [48].		
	Dimethyl ether	[65547-54-8]	
HO	$C_{12}H_{14}O_4$		mol. wt. 222.24

-Preparation from 2,4-dimethoxyacetophenone (68 %) [2677].

-Preparation from 2,4-dimethoxyacetophenone on Claisen condensation with ethyl acetate in the presence of pulverized sodium [48, 3313].

-Preparation from 2,4-dimethoxybenzoyl chloride (75 %) [2914].

-Also refer to: [276, 1193, 1206, 2780].

N.B.: Ratio of the keto and enol forms 1:2 [48].

yellow glistening needles [48]; m.p. 68–69° [48], 67–68° [2914], 58.5° [2677]; ¹H NMR [48, 2914], ¹³C NMR [2914], IR [2914].

BIOLOGICAL ACTIVITY: As bronchodilatator [3313].

Diethyl ether $C_{14}H_{18}O_4$ mol. wt. 250.29

-Obtained by reaction of 2,4-diethoxyacetophenone with ethyl acetate in the presence of sodium [3083].

-Also refer to: [382, 1740].

m.p. 90° [382, 3083]; UV [3083].

1-(2,5-Dihydroxyphenyl)-1,3-butanedione

 $\begin{array}{c} C_{10}H_{10}O_4 & \text{mol. wt. 194.19} \\ OH & Synthesis \\ -Refer to: [3313]. \\ \textbf{Dimethyl ether} & [65547-50-4] \\ C_{12}H_{14}O_4 & \text{mol. wt. 222.24} \end{array}$

-Preparation from 2,5-dimethoxyacetophenone on Claisen condensation with ethyl acetate in the presence of pulverized sodium (49 %) [48, 3313].

N.B.: Ratio of the keto and enol forms 1:2.

-Also refer to: [48, 168, 1206, 3312].

light yellow liquid [48]; b.p._{0.1} 97° [3312]; ¹H NMR [48]; $n_D^{25} = 1.5893$ [3312].

С

BIOLOGICAL ACTIVITY: As bronchodilatator [3313].

Diethyl ether

-Obtained by reaction of 2,5-diethoxyacetophenone with ethyl acetate in the presence of sodium [3083].

-Also refer to: [752].

m.p. 60° [752], 58–59° [3083]; UV [3083].

1-(3,4-Dihydroxyphenyl)-1,3-butanedione

 $C_{10}H_{10}O_4$ mol. wt. 194.19

ОН	Synthesis		
ОН	-Refer to: [3083].		
	Dimethyl ether	[13298-49-2]	
\mathbf{i}	$C_{12}H_{14}O_4$		mol. wt. 222.24
COCH ₂ COCH ₃			

-Obtained by reaction of 3,4-dimethoxyacetophenone with ethyl acetate in the presence of sodium [3083].

b.p._{0.05} 132° [1338]; m.p. 71–72° [3083], 69–71° [464], 62° [1338]; IR [464], UV [464, 3083], MS [1959].

1-(2,4,5-Trihydroxyphenyl)-1,3-butanedione

$$\begin{array}{cccc} & & C_{10}H_{10}O_5 & & \text{mol. wt. 210.19} \\ & & & Synthesis \\ & & & -\text{Refer to: [834].} \\ & & & & \text{Trimethyl ether} & [62406-99-9] \\ & & & & C_{13}H_{16}O_5 & & \text{mol. wt. 252.27} \end{array}$$

-Obtained by gently heating a mixture of 2,4,5-trimethoxyacetophenone, sodium sand and ethyl acetate until a vigorous reaction set in, then to heat the mixture at reflux for 2 h (76 %) [834].

N.B.: Ratio of the keto and enol forms 1:9 -Also refer to: [48].

pale yellow plates [834]; m.p. 89° [834]; ¹H NMR [48, 834], IR [834].

Na salt $C_{13}H_{15}O_5Na$ mol. wt. 274.25

m.p. 201° (d) [834].

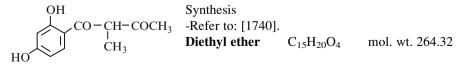
1-(2-Hydroxyphenyl)-2-methyl-1,3-butanedione

[35115-14-1]	$C_{11}H_{12}O_3$	mol. wt. 192.21
OH CO-CH-COCH ₃	Synthesis -Obtained by treatment of dilute alkali [1126]. b.p. ₇ 130° [2288]; ¹ H NM	2,3-dimethylchromone with /IR [2288].

1-(4-Hydroxyphenyl)-2-methyl-1,3-butanedione

[91142-94-8]	$C_{11}H_{12}O_3$	mol. wt. 192.21
CO-CH-COCH ₃	Synthesis -Refer to: [675]. m.p. 59–60° [675].	

1-(2,4-Dihydroxyphenyl)-2-methyl-1,3-butanedione



-Obtained by reaction of ethyl acetate with 2,4-diethoxypropiophenone in the presence of pulverized sodium [1740].

m.p. 72.5° [1740].

1-(2,4,5-Trihydroxyphenyl)-2-methyl-1,3-butanedione

$$\begin{array}{c} C_{11}H_{12}O_5 & \text{mol. wt. } 224.21 \\ OH & CH_3 & Synthesis \\ -Refer to: [834]. \\ \textbf{Trimethyl ether} & [62407-00-5] \\ C_{14}H_{18}O_5 & \text{mol. wt. } 266.29 \end{array}$$

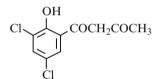
-Obtained by adding 1-(2,4,5-trihydroxyphenyl)-1,3-butanedione in THF to 50 % sodium hydride dispersion in oil. After isolation of the sodium salt, this one was warmed with methyl iodide for 4 h (64 %) [834].

pale yellow plates [834]; m.p. 114.5° [834]; ¹H NMR [834], IR [834].

5.2 Substituted Aromatic Ring

1-(3,5-Dichloro-2-hydroxyphenyl)-1,3-butanedione

C₁₀H₈Cl₂O₃ mol. wt. 247.08



COCH₂COCH₃ Synthesis -Obtained by treatment of 3,5-dichloro-2-hydroxyacetophenone with ethyl acetate and sodium metal [947].

1-(4,5-Dichloro-2-hydroxyphenyl)-1,3-butanedione

[111477-92-0] $C_{10}H_8Cl_2O_3$ mol. wt. 247.08 OH Syntheses -Obtained by treatment of 4,5-dichloro-2-hydroxyacetophenone with ethyl acetate and sodium metal [947]. -Also refer to: [1889, 2472].

m.p. 134–135° [1889, 2472].

1-(5-Bromo-2-hydroxyphenyl)-1,3-butanedione

$$\begin{array}{c} C_{10}H_9BrO_3 & \mbox{mol. wt. } 257.08 \\ Syntheses \\ \mbox{COCH}_2COCH_3 & -Obtained by treatment of 5-bromo-2-hydroxyace-tophenone with ethyl acetate and sodium metal [947].} \\ \ -Also refer to: [1564]. \end{array}$$

mol wt 226.66

1-(5-Chloro-2-hydroxyphenyl)-1,3-butanedione

¹H NMR [2691], ¹³C NMR [2691], IR [2691].

Methyl ether	[861349-33-9]	$C_{11}H_{11}ClO_3$	mol. wt. 226.66
meenyr cuici		Chilleros	11101. 111. 220.00

-Obtained by treatment of 5-chloro-2-methoxyacetophenone with ethyl acetate in the presence of sodium [3324].

m.p. 76.5–77.5° [3324].

1-(3-Chloro-2-hydroxy-5-methylphenyl)-1,3-butanedione

 $C_{11}H_{11}ClO_2$

	emmetos	mon. wt. 220.00
CI CH CH ₃ CH ₃ CH ₂ COCH ₂ COCH ₃	Synthesis -Obtained by treatment of 5-methyl-acetophenone with sodium metal [947].	

1-(5-Chloro-2-hydroxy-4-methylphenyl)-1,3-butanedione

pale yellow crystals [3324]; m.p. 115.5–116.5° [3324, 3325].

1-(2-Hydroxy-5-methylphenyl)-1,3-butanedione

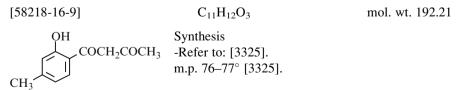
[16636-64-9] C11H12O3 mol. wt. 192.21 **Syntheses** OH COCH₂COCH₃ -Obtained by adding a solution of 2-hydroxy-5-methylacetophenone in ethyl acetate to a suspension of pulverized sodium in ether, [(64 %) 235, 947, (60 %) 3325]. -Also refer to: [268, 919, 1108–1110, 1011, 2745, 2746, 33241. colourless needles [3325]; compact prisms [235]; m.p. 112° [1110], 110° [1108], 102° [1109], 99° [235], 94.5–96° [3324, 3325], 94–96° [268]; ¹H NMR [919]. Dioxime [56686-34-1] $C_{11}H_{14}N_2O_3$ mol. wt. 222.24 m.p. 122–123° [268, 1011]; ¹H NMR [268]. Methyl ether [56290-52-9] C₁₂H₁₄O₃ mol. wt. 206.24 -Obtained condensation of ethyl acetate with by 2-methoxy-5-methylacetophenone [193]. -Also refer to: [942, 1338]. $b.p._{0.1}$ 115° [942, 1338], $b.p._{15}$ 182–183° [193]; $n_D^{17.9} = 1.5758$ [193]. 1-(2-Hydroxy-3-methylphenyl)-1,3-butanedione [58218-17-0] $C_{11}H_{12}O_3$ mol. wt. 192.21

OH COCH₂COCH₃ Synthesis -Refer to: [3325]. m.p. 85–86° [3325], 84–85° [268].

Dioxime	[56686-30-7]	$C_{11}H_{14}N_2O_3$	mol. wt. 222.24

m.p. 150–151° [3325], 148–149° [268]; ¹H NMR [268].

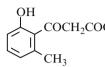
1-(2-Hydroxy-4-methylphenyl)-1,3-butanedione



mol. wt. 445.96

1-(2-Hydroxy-6-methylphenyl)-1,3-butanedione

C11H12O3 mol. wt. 192.21



Synthesis COCH₂COCH₃ -Obtained by adding a solution of 2-hydroxy-6-methylacetophenone in ethyl acetate to a suspension of pulverized sodium in ether (85-90 %) [3325].

colourless needles [3325]; m.p. 85–86° [3325].

1-(4-Hydroxy-3-methylphenyl)-1,3-butanedione

[92016-84-7] C11H12O3 mol. wt. 192.21 Synthesis CH₃ -Preparation from 4-acetoxy-3-methylacetophenone [1538]. m.p. 95–96° [1538]. COCH₂COCH₃

C22H22O6Cu

Copper salt

m.p. 280–282° [1538].

1-(2,4-Dihydroxy-3-methylphenyl)-1,3-butanedione

($C_{11}H_{12}O_4$		mol. wt. 208.21
CH ₃ HO	Synthesis -Refer to: [55]. Dimethyl ether $C_{13}H_{16}O_4$	[124259-63-8]	mol. wt. 236.27

-Obtained by treatment of 2,4-dimethoxy-3-methylacetophenone with ethyl acetate and sodium metal (65 %) [55].

oil [55]; ¹H NMR [55].

1-(2,4-Dihydroxy-6-methylphenyl)-1,3-butanedione

 $C_{11}H_{12}O_4$ mol. wt. 208.21 Synthesis COCH₂COCH₃ -Refer to: [55]. **Dimethyl ether** [53270-35-2] $C_{13}H_{16}O_{4}$ HC mol. wt. 236.27

-Obtained by deacetalisation of 1-(2,4-dimethoxy-6-methylphenyl)-2-(2-methyl-1,3-dioxolan-2-yl)ethanone (m.p. 54°) with concentrated hydrochloric acid in dilute acetone (95 %) [1330].

-Also obtained by decarboxylation of 5-(2,4-dimethoxy-6-methylphenyl)-3,5-dioxo-1-pentanoic acid on heating [2974] in refluxing chloroform for 5 h in a stream nitrogen (95 %) [1330].

-Also obtained by refluxing a mixture of 2,4-dimethoxy-6-methylacetophenone, ethyl acetate and sodium metal [2974] for 1 h $(115-120^{\circ})$ (88 %) [47], (68 %) [55], (62 %) [2813].

-Also refer to: [1168].

yellow needles [47]; m.p. 77–78° [1168], 74–76° [47, 55, 2813], 72–73° [454, 1330, 2974], 65-66.5° [1259]: ¹H NMR [47, 55, 454, 1330, 2974], IR [454, 1330, 2974], UV [1330, 2974], MS [454, 1330, 2974].

N.B.: In the paper [55], this compound is called by error 3-(2',6'-dimethoxy-3'-methylphenyl)-1-methylpropane-1,3-dione.

 $C_{25}H_{24}O_{4}$ **Dibenzyl ether** [82883-60-1] mol. wt. 388.46

-Obtained by deacetalisation of 1-(2.4-dibenzyloxy-6-methylphenyl)-2-(2-methyl-1,3-dioxolan-2-yl)ethanone (m.p. 82–83°) with concentrated hydrochloric acid in dilute acetone (97 %) [454].

-Also refer to: [453, 455].

OH

[101396-11-6]

m.p. 83° [453, 454]; ¹H NMR [453, 454], IR [453, 454], MS [454].

1-(2-Hydroxy-3-methoxyphenyl)-1,3-butanedione

[65547-78-6]	$C_{11}H_{12}O_4$	mol. wt. 208.21
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C11H12O4

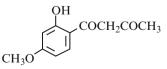
Synthesis

COCH₂COCH₃ -Obtained by adding a solution of 2-hydroxy-3-methoxy-acetophenone in ethyl acetate to a suspension of pulverized sodium in ether, then to warm at reflux for 3 h (48 %) [48].

yellow small needles [48]; m.p. 66–68° [48]; ¹H NMR [48].

1 (2-Hydrovy-4-methovynhenyl)-1.3-butanedione

mol. wt. 208.21



Syntheses COCH₂COCH₃ -Obtained by treatment of 2-acetoxy-4-methoxyacetophenone with metallic sodium in refluxing benzene for 3 h (poor yield) [3234]. -Also obtained from 7-methoxy-2-methyl-4H-1-benzopyran-4-one [1740].

-Also refer to: [234, 276, 2216, 2780].

vellowish brown needles [3234]; m.p. 75° [3234], 71–72° [234], 68° [2216].

Copper salt Refer to: [3234].

1-(2-Hydroxy-5-methoxyphenyl)-1,3-butanedione

[65547-81-1]

C11H12O4

mol. wt. 208.21

OCH3

Syntheses COCH₂COCH₃ -Obtained by adding a solution of 2-hydroxy-5-methoxyacetophenone in ethyl acetate to a suspension of pulverized sodium in ether (64 %) [48, 3312, 3313].

> -Also obtained by catalytic debenzylation of 2-benzyloxy-4,5-dimethoxybenzoylacetone in the presence of 5 % Pd/C in ethyl acetate (quantitative yield) [48].

-Also obtained by irradiation of 4-methoxyphenyl 3-oxobutanoate with a 125 W medium pressure mercury lamp inside a quartz immersion well in for 6 h at r.t. (25 %) [96].

-Also obtained by of 3-(ethylenedioxy)-1-(2-hydroxytreatment 5-methoxylphenyl)-1-butanone with dilute sulfuric acid in the presence of silica gel [96].

-Also obtained by reaction of 2-hydroxy-5-methoxyacetophenone with ethyl acetate in the presence of sodium hydride [1540].

vellow needles [48]: m.p. 102–104° [3312], 102–103° [48], 101–102° [1540]; ¹H NMR [48, 96], IR [48].

BIOLOGICAL ACTIVITY: As bronchodilatator [3313].

Benzyl ether [65547-83-3] $C_{18}H_{18}O_4$ mol. wt. 298.34

-Obtained by Claisen condensation of 2-benzyloxy-5-methoxyacetophenone (m.p. $44-45^{\circ}$) with ethyl acetate (quantitative yield) [48].

vellowish needles [48]; m.p. 59–60° [48].

1-(2-Hydroxy-6-methoxyphenyl)-1,3-butanedione

[65547-63-9] C ₁	$_{1}H_{12}O_{4}$	mol. wt. 208.21
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OН OCH₂

Syntheses COCH₂COCH₃ -Obtained by adding a solution of 2-hydroxy-6-methoxyacetophenone in ethyl acetate to a suspension of pulverized sodium [48], (48 %) [2564].

colourless needles [48, 2564]; m.p. 96–97° [48], 94–95° [2564]; ¹H NMR [2968], ¹³C NMR [2968], MS [2968].

Copper salt green crystals [2564];

m.p. 226° [2564].

Methyl ether	[32085-87-3]	$C_{12}H_{14}O_{4}$	mol. wt. 222.24
menyi culci		012111404	11101. Wt. 222.21

-Preparation from 2,6-dimethoxyacetophenone on Claisen condensation with ethyl acetate in the presence of pulverized sodium (57 %) [48]. **N.B.**: Ratio of the keto and enol forms 1:2.

colourless needles [48]; b.p.₅₋₆ 178–180° [240]; m.p. 49–50° [48]; ¹H NMR [48].

1-(2,4,6-Trihydroxy-3-methylphenyl)-1,3-butanedione

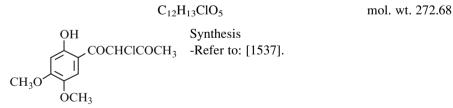
$C_{11}H_{12}O_5$	mol. wt. 224.21
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 $\begin{array}{c} OH \\ CH_3 \\ HO \end{array} COCH_2COCH_3 \\ HO \\ OH \end{array}$

1-(5-Chloro-2-hydroxy-4,6-dimethylphenyl)-1,3-butanedione

 $[855242-08-9] C_{12}H_{13}ClO_3 mol. wt. 240.69$ $OH COCH_2COCH_3 -Refer to: [24]. m.p. 116-117^{\circ} [24].$

1-(2-Hydroxy-4,5-dimethoxyphenyl)-2-chloro-1,3-butanedione



1-(2-Hydroxy-3,5-dimethylphenyl)-1,3-butanedione

[104516-36-1]	$C_{12}H_{14}O_3$	mol. wt. 206.24
CH ₃ COCH ₂ COCH ₃ CH ₃	3,5-dimethyl-acetopheno	solution of 2-hydroxy- one in ethyl acetate to erized sodium in ether

colourless needles [3325]; m.p. 116–117° [3325], 85° [1016].

Methyl ether

C13H16O3

mol. wt. 220.27

-Obtained by adding a solution of 2-methoxy-3,5-dimethylacetophenone in ethyl acetate to a suspension of pulverized sodium in ether (57 %) [3325].

colourless needles [3325]; m.p. 59–60° [3325].

1-(2-Hydroxy-4,6-dimethylphenyl)-1,3-butanedione

[861778-02-1]	$C_{12}H_{14}O_3$	mol. wt. 206.24
CH ₃ CH ₃ OH COCH ₂ COCH ₃ CH ₃	Syntheses -Refer to: [3325]. m.p. 116–117° [3325].	

1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1,3-butanedione

[58530-24-8]	$C_{12}H_{14}O_4$	mol. wt. 222.24

	OH		
	\checkmark	,COC	H ₂ COCH ₃
CH ₃ O	\sim	CH ₃	

Syntheses -Obtained by refluxing a mixture of 2-hydroxy-4-methoxy-6-methylacetophenone, ethyl acetate, ethyl ether and sodium metal for 4 h (65 %) [47]. -Also obtained by decarboxylation of 5-(2-hydroxy-4-methoxy-6-methylphenyl)-3,5-dioxopentanoic acid on heating [2974]. -Also refer to: [53].

yellow needles [47]; m.p. 142–144° [47].

Benzyl ether [68436-75-9] $C_{19}H_{20}O_4$ mol. wt. 312.37

-Obtained by adding a solution of 2-hydroxy-4-methoxy-6-methylacetophenone in ethyl acetate to a suspension of pulverized sodium in ether. The mixture was refluxed on a water bath for 2–3 h (58 %) [53]. N.B.: Ratio of the keto and enol forms 1:5

colourless needles [53]; m.p. 76–77° [53]; ¹H NMR [53].

[75160-45-1] $C_{15}H_{18}O_{4}$ Allyl ether mol. wt. 262.31

-Obtained by adding a solution of 2-allyloxy-4-methoxy-6-methylacetophenone in ethyl acetate to pulverized sodium in ethyl ether.

Then, the mixture was refluxed on a water bath for 3 h (56 %) [54].

-Refer to: [1127, 2559].

b.p._{0.3} 135–136° [2559], b.p.₁ 149–152° [1127]; light brown needles [54]; m.p. 47–49° [54]; ¹H NMR [54].

1-(2-Hydroxy-4-methoxyphenyl)-2-methyl-1,3-butanedione

$$C_{12}H_{14}O_4$$
 mol. wt. 222.24

 CH_{3O} $CH_{$

1-(2-Hydroxy-3,4-dimethoxyphenyl)-1,3-butanedione

$$[65547-75-3] C_{12}H_{14}O_5 mol. wt. 238.24$$

Synthesis

-Obtained by adding a solution of 2-hydroxy-3,4-dimethoxyacetophenone in ethyl acetate to a suspension of pulverized sodium in ether [48, 3313].

yellow needles [48]; m.p. 107–108° [48], 104–107° [3312]; ¹H NMR [48], IR [48].

BIOLOGICAL ACTIVITY: As bronchodilatator [3313].

Methyl ether [65547-48-0] C₁₃H₁₆O₅ mol. wt. 252.27

-Preparation from 2,3,4-trimethoxyacetophenone on Claisen condensation with ethyl acetate in the presence of pulverized sodium [48, 3313]. **N.B.**: Ratio of the keto and enol forms 1:9 [48].

colourless glistening plates [48]; b.p.₂₀ 217° [482]; m.p. 65° [384], 64–65° [48], 59–60° [482]; ¹H NMR [48].

BIOLOGICAL ACTIVITY: As bronchodilatator [3313].

1-(2-Hydroxy-3,6-dimethoxyphenyl)-1,3-butanedione

 $[65547-62-8] C_{12}H_{14}O_5 mol. wt. 238.24$ $CH_3O + COCH_2COCH_3 OCH_3 OC$

*in ethyl ether (33 %) [48];

*in methanol (65 %) [3206].

yellow needles [48]; m.p. 113–114° [48], 112–114° [3206].

Methyl ether [65547-46-8] $C_{13}H_{16}O_5$ mol. wt. 252.27

-Preparation from 2,3,6-trimethoxyacetophenone on Claisen condensation with ethyl acetate in the presence of pulverized sodium [48]. **N.B.**: Ratio of the keto and enol forms 1:2.

vellow needles [48]; m.p. 87–89° [48]; ¹H NMR [48].

1-(2-Hydroxy-4,5-dimethoxyphenyl)-1,3-butanedione

Benzyl ether [65547-70-8] $C_{10}H_{20}O_5$ mol. wt. 328.36

-Obtained by Claisen condensation of 2-benzyloxy-4,5-dimethoxyacetophenone (m.p. 121°) with ethyl acetate (70 %) [48].

yellow needles [48]; m.p. 151° [48].

1-(2-Hydroxy-4,6-dimethoxyphenyl)-1,3-butanedione

[65547-60-6]	$C_{12}H_{14}O_5$	mol. wt. 238.24
CH ₃ O ^{OH} COCH ₂ COCH ₃ CH ₃ O ^{OCH₃}	4,6-dimethoxyacetopher suspension of pulverize	solution of 2-hydroxy- none in ethyl acetate to a ed sodium in ether. Then, ed for 1.5 h (67 %) [48],

(37 %) [3313].

N.B.: Ratio of the keto and enol forms 3:1 [48].

-Also refer to: [2144, 2178].

colourless cubic crystals [48]; white solid [3313]; m.p. 83-85° [3313], 82-83° [48], 82° [1943]; ¹H NMR [48], IR [48].

BIOLOGICAL ACTIVITY: As bronchodilatator [3313].

-Obtained by adding a solution of 2-hydroxy-4,6-dimethoxyacetophenone in ethyl acetate to a suspension of pulverized sodium in ether. The mixture was refluxed on a water bath for 3 h (48 %) [53].

N.B.: Ratio of the keto and enol forms 4:3 [53].

oil [53]; ¹H NMR [53].

Allyl ether	[75160-39-3]	$C_{15}H_{18}O_5$	mol. wt. 278.30
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-Obtained by adding a solution of 2-allyloxy-4,6-dimethoxyacetophenone in ethyl acetate to pulverized sodium in ethyl ether. Then, the mixture was refluxed on a water bath for 3 h (51 %) [54].

oil [54]; ¹H NMR [54].

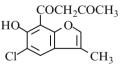
1-(6-Hydroxy-2,3-dimethoxyphenyl)-1,3-butanedione

$$[65547-71-9] C_{12}H_{14}O_5 mtext{mol. wt. 238.24} \\ OH \\ COCH_2COCH_3 \\ OCH_3 \\ OCH_3 \\ OCH_3 \\ COCH_3 \\ OCH_3 \\ COCH_3 \\ OCH_3 \\ COCH_3 \\ OCH_3 \\ COCH_3 \\ COCH$$

liquid [2564]; light yellow needles [48]; m.p. 93–95° [48]; ¹H NMR [48], IR [48].

Copper salt m.p. 234° [2564].

1-(5-Chloro-6-hydroxy-3-methyl-7-benzofuranyl)-1,3-butanedione $C_{13}H_{11}ClO_4$ me



Synthesis

-Obtained by reaction of ethyl acetate with 7-acetyl-5-chloro-6-hydroxy-3-methylcoumarone in the presence of sodium at reflux for 4 h [2828].

mol. wt. 266.7

m.p. 124° [2828].

1-(6-Hydroxy-3-methyl-7-benzofuranyl)-1,3-butanedione

[100397-26-0] C₁₃H₁₂O₄ mol. wt. 232.24

 I₃ Syntheses
 -Obtained by reaction of ethyl acetate with 7-acetyl-6-hydroxy-3-methylcoumarone in the presence of sodium at reflux for 4 h [2828].

-Also obtained by treatment of 7-acetyl-6-acetyloxy-3-methylbenzofuran in pyridine with powdered KOH for 50 min (Baker-Venkataraman rearrangement) (87 %) [2219].

-Also refer to: [1881].

m.p. 105° [2219], 91–92° [1881], 90–91° [2828]; ¹H NMR [2219], IR [2219].

1-[2,4-Dihydroxy-6-(2-propenyloxy)phenyl]-1,3-butanedione

($C_{13}H_{14}O_5$	mol. wt. 250.25
HO OH COCH ₂ COCH ₃ HO OCH ₂ CH=CH ₂	Synthesis -Refer to: [54].	

1-(2-Hydroxy-3,4-dimethoxy-6-methylphenyl)-1,3-butanedione

-Obtained by Claisen condensation of 2,3,4-trimethoxy-6-methylacetophenone with ethyl acetate in the presence of pulverized sodium in ethyl ether. Then, the mixture was refluxed for 1.5 h at $115-120^{\circ}$ (67 %) [52].

colourless needles [52]; $55-56^{\circ}$ [52].

1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-1,3-butanedione

[211751-37-0]	$C_{13}H_{16}O_5$	mol. wt. 252.27
CH ₃ CH ₃ O CH ₃ O OCH ₃	2-hydroxy-4,6-din in the presence of	action of ethyl acetate with nethoxy-3-methylacetophenone sodium powder, on an boiling n, then at r.t. overnight (55 %)
-Also refer to: [2178].		
m.p. 116–117° [2756].		
Methyl ether	$C_{14}H_{18}O_5$	mol. wt. 266.29

Methyl ether

m.p. 70–71° [2756].

OCH₃

OCH3

1-(2,4,6-Trimethoxyphenyl)-1,3-butanedione

(Eugenone)

CH₂O

Syntheses

COCH₂COCH₃ -Obtained from 2,4,6-trimethoxyacetophenone on Claisen condensation with ethyl acetate in the presence of pulverized sodium (58 %) [48], (32 %) [2757].

-Obtained by reaction of ethyl acetate with 2,4,6-trimethoxyacetophenone in the presence of sodium sand [3313].

N.B.: Ratio of the keto and enol forms 1:2 [48].

-Also obtained by adding a solution of tritylpotassium in DME to a solution of 2,4,6-trimethoxy-acetophenone in THF and to keep the mixture at r.t. (37 %) [1690].

-From biogenetic-type synthesis of [2137].

-Also refer to: [50, 53, 263, 1519, 1943, 2040, 2178, 3312].

Isolation from natural sources

-From the essential oil of *Pimpinella acuminata* seed (umbelliferone) (0.6 %) [163]. -From the oil of Eugenia carvophyllata [2040].

white solid [48]; sublimation 100–105°/0.008 Torr [2040]; m.p. 106° [1943], 102–104° [1690], 101–102° [48], 99–100° [2040], 97–99° [2757], 96–97° [3312, 3313], 94–95° [1519]; ¹H NMR [48, 1690], IR [1690], UV [1690, 2757], MS [163, 1690]; GLC [163].

BIOLOGICAL ACTIVITY: As bronchodilatator [3313].

1-(2-Hydroxy-3,4,5-trimethoxyphenyl)-1,3-butanedione

[67231-46-3]	$C_{13}H_{16}O_{6}$	mol. wt. 268.27
CH ₃ O CH ₃ O OCH ₃ O OCH ₃ O	3,4,5-trimethoxyacetoph in the presence of pul- ether. Then, the mixtur	ndensation of 2-hydroxy- enone with ethyl acetate verized sodium in ethyl e was refluxed for 1.5 h solvent elimination

¹H NMR [52].

Methyl ether [67231-43-0] C₁₄H₁₈O₆ mol. wt. 282.29

-Obtained by Claisen condensation of 2,3,4,5-tetramethoxyacetophenone with ethyl acetate in the presence of pulverized sodium in ethyl ether. Then, the mixture was refluxed for 1.5 h at $115-120^{\circ}$, after solvent elimination [52].

¹H NMR [52].

1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1,3-butanedione

[875850-72-9]	$C_{13}H_{16}O_{6}$		mol. wt. 268.27
CH ₃ O CH ₃ O CH ₃ O OCH ₃	Syntheses -Obtained by 3,4,6-trimethox in the presence -Also refer to: [5	y-acetophenone of sodium (68 %	with ethyl acetate (6) [595].

light fawn-coloured plates [595]; m.p. 123–124° [595], 117–119° [3312].

BIOLOGICAL ACTIVITY: As bronchodilatator [3313].

Methyl ether	[67231-33-8]	$C_{14}H_{18}O_{6}$	mol. wt. 282.29
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-Obtained by Claisen condensation of 2,3,4,6-tetramethoxyacetophenone with ethyl acetate in the presence of pulverized sodium in ethyl ether. Then, the mixture was heated on a boiling water bath for 3 h (60 %) [52]. -Also refer to: [3312, 3313].

yellow solid [52]; m.p. 76–78° [3312], 55–56° [52]; ¹H NMR [52].

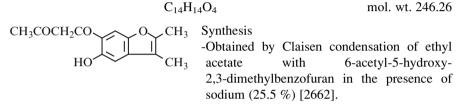
BIOLOGICAL ACTIVITY: As bronchodilatator [3313].

1-(2-Hydroxy-4,5,6-trimethoxyphenyl)-1,3-butanedione

$$\begin{array}{c} C_{13}H_{16}O_6 & \text{mol. wt. } 268.27 \\ OH & Synthesis \\ COCH_2COCH_3 & -Obtained by condensation of 2-hydroxy- \\ 4,5,6-trimethoxy-acetophenone with ethyl acetate \\ in the presence of sodium (68 %) [595]. \end{array}$$

pale cream-coloured glistening plates [595]; m.p. 141–142° [595].

1-(5-Hydroxy-2,3-dimethyl-6-benzofuranyl)-1,3-butanedione



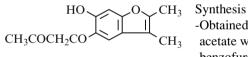
m.p. 151° [2662].

1-(6-Hydroxy-2,3-dimethyl-5-benzofuranyl)-1,3-butanedione

[4196-61-6]

 $C_{14}H_{14}O_4$

mol. wt. 246.26



-Obtained by Claisen condensation of ethyl

acetate with 5-acetyl-6-hydroxy-2,3-dimethylbenzofuran in the presence of sodium (14.5 %) [2662].

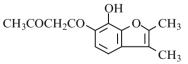
m.p. 163-164° [2662].

1-(7-Hydroxy-2,3-dimethyl-6-benzofuranyl)-1,3-butanedione

[4349-54-6]



mol. wt. 246.26



Synthesis -Obtained by Claisen condensation of ethyl acetate with 6-acetyl-7-hydroxy-2,3-dimethylbenzofuran in the presence of sodium (7 %) [2662].

m.p. 141-142° [2662].

1-(6-Hydroxy-3-methyl-7-benzofuranyl)-2-methyl-1,3-butanedione

 $C_{14}H_{14}O_{4}$ mol. wt. 246.26

Synthesis CH₃ CO-CH-COCH₃ -Obtained by reaction of ethyl acetate with 6-hydroxy-3-methyl-7-propionylcoumarone in the presence of HO sodium at reflux for 6 h [2828]. m.p. 96° [2828].

1-[4-Hvdroxy-2-methyl-6-(2-propenyloxy)phenyl]-1,3-butanedione



1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-2-methyl-1,3-butanedione

[854460-42-7]		$C_{14}H_{18}O_6$	mol. wt. 282.29
	~~~	a 1 i	

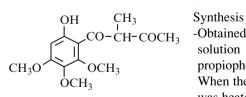
$$CH_{3}O \xrightarrow{OH} CO-CH-COCH_{3}$$

$$CH_{2}O \xrightarrow{CH_{3}} OCH_{2}$$

Synthesis -Obtained by adding powder sodium to a solu-2-hydroxy-3,4,6-trimethoxytion of propiophenone in ethyl acetate and ethyl ether. When the initial reaction was over, the mixture was heated under reflux for 4 h (62 %) [2177].

colourless rectangular prisms [2177]; m.p. 133–134° [2177].

# 1-(2-Hydroxy-4,5,6-trimethoxyphenyl)-2-methyl-1,3-butanedione C14H18O6



CO-CH-COCH₃ -Obtained by adding powder sodium to a 2-hydroxy-4,5,6-trimethoxysolution of propiophenone in ethyl acetate and ethyl ether. When the initial reaction was over, the mixture was heated under reflux for 4 h (25 %) [2177].

colourless rectangular prisms [2177]; m.p. 204–206° [2177].

mol. wt. 248.28

mol. wt. 282.29

#### 1-(2-Hydroxy-3,4,5,6-tetramethoxyphenyl)-1,3-butanedione

[67231-48-5]	$C_{14}H_{18}O_7$	mol. wt. 298.29
CH ₃ O CH ₃ O CH ₃ O OCH ₃ OCH ₃	Synthesis -Obtained by Claisen condensati 3,4,5,6-tetramethoxyacetopheno acetate in the presence of pulv ethyl ether. Then, the mixture 1.5 h at 115–120°, after sol (43 %) [52].	ne with ethyl erized sodium in was refluxed for
1		

¹H NMR [52], IR [52].

Methyl ether	[67231-44-1]	C-H-O-	mol. wt. 312.32
wieuryr euler	0/231-44-1	$C_{15}H_{20}O_7$	11101. WL 512.52

-Obtained by Claisen condensation of 2,3,4,5,6-pentamethoxyacetophenone with ethyl acetate in the presence of pulverized sodium in ethyl ether. Then, the mixture was refluxed for 1.5 h at  $115-120^{\circ}$ , after solvent elimination (61 %) [52].

m.p. 70–71° [52].

#### 1-(2-Hydroxy-5-methylphenyl)-1,3-bis-1,3-butanedione

 $\begin{array}{c} C_{15}H_{16}O_5 \\ OH \\ CH_3COCH_2CO \\ CH_3 \\ CH_3 \\ CH_3 \end{array} \begin{array}{c} OH \\ CH_3 \\ CH_3 \\ CH_3 \\ CH_3 \end{array} \begin{array}{c} OH \\ COCH_2COCH_3 \\ COCH_2COCH_3 \\ CH_3 \\ CH_3$ 

-Also obtained [931] by conversion of 2,6-diacetyl-4-methylphenol by slightly modification of the procedure [230].

bright yellow powder [230]; m.p. 129° [230].

#### Copper (II) complex

C₃₀H₂₈O₁₀Cu

mol. wt. 612.09

-Preparation by treatment of tetraketone with cupric acetate in refluxing ethanol for 1 h (70 %) [230].

## 1-(3,4-Dihydro-7-hydroxy-2,2-dimethyl-2H-1-benzopyran-6-yl)-1,3-butanedione

[83805-59-8]

 $C_{15}H_{18}O_{4}$ mol. wt. 262.31

Syntheses

-Obtained by refluxing a mixture of 6-acetyl-3,4-dihydro-7-hydroxy-2,2-dimethyl-2H-1-benzo-pyran, ethyl acetate and sodium metal for 4 h (75 %) [51].

-Also refer to: [2518, 3223 (15 %)].

m.p. 108–109° [51, 2518], 107° [3223]; ¹H NMR [51, 2518, 3223], IR [3223].

#### 1-[2-Hydroxy-4-(2-tetrahydropyranyl)oxyphenyl]-1,3-butanedione

C15H18O5

mol. wt. 278.30

Synthesis

COCH₂COCH₃ -Preparation by reaction of ethyl acetate with resacetophenone 4-tetrahydropyranyl ether in the presence of powdered sodium at r.t. for 12 h (75 %) [1100].

by refluxing

and sodium metal for 3 h (70 %) [51].

m.p. 97–98° [2786].

# 1-(3,4-Dihydro-7-hydroxy-5-methoxy-2,2-dimethyl-2H-1-benzopyran-6-yl)-**1.3-butanedione**

C₁₆H₂₀O₅

-Obtained

[83805-67-8]

C₆H₅CH₂O

CH₃ Synthesis HO CH₂ CH₃COCH₂CO OCH₂

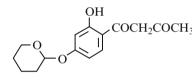
¹H NMR [51]. m.p. 104–105° [51];

#### 1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1,3-butanedione

C17H16O4 [34128-24-0] mol. wt. 284.31 Synthesis

COCH₂COCH₃ -Obtained by refluxing for 3 h a mixture of 4-benzyloxy-2-hydroxyacetophenone, ethyl acetate and sodium metal (46 %) [51].

shining needles [51]; m.p. 120–121° [51]; ¹H NMR [51].



mixture 6-acetyl-3,4-dihydro-7-hydroxy-5-methoxy-2,2-dimethyl-2*H*-1-benzopyran, ethyl acetate

a

mol. wt. 292.33

of

#### 1-[2,6-Dihydroxy-4-(phenylmethoxy)phenyl]-1,3-butanedione

[52751-45-8] C17H16O5 mol. wt. 300.31 Synthesis COCH₂COCH₃ -Obtained (by-product) by refluxing benzyl chloride and 4-acetyloxy-2,6-dihydroxy-C₆H₅CH₂O acetophenone for 24 h (0.5 %) [67]. m.p. 162–164° [67]; ¹H NMR [67], UV [67].

#### 1-[2-Hydroxy-4-methoxy-5-(phenylmethoxy)phenyl]-1,3-butanedione

[101790-57-2] C18H18O5 mol. wt. 314.34 Syntheses COCH₂COCH₃ -Preparation by treatment of 2-acetoxy-5-benzoyloxy-4-methoxyacetophenone with sodium hydride in refluxing pyridine for 15 min CH₂C (70 %) [835]. -Also obtained by reaction of ethyl acetate with 2-hydroxy-4-methoxy-5-benzyloxyacetophenone in the presence of sodium at reflux for 6 h (52 %) [835]. m.p. 124° [835].

#### 1-[2-Hydroxy-6-methoxy-4-(phenylmethoxy)phenyl]-1,3-butanedione

[83805-61-2] COCH₂COCH₃ -Obtained by refluxing for 3 h a mixture of 4-benzular 2 h i of C₄H₅CH₂ acetophenone, ethyl acetate and sodium

C₂₄H₁₈O₄

m.p. 135–136° [51]; ¹H NMR [51].

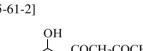
#### 1-(5-Hydroxy-2,3-diphenyl-6-benzofuranyl)-1,3-butanedione

C₆H₅ Synthesis CH₃COCH₂CO HO

-Obtained by reaction of 6-acetyl-5-hydroxy- $C_6H_5$  2,3-diphenylbenzofuran with ethyl acetate in the presence of sodium at reflux for 1 h (82 %) [2200].

m.p. 159° [2200].

[2035-55-4]



mol. wt. 314.34

mol. wt. 370.40



metal (52 %) [51].

 Methyl ether
 [78481-50-2]  $C_{25}H_{20}O_4$  mol. wt. 384.43

 -Refer to: [2-4].
 m.p. 158° [3, 4];
 1

 ¹H NMR [3, 4], IR [3], MS [3, 4].
 MS [3, 4].

# 6 Aromatic Hydroxy-1,2,3-Butanetrione

#### 1-(2,4-Dihydroxyphenyl)-1,2,3-butanetrione

 $\begin{array}{c} C_{10}H_8O_5 \\ OH \\ COCOCOCH_3 \\ HO \end{array} \begin{array}{c} \text{Synthesis} \\ -\text{Refer to: [2677].} \\ \text{Dimethyl ether} \\ C_{12}H_{12}O_5 \\ \text{mol. wt. 236.22} \end{array}$ 

-Refer to: [2677]; m.p. 97° [2677].

# 7 Aromatic Hydroxyketones Derived from Diphenyle

#### 1,1'-(4,4'-Dihydroxy[1,1'-biphenyl]-3,3'-diyl)bis-1-butanone

*in the presence of sodium chloride at 140° (93 %) [2091];
*in refluxing chlorobenzene for 24 h (89 %) [2377].
-Also refer to: [2365].

m.p. 125–126° [2377]; IR [2377].

#### 1,1'-(4,4'-Dihydroxy[1,1'-biphenyl]-3,3'-diyl)bis-3-methyl-1-butanone

 $\begin{array}{ccc} C_{22}H_{26}O_4 & \text{mol. wt. 354.45} \\ (CH_3)_2CHCH_2CO & COCH_2CH(CH_3)_2 \\ HO & & & & \\ HO & & \\ HO & & \\ HO & &$ 

chloride

at

 $140^{\circ}$ 

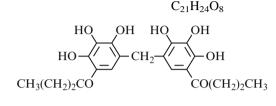
sodium

(99 %) [2091].

m.p. 94-95° [2091].

# 8 Aromatic Hydroxyketones Derived from Diphenylmethane

#### 1,1'-[Methylenebis(2,3,4-trihydroxy-5,1-phenylene)]bis-1-butanone



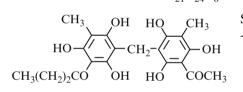
mol. wt. 404.42

Synthesis -Obtained by treatment of 4-butyroyl-pyrogallol with 35 % aqueous solution of formaldehyde in refluxing ethanol for 10 min [506].

m.p. 181–182° [506].

# 3-[(3-Acetyl-2,4,6-trihydroxy-5-methylphenyl)methyl]-(2,4,6-trihydroxy-5-methyl-3-ylphenyl)-1-butanone

 $C_{21}H_{24}O_8$ 



mol. wt. 404.42

Synthesis

-Obtained by reaction of paraformaldehyde with a mixture (1:1) of 3-methylphlorobutyro-phenone and 3-methylphloroacetophenone in dilute ethanol in the presence of concentrated sulfuric acid (10 drops) at r.t. for 48 h [2034].

pale yellow needles [2034]; m.p. 246–248° [2034].

## 1-(4-Butyryl-2-fluoro-5-hydroxyphenyl)-1-(3-butyryl-5-fluoro-2-hydroxyphenyl)ethane

3',4^{'''}-Ethylidenebis[5'-fluoro-2'-hydroxybutyrophenone] (Sadtler)

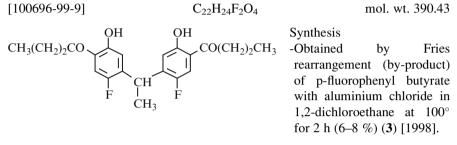
mol. wt. 390.43

Synthesis -Obtained by Fries rearrangement (by-product) of p-fluorophenyl butyrate with aluminium chloride in 1,2-dichloroethane at  $100^{\circ}$ for 2 h (1–2 %) (5) [1998]. **N.B.**: In tar of the industrial preparation from 500 kg of the ester (Sanofi industries, Aramon, Fr.).

m.p. 93° (Sadtler standard N° 38634M) [1998]; ¹H NMR (Sadtler standard N° 38634M) [1998], IR (Sadtler standard N° 65683K) [1998], UV [1998], MS [1998].

#### 1,1'-[Ethylidenebis(2-fluoro-5-hydroxy-4,1-phenylene)]bis-1-butanone

Bis(butyryl-4-fluoro-2-hydroxy-5-phenyl)-1,1-ethane [1998] 4',4^{'''}-Ethylidenebis[5'-fluoro-2'-hydroxybutyrophenone] (Sadtler)



**N.B.**: In tar of the industrial preparation from 500 kg of the ester (Sanofi industries, Aramon, Fr.).

m.p. 114° [1998];

¹H NMR (Sadtler standard  $N^{\circ}$  38501M) [1998],

IR (Sadtler standard N° 65539K) [1998], UV [1998], MS [1998].

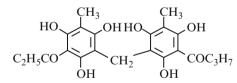
# 1-[3-(5-methyl-3-propionyl-2,4,6-trihydroxyphenylmethyl)-2,4,6-trihydroxy-5-methyl-phenyl]-1-butanone

(Abbreviatin PB)

[84633-06-7]

 $C_{22}H_{26}O_8$ 

mol. wt. 418.44



Isolation from natural sources OH -From *Dryopteris abbreviata* (DC.) NEWMAN (Aspidiaceae) [724]. COC₃H₇ pale yellow powder [724]; m.p. 206–208° [724];

¹H NMR [724], IR [724], UV [724], MS [724]; TLC [724]; HPLC [724].

# **3,5-Dihydroxy-4,4-dimethyl-2-(1-oxopropyl)-6-[[2,4,6-trihydroxy-3-methyl-5-(1-oxobutyl)phenyl]methyl]-2,5-cyclohexadien-1-one** (*Flavaspidic Acid PB*)

[3773-25-9]	$C_{23}H_{28}O$	mol. wt. 432.48
$\begin{array}{c} CH_3 \\ HO \\ HO \\ C_2H_5CO \\ O \\ O \\ CH_2 \\ O \\ $	CO(CH ₂ ) ₂ CH ₃ OH CH ₃ OH	Isolation from natural sources -From <i>Dryopteris abbreviata</i> (DC.) NEWMAN (Aspidiaceae) [724]. -From rhizomes of <i>Dryopteris</i> <i>crassirhizoma</i> (Dryopteridaceae) [1852, 2208].

-Also refer to: [2447].

pale yellow powder [724]; m.p. 170–171° [2447], 156–158° [724], 148° [1852]; ¹H NMR [724, 2208], IR [724], UV [724, 1852], MS [724]; TLC [724]; HPLC [724].

BIOLOGICAL ACTIVITY: Antioxidant [1852]; Inhibition of enzyme [2208].

**1,1'-[Methylenebis(2,4-dihydroxy-6-methoxy-3,1-phenylene)]bis-1-butanone** (*Methylene-bis-desaspidinol)* (*Methylene-bis-desaspidinol-BB*)

 $[32190-32-2] C_{23}H_{28}O_8 mol. wt. 432.48$ 

Syntheses -Preparation by reaction of formaldehyde with desaspidinol [2446, 2450]. -Also obtained (by-product) from

phloraspin [2444].

-Also obtained (by-product) from desaspidin [2450].

-Also refer to: [1914, 2451].

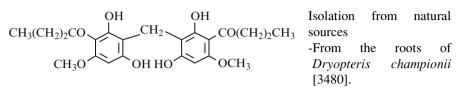
Isolation from natural sources

-From *Dryopteris austriaca* [2446]. -Also refer to: [3302].

m.p. 176–179° [3302], 174–175° [2446, 2451]; ¹H NMR [2451], MS [1914].

# 1,1'-[Methylenebis(2,6-dihydroxy-4-methoxy-3,1-phenylene)]bis-1-butanone

C23H28O8



# **1,1'-[Methylenebis(2,4,6-trihydroxy-5-methyl-3,1-phenylene)]bis-1-butanone** (*Abbreviatin BB*)

C23H28O8

 $\begin{array}{c} CH_3 & OH & HO & CH_3 \\ HO & & CH_2 & -CH_2 & -OH \\ CH_3(CH_2)_2 CO & OH & HO & CO(CH_2)_2 CH_3 \end{array}$ 

Synthesis

-Obtained by reaction of *para*formaldehyde with 3-methylphlorobutyrophenone in dilute methanol in the presence of concentrated sulfuric acid (10 drops) at r.t. for 24 h [2034].

mol. wt. 432.48

-Also refer to: [725, 1610, 1913, 2208, 2451].

Isolation from natural sources

-From Dryopteris abbreviata (DC.) NEWMAN (Aspidiaceae) [724, 725].

-From Dryopteris filix-mas var. rigidiformis [3302].

-From Dryopteris athamantica [3302].

-From Dryopteris crassirhizoma (Dryopteridaceae) [2208].

pale yellow needles [725, 2034];

m.p. 213–215° [2451], 212° [1610, 2034], 211–212° [3302], 200–202° [725];
 ¹H NMR [724, 725, 2208], ¹³C NMR [1913], IR [725], UV [725], MS [725, 2208].

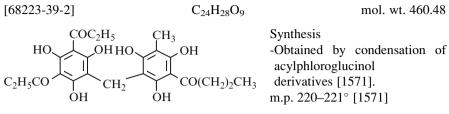
BIOLOGICAL ACTIVITY: Ecotoxicology [2208]; Fatty acid synthase inhibition [2208].

# 1-[3-(3-Acetyl-5-butyryl-2,4,6-trihydroxyphenylmethyl)-2,4,6-trihydroxy-5-methyl-phenyl]-1-butanone

[68223-50-7]	$C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{24}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}C_{28}H_{28}H_{28}C_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{28}H_{2$	<b>D</b> 9		mol. wt. 460.48
HO C ₃ H ₇ CO OH		Synthesis -Obtained acylphlorog m.p. 193–19	glucino	condensation of derivatives [1571]. 571].
BIOLOGICAL ACTI	VITY: Antimalarial [	1571].		

[4069-49-2]

# 1-[3-[(3,5-Dipropionyl)-2,4,6-trihydroxyphenylmethyl]-2,4,6-trihydroxy-5-methyl-phenyl]-1-butanone



**BIOLOGICAL ACTIVITY:** Antimalarial [1571].

# 1-[3-(3-Butyryl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl-2,6-dihydroxy-4-methoxyphenyl]-1-butanone

(Phloraspidinol-BB, Phloraspidinol)

CH₃(CH₂)₂CO HO OCH₃ CH₂ OCH₃ CH₃O

[1509-10-0]

$$C_{24}H_{30}O_8$$

mol. wt. 446.50

Syntheses

-Obtained by reaction of 4 % formaldehyde with a mixture of aspidinol* and desaspidinol* in

the presence of 1 % potassium hydroxide for 1 min at r.t., then acidification with dilute hydrochloric acid [2446].

*Aspidinol: 2,6-dihydroxy-4-methoxy-3-methylbutyrophenone.

*Desaspidinol: 2,6-dihydroxy-4-methoxybutyrophenone.

-Also refer to: [1914, 3301].

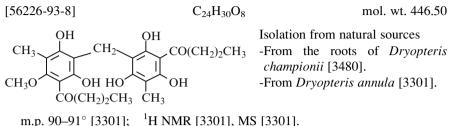
Isolation from natural sources

-From *Dryopteris austriaca* (ferns) [2446, 2451]. -From *Dryopteris athamantica* [3302]. -From *Dryopteris arwanda* [3302].

m.p. 193–194° [2446, 2451, 3302], 190–192° [3301]; ¹H NMR [3301], MS [1914].

# 1-[3-[[2,6-Dihydroxy-4-methoxy-3-methyl-5-(1-oxobutyl)phenyl]methyl]-2,4,6-trihydroxy-5-methylphenyl]-1-butanone

(Aemulin BB)



# 1-[3-[[2,4-Dihydroxy-6-methoxy-5-methyl-3-(1-oxobutyl)phenyl]methyl]-2,4,6-trihydroxy-5-methylphenyl]-1-butanone

(Margaspidin) (Margaspidin BB)

[1867-82-9]	$C_{24}H_{30}O_8$	mol. wt. 446.50
$CO(CH_2)_2CH_3$ $CO(CH_2)_2$		
но он но он	-Obtained by treatm	nent of a 2,4,6-tri-
		ylbutyrophenone and
CH ₃ CH ₂ CH ₃	2,6-dihydroxy-4-n	nethoxy-3-methyl-
OCH ₃ OH	butyrophenone mix	ture with bis(sulfo-

butvrophenone mixture with bis(sulfonium ylide) in the presence of silver oxide (84 %) [2414].

-Also prepared by treatment of a 3-methyl-2,4,6-trihydroxybutyrophenone and aspidinol mixture with 4 % formaldehyde in the presence of 1 % aqueous sodium hydroxide [2443].

-Also refer to: [80, 206, 438, 1914].

Isolation from natural sources

-From Dryopteris marginata (Wall.) CHRIST (Aspidiaceae) [2527].

-From Dryopteris arwanda [3302].

-From Dryopteris annula [3301].

-From Dryopteris marginalis [2451].

-From Dryopteris bissetiana (Aspidiaceae) [1332].

yellow crystals [2443];

m.p. 189–191° [1332], 179–181° [3301], 178–180° [2443, 2451, 3302], 175–176° [2527], 175° [2527];

¹H NMR [1332, 3301], ¹³C NMR [206], UV [1332, 2443], MS [1914, 2527].

BIOLOGICAL ACTIVITY: Dihydrofolate reductase inhibition screening using protein flexibility and species specificity in structure-based drug discovery [438]; Anthelmintic [2414].

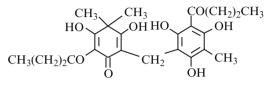
# 3,5-Dihydroxy-4,4-dimethyl-2-(1-oxobutyl)-6-[[2,4,6-trihydroxy-3-methyl-5-(1-oxobutyl)phenyl]methyl]-2,5-cyclohexadien-1-one

3'[(5-Butyryl-2,4-dihydroxy-3,3-dimethyl-6-oxo-1,4-cyclohexadien-1-vl)methyl]-5'-methyl-phlorobutyrophenone.

(Flavapidic Acid) (Polystichocitrin) (Toxifren)

$$C_{24}H_{30}O_8$$

mol. wt. 446.50



-Refer to: [35, 70, 2033, 2617].

Syntheses

Isolation from natural sources

-From the rhizomes of male fern [388, 389]. -From Dryopteris ferns [959].

 $(\alpha$ -form) orthorhombic crystals; m.p. 157–158.5° [36], 157–158° [2033], 156° [959, 2449, 2451, 3102], 154° [2618], 93° [2618], 92° [959, 2033], 90° [36]; m.p.  $92^{\circ}$ , solidifies again at  $110^{\circ}$  and melts again at  $156^{\circ}$  [959, 3102]; ¹H NMR [3302], IR [3302]. (β-form) monoclinic crystals; X-ray data [959].

BIOLOGICAL ACTIVITY: Anthelmintic [70]; Toxicity [70];

LD₅₀ [3102].

# 1,1'-[Methylenebis(2,4,6-trihydroxy-3-acetyl-5,1-phenylene)]bis-1-butanone C₂₅H₂₈O₁₀

CO(CH₂)₂CH₃

[68223-37-0]

HO

CH₃(CH₂)₂CO

OH HO COCH₃ CH₃CO CH₂· OH

HO

mol. wt. 488.49

Syntheses

-Preparation by condensation of two molecules of acylphloroglucinol with formaldehyde or methoxymethyl acetate [3391].

-Also refer to: [1571].

m.p. 166–168° [1571], 156–158° [3391].

OH

#### Hexamethyl ether

C₃₁H₄₀O₁₀

mol. wt. 572.65

-Preparation by condensation of two molecules of acylphloroglucinol with formaldehyde or methoxymethyl acetate [3391].

m.p. 113-115° [3391].

## 2-[[2,6-Dihydroxy-4-methoxy-3-methyl-5-(1-oxobutyl)phenyl]methyl]-3,5-dihydroxy-4,4-dimethyl-6-(1-oxobutyl)-2,5-cyclohexadien-1-one still named

3'-[(5-Butyryl-2,4-dihydroxy-3,3-dimethyl-6-oxo-1,4-cyclohexadien-1-yl) methyl]-2',4'-dihydroxy-6'-methoxy-5'-methyl-1-butanone (Aspidin BB) [3102], (Polystichin) [3102], (para-Aspidin) [2408, 2445].

[584-28-1]

C₂₅H₃₂O₈

Syntheses

mol. wt. 460.52

CO(CH₂)₂CH₃ CH₃O OH CH₃  $CH_2$ CO(CH₂)₂CH₃ OH HC CH₃CH₃

-Obtained by adding formaldehyde to a solution of 3-butyrylfilicinic acid and aspidinol in aqueous potassium hydroxide. The mixture was kept at r.t. for 15 min [2445]. -Also refer to: [36].

Isolation from natural sources

-From *Hypericum uliginosum* HBK (XX) [2408]. -From Dryopteris austriaca (Jacq.) Woynar (Polypodiaceae) [36, 2445, 3102].

m.p. 123–125° [3300], 124–125° [2445, 3102], 122–124° [3302], 121–123° [36, 387, 390, 2617]; UV [2445, 3102], Ms [1914].

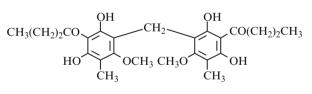
# 1,1'-[Methylenebis(2,6-dihydroxy-4-methoxy-5-methyl-3,1-phenylene)]bis-1-butanone

C25H32O8

(Methylene-bis-aspidinol)

[5377-72-0]

mol. wt. 460.52



sources -Obtained from the

Isolation from natural

leaves and twigs of Calyptranthes pallens (Myrtaceae) [1898].

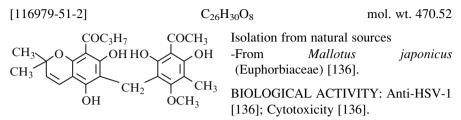
-Also obtained from the rhizomes of *Ctenitis apiciflora* (Aspidiaceae) [3295]. -Also obtained from the rhizomes of Ctenitis nidus (Aspidiaceae) [3295]. -Also obtained from the from genus Dryopteris marginalis [3297]. -Also refer to: [245].

yellow needles [1898]; m.p. 188–190° [3297], 187–190° [3295], 186–187° [1898]; ¹H NMR [1898], ¹³C NMR [1898], IR [1898], UV [1898], MS [1898, 3295].

**BIOLOGICAL ACTIVITY: Cytotoxicity** [1898].

# 1-[6-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-1-butanone

(Butyrylmallotochromene)



# 3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-2,4,6-trihydroxy-5-[(3,3-dimethylallyl)phenyl]-1-butanone

(Butyrylmallotojaponin)

[96853-73-5]

C₂₆H₃₂O₈

mol. wt. 472.54

ОН	но	COC ₃ H ₇	
CI	H ₂ -{/_	Он	
ОСН	3 НО	CH ₂ CH=C(C	H ₃ ) ₂
	-Сі	ОН НО - СН ₂ -СН ₂ -СН ₂ -СН ₂ -СН ₂ -СН ₃ -СП	CH ₂ -CH ₂ -OH

Isolation from natural sources -From the fruits of *Mallotus japonicus* (Euphorbiaceae) [1746]. -From the pericarps of *Mallotus japonicus* (Euphorbiaceae) [136, 138, 139].

Yellow needles [1746]; m.p. 157–158° [1746]; ¹H NMR [1746], ¹³C NMR [1746], IR [1746], MS [1746].

BIOLOGICAL ACTIVITY: Anti-HSV-1 [136]; Cytotoxicity [136, 139].

# Pentaacetate

 $C_{36}H_{42}O_{13}$ 

mol. wt. 682.72

-Obtained by reaction of acetic anhydride with the title ketone in the presence of pyridine at r.t. for 24 h [1746].

¹H NMR [1746], MS [1746]; HPLC [1746].

BIOLOGICAL ACTIVITY: Cytotoxicity [139].

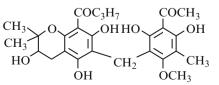
1-[6-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-3,4-dihydro-3,5,7-trihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-1-butanone

(Butyrylmallotochromanol)

[129399-52-6]

 $C_{26}H_{32}O_9$ 

mol. wt. 488.53

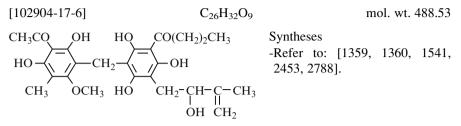


Isolation from natural sources-FromMallotusjaponicus(Euphorbiaceae) [136].

BIOLOGICAL ACTIVITY: Anti-HSV-1 [136]; Cytotoxicity [136].

# 1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-2,4,6-trihydroxy-5-(2-hydroxy-3-methyl-3-butenyl)phenyl]-1-butanone

(Mallotolerin) (Butyrylmallotolerin)



Isolation from natural sources

-From in pericarps of Mallotus japonicus (Euphoebiaceae) [136-138, 1462, 2232].

yellow needles [138]; m.p. 197–198° [138]; ¹H NMR [138], ¹³C NMR [137], IR [138], UV [138], MS [138].

USE: Biocompatible, biostable coating of medical surfaces composed of polysulfone and hydrophilic polymers [1359].

BIOLOGICAL ACTIVITY: Expandable medical device comprising gemcitabine for treatment and prevention of cardiovascular diseases [2453]; Anti-HSV-1 [136]; Cytotoxicity [136–138].

# Hexaacetate $C_{38}H_{44}O_{15}$ mol. wt. 744.23

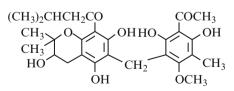
-Obtained by reaction of acetic anhydride with mallotolerin in the presence of pyridine at r.t. [138].

colourless oil [138]; ¹H NMR [138].

# 1-[6-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-3,4-dihydro-3,5,7-trihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-3-methyl-1-butanone

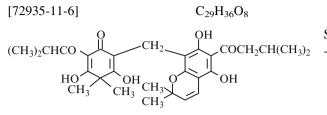
 $C_{27}H_{34}O_{9}$ 

mol. wt. 502.55



Isolation from natural sources -From the pericarps of *Mallotus japonicus* (Euphorbiaceae) [1461].

# 2-(5,7-Dihydroxy-6-isovaleryl-2,2-dimethyl-2H-chromen-8-ylmethyl)-3,5-dihvdroxy-6-isobutyryl-4,4-dimethyl-2,5-cyclohexadiene-1-one (Isouliginosin B-iBiV)



mol. wt. 512.60

Synthesis -Obtained by treatment of uliginosin A-iBiV with DDQ (13 %)[2041].

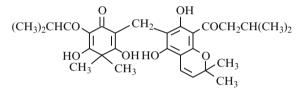
vellow prisms [2041]; m.p. 150–151° [2041]; ¹H NMR [2041], IR [2041], MS [2041].

# 2-(5,7-Dihydroxy-8-isovaleryl-2,2-dimethyl-2H-chromen-6-ylmethyl)-3,5-dihydroxy-6-isobutyryl-4,4-dimethyl-2,5-cyclohexadiene-1-one (Uliginosin B-iBiV)

[72935-10-5]

#### C20H36O8

mol. wt. 512.60



-Obtained by treatment of uliginosin A-iBiV with DDQ (26 %) [2041].

vellow prisms [2041]; m.p. 143–145° [2041]; ¹H NMR [2041], IR [2041], MS [2041].

#### 1,1'-[Methylenebis(2,4,6-trihydroxy-3,5-phenylene)]bis-1-butanone

[68223-30-3] C29H36O10  $CH_3(CH_2)_2CO$  $CO(CH_2)_2CH_3$ OHHO HO OH  $CO(CH_2)_2CH_3$  with CH₃(CH₂)₂CO CH₂ ÒН ÒН

mol. wt. 544.61

Synthesis

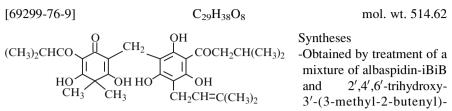
Synthesis

-Preparation by condensation of 40 % formaldehyde 3,5-dibutyrylphloroglucinol (65 %) [3391].

-Also refer to: [1571].

m.p. 178.5–179.5° [1571], 174–176° [3391].

### Uliginosin A-iBiV



isovalerophenone with sodium hydride in refluxing methanol for 1.5 h (59 %) [2042].

-Also refer to: [2041].

m.p. 140.5–142° [2042]; ¹H NMR [2042], IR [2042], MS [2042].

# 1-[3,5-Bis[[2,6-dihydroxy-4-methoxy-3-methyl-5-(1-acetyl)phenyl]methyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-butanone

(Agrimol E)

[55576-68-6] C33H38O12 Syntheses CH₃ CO-CH-C₂H₅ CH₃ CH₃ paper). CH₃C OHHO он но OCH₃ CH₃CC CH₂ CH₂ COCH₃ OH OH

mol. wt. 626.66

-Refer to: [1866] (Chinese

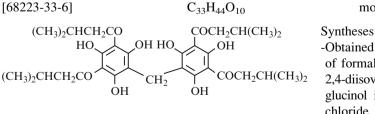
Isolation from natural sources

-From the Chinese herb medicine Agrimonia pilosa [632, 2860] (Chinese paper).

m.p. 240–242° [2860], 239–241° [1866]; ¹H NMR [632], IR [632, 1866], UV [632], MS [632].

# 1,1',1"',1"'-[Methylenebis(2,4,6-trihydroxy-5,1,3-benzenetriyl)]bis-3-methyl-1-butanone

Methylene-bis-(3,5-diisopentanoyl-2,4,6-trihydroxybenzene)



-Obtained by reaction of formaldehyde with 2,4-diisovalerylphloroglucinol in methylene chloride at  $60^{\circ}$  for 8-10 h (85 %) [338].

mol. wt. 600.71

-Also obtained by irradiation of a 2,4-bis(isovaleryl)phloroglucinol and formaldehyde mixture with microwave radiations (750 W) in domestic microwave oven for 10–15 min (38 %) [621]. -Also refer to: [1571].

yellow solid [621]; m.p. 165–167° [1571], 158–160° [621]; ¹H NMR [621], ¹³C NMR [621], IR [621], UV [621],

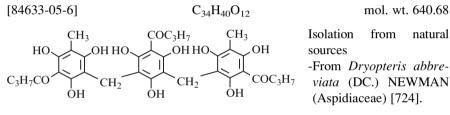
MS [621].

BIOLOGICAL ACTIVITY: Cytotoxicity [621].

-Also refer to: [338].

# 1-[3,5-Bis[[2,4,6-trihydroxy-3-methyl-5-(1-oxobutyl)phenyl]methyl]-2,4,6-trihydroxyphenyl]-1-butanone

(Trisabbreviatin BBB)



pale yellow powder [724]; ¹H NMR [724], IR [724], UV [724], MS [724]; TLC [724]; HPLC [724].

COC₃H₇

ÓН

OH HO

 $CH_2$ 

1-[3-[(3-Acetyl-2,6-dihydroxy-4-methoxy-5-methylphenyl)methyl]-5-[[2,6-dihydroxy-4-methoxy-3-methyl-5-(1-oxobutyl)phenyl]methyl]-2,4,6-trihydroxyphenyl]-1-butanone

(Agrimol F)

CH₃O

CH₃CO

[121693-16-1]

CH₃

ĠН

OH HO.

 $CH_2$ 

 $C_{34}H_{40}O_{12}$ 

CH₃

OH

OCH₃

COC₃H₇

mol. wt. 640.68

Isolation from natural sources

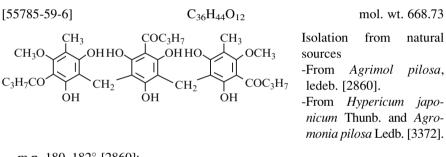
-From Hypericum japonicum Thunb. and Agromonia pilosa Ledb. [3372].

BIOLOGICAL ACTIVITY: Antimicrobial activity of, for *Staphylococcus aureus* [3372].

Trisaspidinol (A mixture of 3 homologous)

BBB  $(R = R' = C_3H_7)$ [49582-13-0]  $C_{36}H_{44}O_{12}$ mol. wt. 668.74  $(R = C_2H_5, R' = C_3H_7)$  [49582-14-1] PBB  $C_{35}H_{42}O_{12}$ mol. wt. 654.71 PBP  $(R = R' = C_2 H_5)$ [49582-15-2]  $C_{34}H_{40}O_{12}$ mol. wt. 640.68 Isolation from CH₃ COC₃H₇ CH₃ natural HO OCH₃ HO. OH CH₃O. OH sources -From Dryopteris inaequalis RCO CH₂ COR' [3297, 3300].  $CH_2$ òн ÓН m.p. 170–172° [3297].

**1,1'-[[2,4,6-Trihydroxy-5-(1-oxobutyl)-1,3-phenylene]bis[methylene-**(**2,4-dihydroxy-6-methoxy-5-methyl-3,1-phenylene)]]bis-1-butanone** (*Agrimol C*)



m.p. 180–182° [2860]; ¹H NMR [2860], IR [2860], UV [2860], MS [2860].

BIOLOGICAL ACTIVITY: Antimicrobial activity of, for *Staphylococcus aureus* [3372].

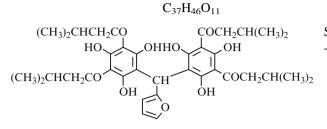
#### Heptaacetyl derivative

C₅₀H₅₈O₁₉

mol. wt. 962.95

-Refer to: [2860].

# 1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl)furan-2-yl-methyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone



mol. wt. 666.77

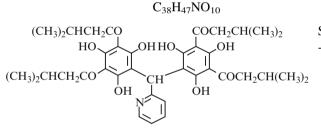
Synthesis -Obtained by irradiation of a 2,4-bis(isovaleryl) phloro-glucinol in chloroform, p-TsCl and furfural mixture with microwave radiations

(750 W) in domestic microwave oven for 10-15 min (48 %) [621].

yellow oil [621]; ¹H NMR [621], ¹³C NMR [621], IR [621], UV [621], MS [621].

**BIOLOGICAL ACTIVITY: Cytotoxicity [621].** 

# 1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl)pyridin-2-yl-methyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone



mol. wt. 677.80

Synthesis

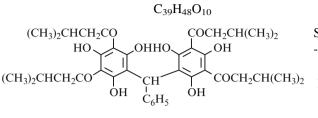
-Obtained by irradiation of a 2,4-bis(isovaleryl) phloro-glucinol in chloroform, p-TsCl and 2-formyl-pyridine mixture with microwave

radiations (750 W) in domestic microwave oven for 10–15 min (46 %) [621].

yellow solid [621]; m.p. 190–192° [621]; ¹H NMR [621], ¹³C NMR [621], IR [621], UV [621], MS [621].

BIOLOGICAL ACTIVITY: Cytotoxicity [621].

# 1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl) phenylmethyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone



mol. wt. 676.80

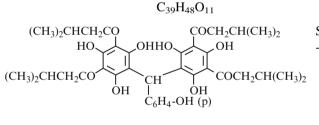
Synthesis -Obtained by irradiation of a 2,4-bis(isovaleryl) phloro-glucinol in chloroform, p-TsCl and benzaldehyde mixture with microwave

radiations (750 W) in domestic microwave oven for 10-15 min (43 %) [621].

yellow solid [621]; m.p. 180–182° [621]; ¹H NMR [621], ¹³C NMR [621], IR [621], UV [621], MS [621].

BIOLOGICAL ACTIVITY: Cytotoxicity [621].

# 1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl)-(4-hydroxyphenyl)-methyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone



mol. wt. 692.80

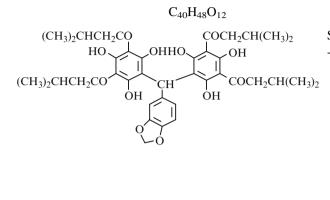
Synthesis -Obtained by irradiation of a 2,4-bis(isovaleryl) phloro-glucinol in chloroform, p-TsCl and 4-hydroxy-benzaldehyde mixture with microwave

radiations (750 W) in domestic microwave oven for 10–15 min (50 %) [621].

brown oil [621]; ¹H NMR [621], ¹³C NMR [621], IR [621], UV [621], MS [621].

BIOLOGICAL ACTIVITY: Cytotoxicity [621].

# 1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl)-3,4-methylenedioxy-phenylmethyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone



mol. wt. 720.81

Synthesis -Obtained by irradiation of a 2,4-bis(isovaleryl) phloro-glucinol in chloroform, p-TsCl and piperonal (3,4-methylene-dioxybenzaldehyde) mixture with microwave radiations (750 W) in domestic microwave oven for 10-15 min (45 %) [621].

-Also refer to: [620 (41 %)].

yellow crystalline solid [621]; yellow solid [620]; m.p. 186–188° [620], 172–174° [621]; ¹H NMR [620, 621], ¹³C NMR [620, 621], IR [621], UV [621], MS [620, 621].

BIOLOGICAL ACTIVITY: Cytotoxicity [621].

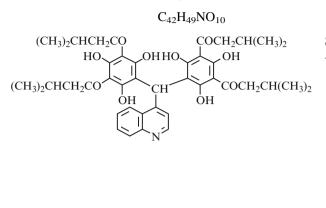
# 1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl) phenylethylmethyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone

C₄₁H₅₂O₁₀ mol. wt. 704.86 (CH₃)₂CHCH₂CO COCH₂CH(CH₃)₂ Synthesis OHHO OH -Obtained by irradiation HO of a 2,4-bis(isovaleryl) COCH₂CH(CH₃)₂ (CH₃)₂CHCH₂CO phloro-glucinol and СН́ ÓН ÓН hydrocinna-maldehyde ĊH₂ (also named 3-phenylpropionaldehyde) mix-GH5 ture with microwave radiations (750 W) in domestic microwave oven for 10-15 min (32 %) [621]. yellow solid [621];

yenow sond [021]; m.p. 138–140° [621]; ¹H NMR [621], ¹³C NMR [621], IR [621], UV [621], MS [621].

BIOLOGICAL ACTIVITY: Cytotoxicity [621].

# 1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl)quinolin-4-yl-methyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone



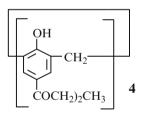
mol. wt. 727.85

**Synthesis** -Obtained by irradiation of a 2,4-bis(isovaleryl) phloro-glucinol in chloroform. p-TsCl and 4-quinolinecarboxaldehyde mixture with microwave radiations (750 W) in domestic microwave oven for 10-15 min (48 %) [621].

cream coloured solid [621]; m.p. 200–202° [621]; ¹H NMR [621], ¹³C NMR [621], IR [621], UV [621], MS [621].

BIOLOGICAL ACTIVITY: Cytotoxicity [621].

# 5,11,17,23-Tetrabutyryl-25,26,27,28-tetrahydroxycalix[4]arene



 $C_{44}H_{48}O_8$ 

Synthesis

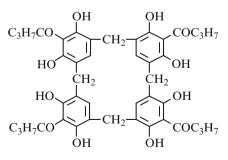
mol. wt. 704.86

-Obtained by Fries rearrangement of 25,26,27,28-tetra-(butyryloxy)calix[4]arene (7) with aluminium chloride in nitrobenzene at r.t. overnight (74 %) (12) [2293]. m.p. 230–232° [2293]; ¹H NMR [2293], IR [2293].

Calix[4]resorcinarene

C44H48O12

mol. wt. 768.86



Synthesis -Obtained by reaction of 2-butyrylresorcinol with 95 % paraformaldehyde in the presence of potassium tert-butoxide in THF at 60° for 24 h under an argon atmosphere (58 %) [1732].

yellow needles [1732];

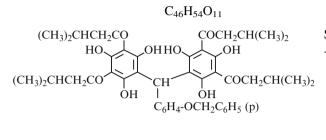
m.p. 256–259° (d) [1732];

¹H NMR [1732], ¹³C NMR [1732], IR [1732], MS [1732].

#### Monohydrate

MS [1732].

# 1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl)-4-benzyloxyphenyl-methyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone



mol. wt. 782.93

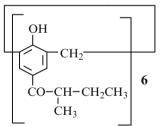
mol. wt. 786.87

Synthesis -Obtained by irradiation of a 2,4-bis(isovaleryl) phloro-glucinol in chloroform, p-TsCl and 4-benzyl-oxyoxybenzaldehyde mixture with microwave radiations (750 W) in domestic microwave oven for 10–15 min (42 %) [621].

brown crystals [621]; m.p. 152–154° [621]; ¹H NMR [621], ¹³C NMR [621], IR [621], UV [621], MS [621].

BIOLOGICAL ACTIVITY: Cytotoxicity [621].

## 5,11,17,23,29,35-Hexakis(2-methylbutanoyl)-37,38,39,40,41,42-hexahydroxycalix[6]arene



 $C_{72}H_{84}O_{12}$ 

mol. wt. 1141.43

Synthesis -Obtained by Fries rearrangement of 37,38,39,40,41,42-hexakis[(2-methylbutanoyl)oxy]calix[6]arene (**2b**) with aluminium chloride in chlorobenzene at 45–50° for 17 h under a nitrogen atmosphere (34 %) (**3b**) [135]. m.p. >360° [135]; ¹H NMR [135], IR [135].

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# 9 Aromatic Hydroxyketones Derived from 1,3-Butanedioic Acid

## 1-(2-Hydroxyphenyl)-4-phenyl-1,3-butanedione

[16850-82-1]  $C_{16}H_{14}O_3$  mol. wt. 254.29 HO-COCH₂COCH₂-COCH₂-COCH₂-COCH₂COCH₂-COCH₂ synthesis -Refer to: [301, 302, 916]. m.p. 107–108° [302, 916]; ¹H NMR [916], ¹³C NMR [916], MS [916].

# 1-(4-Hydroxyphenyl)-4-phenyl-1,3-butanedione

$$C_{16}H_{14}O_{3} mtext{mol. wt. 254.29}$$

$$HO - COCH_{2}COCH_{2} - COCH_{2} - COCH_{2}COCH_{2} - COCH_{2} - COCH_{$$

-Obtained by reaction of 4-methoxyacetophenone with ethyl phenylacetate in the presence of sodium [3083].

m.p. 75–76° [3083]; UV [3083].

#### 1-(2,4-Dihydroxyphenyl)-4-phenyl-1,3-butanedione

$$C_{16}H_{14}O_{4} mol. wt. 270.28$$
OH
OH
COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂COCH₂-COCH₂-COCH₂COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-COCH₂-C

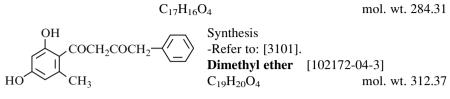
m.p. 91° [1297, 1298].

# **Diethyl ether** $C_{20}H_{22}O_4$ mol. wt. 326.39

-Obtained by reaction of 2,4-diethoxyacetophenone with ethyl phenylacetate in the presence of sodium [3083].

m.p. 75° [1234], 74–75° [3083]; UV [3083].

#### 1-(2,4-Dihydroxy-6-methylphenyl)-4-phenyl-1,3-butanedione



-Refer to: [3101].

b.p._{0.5} 225–230° [3101]; m.p. 69–70° [3101].

### Na salt

-Obtained by Claisen-condensation of orcacetophenone dimethyl ether with ethyl phenylacetate in xylene in the presence of pulverized sodium. Then, the reaction mixture was heated at 120° for 2 h [3101].

Cu (II) salt of the dimethyl ether	$(C_{19}H_{19}O_4)_2Cu$	mol. wt. 686.26
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m.p. 145-146° [3101].

### 1-(2,4-Dihydroxy-6-methylphenyl)-2-methyl-4-phenyl-1,3-butanedione

-Obtained by reaction of methyl iodide with the sodium salt of 1-(2,4-dimethoxy-6-methylphenyl)-4-phenyl-1,3-butanedione in refluxing acetone for 5 h [3101].

oil [3101].

# 10 Aromatic Hydroxyketones Derived from 1,4-Butanedioic Acid

#### 1,4-Bis(5-hydroxy-2-nitrophenyl)-2-bromo-1,4-butanedione

mol. wt. 428.08

#### 1,4-Bis(3-bromo-4-hydroxyphenyl)-1,4-butanedione

$$C_{16}H_{12}Br_2O_4$$



Synthesis -Obtained by bromination of 1,4-bis (4-hydroxyphenyl)-1,4-butanedione in acetic acid [3319].

m.p. 134–136° [3319].

# 1,4-Bis(5-bromo-2-hydroxyphenyl)-1,4-butanedione

[16197-58-3]  $C_{16}H_{12}Br_2O_4$  mol. wt. 428.08 OH OH Syntheses -Obtained by Fries rearrangement of di (4 because benefit) supported with aluminium

di-(4-bromophenyl) succinate with aluminium chloride at 130–140° for 3–4 h [594].
 r -Also refer to: [592].

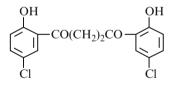
m.p. 180° [594].

**Di-2,4-dinitrophenylhydrazone** [16286-63-8] C₂₈H₂₀Br₂N₈O₁₀ mol. wt. 788.32 m.p. 250° [594].

# 1,4-Bis(5-chloro-2-hydroxyphenyl)-1,4-butanedione

[16197-54-9]

C₁₆H₁₂Cl₂O₄ mol. wt. 339.17



Syntheses -Obtained by Fries rearrangement of di-pchlorophenyl succinate with aluminium chloride at 130–140° for 3–4 h (good yield) [594]. -Also refer to: [592].

m.p. 182° [594].

# **Di-2,4-dinitrophenylhydrazone** [16197-55-0] C₂₈H₂₀Cl₂N₈O₁₀ mol. wt. 699.42 m.p. 210° [592, 594].

**Dioxime** [16197-64-1] C₁₆H₁₄Cl₂N₂O₄ mol. wt. 369.20 m.p. 175° [594].

#### 1-(4-Chlorophenyl)-4-(4-hydroxyphenyl)-1,4-butanedione

$$\begin{array}{cccc} C_{16}H_{13}ClO_3 & \text{mol. wt. } 288.73 \\ OH & Synthesis \\ -Refer to: [2953]. \\ \textbf{Methyl ether} & [67756-16-5] \\ C_{17}H_{15}ClO_3 & \text{mol. wt. } 302.76 \end{array}$$

-Obtained from 4-chlorobenzaldehyde and 1-(4-methoxyphenyl)-2-propen-1-one (77 %) [2953].

-Also refer to: [1055, 2452, 2951, 3458].

m.p. 145° [2953], 136° [2951]; ¹H NMR [1055, 2951, 2953, 3458], ¹³C NMR [2452], IR [1055, 2951, 2953], MS [2452].

#### 1-(4-Hydroxyphenyl)-4-phenyl-1,4-butanedione

	$C_{16}H_{14}O_3$	mol. wt. 254.29
он Т	Synthesis -Refer to: [2953].	
	<b>Methyl ether</b> [60755-22-8]	
$\Upsilon$ CO(CH ₂ ) ₂ COC ₆ H ₅	$C_{17}H_{16}O_3$	mol. wt. 268.31

-Obtained from benzaldehyde and 1-(4-methoxyphenyl)-2-propen-1-one (63 %) [2953].

-Also refer to: [1055, 1268, 1295, 1658, 2275, 2951, 3358, 3393, 3458].

m.p. 107° [2953], 106–107° [2275], 100–101° [1268, 1295], 98–100° [3393, 3436], 58–60° [3358]; ¹H NMR [1055, 1658, 2275, 2951, 2953, 3358, 3393, 3458], ¹³C NMR [3358, 3393, 3436], IR [1055, 1268, 2275, 2951, 2953, 3358, 3393, 3436], UV [2275], MS [3393, 3436].

#### 1-(3,4-Dihydroxyphenyl)-4-phenyl-1,4-butanedione

	$C_{16}H_{14}O_4$	mol. wt. 270.28
OH OH COCH ₂ CH ₂ CO-	Synthesis -Refer to: [2953]. <b>Dimethyl ether</b> [67756-25-6] $C_{18}H_{18}O_4$	mol. wt. 298.34

-Obtained from benzaldehyde and 1-(3,4-dimethoxyphenyl)-2-propen-1-one (60 %) [2953].

-Also refer to: [321].

m.p. 109° [2953]; ¹H NMR [321, 2953], IR [321, 2953].

**BIOLOGICAL ACTIVITY:** Antiprotozoal [321].

#### 1,4-Bis(2-hydroxyphenyl)-1,4-butanedione

$$C_{16}H_{14}O_4 \qquad \text{mol. wt. 270.28}$$

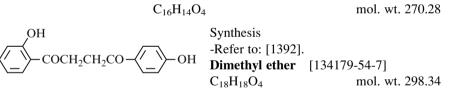
$$OH \qquad HO \qquad Synthesis \\ -\text{Refer to: [579].} \\ \textbf{Dimethyl ether} \qquad [134179-55-8] \\ C_{18}H_{18}O_4 \qquad \text{mol. wt. 298.34}$$

-Obtained by adding zinc dust and a small amount of iodine to a solution of 2-bromo-2'-methoxy-acetophenone. Then, the reaction mixture was heated at 65° for 16 h (26 %) [579].

-Also obtained by reduction of 1,2-bis(2-methoxybenzoyl)ethene with zinc dust in acetic acid (2 %) [1392].

colourless crystals [579]; m.p. 102–104° [1392], 98–100° [579]; ¹H NMR [579, 1392], ¹³C NMR [579], IR [579].

#### 1-(2-Hydroxyphenyl)-4-(4-hydroxyphenyl)-1,4-butanedione



-Refer to: [1392 (8 %)].

m.p. 89–90° [1392], ¹H NMR [1392].

**N.B.**: Its name was 1-(3-methoxybenzoyl)-2-(4-methoxybenzoyl)ethane (in the paper, formula **6e** page 4813) in contradiction with formula represented page 4812.

#### 1-(3-Hydroxyphenyl)-4-(4-hydroxyphenyl)-1,4-butanedione

$$\begin{array}{c} C_{16}H_{14}O_{4} & \text{mol. wt. 270.28} \\ HO & \\ \hline \\ - COCH_{2}CH_{2}CO & \\ \hline \\ - OH & \\ \hline \\ Dimethyl \ ether \\ C_{18}H_{18}O_{4} & \text{mol. wt. 298.34} \\ \end{array}$$

-Refer to: [699, 1156].

¹H NMR [121], IR [121].

### 1,4-Bis(3-hydroxyphenyl)-1,4-butanedione

 $C_{16}H_{14}O_{4}$ HO Synthesis -Refer to: [579]. Dimethyl ether COCH₂CH₂CO  $C_{18}H_{18}O_4$ mol. wt. 298.34

-Obtained by adding zinc dust and a small amount of iodine to a solution of 2-bromo-3'-methoxy-acetophenone. Then, the reaction mixture was heated at 65° for 16 h (60 %) [579].

-Also refer to: [2401].

colourless crystals [579]; m.p. 128° [579]; ¹H NMR [579], ¹³C NMR [579], IR [579], MS [579].

#### 1,4-Bis(4-hydroxyphenyl)-1,4-butanedione

$$[108791-64-6] \qquad C_{16}H_{14}O_4$$

$$HO \longrightarrow COCH_2CH_2CO \longrightarrow OH \qquad S$$

Syntheses

-Obtained (by-product) by reaction of succinic anhydride with anisole in the presence of aluminium chloride in nitrobenzene or in tetrachloroethane [858].

-Preparation by treatment of dimethyl ether with aluminium chloride in nitrobenzene [3319].

-Also refer to: [2567].

m.p. 147° [2567, 3319].

mol. wt. 270.28

mol. wt. 270.28

# **Dimethyl ether** [15982-64-6] $C_{18}H_{18}O_4$ mol. wt. 298.34

-Obtained by Friedel-Crafts reaction of succinyl chloride with anisole in the presence of aluminium chloride [2142],

- *in carbon disulfide for 45 min at 60° (18–21 %) [478] or for 36 h (8 %) [432];
- *in petroleum ether at  $0^{\circ}$ , then 2 h at r.t. [3319].
- -Also obtained from 4-methoxybenzaldehyde and 1-(4-methoxyphenyl)-2-propen-1-one (47 %) [2953].
- -Also prepared by reaction of trimethylsilyl enol ether obtained from p-methoxyacetophenone with lead tetraacetate in methylene chloride and tetrahydrofuran at  $-78^{\circ}$  (50 %) [2148].
- -Also obtained by reaction between 2'-bromo-4-methoxyacetophenone and methylmagnesium iodide (10 %) [761].
- -Preparation: Ethyl p-anisoylacetate in THF was added to a suspension of NaH in THF. After hydrogen evolution had stopped, there was added in succession potassium iodide, acetone and p-methoxyphenacyl bromide. The mixture was stirred for 1 h at r.t. (75 %) [1839].
- -Also obtained by reaction of  $\alpha$ -bromo-p-phenacyl bromide with TDAE in THF in the presence of iodine at 67° for 30 min (69 %) [2291].
- -Also obtained by indium (I) bromide-mediated reductive coupling of  $\alpha$ , $\alpha$ -dichlorop-methoxy-acetophenone in THF at 20° for 24 h (60 %) [2452].
- -Also obtained by reduction of 1,2-bis(4-dimethoxybenzoyl)ethene with zinc dust in acetic acid [1392].
- -Also obtained by hydrolysis of 2,5-bis(dimethylamino)-2,5-bis(4-methoxyphenyl) adiponitrile in refluxing THF and 30 % aqueous oxalic acid mixture (1:1) for 90 min (76 %) [3026].
- -Also refer to: [93, 252 (74 %), 446, 536, 579 (32 %), 699, 1058, 1059, 1342, 1343, 1423, 1431 (84 %), 1533, 1582–1584, 1784, 1815, 1821, 2546, 2304].

colourless crystals [252, 579];

- m.p. 156–157° [1583], 155–159° [579], 154–156° [1815], 154–155° [446, 2304], 154° [478, 1342, 1343, 3319], 153–155° [3026], 153–154.5° [1839], 152° [1821], 151° [252, 2953], 150–152° [2452], 150–151° [536, 1784, 2148], 149–150° [761];
- ¹H NMR [93, 579, 761, 1533, 1583, 1815, 2452, 2546, 2953],
- ¹³C NMR [579, 1583, 2452],
- IR [252, 579, 761, 1533, 1583, 1815, 2452, 2546, 2953],
- MS [252, 761, 1815, 2452], UV [2142];
- luminescence spectroscopy [2142].

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m.p. 197–198° [432].

#### 1,4-Bis(2,4-dihydroxyphenyl)-1,4-butanedione

CO(CH₂)₂CO

 $[16290-14-5] C_{16}H_{14}O_6 mol. wt. 302.28$ OH HO Syntheses

OH

-Obtained by reaction of succinic anhydride with resorcinol in the presence of zinc chloride at  $170^{\circ}$  for 2 h (25 %) [651].

-Also obtained by reaction of succinic acid with resorcinol in the presence of zinc chloride [2256].

-Also obtained by reaction of 4-(2,4-dihydroxyphenyl)-4-oxo-1-butanoic acid with resorcinol in the presence of zinc chloride [651].

-Also obtained by condensation of  $\beta$ -(2-hydroxy-4-methoxybenzoyl)propionic acid methyl ester with resorcinol in the presence of a mixture of aluminium chloride and sodium chloride at 190–200° for 50 min (71 %) [1669, 1670].

m.p. 319° [651], 310–312° [2567], 295–296° [1669, 1670], 142–143° [2515]; **N.B.**: One of the reported melting point is obviously wrong.
¹H NMR [1670, 2515], IR [651, 2515],
UV [651, 1669, 1670], MS [1670, 2515].

Isolation from natural sources

-From Gnetum ula (Gnetaceae) [2515].

Tetraacetate	[16290-15-6]	$C_{24}H_{22}O_{10}$	mol. wt. 470.43
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-Obtained by reaction of acetic anhydride with the title diketone in the presence of pyridine [651].

-Also refer to: [2567].

m.p. 166° [651], 140–141° [2567].

N.B.: One of the reported melting point is obviously wrong.

#### Dioxime

mol. wt. 388.42

m.p. 218-220° [2567].

Tetramethyl ether	[16290-20-3]	$C_{20}H_{22}O_{6}$	mol. wt. 358.39
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-Obtained by reaction of dimethyl sulfate with the title diketone in the presence of potassium carbonate in refluxing acetone for 10 h (63 %) [651].

-Obtained by heating a mixture of 1-(2,4-dimethoxyphenyl)-2-propen-1-one (0.026 mol), 2,4-dimethoxybenzaldehyde (0.030 mol), 3-benzyl-5-(2-hydroxyethyl)-4-methylthiazolium chloride (0.0021 mol) and triethylamine (0.014 mol) in DMF at  $65^{\circ}$  for 4.5 h (78 %) [241].

-Also obtained by Friedel-Crafts reaction of succinyl chloride with resorcinol dimethyl ether in the presence of aluminium chloride (7 %) [2142].

-Obtained: 2,4-dimethoxyacetophenone silyl enol ether was oxidatively coupled at  $97^{\circ}$  (74 %) [911].

-Also refer to: [2567].

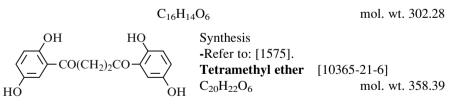
colourless fine needles [2142]; m.p. 148–150° [2567], 148° [651], 146° [241], 145° [911], 128–129° [2142]; ¹H NMR [241, 911, 2142], ¹³C NMR [241, 911, 2142], IR [241, 651, 911, 2142], UV [651, 2142], MS [241, 911, 2142]; luminescence spectroscopy [2142].

# (E,E)-Di-O-methyl oxime of the tetramethyl ether

[125304-88-3] C₂₂H₂₈N₂O₆ mol. wt. 416.47 -Refer to: [911 (25 %)]. m.p. 122° [911];

¹H NMR [911], ¹³C NMR [911], IR [911], MS [911].

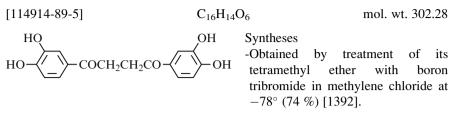
# 1,4-Bis(2,5-dihydroxyphenyl)-1,4-butanedione



-Obtained by reaction of succinic acid dichloride (1 mol) with hydroquinone dimethyl ether (2.5 mol) in the presence of aluminium chloride (1 mol) [1575].

m.p. 150° [1575].

#### 1,4-Bis(3,4-dihydroxyphenyl)-1,4-butanedione



-Also refer to: [349].

m.p. 280–283° [1392]; ¹H NMR [1392], IR [1392].

**Tetramethyl ether** [4650-71-9] C₂₀H₂₂O₆ mol. wt. 358.39

-Obtained by adding 3,4-dimethoxyacetophenone to lithium diisopropylamide (LDA) in tetrahydrofuran at  $-40^{\circ}$  under nitrogen. After 1 h, cupric chloride in DMF was added and stirring was continued overnight (25 %) [349].

-Also obtained by reduction of 1,2-bis(3,4-dimethoxybenzoyl)ethene with zinc dust in acetic acid (0.6 %) [1392]. The mixture was heated at 80° for 1 h (39 %) [1392]. -Also refer to: [321, 1279, 1533, 2379, 3088, 3133, 3363, 3364].

m.p. 181–182° [349], 180–181° [1279, 3133], 176–178° [3088], 154–156° [1392], 115.2–116.2° [3363].
N.B.: One of the reported melting point is obviously wrong.
¹H NMR [321, 349, 1392, 1533, 2379, 3088], ¹³C NMR [321, 3088], IR [1392, 1533, 3364], MS [3088].

Synthesis

BIOLOGICAL ACTIVITY: Antiprotozoal [321].

#### 1,4-Bis(2,3,4-trihydroxyphenyl)-1,4-butanedione

HO OH HO OH HO CO(CH₂)₂CO OH mol. wt. 334.28

-Obtained by reaction of succinic anhydride with pyrogallol in the presence of zinc chloride [1574].

Hexamethyl ether

[10388-38-2]

 $C_{16}H_{14}O_8$ 

C₂₂H₂₆O₈ mol. wt. 418.44

-Obtained by reaction of dimethyl sulfate with 1,4-bis(2,3,4-trihydroxyphenyl)-1,4-butanedione in the presence of 20 % sodium hydroxide (43 %) [1574].

m.p. 150° [1574].

mol. wt. 334.28

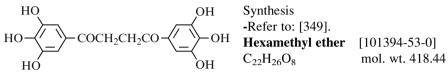
## 2,4-Dinitrophenylhydrazone of the hexamethyl ether

 $[10388-39-3] C_{34}H_{34}N_8O_{14} mol. wt. 778.69$ 

m.p. 160° [1574].

# 1,4-Bis(3,4,5-trihydroxyphenyl)-1,4-butanedione

 $C_{16}H_{14}O_8$ 



-Obtained by adding 3,4,5-trimethoxyacetophenone to lithium diisopropylamide (LDA) in tetrahydrofuran at  $-70^{\circ}$  under nitrogen. After 2 h, cupric chloride in DMF was added and stirring was continued for 2 more hours (49 %) [349]. -Also refer to: [321].

white solid [349]; m.p. 189–190° [349]; ¹H NMR [321], ¹³C NMR [321].

BIOLOGICAL ACTIVITY: Antiprotozoal [321].

# 1,4-Bis(3-bromo-2-hydroxy-5-methylphenyl)-1,4-butanedione

[128733-94-8] C₁₈H₁₆Br₂O₄ mol. wt. 456.13 Syntheses OH OH -Obtained by reaction of bromine with Br CO(CH₂)₂CC 1.1'-bis (2-hydroxy-5-methylphenyl)-1,4-butanedione in acetic acid [1035]. ĊH₃ ĊH3 -Also obtained by treatment of 1,4-bis (2-hydroxy-5-methylphenyl)-1,4-butanedione with 2,4,4,6-tetrabromocyclohexa-2,5-dienone first at  $0^{\circ}$ , then at  $25^{\circ}$  for 13 h (88 %) [473].

slightly yellow solid [473]; m.p. 238–240° [473], 232° [1035]; ¹H NMR [473], MS [473].

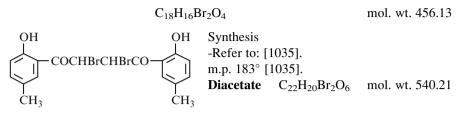
# Diacetate $C_{22}H_{20}Br_2O_6$

mol. wt. 540.21

-Obtained by reaction of acetic anhydride with the title ketone in the presence of sodium acetate [1035].

m.p. 140° [1035].

#### 1,4-Bis(2-hydroxy-5-methylphenyl)-2,3-dibromo-1,4-butanedione



-Obtained by reaction of acetic anhydride with the title diketone in the presence of sulfuric acid [1035].

m.p. 187° [1035].

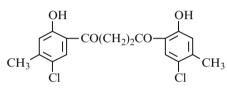
## 1,4-Bis(5-chloro-2-hydroxy-4-methylphenyl)-1,4-butanedione

[16197-59-4]



Synthesis

mol. wt. 367.23



-Obtained by Fries rearrangement of di-(4-chloro-3-methylphenyl) succinate with aluminium chloride at 130– 140° for 3–4 h [594]. m.p. 194° [594].

Di-2,4-dinitrophenylhydrazone [16197-60-7] C₃₀H₂₄Cl₂N₈O₁₀ mol. wt. 727.47

-Refer to: [592, 594].

m.p. 250° [594].

# 1,4-Bis(2-hydroxy-4-methylphenyl)-1,4-butanedione

(Hofmeisterin II)

[122427-50-3]



mol. wt. 298.34



Isolation from natural sources -From the aerial parts of *Hofmeisteria schaffneri* (Asteraceae) [2455].

colourless crystalline needles [2455]; m.p. 193° [2455]; ¹H NMR [2455], ¹³C NMR [2455], IR [2455], UV [2455], MS [2455]; X-ray data [2455].

BIOLOGICAL ACTIVITY: Phytotoxicity [2455].

#### 1,4-Bis(2-hydroxy-5-methylphenyl)-1,4-butanedione

[13282-23-0]  $C_{18}H_{18}O_{4}$ mol. wt. 298.34 OH OH Syntheses -Obtained by Fries rearrangement of di CO(CH₂)₂CO (4-methylphenyl) succinate with aluminium chloride. *without solvent, -at 130° for 4 h [1035], (44 %) [3107, 3465]; -at 130–140° for 3–4 h [594], (25 %) [2013]; -at 180–185° for 2.5 h (49 %) [473]. *in refluxing chlorobenzene for 6 h (43 %) [3107]. light brown solid [2013]; light, off-white solid [473]; m.p. 189° [594, 3107], 187–188° [3465], 187° [1035], 185° [1966], 184–186° [2013], 180–189° [473]; ¹H NMR [473, 3465], IR [2013, 3107, 3465],

UV [2013], MS [1966].

Isolation from natural sources

-From *Berberis coriaria* (Berberidaceae) [1966]. -From *Berberis acanthifolium* (Berberidaceae) [2013].

**Di-2,4-dinitrophenylhydrazone** [16197-56-1] C₃₀H₂₆N₈O₁₀ mol. wt. 658.58

m.p. 239° [594].

Dioxime	[16197-57-2]	$C_{18}H_{20}N_2O_4$	mol. wt. 328.37
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-Refer to: [592, 594].

m.p. 269° [594].

Diacetate	[69618-10-6]	$C_{22}H_{22}O_{6}$	mol. wt. 382.40
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-Obtained by reaction of acetic anhydride with the title diketone in the presence of sulfuric acid [1035].

-Also refer to: [1966].

m.p. 163° [1035], 141° [1966]; ¹³C NMR [1966], ¹H NMR [1966], IR [1966], UV [1966], MS [1966]. **Dimethyl ether** [69618-11-7] C₂₀H₂₂O₄ mol. wt. 326.39

-Obtained by reaction of methyl iodide with the title diketone in the presence of potassium carbonate [1966], (85 %) [2013].

m.p. 68° [1966]; ¹H NMR [1966, 2013], ¹³C NMR [1966], IR [1966, 2013], UV [1966, 2013], MS [1966, 2013].

 $C_{18}H_{18}O_4$ 

# 1,4-Bis(4-hydroxyphenyl)-2,3-dimethyl-1,4-butanedione

 HO
  $CH_3 CH_3$  $CH_-CH-CH-CO$  Synthesis -Refer to: [2461].

 Dimethyl ether (racemic)
  $C_{20}H_{22}O_4$  mol. wt. 326.39

 -Obtained by condensation of  $\alpha$ -bromo-p-methoxypropiophenone (m.p. 66–69°) with p-methoxy-propiophenone (67 %) [2461].
 m.p. 124–127° [2461]; ¹H NMR [2461], IR [2461], MS [2461].

 Dimethyl ether
 [81096-36-8]
  $C_{20}H_{22}O_4$  mol. wt. 326.39

 -Refer to: [2671].
 m.p. 166–168° [2671]; ¹H NMR [2671], IR [2671].
 mol. wt. 326.39

 Dimethyl ether (meso)
 [222158-42-1]
  $C_{20}H_{22}O_4$  mol. wt. 326.39

 -Refer to: [2671].
 1H NMR [2671], IR [2671].
 mol. wt. 326.39

# 1, 4-Bis (3, 4-dihydroxyphenyl)-2, 3-dimethyl-1, 4-but anedione

C₁₈H₁₈O₆

HO HO HO CO-CH-CH-CO CH₃ CH₃ OH HO OH OH

H Synthesis -Refer to: [1474]. -OH **Tetramethyl ether** [4440-92-0] C₂₂H₂₆O₆ mol. wt. 386.45

mol. wt. 330.34

-Obtained by reaction* of a THF solution of lithium enolate of 3,4-dimethoxypropiophenone with  $CuCl_2$  in DMF at  $-78^{\circ}$  (31 %) [1474].

*(Oxidative dimerization of 3,4-dimethoxypropiophenone).

-Also obtained by treatment of bis-methyl ethyl veratroylacetate with sodium hydroxide in ethanol at r.t. for 24 h (48 %) [232].

-Also obtained by treatment of  $\beta$ -bromopropioveratrone with copper powder in refluxing xylene for 24 h [171].

-Also refer to: [85 (90 %), 678, 2535].

367

mol. wt. 298.34

			-	
m.p. 189–190°	bic prisms [171]; [171, 232], 177–178° [ ], IR [1474]; TLC [1			
-		-		
(meso-isomer)	[36287-37-3]	$C_{22}H_{26}O_{6}$	mol. wt. 386.45	
-Refer to: [349, 24	61 (3.8 %)].			
white crystals [ m.p. 189–190° ¹ H NMR [2461	[171], 184.5–187.5° [2	461];		
(racemic)	[27686-81-3]	$C_{22}H_{26}O_{6}$	mol. wt. 386.45	
-Refer to: [349, 35	0, 2461 (90.3 %)].			
m.p. 145–146°	[2461]; ¹ H NMR [24	61], IR [2461], MS	[2461].	
Monophenylhydr	azone of the tetramet	hyl ether (racemic)		
[116588-92-2]	C ₂₈ H ₃₂	$N_2O_5$	mol. wt. 476.57	
-Obtained by reaction of phenylhydrazine with the tetramethyl ether in refluxing ethanol for 1 h [85].				
yellow needles ¹ H NMR [85], 1	[85]; m.p. 140° [85]; IR [85].			
$\begin{array}{llllllllllllllllllllllllllllllllllll$				
-Refer to: [232].				
1,4-Bis(2-hydroxy-4-methoxyphenyl)-1,4-butanedione				
[16290-18-9]	$C_{18}H_{1}$	₈ O ₆	mol. wt. 330.34	
ОН	но	Syntheses -Obtained	by methylation of	

CH₃O- $\rightarrow$  COCH₂CH₂CO-·OCH₃

1,4-bis (2,4-dihydroxyphenyl)-1,4-butanedione with excess diazomethane in ethyl ether at r.t. overnight (31 %) [1669, 1670].

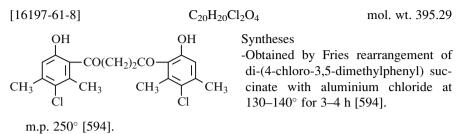
-Also refer to: [651].

Isolation from natural sources

-From Gnetum ula [1670, 2515].

yellow needles [1670]; yellow crystals [1669]; m.p. 200–202° [1669, 1670], 192° [651]; ¹H NMR [1669, 1670], IR [651], UV [651], MS [1669, 1670].

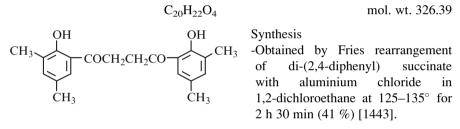
#### 1,4-Bis(3-chloro-6-hydroxy-2,4-dimethylphenyl)-1,4-butanedione



# $\label{eq:2.1} \textbf{Di-2,4-dinitrophenylhydrazone} ~~ [16197-62-9] ~~ C_{32}H_{28}Cl_2N_8O_{10} ~~ mol. ~~ wt. ~~ 755.53$

m.p. 220° [594].

# 1,4-Bis(2-hydroxy-3,5-dimethylphenyl)-1,4-butanedione



C20H24N2O4

 $C_{24}H_{26}O_{6}$ 

m.p. 206–208° [1443].

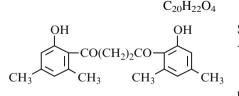
#### Dioxime

m.p. 225-230° [1443].

#### Diacetate

m.p. 161° [1443].

# 1,4-Bis(2-hydroxy-4,6-dimethylphenyl)-1,4-butanedione



mol. wt. 326.39

mol. wt. 356.42

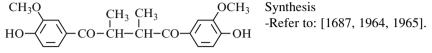
mol. wt. 410.47

Synthesis

-Obtained by Fries rearrangement of bis (3,5-dimethylphenyl) succinate with aluminium chloride [1035]. m.p. 141° [1035].

#### 1,4-Bis(4-hydroxy-3-methoxyphenyl)-2,3-dimethyl-1,4-butanedione

 $\begin{array}{cccc} [1173986-38-3] \ (+) & C_{20}H_{22}O_6 & \mbox{mol. wt. } 358.39 \\ [1173986-37-2] \ (-) & \\ [1204225-56-8] \ \mbox{meso} & \end{array}$ 



(+)-7,7'-Dioxodihydroguaiaretic [1173986-38-3]  $C_{20}H_{22}O_6$  mol. wt. 358.39 -Refer to: [3380].

¹H NMR [3380], ¹³C NMR [3380], MS [3380];  $(\alpha)_D^{20} = +102^{\circ}$  [3380].

BIOLOGICAL ACTIVITY: Antibacterial [1642]; Antifungal [1642]; Inhibition of enzyme activity [3380].

(-)-7,7'-Dioxodihydroguaiaretic [1173986-37-2]  $C_{20}H_{22}O_6$  mol. wt. 358.39 -Refer to: [1642].

 $(\alpha)_{\rm D}^{20} = -102^{\circ}$  [3380].

BIOLOGICAL ACTIVITY: Antibacterial [1642]; Antifungal [1642]; Inhibition of enzyme activity [3380].

Meso -7,7'-Dioxodihydroguaiaretic [1204225-56-8] C₂₀H₂₂O₆ mol. wt. 358.39

-Refer to: [1642].

colourless cristal [1642]; m.p. 175–177° [1642]; ¹H NMR [1642], ¹³C NMR [1642], MS [1642]

BIOLOGICAL ACTIVITY: Antibacterial [1642]; Antifungal [1642].

**Dimethyl ether** [4440-92-0] C₂₂H₂₆O₆ mol. wt. 386.45

-Refer to: [85, 171, 232, 678, 1474, 2535].

m.p. 189–190° [171, 232]; 177–178° [1474]; 165° [85]; ¹H NMR [1474], ¹³C NMR [1474], IR [1474], MS [1474].

*Meso isomer* [36287-37-3]  $C_{22}H_{26}O_6$  mol. wt. 386.45

-Refer to: [349, 2461].

m.p. 186–190° [2461]; ¹H NMR [2461], MS [2461].

-Refer to: [349, 350, 2461].

m.p. 145–146° [2461]; ¹H NMR [2461], IR [2461], MS [2461].

**Diethyl ether** [986-89-0]  $C_{24}H_{30}O_6$  mol. wt. 414.50

-Obtained by heating  $\alpha$ -bromo-4-ethoxy-3-methoxypropiophenone with freshly precipitated copper in xylene for 24 h (7 %) [1687]. -Also refer to: [1964, 1965].

needles [1687]; m.p. 185–186° [1687], 185° [1964, 1965]; ¹H NMR [1964, 1965], IR [1964, 1965], UV [1687, 1964], MS [1964, 1965].

**Methyl and ethyl ether** [749-41-7] C₂₃H₂₈O₆ mol. wt. 400.47

-Following the conditions used for the benzylation of propiophenone, sodium O-ethylpropio-vanillone and  $\alpha$ -bromopropioveratrone afforded two products separated after alumina chromatography by crystallisation from methanol. Only the minor more-soluble product consisted of the titled diketone [1687].

m.p. 161° [1687].

#### 1,4-Bis(2-hydroxy-3,4-dimethoxylphenyl)-1,4-butanedione

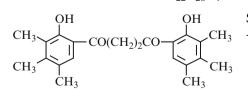
[10351-89-0]  $C_{20}H_{22}O_8$  mol. wt. 390.39  $CH_3O$  OH HO OCH₃ Synthesis  $CH_3O$  CO(CH₂)₂CO OCH₃ OCH₃ -Obtained by reaction of succinic acid dichloride with pyrogallol trimethyl ether in the presence of aluminium chloride in tetrachloroethane [1574].

m.p. 208° [1574].

#### 1,4-Bis(2-hydroxy-3,4,5-trimethylphenyl)-1,4-butanedione

C22H26O4

mol. wt. 354.45



Synthesis

,CH₃ -Obtained (by-product) by Fries rearrangement of 2,3,5-trimethyl, CH₃ phenyl succinate with aluminium chloride (2 mol) in tetrachloroethane at 140° for several hours (20 %) [2908].

m.p. 246-248° [2908].

Diacetate

C26H30O6

mol. wt. 438.52

m.p. 213–214° [2908].

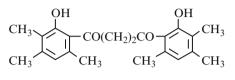
#### 1,4-Bis(2-hydroxy-3,4,6-trimethylphenyl)-1,4-butanedione

[860705-13-1]

 $C_{22}H_{26}O_4$ 

**Syntheses** 

mol. wt. 354.45



-Obtained by Fries rearrangement of 2,3,5-trimethylphenyl succinate with aluminium chloride for 1.5 h at  $140-150^{\circ}$ ,

*in tetrachloroethane (31 %) [2908];

*in stannic chloride (24 %) [2908];

*in o-dichlorobenzene (20 %) (very impure) [2908];

*without solvent (10 %) [2908].

-Also obtained by Friedel-Crafts reaction of succinic acid with 2,3,5-trimethylphenol in the presence of boron trifluoride etherate (9 %) [2908].

-Also obtained by hydrolysis of the acetate by action for 48 h of boiling aqueous methanol containing hydrochloric acid (79 %) [2908].

m.p. 207-209° [2908].

#### Diacetate

C₂₆H₃₀O₆ mol. wt. 438.52

-Obtained by reaction of acetic anhydride with the title diketone in boiling pyridine for 2 h (60 %) [2908].

m.p. 154-156° [2908].

# **Dimethyl ether**

 $C_{24}H_{30}O_4$ 

mol. wt. 382.50

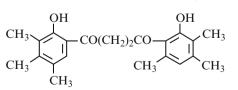
-Obtained by reaction of methyl iodide with the disodium salt of the title diketone in xylene (50 %) [2908].

m.p. 124-125° [2908].

# 1-(2-Hydroxy-3,4,5-trimethylphenyl)-4-(2-hydroxy-3,4,6-trimethylphenyl)-1,4-butanedione

C22H26O4

mol. wt. 354.45



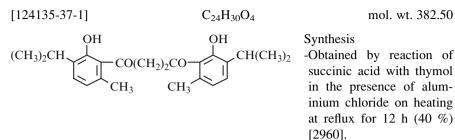
Synthesis

-Obtained (by-product) by Fries rearrangement of 2,3,5-trimethylphenyl succinate with aluminium chloride (2 mol) in tetrachloroethane at  $140^{\circ}$  for several hours [2908].

m.p. 246-248° [2908].

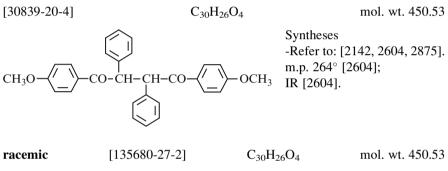
# 1,4-Bis[2-hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1,4-butanedione

(Succinothymon)



m.p. 44° [2960].

#### 1,4-Bis(4-methoxyphenyl)-2,3-bis(phenyl)-1,4-butanedione



colourless microcrystalline powder [2142]; m.p. 136–137° [2142]; ¹H NMR [2142, 2143], ¹³C NMR [2142], IR [2142], UV [2142], MS [2142]; luminescence spectroscopy [2142].

#### meso

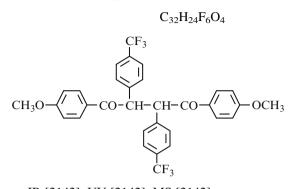
#### $C_{30}H_{26}O_{4}$

mol. wt. 450.53

colourless powder [2142]; m.p. 258–260° [2142]; ¹H NMR [2142], ¹³C NMR [2142], IR [2142], UV [2142], MS [2142], luminescence spectroscopy [2142].

mol. wt. 586.53

## 1,4-Bis(4-methoxyphenyl)-2,3-bis[(4-trifluoromethyl)phenyl]-1,4-butanedione



Synthesis -Refer to: [2142]. **racemic**   $C_{32}H_{24}F_6O_4$  mol. wt. 586.53 colourless powder [2142]; ¹H NMR [2142], ¹³C NMR [2142],

IR [2142], UV [2142], MS [2142]; luminescence spectroscopy [2142].

#### meso

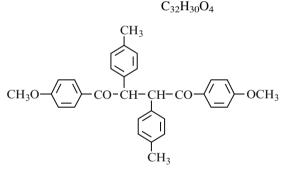
## $C_{32}H_{24}F_6O_4$

mol. wt. 586.53

colourless powder [2142];

¹H NMR [2142], ¹³C NMR [2142], UV [2142], IR [2142], MS [2142]; luminescence spectroscopy [2142].

## 1,4-Bis(4-methoxyphenyl)-2,3-bis(4-methylphenyl)-1,4-butanedione



mol. wt. 478.59

Synthesis -Refer to: [2142]. **racemic**   $C_{32}H_{30}O_4$  mol. wt. 478.59 colourless plates [2142]; m.p. 196–198° [2142]; ¹H NMR [2142], ¹³C NMR [2142],

IR [2142], UV [2142], MS [2142]; luminescence spectroscopy [2142].

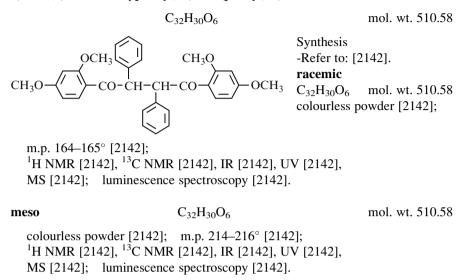
#### meso

 $C_{32}H_{30}O_4$ 

mol. wt. 478.59

colourless crystals [2142]; m.p. 229–230° [2142]; ¹H NMR [2142], ¹³C NMR [2142], IR [2142], UV [2142], MS [2142]; luminescence spectroscopy [2142].

#### 1,4-Bis(2,4-dimethoxyphenyl)-2,3-bis(phenyl)-1,4-butanedione



#### 1,4-Bis(4-methoxyphenyl)-2,3-(4-methoxyphenyl)-1,4-butanedione

 $\begin{bmatrix} 103326-23-4 \end{bmatrix} \qquad \begin{array}{c} C_{32}H_{30}O_{6} \\ & & \\ & & \\ & & \\ & & \\ CH_{3}O - & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & &$ 

Syntheses -Refer to: [781, 2142, 2157]. m.p. 208° [781]; ¹H NMR [2157], ¹³C NMR [2157], UV [781].

### racemic

## C32H30O6

mol. wt. 510.58

mol. wt. 510.58

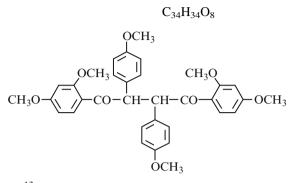
colourless needles [2142]; m.p. 151–152° [2142]; ¹H NMR [2142], ¹³C NMR [2142], IR [2142], UV [2142], MS [2142]; luminescence spectroscopy [2142].

meso	$C_{32}H_{30}O_{6}$	mol. wt. 510.58
111050	$C_{32} I_{30} O_6$	1101. wt. 510.38

colourless microcrystalline powder [2142]; m.p. 203–204° [2142]; ¹H NMR [2142], ¹³C NMR [2142], IR [2142], UV [2142], MS [2142]; luminescence spectroscopy [2142].

mol. wt. 570.64

## 1,4-Bis(2,4-dimethoxyphenyl)-2,3-(4-methoxyphenyl)-1,4-butanedione



Synthesis -Refer to: [2142]. **racemic**   $C_{34}H_{34}O_8$  mol. wt. 570.64 colourless needles [2142]; m.p. 133–134° [2142]; ¹H NMR [2142],

¹³C NMR [2142], IR [2142], UV [2142],
MS [2142]; luminescence spectroscopy [2142].

meso

CH₃CC

HC

 $C_{34}H_{34}O_8$ 

mol. wt. 570.64

colourless crystals [2142]; m.p. 172–173° [2142]; ¹H NMR [2142], ¹³C NMR [2142], IR [2142], UV [2142], MS [2142]; luminescence spectroscopy [2142].

## 11 Diketones Derived from Acetic and 1-Butanoic Acids

## 1-(3-Bromo-5-acetyl-2,4,6-trihydroxyphenyl)-1-butanone

$C_{12}H$	$I_{13}BrO_5$	mol. wt. 317.14
Н	Synthesis	
CO(CH ₂ ) ₂ CH ₃	-Obtained by acylating the	phloroglucinol with
1	appropriate acyl chloride o	or acid anhydride in
∕ _{OH}	the presence of boron trifluo	oride [3391].
r	m.p. 109–110° [3391].	

-Obtained by reaction of butyryl chloride with 2-hydroxyacetophenone in the presence of aluminium chloride in carbon

## 1-[3-Acetyl-4-hydroxyphenyl]-1-butanone

Synthesis

 $[173469-74-4] \qquad \qquad C_{12}H_{14}O_3 \qquad \qquad \text{mol. wt. } 206.24$ 

OH COCH₃ CO(CH₂)₂CH₃

m.p. 57–58° [345]; ¹H NMR [345], IR [345].

disulfide (90 %) [345].

## 1-(5-Acetyl-2-hydroxyphenyl)-1-butanone

m.p. 54–55° [1305]; ¹H NMR [1305], IR [1305].

## 1-(3-Acetyl-2-hydroxy-5-methylphenyl)-1-butanone

 $\begin{array}{c} \mbox{[213622-17-4]} & C_{13}H_{16}O_3 & \mbox{mol. wt. 220.27} \\ OH & Synthesis \\ CH_3CO & CO(CH_2)_2CH_3 & -Obtained by reaction of butyryl chloride with 2-hydroxy-5-methylacetophenone in the presence of aluminium chloride in carbon disulfide \\ \end{array}$ 

at r.t. for 1 h (90 %) [344].

m.p. 53–55° [344]; ¹H NMR [344], IR [344].

Benzoate [213622-22-1] C₂₀H₂₀O₄ mol. wt. 324.38

-Obtained by reaction of benzoyl chloride with 1-(3-acetyl-2-hydroxy-5-methylphenyl)-1-butanone in the presence of pyridine (95 %) [344].

m.p. 90–91° [344].

**p-Chlorobenzoate** [213622-23-2] C₂₀H₁₉ClO₄ mol. wt. 358.82

-Obtained by reaction of p-chlorobenzoic acid and phosphorous oxychloride with 1-(3-acetyl-2-hydroxy-5-methylphenyl)-1-butanone in the presence of pyridine (90 %) [344].

m.p. 101-102° [344].

**p-Methylbenzoate** [213622-24-3] C₂₁H₂₂O₄ mol. wt. 338.40

-Obtained by reaction of p-toluic acid and phosphorous oxychloride with 1-(3-ace-tyl-2-hydroxy-5-methylphenyl)-1-butanone in the presence of pyridine (95 %) [344].

m.p. 65–66° [344].

## **p-Methoxybenzoate** [213622-25-4] C₂₁H₂₂O₅ mol. wt. 354.40

-Obtained by reaction of p-toluic acid and phosphorous oxychloride with 1-(3-acetyl-2-hydroxy-5-methylphenyl)-1-butanone in the presence of pyridine (95 %) [344].

m.p. 73-75° [344].

### 1-(5-Acetyl-2-hydroxyphenyl)-3-methyl-1-butanone

(Desmethylespeleton)

[62458-64-4]

C₁₃H₁₆O₃ mol. wt. 220.27

Syntheses -Preparation by Fries rearrangement of 4-acetylphenyl isovalerate in the presence of aluminium chloride without solvent at 140–160° for 2.5 h (51 %) [402] or at 150° for 3 h (32 %) [1305].

-Also obtained by Friedel-Crafts acylation of 4-hydroxyacetophenone with isovaleryl chloride (4 mol) in tetrachlroethane at  $130^{\circ}$  for 4 h (37 %) [1305].

-Also obtained by treatment of its methyl ether with 48 % hydrobromic acid in refluxing acetic acid for 6 h (65 %) [2220].

-Also obtained by treatment of 2-isovaleroyl-4-(1-hydroxyethyl)phenol with manganese dioxide in ethyl ether for 2 h [3444].

Isolation from natural sources

-From the genus *Flourensia cernua* DC in Sub-tribe *Helianthinae* (Tribe Heliantheae) (Compositae) [396].

-From the aerial parts of *Ophryosporus floribundus* (Compositae, tribe Eupatorieae) [3443].

-From sliced yacon tubers after inoculation with the bacterium *Pseudomonas cichorii* and incubation at 20° for 3 days, then extraction with acetone. Yacon (*Polymnia sonchifolia*) (Compositae) is cultivated in South America and has recently been introduced in Japan [3035].

-From the aerial parts of *Ophryosporus axilliflorus* (Griseb.) Hieron (Asteraceae) [979, 2353].

-From Helichrysum argyrophyllum DC (86/18, Hogsback) [1488].

-From Brachyclados megalanthus [3444].

colourless oil [396]; white crystals [2353]; b.p.₄ 156–158° [2220]; m.p. 94.5–96° [3035], 64–66° [1305, 2353], 61.5° [402]. **N.B.**: One of the reported melting points is obviously wrong. ¹H NMR [396, 1305, 2353, 3035, 3444], ¹³C NMR [2353, 3035], IR [396, 1305, 3035], UV [3035], MS [396, 3035]; TLC [3444].

BIOLOGICAL ACTIVITY: Antiinflammatory [979, 2353].

Methyl ether	[51995-98-3]	$C_{14}H_{18}O_3$	mol. wt. 234.30
(Espeleton)			

Syntheses

-Obtained by oxidation of 1-[5-(1-hydroxyethyl)-2-methoxyphenyl]-3-methyl-1-butanone with chromium trioxide in pyridine at 25° for 15 h [399, 409].

-Also obtained by treatment of 5-ethyl-2-methoxyisovalerophenone,

*with DDQ in refluxing dioxane for 70 h (60 %) [2220];

*with BTAP (benzyltriethylammonium permanganate) in acetic acid at  $30^{\circ}$  for 2 days (26 %) [2701].

Isolation from natural sources

-From Espeletia Schultzii Wedd. (Tribe Heliantheae; Compositae) [399].

-From Heteroplexis microcephala (Compositae) [971].

-From Otanthus maritimus (Asteraceae) [662].

-From the aerial parts of Eupatorium aschenborniana Schauer (Asteraceae) [2626].

-From Ageratina glabrata (HBK) King et Rob (Eupatorium Groupe) [409].

-From aerial parts of *Ageratina pichinchensis* var. bustamenta (DC) R.M. King and H. Rob., Axihuitl [46].

-From Bahianthus viscidus [408].

-From aerial parts of Verbesina luetzelburgl [407].

colourless oil [399]; b.p.₁ 140–142° [2701], b.p.₅ 151–153° [2220]; ¹H NMR [399, 2701], IR [399, 2701], UV [399], MS [399].

USE: Antifungal [2626]; Insecticidal [662]; Cytotoxic activity [971]; Active against dermatophytes [2626]; Antimicrobial [2626].

## Glucoside 1-[5-Acetyl-2-(β-D-glucopyranosyloxy)phenyl]-3-methyl-1-butanone

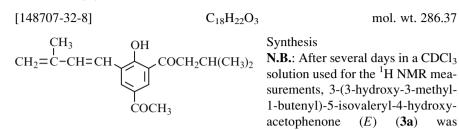
[934302-22-4]  $C_{19}H_{26}O_8$  mol. wt. 382.41 -From the aerial parts of *Ophryosporus axilliflorus* (Griseb) Hieron

-From the aerial parts of *Ophryosporus axilliflorus* (Griseb.) Hieron (Asteraceae) [2353].

-Also obtained by treatment of 1-(5-acetyl-2-hydroxyphenyl)-3-methyl-1-butanone with Brassica napus hairy roots.

pale yellow crystals [2353]; ¹H NMR [2353], ¹³C NMR [2353];  $(\alpha)_D^{25} = -40.8^{\circ}$  (methanol) [2353].

## 1-[5-Acetyl-2-hydroxy-3-(3-methyl-1,3-butadienyl)phenyl]-3-methyl-**1-butanone** (E)



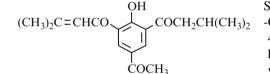
¹H NMR [2880].

## 1-[5-Acetyl-2-hydroxy-3-(3-methyl-1-oxo-2-butenyl)phenyl]-3-methyl-1-butanone

$$C_{18}H_{22}O_4$$

mol. wt. 302.37

was



**Synthesis** 

(**3b**) [2880].

-Obtained by oxidation of 4-hydroxy-3-(1-hydroxy-3,3-dimethylallyl)-5-isovalerylacetophenone with manganese dioxide in ethyl ether for 30 min (60 %) [395].

converted into the titled substance

## Isolation from natural source

-From the roots of Gerbera piloselloides Cass. (Family of Compositae, Tribe Arctotideae) [395].

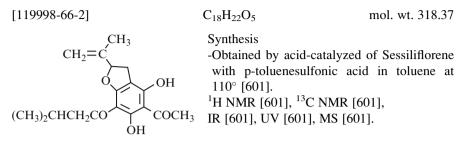
colourless oil [395]; ¹H NMR [395], IR [395], MS [395].

## 1-[7-Acetyl-2,3-dihydro-4,6-dihydroxy-2-(1-methylethenyl)-5-benzofuranyl]-3-methyl-1-butanone

COCH₃ HO OH  $COCH_2CH(CH_3)_2$  C18H22O5 mol. wt. 318.37

**Synthesis** -Obtained by acid-catalyzed of Sessiliflorene with p-toluenesulfonic acid in toluene at 110° [601]. ¹H NMR [601], ¹³C NMR [601], IR [601], UV [601], MS [601].

## 1-[5-Acetyl-2,3-dihydro-4,6-dihydroxy-2-(1-methylethenyl)-7-benzofuranyl]-3-methyl-1-butanone



# 1-[5-Acetyl-2-hydroxy-3-(1-hydroxy-3-methyl-2-butenyl)phenyl]-3-methyl-1-butanone

C₁₈H₂₄O₄

(Hydroxypiloselloidon)

[94413-27-1] [54963-61-0]

$$(CH_3)_2C = CH - CH - CH + OH - COCH_2CH(CH_3)_2 - COCH_3CH(CH_3)_2 - COCH_3CH(CH_3)CH(CH_3)CH(CH_3)CH(CH_3)CH(CH_3)CH(CH_3)CH(CH_3)CH(CH_3)CH(C$$

Isolation from natural sources

mol. wt. 304.39

-From the aerial parts of *Ophryosporus peruvianus* Gmel. K. et R. (Compositae) [410]. -From the roots of *Gerbera piloselloides* Cass. (Compositae) [395].

colourless oil [395]; ¹H NMR [395], IR [395], MS [395];  $(\alpha)_D^{24} = -50.4^{\circ}$  (chloroform) [395].

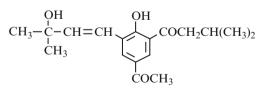
# 1-[5-Acetyl-2-hydroxy-3-(3-hydroxy-3-methyl-1-butenyl)phenyl]-3-methyl-1-butanone $({\cal E})$

(Hydroxyisopiloselloidon)

[54963-60-9]



mol. wt. 304.39



OH COCH₂CH(CH₃)₂ Isolation from natural sources -From the aerial parts of *Ophryosporus charua* Griseb. Hieron (Compositae) [831].

-From the aerial parts of *Ophryosporus macrodon* Griseb. (Compositae, tribe Eupatorieae) [2880].

-From the leaves of *Ophryosporus axilliflorus* (Griseb.) Hieron (Asteraceae-Asteroideae/Eupatorieae) [979].

-From the roots of *Gerbera piloselloides* Cass. (Family of Compositae, Tribe Arctotideae) [395].

colourless crystals [395]; m.p. 103–105° [2880], 97° [395]; ¹H NMR [395, 979, 2880], ¹³C NMR [979], IR [395]; UV [395], MS [395, 2880].

**BIOLOGICAL ACTIVITY:** Antiinflammatory [979].

## 1,1'-[5-Acetyl-2-hydroxy-1,3-phenylene]bis-3-methyl-1-butanone

[94413-28-2] Isolation from the natural sources OH  $COCH_2CH(CH_3)_2$ -From the aerial parts (CH₃)₂CHCH₂CC Ophryosporus peruvianus Gmel. K. et R. (Compositae) [410]. ĊOCH₂

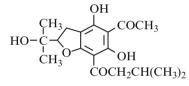
¹H NMR [410], IR [410], MS [410]; TLC [410].

## 1-[5-Acetyl-2,3-dihydro-4,6-dihydroxy-2-(1-hydroxy-1-methylethyl)-7-benzofuranyl]-3-methyl-1-butanone

 $C_{18}H_{24}O_{6}$ 

(Sessiliflorol A)

[119998-60-6]



Isolation from natural sources -From the Plant melicope sessiliflora (Rutaceae) [601]. m.p. 105–107° [601]; ¹H NMR [601], ¹³C NMR [601], IR [601].

UV [601], MS [601]; X-ray data [601].

BIOLOGICAL ACTIVITY: In vitro antiherpes activity [601].

## 1-[7-Acetyl-2,3-dihydro-4,6-dihydroxy-2-(1-hydroxy-1-methylethyl)-5-benzofuranyl]-3-methyl-1-butanone

(Sessiliflorol B)

[119998-61-7]	$C_{18}H_{24}O_{6}$		mo	ol. wt. 336.38
CH ₃ -C CH ₃ -C O COCH ₃ HO COCH ₂ CH(CH ₃ ) ₂	Isolation from -From the (Rutaceae) [6 ¹ H NMR [601 ¹³ C NMR [60 UV [601], MS	Plant 01]. ], 1], IR [601	melicope	sessiliflora

BIOLOGICAL ACTIVITY: In vitro antiherpes activity [601].

 $C_{18}H_{24}O_{4}$ 

mol. wt. 304.39

mol. wt. 336.38

of

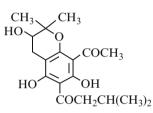
## 1-(6-Acetyl-3,4-dihydro-3,5,7-trihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl)-3-methyl-1-butanone

[119998-64-0]	$C_{18}H_{24}O_{6}$	mol. wt. 336.38
$\begin{array}{c} CH_3 \\ HO \\ HO \\ HO \\ COCH_2 CH(CH_3)_2 \\ HO \\ COCH_3 \end{array}$	Synthesis -Obtained by acid-catalyzed with p-toluenesulfonic acid 110° [601]. ¹ H NMR [601], ¹³ C NMR [601] IR [601], UV [601], MS [601]	1 in toluene at

1-(8-Acetyl-3,4-dihydro-3,5,7-trihydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-3-methyl-1-butanone

[119998-63-9]

 $C_{18}H_{24}O_6$  mol. wt. 336.38



Synthesis -Obtained by acid-catalyzed of Sessiliflorene with p-toluenesulfonic acid in toluene at 110° [601]. ¹H NMR [601], ¹³C NMR [601], IR [601], UV [601], MS [601].

## 1-[3-Acetyl-2,4,6-trihydroxy-5-(2-hydroxy-3-methyl-3-butenyl)phenyl]-3-methyl-1-butanone

(Sessiliflorene)

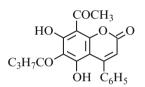
BIOLOGICAL ACTIVITY: *In vitro* antiherpes [601]; Antiprotozoal and antimicrobial [337].

## 1-[3-Acetyl-2,6-dihydroxy-4-methoxy-5-(3-methyl-2-butenyl)phenyl]-3-methyl-1-butanone

(Acronyculatin C)

## **5,7-Dihydroxy-6-(1-butanoyl)-8-acetyl-4-phenyl-2***H***-1-benzopyran-2-one** (*Racemosone*)

C₂₁H₁₈O₆ mol. wt. 366.37



Synthesis

-Obtained by reaction of acetyl chloride with 5,7-dihydroxy-6-(1-butanoyl)-4-phenyl-2*H*-1-benzopyran-2-one in the presence of aluminium chloride in carbon disulfide, then in refluxing nitromethane (11 %) [2146].

Isolation from natural sources

-From the leaves of Mesua racemosa (Clusiaceae) [2146].

m.p. 149–150° [2146]; ¹H NMR [2146], ¹³C NMR [2146], IR [2146], UV [2146], MS [2146].

## 1-[3-Acetyl-4-[[(2*E*)-3,7-dimethyl-2,6-octadienyl]oxy]-2,6-dihydroxy-5-methylphenyl]-2-methyl-1-butanone

[267009-82-5] C24H34O5 mol. wt. 402.53 Isolation from CH₃ OH natural sources CH₃CO CO-CH-C₂H₅ -From *Hypericum* CH₃-C=CHCH₂CH₂-C=CHCH₂O^{*} CH₃ CH₃ japonicum (Gutti-OH ferae) [1386]. CH₃ colourless oil [1386]; ¹H NMR [1386], ¹³C NMR [1386], IR [1386], UV [1386], MS [1386];  $(\alpha)_{D}^{31.2} = -7.02$  (methanol) [1386].

## 12 Diketones Derived from Propionic and 1-Butanoic Acids

### 1-[4-Hydroxy-3-(1-oxopropyl)phenyl]-1-butanone

 $\begin{array}{c} [173469-67-5] \\ OH \\ OH \\ COCH_2CH_3 \\ CO(CH_2)_2CH_3 \end{array} \begin{array}{c} C_{13}H_{16}O_3 \\ Obtained by reaction of butyryl chloride with 2-hydroxy-propiophenone in the presence of aluminium chloride in carbon disulfide (90 \%) [345]. \end{array} \right.$ 

## m.p. 81–82° [345]; ¹H NMR [345], IR [345].

## 1-[2,4,6-Trihydroxy-3-(1-oxopropyl)phenyl]-1-butanone

[96573-40-9]

 $C_{13}H_{16}O_5$ 

Syntheses

mol. wt. 252.27

CH₃CH₂CO HO OH CO(CH₂)₂CH₃

CO(CH₂)₂CH₃ -Obtained by reaction of butyryl chloride with phloropropiophenone in the presence of aluminium chloride in nitrobenzene for 3 days at r.t. (5–10 %) [421].

-Also refer to: [3033].

m.p. 135° [421]; ¹H NMR [421], IR [421], MS [421].

BIOLOGICAL ACTIVITY: Inhibitory effects of treatment with this acylphenol on TPA induced EBV-EA activation [3033].

## 1-[4-Hydroxy-3-(1-oxo-3-phenyl-2-propenyl)phenyl]-1-butanone

[173469-75-5]	$C_{19}H_{18}O_3$	mol. wt. 294.35
$\bigcup_{CO(CH_2)_2CH_3}^{OH} COCH=CHC_6H_5$	Synthesis -Obtained by reaction of benzalde 4-hydroxyphenyl)-1-butanone i potassium hydroxide in methano	n the presence of

m.p. 99–100° [345]; ¹H NMR [345], IR [345].

## 13 Diketones Derived from Benzoic and 1-Butanoic Acids

## 1-[5-Fluoro-3-(4-fluorobenzoyl)-2-hydroxyphenyl]-1-butanone

	nuorobenzoyr)-2-ny	• • • •	
[2250-71-7]	C ₁₇ H	$_{14}F_{2}O_{3}$	mol. wt. 304.29
F-CO	OH CO(CH ₂ ) ₂ CH ₃	2-butyryl-4-fluorop	es rearrangement of henyl 4-fluorobenzoate aluminium chloride for 6 h [1550].
m.p. 150° [155	0].		
2,4-Dinitropheny	lhydrazone	$C_{23}H_{18}F_2N_4O_6\\$	mol. wt. 428.39
m.p. 147° [155	0].		
1-(5-Benzoyl-2-hy	ydroxy-3-methylphe	enyl)-1-butanone	
[101789-78-0]	C ₁₈	$H_{18}O_3$	mol. wt. 282.34
OH	Synthese CH ₂ ) ₂ CH ₃ -Obtaine 2-methy (50 %)   -Also ob 2-butyry	s d by Fries rearrang 1-phenyl butyrate wi [106]. tained by reaction of	mol. wt. 282.34 gement of 4-benzoyl- th aluminium chloride benzoyl chloride with the presence of alu-
CH ₃ CO(C	Synthese CH ₂ ) ₂ CH ₃ -Obtaine 2-methy (50 %)   -Also ob 2-butyry minium	s d by Fries rearrang [-phenyl butyrate wi [106]. tained by reaction of vl-6-methylphenol in	gement of 4-benzoyl- th aluminium chloride benzoyl chloride with
CH ₃ CO(C COC ₆ H ₅	Synthese CH ₂ ) ₂ CH ₃ -Obtaine 2-methy (50 %)   -Also ob 2-butyry minium	s d by Fries rearrang [-phenyl butyrate wi [106]. tained by reaction of vl-6-methylphenol in	gement of 4-benzoyl- th aluminium chloride benzoyl chloride with

## 14 Diketones Derived from Formic and 1-Butanoic Acids

## 2,6-Dihydroxy-3-(1-oxobutyl)benzaldehyde

[855875-24-0]	$C_{11}H_{12}O_4$	mol. wt. 208.21
H O HO HO CO(CH ₂ ) ₂ CH ₃		resbutyrophenone; zinc cyanide, de; aluminium chloride and hydro- %) [2821].

yellow needles [2821]; m.p. 42–43° [2821].

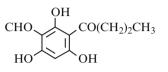
Semicarbazone

$$C_{12}H_{15}N_3O_4$$

m.p. 242–245° (d) [2821].

## 2,4,6-Trihydroxy-3-(1-oxobutyl)benzaldehyde

$$[96573-30-7] C_{11}H_{12}O_5 mtext{mol. wt. } 224.21$$



Synthesis CO(CH₂)₂CH₃ -Obtained by reaction of ethyl orthoformate with 2,4,6-trihydroxybutyrophenone in the presence of aluminium chloride in methylene chloride with cooling in an ice bath for 30 min (70 %) [421].

m.p. 163–165° (d) [421]; ¹H NMR [421], IR [421], MS [421].

BIOLOGICAL ACTIVITY: As photosynthetic electron transport (PET) [3405].

## 2,4,6-Trihydroxy-3-(3-methyl-( $d_3$ )-1-oxobutyl)benzaldehyde

$$C_{12}H_{11}D_3O_5$$
 mol. wt. 238.24

HO OH

 $CD_3$  Syntheses  $CO-CH_2-CH-CH_3$  -Obtained by reaction of ethyl orthoformate with phloroisovalerophenone in the presence of aluminium chloride in methylene chloride at 0° for 30 min (70 %) [421, 3415].

m.p. 74° [3415]; ¹H NMR [3415], IR [3415], MS [3415].

## 2,4,6-Trihydroxy-3-(3-methyl-1-oxobutyl)benzaldehyde

[78423-49-1]	$C_{12}H_{14}O_5$	mol. wt. 238.24
ОН	Syntheses	
COCH ₂ CH(CH ₃ ) ₂	-Obtained by reaction of	ethyl orthoformate with
	2,4,6-trihydroxyisobutyro	phenone in the presence
но Он	of aluminium chloride in	methylene chloride with
СНО	cooling in an ice bath for	30 min (70 %) [421].

-Also obtained by adding DMF and phosphoryl chloride (Vilsmeier-Haack reagent) to a solution of phloroglucinol in ethyl acetate. The reaction mixture was further stirred for 30 min at r.t. [334], (70 %) [335, 337, 621].

-Also obtained by reaction of zinc cyanide with 2,4,6-trihydroxyisovalerophenone (Houben-Hoesch reaction) [2534].

-Also refer to: [336, 3033, 3405, 3408].

m.p. 137–139° [2534], 74° [421]; **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [421, 2534], IR [421], MS [421, 2534].

mol. wt. 265.27

BIOLOGICAL ACTIVITY: Effects on transpiration and stomatal closure [3408]; Germination inhibition [421]; Inhibitory effects of treatment with this acylphenol on TPA induced EBV-EA activation [3033]; As photosynthetic electron transport (PET) [3405].

## 2,4,6-Trihydroxy-3-methyl-(d₃)-5-(3-methyl-1-oxobutyl)benzaldehyde

Synthesis

(Deuterated grandinol)

$$C_{13}H_{13}D_3O_5$$
 mol. wt. 255.24

 $COCH_2CH(CH_3)_2$ CD₃ HO CHO

-Obtained by heating mixture of a 3-formylphloro-isovalerophenone, potassium hydroxide,  $d_3$ -iodomethane,  $d_4$ -methanol and deuterium oxide at  $70^{\circ}$  for 4 h (28 %) [3415]. m.p. 125–130° [3415];

¹H NMR [3415], UV [3415], IR [3415], MS [3415].

## 2,4,6-Trihydroxy-5-(3-methyl-1-oxobutyl)-1,3-benzenedicarboxaldehyde (Jensenone)

[96573-43-2]

C13H14O6 mol. wt. 266.25

CHO HO ĊHO

Syntheses  $COCH_2CH(CH_3)_2$  -Obtained by reaction of hexamethylenetetramine with 2,4,6-trihydroxyisovalerophenone in the presence of trifluoroacetic acid at  $70^{\circ}$ (40 %) [335].

-Also obtained by reaction of 3-methyl-1-(2,4,6-trihydroxy-3-methylphenyl)-1-butanone with dichloromethyl methyl ether in the presence of titanium tetrachloride in methylene chloride at  $-20^{\circ}$  under argon. Then the mixture was allowed to stand at  $-20^{\circ}$  for 2 h (41.6 %) [642].

Isolation from natural sources

-From fresh leaves of Eucalyptus grandis (Myrtaceae) [337, 1754].

-From Eucalyptus jensenii Maiden (Myrtaceae) [337, (approximatively 70 % of the oil) 419, 1106, 1453, 2037].

-From the leaf essential oil of Eucalyptus apodophylla (Myrtaceae) [2046]. -Also refer to: [1107, 3033].

BIOLOGICAL ACTIVITY: Antiprotozoal and antimicrobial [337]; Antifeedant [337]; A marsupial antifeedant [2037]; Inhibitory effects of treatment with this acylphenol on TPA induced EBV-EA activation [3033].

white solid [419]; pink crystals [642];

m.p. 198–200° [421], 113–114° [642], 102° [419];

**N.B.**: One of the reported melting point is obviously wrong.

¹H NMR [419, 421, 642], ¹³C NMR [419], IR [419, 421, 642], UV [642], MS [419, 421, 642, 2046]; GC-FID [2037]; GC-MS [419, 2037, 2046]; LC-UV [2037]; LC-MS [2037].

**Triacetate** [143183-56-6] C₁₉H₂₀O₉ mol. wt. 392.36

-Obtained by reaction of acetic anhydride with Jensenone in the presence of pyridine at r.t. for 48 h [419].

¹H NMR [419], ¹³C NMR [419], IR [419], MS [419].

## **2,4,6-Trihydroxy-3-methyl-5-(3-methyl-1-oxobutyl)benzaldehyde** (*Grandinol*)

Syntheses

[63861-11-0]

C₁₃H₁₆O₅ mol. wt. 252.27

$$\begin{array}{c} OH \\ CH_3 \\ HO \\ CHO \\ \end{array} \begin{array}{c} OH \\ OH \\ CHO \\ \end{array} \begin{array}{c} OH \\ OH \\ CHO \\ \end{array}$$

-Obtained by reaction of methyl iodide with 3-formyl-phloroisovalerophenone in the presence of potassium hydroxide in methanol [334, 335, 421, 3415].

-Also obtained by reaction of triethyl orthoformate with 3-isovaleroyl-2,4,6-trihydroxytoluene [3176] according to [641].

-Also obtained by formylation of 3-methyl-1-(2,4,6-trihydroxy-3-methylphenyl)-1-butanone using dichloromethyl methyl ether reagent and titanium tetrachloride as catalyst in methylene chloride at 0° for 1 h (25%) [3437].

-Also obtained by reaction of 3-methyl-1-(2,4,6-trihydroxy-3-methylphenyl)-1-butanone with dichloromethyl methyl ether in the presence of titanium tetrachloride in methylene chloride at  $-20^{\circ}$  under argon. Then the mixture was allowed at  $-20^{\circ}$  for 2 h (41.6 %) [642].

Isolation from natural sources

-From fresh leaves of *Eucalyptus grandis* (Myrtaceae) [337, 419, 421, 769, 1346, 1754, 3405, 3406, 3413–3415, 3437].

-From Eucalyptus grandis W. Hill ex Maiden [1106, 1107].

-From fresh leaves and stems of Eucalyptus pulverulenta [419-421] Sims [1106].

-From the leaves of Eucalyptus perriniana [2235] F. Muell. ex Rodway [1106].

-From Eucalyptus jensenii Maiden (Myrtaceae) [419].

-From *Eucalyptus* [642].

-Also refer to: [1753, 3033].

colourless crystals; yellowish powder [2235]; pink crystals [642];

m.p. 131–133° [420], 130–132° [421, 769, 3413, 3437], 130° [2235], 113–114° [642];

¹H NMR [420, 421, 642, 769, 2235, 3413], IR [421, 642, 769, 2235, 3413],

UV [642, 769, 2235, 3413], MS [420, 421, 642, 769, 2235, 3413]; X-ray analysis [421]; GC-MS [420]; TLC [2235]; HPLC [3413].

BIOLOGICAL ACTIVITY: Antiprotozoal and antimicrobial [337]; G-inhibitor (plant growth regulator) [419, 1107, 3413]; A rooting inhibitor in adult tissues of *Eucalyptus grandis* [769, 3415]; A potent germination inhibitor was found in *Eucalyptus pulverulenta* [420]; Antibacterial [2235]; Effects on transpiration and stomatal closure [3408]; Germination inhibition [421, 3414]; Inhibitory effects of treatment with this acylphenol on TPA induced EBV-EA activation [3033]; Strongly inhibit photosynthesis as well as germination of cress seeds [1346]; As photosynthetic electron transport (PET) [3405]; PET inhibitory activity [3414].

## 2,4,6-Trihydroxy-3-(hydroxymethyl)-5-(3-methyl-1-oxobutyl)benzaldehyde

 $\begin{array}{c} C_{13}H_{16}O_6 & \text{mol. wt. } 268.27 \\ O & OH & Synthesis \\ H & C & COCH_2CH(CH_3)_2 & \text{-Refer to: [1106].} \\ H & OH & OH \\ CH_2OH & \end{array}$ 

#### 2,4-Dihydroxy-6-methoxy-5-methyl-3-(3-methyl-1-oxobutyl)benzaldehyde

[63861-20-1]	$C_{14}H_{18}O_5$ mol. wt. 266.29
CHO CHO CHO CHO CHO COCH ₂ CH(CH ₃ ) ₂	Synthesis -Obtained by treatment of Grandinol with diazomethane [769].
CH ₃ O CH ₃ OH	m.p. 89–89.5° [769]; ¹ H NMR [769], ¹³ C NMR [769], UV [769], MS [769]; X-ray data [769].

BIOLOGICAL ACTIVITY: Germination inhibition [421].

### 2,6-Dihydroxy-4-methoxy-5-methyl-3-(3-methyl-1-oxobutyl)benzaldehyde

[63861-21-2]	$C_{14}H_{18}O_5$	mol. wt. 266.29
$\begin{array}{c} OH \\ CHO \\ HO \\ CH_3 \\ $	Synthesis -Obtained by treatment of diazomethane [769]. m.p. 78–78.5° [769]; ¹ H NMR [769], UV [769], MS	

BIOLOGICAL ACTIVITY: Germination inhibition [421].

#### 3-Ethyl-2,4,6-trihydroxy-5-(3-methyl-1-oxobutyl)benzaldehyde

mol. wt. 266.29 [96573-34-1] C14H18O5 Syntheses OH COCH₂CH(CH₃)₂ -Obtained by reaction of ethyl iodide with CHO. 2,4,6-tri-hydroxy-3-(3-methyl-1-oxobutyl)benz-HO OH aldehyde in the presence of potassium hydroxide CH₂CH₃ in aqueous methanol at  $65^{\circ}$  for 24 h (30 %) [421].

-Also refer to: [3033].

m.p. 126–126.5° [421]; ¹H NMR [421], IR [421], MS [421].

BIOLOGICAL ACTIVITY: Germination inhibition [421]; Inhibitory effects of treatment with this acylphenol on TPA induced EBV-EA activation [3033].

### 2-Hydroxy-4,6-dimethoxy-3-(3-methyl-1-oxobutyl)benzaldehyde

[179630-54-7]

C14H18O5

Syntheses

mol. wt. 266.29

$$\begin{array}{c} H \\ O = C \\ CH_{3}O \end{array} \begin{array}{c} OH \\ COCH_{2}CH(CH_{3})_{2} \\ OCH_{3} \end{array}$$

-Obtained by reaction of dichloromethyl methyl

ether with 2-hydroxy-4,6-dimethoxyisovalerophenone in the presence of titanium tetrachloride in methylene chloride at  $-10^{\circ}$ . Then, the reaction mixture was stored at r.t. for 1 h (98 %) [643].

-Obtained by reaction of isovaleryl chloride with 2,4-dimethoxy-6-hydroxybenzaldehyde in the presence of aluminium chloride in methylene chloride for 20 h at r.t. (80 %) [2576].

-Also refer to: [270].

colourless solid [2576]; m. p. 91° [2576], 62° [643]; **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [643, 2576], ¹³C NMR [643, 2576], IR [643, 2576], UV [643], MS [643]; X-ray data [270]; TLC [2576].

#### Tert-Butyldimethylsilyl [233751-78-5] C₂₀H₃₂O₅Si mol. wt. 380.56 derivative

-Obtained by reaction of TBSCl (tert-butyldimethylsilyl chloride) with the title ketone in the presence of ethyldimethylamine and DMAP (4-dimethylaminopyridine) in methylene chloride for 20 h at r.t. (76 %) [2576].

colourless solid [2576]; m. p.105.8° [2576]; ¹H NMR [2576], ¹³C NMR [2576], IR [2576]; TLC [2576].

## 2,4-Dihydroxy-3-(3-methyl-1-oxobutyl)-6-(2-propen-1-yloxy)benzaldehyde

[918814-59-2]  $C_{15}H_{18}O_5$  mol. wt. 278.30 OH CHO CHO COCH₂CH(CH₃)₂ Obtained from 1-(2,6-dihydroxy-4-allyloxy)-isovalerophenone further upon Vilsmeier-Haack formylation (40 %) [337].

light violet solid [337]; m.p. 96–98° [337]; ¹H NMR [337], ¹³C NMR [337], IR [337], UV [337], MS [337].

BIOLOGICAL ACTIVITY: In vitro antileishmanial [337]; Cytotoxicity [337].

## 2,4,6-Trihydroxy-3-(3-methyl-1-oxobutyl)-5-propylbenzaldehyde

[96573-35-2]	$C_{15}H_{20}O_5$	mol. wt. 280.32
CHO HO CHO HO CH2CH(CH ₃ ) ₂ OH CH ₂ CH ₂ CH(CH ₃ ) ₂	Syntheses -Obtained by reaction of with 2,4,6-trihydroxy-3-(3-me benzaldehyde in the present hydroxide in aqueous methano (30 %) [421].	ethyl-1-oxobutyl) e of potassium

-Also refer to: [3033].

m.p. 124–125° [421]; ¹H NMR [421], IR [421], MS [421].

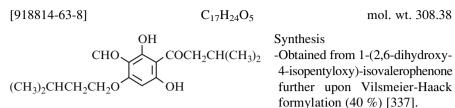
BIOLOGICAL ACTIVITY: Inhibitory effects of treatment with this acylphenol on TPA induced EBV-EA activation [3033].

## 3-Butyl-2,4,6-trihydroxy-5-(3-methyl-1-oxobutyl)benzaldehyde

[96573-36-3]	$C_{16}H_{22}O_5$	mol. wt. 294.35
$\begin{array}{c} OH \\ CHO \\ HO \\ HO \\ (CH_2)_3CH_3 \end{array} OH$	Synthesis -Obtained by reaction of 2,4,6-tri-hydroxy-3-(3-mo benzaldehyde in the prese hydroxide in aqueous metha (30 %) [421].	ethyl-1-oxobutyl) ence of potassium

m.p. 101–102° [421]; ¹H NMR [421], IR [421], MS [421].

#### 2,4-Dihydroxy-6-(3-methylbutoxy)-3-(3-methyl-1-oxobutyl)-1-butanone



off white solid [337]; m.p. 85–87° [337]; ¹H NMR [337], ¹³C NMR [337], IR [337], UV [337], MS [337].

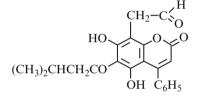
BIOLOGICAL ACTIVITY: In vitro antileishmanial [337]; Cytotoxicity [337].

## [5,7-Dihydroxy-6-(3-methylbutyryl)-2-oxo-4-phenyl-2*H*-chromen-8-yl] acetaldehyde

[37972-56-8]



mol. wt. 380.40

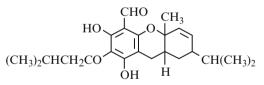


Synthesis -Formation by simple fission of the double bond of 5,7-dihydroxy-8-(3-methyl-2-butenyl)-6-(3-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one obtained by ozonization in acetic acid at 15° (64 %) [1007].

pale yellow needles [1007]; m.p. 175–183° [1007]; IR [1007], UV [1007].

### **Euglobal-IIc**

[77794-62-8]



mol. wt. 386.49

Isolation from natural sources -From the flower buds of *Eucalyptus globulus* LABILL [1753, 3033].

colourless oil [1754];  $(\alpha)_D^{20} = -137^{\circ}$  (chloroform) [1754];  $(\alpha)_D^{20} = -144^{\circ}$  (chloroform) [1753]; ¹H NMR [1753, 1754], ¹³C NMR [1753], IR [1753, 1754], UV [1753, 1754], MS [1754]; circular dichroism [1753].

 $C_{23}H_{30}O_5$ 

Refer to: [1754 (5), 3033 (7)].

BIOLOGICAL ACTIVITY: Inhibitory effects of treatment with this acylphenol on TPA induced EBV-EA activation [3033]; Active in the fertile egg test [1753].

## 1,1'-Methylenebis[2,4,6-trihydroxy-3-(1-oxomethyl)-5,1-phenylene]bis-3-methyl-1-butanone

Methylenebis(3-formyl-5-isopentanoyl)-2,4,6-trihydroxybenzene (*Robustaol A*)

$$\begin{array}{cccc} C_{25}H_{28}O_{10} & \text{mol. wt. 488.49} \\ & & & \text{CHO} & \text{CHO} & \text{Syntheses} \\ HO & OH HO & OH & -\text{Refer to: [621} \\ (CH_3)_2CHCH_2CO & OH & OH \end{array}$$

Isolation from natural sources

-From the leaves of *Eucalyptus robusta*, a plant used in Chinese herbal medicine [1819].

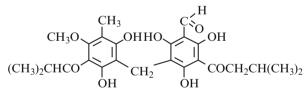
cream coloured solid [621]; m.p. 228–230° [621]; ¹H NMR [621], ¹³C NMR [621], IR [621], UV [621], MS [621].

BIOLOGICAL ACTIVITY: Antimalarial [1819, 2534].

-Also refer to: [621].

## **5-[2,6-Dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl)phenyl] methyl-3-(3-methyl-1-oxobutyl)-2,4,6-trihydroxybenzaldehyde** (*Robustaol A*)

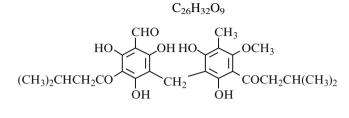
 $C_{25}H_{30}O_9$  mol. wt. 474.50



Isolation from natural sources -From the leaves of *Eucalyptus robusta* Sm. (Myrtaceae) [2534].

m.p. 163–164° [2534]; ¹H NMR [2534], IR [2534], UV [2534], MS [2534]. **N.B.**: Identification from its alkaline degradation products [2534].

## 2,4,6-Trihydroxy-5-(3-methyl-1-oxobutyl)-3-[2,6-dihydroxy-4-methoxy-3-(3-methyl-1-oxobutyl)-5-methylphenyl]methylbenzaldehyde (*Robustaol A*)



mol. wt. 488.53

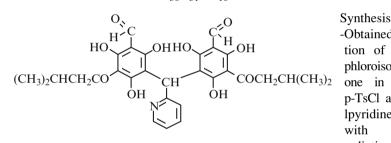
Isolation from natural sources -From the leaves of *Eucalyptus robusta* Smith (Myrtaceae) [1106].

BIOLOGICAL ACTIVITY: Antimalarial [1106].

## 1-[3-Formyl-5-[(5-formyl-3-isopentanoyl-2,4,6-trihydroxyphenyl)pyridin-2-yl-methyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone

C₃₀H₃₁NO₁₀

mol. wt. 565.58



-Obtained by irradiation of a 3-formyl phloroiso-valerophenone in chloroform, p-TsCl and 2-formylpyridine mixture with microwave radiations (750 W) in domestic microwave oven for 10– 15 min (44 %) [621].

yellow solid [621]; m.p. 148–149° [621]; ¹H NMR [621], ¹³C NMR [621], IR [621], UV [621], MS [621].

BIOLOGICAL ACTIVITY: Cytotoxicity [621].

## 1-[3-Formyl-5-[(5-formyl-3-isopentanoyl-2,4,6-trihydroxyphenyl)-4-benzyloxyphenyl-methyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone

 $C_{38}H_{38}O_{11}$   $O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O_{11}O$ 

Syntheses -Obtained by irradiation of a 3-formyl phloroiso-valerophenone in chloroform, p-TsCl and 4-benzyloxybenzaldehyde mixture

mol. wt. 670.71

with microwave radiations (750 W), in domestic microwave oven for 10–15 min (40 %) [621].

-Also refer to: [620 (33 %)].

cream colour solid [620, 621]; m.p. 195–197° [620], 180° [621]; ¹H NMR [620, 621], ¹³C NMR [620, 621], IR [621], UV [621], MS [620, 621].

BIOLOGICAL ACTIVITY: Cytotoxicity [621].

## 15 Aromatic Hydroxy-4-Oxo-1-Butanoic Acids

## 15.1 Unsubstituted Hydroxyketones

## 4-(4-Hydroxyphenyl)-2,3-dibromo-4-oxo-1-butanoic acid

	$C_{10}H_8Br_2O_4$	mol. wt. 351.98
ОН	Synthesis	
$\checkmark$	-Refer to: [2593].	
	Ethyl ether	
$\mathbf{Y}$	$C_{12}H_{12}Br_2O_4$	mol. wt. 380.03
COCHBrCHB	rCO ₂ H	

-Obtained by treatment of p-ethoxybenzoylacrylic acid (m.p.  $141-142^{\circ}$ ) with bromine in acetic acid [2593].

m.p. 151° (d) [2593].

Methyl ether	[103862-60-8]	$C_{11}H_{10}Br_2O_4$	mol. wt. 366.01
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-Refer to: [1095, 2592, 2753, 2794].

m.p. 164° (higher-melting form) [2592, 2794], 140–141° [2794], 140° (lower-melting form) [2592].

BIOLOGICAL ACTIVITY: Antifungal [1095].

-Also refer to: [2753].

### Methyl ester of the methyl ether

m.p. 115–116° (erythro) [255, 2795], 115° [2592], 68–69° (threo) [255], 57–58.5° [2795].

### 4-(2,4-Dihydroxyphenyl)-2,4-dioxo-1-butanoic acid

 $C_{10}H_8O_6$ OH Synthesis COCH₂COCO₂H -Refer to: [2456].

Ethyl ester of the dimethyl ether [80081-75-0] C₁₄H₁₆O₆ mol. wt. 280.28 -Obtained by reaction of resacetophenone dimethyl ether with ethyl oxalate in the presence of sodium [2456].

yellow crystals [2456]; m.p. 86–87° [2456].

## Methyl ester of the dimethyl ether [39757-32-9] C₁₃H₁₄O₆ mol. wt. 266.25

m.p. 100–101° [117].

Methyl ester of the dibenzyl ether	$C_{25}H_{22}O_{6}$	mol. wt. 418.45
m.p. 160–164° [167].		
Ethyl ester of the diethyl ether	$C_{16}H_{20}O_{6}$	mol. wt. 308.33
m.p. 152° [1742].		

mol. wt. 224.17

Ethyl ester of the dibenzyl ether [170283-09-7]  $C_{26}H_{24}O_6$  mol. wt. 432.47

## 4-(2,4,5-Trihydroxyphenyl)-2,4-dioxo-1-butanoic acid

$$\begin{array}{ccc} C_{10}H_8O_7 & \mbox{mol. wt. } 240.17 \\ OH & Synthesis \\ +OCCH_2COCO_2H & -Refer to: [2196]. \\ Triethyl ether & [63213-34-3] \\ C_{16}H_{20}O_7 & \mbox{mol. wt. } 324.33 \\ 2,4,5-Triethoxy-\alpha,\gamma-dioxobenzenebutanoic acid \end{array}$$

-Preparation: An oil dispersion of sodium hydride was added to a mixture of 2,4,5-triethoxy-acetophenone, diethyl oxalate and benzene. The mixture was stirred at  $65-70^{\circ}$  for 1 h. The ethyl ester obtained (90 %, m.p. 153–155°) was dissolved in sulfuric acid at r.t. for 2 h (65 %) [2196].

m.p. 178-181° [2196].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

Ethyl ester of the triethyl ether [63213-44-5]  $C_{18}H_{24}O_7$  mol. wt. 352.38

-Refer to: [2196].

[39560-34-4]

m.p. 153-155° [2196].

## 4-(2-Hydroxyphenyl)-4-oxo-1-butanoic acid

γ-o-hydroxyphenyl-γ-ketobutyric acid

 $C_{10}H_{10}O_4$  mol. wt. 194.19



Syntheses

-Preparation by reaction of succinic anhydride with phenol in the presence of aluminium chloride in tetrachloroethane at  $130-140^{\circ}$  for 2 h (62 %) [2100], (55 %) [197], (30-35 %) [2571], (26-28 %) [222], (20 %) [990].

-Also obtained by Fries rearrangement of phenyl hydrogen succinate with aluminium chloride at  $117^{\circ}$  for 2.5 h in tetrachloroethane (58 %) or in chlorobenzene (41 %) [1987].

-Also obtained from 4-(2-hydroxyphenyl)-4-oxobutyronitrile [2649]. -Also refer to: [222 (54 %), 676, 997 (2 %), 3291 (0.4 %)].

colourless crystals [2100]; long colourless needles [990]; m.p. 146° [197, 2097, 2571], 145–146° [222], 145° [2100, 2649], 139–140° [222, 990, 3291], 127–130° [997]; ¹H NMR [676, 997, 3291], IR [676, 997, 3291]. **Methyl ether** [103987-16-2] C₁₁H₁₂O₄ mol. wt. 208.21

-Obtained by reaction of methyl iodide with o-benzoylpropionic acid in the presence of potassium carbonate in refluxing acetone for 8 h. Then, hydrolysis of the methyl  $\beta$ -o-anisoylpropionate obtained [222].

-Also obtained by methylation of the title ketone [2571].

-Also obtained by Robinson's method [2571].

-Also refer to: [391, 805 (86 %), 1032, 1131, 3139].

m.p. 100–101° [222], 98° [3139], 97–98° [2571], 95.6–96.6° [805], 86–88° [1032]; ¹H NMR [1032], IR [391].

BIOLOGICAL ACTIVITY: Human zinc insulin; Delivery of [1131].

Methyl ester of the methyl ether [99046-13-6]  $C_{12}H_{14}O_4$  mol. wt. 222.24

-Obtained from o-bromoanisole and  $\beta$ -carbomethoxypropionyl chloride (Grignard reaction) (43 %) [805].

b.p._{1,5} 161–162° [805], 160° [3139].

**Methyl ester** [56871-93-3] C₁₁H₁₂O₄ mol. wt. 208.21

-Refer to: [915, 997, 3122].

b.p._{0.4} 135° [3122]; m.p. 33–34° [997]; ¹H NMR [915, 997], ¹³C NMR [915], IR [997], MS [915].

**Ethyl ester** [39496-84-9] C₁₂H₁₄O₄ mol. wt. 222.24

-Refer to: [216, 2571].

colourless liquid [2571]; b.p._{0.1} 112–113° [216], b.p.₁₈₄ 255° [2571].

#### 4-(3-Hydroxyphenyl)-4-oxo-1-butanoic acid

m.p. 146.5° [1706], 146-147° [665], 144-145° [1993].

## 4-(4-Hydroxyphenyl)-4-oxo-1-butanoic acid

γ-p-hydroxyphenyl-γ-ketobutyric acid

[56872-39-0]

$$C_{10}H_{10}O_4$$

mol. wt. 194.19

OH COCH₂CH₂CO₂H

Syntheses

-Preparation by reaction of succinic anhydride with phenol in the presence of aluminium chloride in tetrachloroethane at  $130-140^{\circ}$  for 2 h [222], (6 %) [197], (2–3 %) [2571].

 -Also obtained by treatment of its methyl ether with hydrogen bromide in refluxing acetic acid (86 %) [2652].

-Also refer to: [43, 496, 990 (3 %), 1208, 1341, 1482, 1561, 1706, 1779, 1939, 2100, 2438, 3007].

lustrous needles [222]; slightly yellow [990]; m.p. 160–161° [222], 159–160° [3007], 159° [2652], 157° [1706], 156–159° [1561], 156° [2571], 155–156° [1341], 154–156° [990]; ¹H NMR [1208, 1341], ¹³C NMR [1341].

BIOLOGICAL ACTIVITY: Feeble choleretic [496].

**Methyl ether** [3153-44-4] C₁₁H₁₂O₄ mol. wt. 208.21

-Preparation by reaction of succinic anhydride with anisole in the presence of aluminium chloride [257, 258, (90 %) 2392, 3401],

- *in tetrachloroethane [858] first at  $0^{\circ}$ , then at r.t. overnight (83 %) [2649], (80 %) [2100];
- *in tetrachloroethane and nitrobenzene mixture (85 %) [987], at r.t. for 2.5–3 h (75 %) [2579];
- *in nitrobenzene: [858, 1281], first at  $0^{\circ}$ , then at r.t. overnight (72 %) [606] or at  $60^{\circ}$  for 3 h (77 %) [1745];
- *in benzene at 60° for 30 min (77 %) [1302];
- *in carbon disulfide [257, 891, 1209];
- *in methylene chloride with ice-ethanol bath cooling and stirring for 3 h (55 %) [878].

-Also obtained by basic hydrolysis of its methyl ester [1093].

-Also refer to: [71, 72, 99, 262, 437, 806, 837, 945, 977, 980, 991, 1061, 1131, 1169, 1410, 1503, 1520, 1529, 1635, 1664, 1667, 1668, 1779, 1854, 1953, 1955, 2015, 2128, 2129, 2358, 2359, 2383, 2427, 2529, 2546, 2733 (80 %), 2817, 2831]

large hexagonal prisms [1209]; white flaky solid [2546]; b.p. 154° [2579]; m.p. 148–151° [1302], 148–150° [2579], 147.5–148.5° [606], 147–149° [2733], 147–148° [1281, 1766, 2392], 147° [1209], 146–148° [878], 146–147° [987, 2358, 2359, 2546], 146° [2099, 2100, 2649], 145–146° [3401], 144.5–146.5° [1529], 144–146° [1093], 144–145° [257, 258], 142–143° [1745]; ¹H NMR [606, 891, 1061, 1131, 1410, 1520, 1635, 1668, 1745, 1953, 2427, 2546, 2831],
¹³C NMR [1520, 1635, 1745],
IR [837, 1520, 1635, 1745, 1953, 2427, 2546, 2817], UV [806, 1169],
MS [606, 1520, 1635, 1745, 2546].

BIOLOGICAL ACTIVITY: Feeble choleretic [496]; Human zinc insulin, delivery of [1131]; Inhibition of aggregation of thrombocytes [1779]; Antinociceptive [891].

Methyl ester of the methyl ether [5447-74-5]  $C_{12}H_{14}O_4$  mol. wt. 222.24

-Obtained by bubbling hydrogen chloride in 1-(4-methoxyphenyl)-4-oxo-1-butanoic acid in methanol for 1.5 h [257, 258].

-Also obtained by reaction between anisole and 3-carbomethoxypropanoic acid in the presence of PPA for 2.5 h at  $45^{\circ}$  (75 %) [1093].

-Also obtained by treatment of the methyl ether with diazomethane in ethyl ether [339, 806, 1169, 1189, 1512, 1635, 1745 (99 %), 2668].

b.p._{0.6} 147–152° [1274], b.p.₁ 160–161° [1189]; m.p. 48–49° [1093], 47–48.5° [1189], 46–47° [257, 258, 1169, 1512], 46° [1745]; ¹H NMR [339, 1512, 1635, 1745, 2668], ¹³C NMR [1635, 1745, 2668], IR [339, 1189, 1512, 1635, 1745, 2668], UV [806, 1169], MS [1512, 1635, 1745, 2668].

Ethyl ester of the methyl ether [15118-67-9]  $C_{13}H_{16}O_4$  mol. wt. 236.27

-Obtained by reaction of ethyl succinyl chloride with anisole in the presence of aluminium chloride in methylene chloride first at  $0^{\circ}$ , then at r.t. for 15 h (67 %) [1436].

-Also refer to: [312, 1055, 1209, 1485, 1840, 1954, 1955, 2592, 2644, 2689 (26 %), 2733 (87 %), 3139].

b.p._{0.04} 160° [312]; m.p. 57° [3139], 55–57° [2733], 52° [1209], 51–53° [2689], 49–50° [1436], 33–35° [1955]; ¹H NMR [1055, 1436, 1485, 1840, 1954, 1955], ¹³C NMR [1485, 1840], IR [1055, 1436, 1485, 1840, 1954].

**Ethyl ether** [53623-37-3]  $C_{12}H_{14}O_4$  mol. wt. 222.24

-Obtained by Friedel-Crafts reaction of succinic anhydride and phenetole (82 %) [2579], (59 %) [2593].

-Also refer to: [1075, 2392 (88 %), 3140].

b.p. 172° [2579]; m.p. 140–142° [1667], 138–139° [1075, 2392, 2593], 137–139° [2579], 137–138° [3140], 136–137° [2794]; MS [1567, 3038]. Methyl ester of the ethyl ether [319494-44-5]  $C_{13}H_{16}O_4$  mol. wt. 236.27 m.p. 53° [2593]; ¹H NMR [3038], MS [1567, 3038].

**Ethyl ester of the ethyl ether**  $C_{14}H_{18}O_4$  mol. wt. 250.29

m.p. 52° [3140], 51° [2593].

**Ethyl ester** [66123-43-1]  $C_{12}H_{14}O_4$  mol. wt. 222.24

-Obtained by treatment of ethyl 4-(4-methoxyphenyl)-4-oxobutanoate,

*with boron tribromide in methylene chloride, first at  $0^{\circ}$ , then at r.t. for 15 h (62 %) [1436];

*with 48 % HBr in refluxing acetic acid for 20 h (33 %) [878]. -Refer to: [1399, 1482, 2571].

m.p. 111–112° [1436], 111° [2571], 108–110° [878]; ¹H NMR [1436], IR [1436], MS [1399].

**Butyl ether** [63471-88-5] C₁₄H₁₈O₄ mol. wt. 250.29

-Obtained by Friedel-Crafts reaction of succinic anhydride and n-butyl phenyl ether (72 %) [2579].

b.p. 210° [2579]; m.p. 112° [3140], 110–112° [2579].

**Phenyl ether** [36330-86-6] C₁₆H₁₄O₄ mol. wt. 270.28

-Obtained by Friedel-Crafts reaction of succinic anhydride and phenyl ether in the presence of aluminium chloride (94 %) [2579], in carbon disulfide (93 %) [1692].

b.p. 252° [2579]; m.p. 185° [1667], 172° [286, 1115], 119–120° [1396, 1646, 2579], 119° [2818], 118–119° [2308, 2491, 2594], 117–119° [648, 649], 117° [1692]; ¹H NMR [286, 1115, 1352], ¹³C NMR [1352].

BIOLOGICAL ACTIVITY: Antiinflammatory [649]; Hypolipidemic [1646].

m.p. 203° [2308].

## 4-(2,4-Dihydroxyphenyl)-4-oxo-1-butanoic acid

[13335-54-1]	$C_{10}H_{10}O_5$		mol. wt. 210.19
reaction) [1669]. -Also obtained by condensa	resorcinol in [651, 1669]. -Also obtained with hydroge (47 %) [2652 n of succino	the presence of l by treatment on n bromide in r ]. nitrile with	
reaction) [2187]. -Also refer to: [351, 445, 796, 3	815 856 999	1790 2186 324	5 34851
<ul> <li>m.p. 206–207° [3245], 205°</li> <li>199–200° [856, 1790, 2186,</li> <li>¹H NMR [999, 3344], IR [99</li> </ul>	[351, 796], 203 2187], 197° [4	3° [794, 2652], 1	201.5–203° [3485],
Na salt $C_{10}$	$H_9O_5Na, 3 H_2C$	)	mol. wt. 263.22
m.p. 199–200° [2187].			
Ba, Ca, Pb and Ag salts insolu	ble in water [79	94].	
<b>4-Nitrophenylhydrazone</b> m.p. 194° [856].	C ₁₆ H ₁	₅ N ₃ O ₆	mol. wt. 345.31
<b>Dibenzoate</b> m.p. 146–147° [2187].	$C_{24}H_{18}O_7$		mol. wt. 418.40
m.p. 140–147 [2187].			
<b>Dimethyl ether</b> [1461]	7-06-2]	$C_{12}H_{14}O_5$	mol. wt. 238.24
<ul> <li>-Preparation by a Friedel-Crafts reaction with succinic anhydride and resorcinol dimethyl ether [257, 258, 815, (49 %) 2579].</li> <li>-Preparation by a Friedel-Crafts reaction with succinic anhydride and resorcinol dimethyl ether in the presence of aluminium chloride in nitrobenzene (60 %) [2652].</li> <li>-Obtained by reaction of succinic anhydride with resorcinol dimethyl ether in the presence of aluminium chloride,</li> <li>*in tetrachloroethane at 50–60° for 3 h, then at r.t. overnight (79 %) [2100];</li> <li>*in refluxing carbon disulfide for 3 h [2456].</li> <li>-Also obtained by treatment of 4-(2,4-dihydroxyphenyl)-4-oxo-1-butanoic acid with dimethyl sulfate [2187].</li> </ul>			

-Also obtained by refluxing 2,4-dimethoxyphenyl- $\gamma$ -ketobutyric acid ethyl ester (C₁₄H₁₈O₅ mol. wt. 266.29; b.p.₅ 198–200°, m.p. 66°) with 10 % caustic potash for 3 h (92 %) [2100]. -Also refer to: [223, 258, 262, 794, 795, 1149, 1246, 1293, 1790, 2100, 2186, 2187,

-Also refer to: [223, 258, 262, 794, 795, 1149, 1246, 1293, 1790, 2100, 2186, 2187, 2409, 2427, 2458, 2579, 2652, 3485].

b.p. 214° [2579]; m.p. 150–151.5° [2579], 150° [2652], 149.5° [3485], 148° [794, 795, 2186, 2187], 147° [1790, 2100], 146–148° [2456, 2458], 146° [257, 258], 124–125° [2187], 106–107° [223]. ¹H NMR [2427], IR [2427].

**Oxime of the dimethyl ether**  $C_{12}H_{15}NO_5$  mol. wt. 253.25

m.p. 155–156° [2187].

Methyl ester of the dimethyl ether [14563-41-8]  $C_{13}H_{16}O_5$  mol. wt. 252.27

-Obtained from dimethyl ether in refluxing methanol for 2 h in the presence of 20 % sulfuric acid [2456].

m.p. 58° [2456], 57.5–58.5° [3485], 49° [2595].

Ethyl ester of the dimethyl ether [858445-94-0] C₁₄H₁₈O₅ mol. wt. 266.29

-Preparation by reaction of ClCH₂CH₂CO₂C₂H₅ with resorcinol dimethyl ether in the presence of aluminium chloride [2456]. -Also refer to: [2499].

b.p.₅ 198–200° [2100]; 70° [795], 68.5° [2595], 66° [2100], 44° [2595].

**Diethyl ether** [39560-29-7] C₁₄H₁₈O₅ mol. wt. 266.29

2,4-Diethoxy-y-oxo-benzenebutanoic acid

-Obtained by condensation of resorcinol diethyl ether with succinic anhydride (65 %) [2579], (40 %) [1451]. -Also refer to: [2196].

b.p. 235° [2579]; m.p. 148.5–150.5° [2579], 146–147° [1451].

BIOLOGICAL ACTIVITY: Choleretic action [2196].

## **Dipropyl ether** $C_{16}H_{22}O_5$ mol. wt. 294.35

-Obtained by condensation of resorcinol dipropyl ether with succinic anhydride [1451].

m.p. 111° [1451].

## **Dibutyl ether** $C_{18}H_{26}O_5$ mol. wt. 322.40

-Obtained by condensation of resorcinol dibutyl ether with succinic anhydride (69 %) [2579], (50 %) [1451].

b.p. 290° [2579]; m.p. 118–119° [2579], 117° [1451].

**Di-iso-amyl ether**  $C_{20}H_{30}O_5$  mol. wt. 350.46

-Obtained by condensation of resorcinol di-*iso*-amyl ether with succinic anhydride (85 %) [1451].

m.p. 111° [1451].

Methyl ester	[13335-55-2]	$C_{11}H_{12}O_5$	mol. wt. 224.21
m.p. 138° [794	], 130–131° [3485], 130° [3	3245], 129° [445];	¹ H NMR [3344].
Ethyl ester	[133535-21-4]	$C_{12}H_{14}O_5$	mol. wt. 238.24
b.p. ₃ 193–195°	[3370]; m.p. 100° [794];	IR [3370].	

4-(2,5-Dihydroxyphenyl)-4-oxo-1-butanoic acid

[59701-65-4]  $C_{10}H_{10}O_5$ mol. wt. 210.19 Syntheses OH -Obtained by treatment of its dimethyl ether, CO(CH₂)₂CO₂H *with hydrogen iodide (81 %) [2331]; *with hydrogen bromide (85 %) [2652]; OH *with pyridinium chloride for 40 min at 200° (63 %) [1349]. obtained by photo-Fries rearrangement of 1,4-dihydroxyphenyl -Also monosuccinate (55 %) [2004]. -Also refer to: [696 (25-30 %), 747, 997 (55 %), 1878, 1891].

yellow columns [2331]; dark orange crystals [1891];
m.p. 186.5° [747], 183.5–184.5° [997, 2004], 181–182° [1349, 2652], 180–182° [2331],
179–181.5° [1891], 177° [696, 2652], 159–162° [1878],
¹H NMR [997, 1878, 1891, 2004], ¹³C NMR [1878],
IR [997, 1349, 1891, 2004], MS [1878, 1891];
X-ray crystallographic analysis [1891].

## **Dimethyl ether** [1084-74-8] $C_{12}H_{14}O_5$ mol. wt. 238.24

-Preparation by reaction of succinic anhydride with hydroquinone dimethyl ether in the presence of aluminium chloride [257, 258, 622, (96 %) 696, (80 %) 2566, (58 %) 3387],

*in nitrobenzene first at  $0^{\circ}$ , then was allowed to warm from 5 to  $29^{\circ}$  over a 3.5 h period [(81 %) 2140, (79 %) 3320];

*in methylene chloride first at  $10^\circ$ , then 3 days at r.t. (67 %) [1454];

*in nitromethane first at  $0^{\circ}$ , then at  $25^{\circ}$  for 12 h (81 %) [3343];

*in dichloroethane first from 10 to  $57^{\circ}$ , then at  $57^{\circ}$  for 2.5 h (95 %) [304].

-Also obtained by treatment of its methyl ester with boiling dilute alcoholic potassium hydroxide for 2 h (90 %) [2661].

-Also refer to: [99, 157, 258, 459, 747, 768, 795, 990 (6.3 %), 1349, 1503, 1937 (59 %), 1953, 2062, 2331, 2462, 2708, 3289, 3485].

very pale yellow solid [3320];

m.p. 107° [258, 795], 103–105° [2062], 103–104° [157], 103° [747], 102.5–103.5° [1937], 102–104° [3485], 102–103° [2661], 102° [304, 1454], 101–102° [990, 2140, 2331], 101–101.5° [3387], 100–103° [2566], 99–100° [257, 258], 96–97° [768], 94–97° [3289], 86–88° [2708], ¹H NMR [768, 1454, 1953, 2566, 2708, 3320, 3387], IR [1454, 1953, 2566, 2708], MS [2708, 3387].

Methyl ester of the dimethyl ether [1086-77-7]  $C_{13}H_{16}O_5$  mol. wt. 252.27

-Preparation by treatment of 4-(2,5-dihydroxyphenyl)-4-oxo-1-butanoic acid with diazomethane (72 %) [1454].

-Also obtained by treatment of 2,5-dimethoxybenzoylpropionic acid with methanol in the presence of hydrogen chloride in refluxing benzene for 5 h [696].

-Also obtained by reaction of succinic acid monomethyl ester with hydroquinone dimethyl ether in the presence of stannic chloride in benzene for 17 h at r.t. (47.5 %) [2661].

-Also refer to: [1040].

b.p.₁ 150° [696], b.p.₁₄ 235° [696], b.p.₂₀ 223–224° [2661]; m.p. 55° [1454], 54° [795], 52–53° [3485], 49.5–50° [2661], 49–50° [696]; ¹H NMR [1454], IR [1454], MS [1454].

BIOLOGICAL ACTIVITY: Radioprotector [1040].

LD₅₀ [1040].

### **Ethyl ester of the dimethyl ether** $C_{14}H_{18}O_5$ mol. wt. 266.29

-Obtained by treatment of 2,5-dimethoxybenzoylpropionic acid with ethanol in the presence of hydrogen chloride in refluxing benzene for 5 h [696]. -Also refer to: [2499].

pale yellow oil; b.p.₁₃ 202° [696]; m.p. 46° [795].

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-Preparation by reaction of liquid isobutene with 4-(2,5-dihydroxyphenyl)-4-oxo-1-butanoic acid in the presence of sulfuric acid in methylene chloride at r.t. (62 %) [1454].

m.p. 70–72° [1454]; ¹H NMR [1454], IR [1454].

**Diethyl ether** [63213-46-7] 
$$C_{14}H_{18}O_5$$
 mol. wt. 266.29

2,5-Diethoxy-y-oxobenzenebutanoic acid

-Obtained by condensation of hydroquinone diethyl ether with succinic anhydride (62 %) [1451], (57 %) [2579]. -Also refer to: [747, 768, 989, 1451].

m.p. 151° [747], 148–150° [2579], 147.4–148.6° [989], 145° [1451]; ¹H NMR [768].

BIOLOGICAL ACTIVITY: Choleretic action [2196].

Dipropyl ether	$C_{16}H_{22}O_5$	mol. wt. 294.35
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-Obtained by condensation of hydroquinone dipropyl ether with succinic anhydride (68 %) [1451].

m.p. 95° [1451].

#### **Dibutyl ether**

C₁₈H₂₆O₅ mol. wt. 322.40

-Obtained by condensation of hydroquinone dibutyl ether with succinic anhydride (71 %) [1451].

m.p. 89° [1451].

- **2-Ethoxy-5-methoxy** [107327-72-0] (di-ether mix) C₁₃H₁₆O₅ mol. wt. 252.27 needles [747]; m.p. 137.5° [747].
- **5-Ethoxy-2-methoxy** [107327-65-1] (di-ether mix) C₁₃H₁₆O₅ mol. wt. 252.27 needles [747]; m.p. 105.5° [747].

Methyl ester [127275-13-2]

 $C_{11}H_{12}O_5$ 

mol. wt. 224.21

-Refer to: [997].

m.p. 129–130° [997]; ¹H NMR [997], IR [997].

## 4-(2,6-Dihydroxyphenyl)-4-oxo-1-butanoic acid

	$C_{10}H_{10}O_5$	mol. wt. 210.19
OH CO(CH ₂ ) ₂ COOH	Synthesis -Refer to: [3141]. m.p. 198–199° [3141].	

## 4-(3,4-Dihydroxyphenyl)-4-oxo-1-butanoic acid

3-protocatechuoylpropanoic acid

Dimethyl ether	[5333-34-6]	$C_{12}H_{14}O_5$	mol. wt. 238.24
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3,4-Dimethoxy-y-oxobenzenebutanoic acid

-Preparation by reaction of succinic anhydride with veratrole in the presence of aluminium chloride, [257, 258, (60 %) 2579],
*in nitrobenzene,
-first at 0–10° for 24 h, then at 45° for 1 h (93 %) [1246];
-for 24 h at r.t. (85 %) [1280];
-at 60° for 3 h (81.4 %) [1745].
*in methylene chloride first at 0°, then at reflux for 4–8 h (53 %) [3190].
*in tetrachloroethane and nitrobenzene mixture at 0° for 3 days (67 %) [987].
-Also refer to: [537, 1269, 1451, 2071 (78.6 %), 2196].
colourless solid [2071];
b.p. 206° [2579];
m.p. 165° [1246], 163–164° [2579], 162–163° [1745], 161.6–162° [2071], 160–161° [257, 258, 1246, 1280], 159–161° [3190], 157–159° [987].

¹H NMR [1745, 2071, 3190], ¹³C NMR [1745, 2071],

IR [1745, 2071], MS [2071].

BIOLOGICAL ACTIVITY: Choleretic action [2196].

Methyl ester of the dimethyl ether [14563-40-7]  $C_{13}H_{16}O_5$  mol. wt. 252.27

-Obtained by treatment of the methyl ether with diazomethane in ethyl ether (99 %) [1745].

-Preparation by reaction of 3-carbomethoxypropanoic acid with veratrole in the presence of PPA at  $45-50^{\circ}$  for 1 h, then at r.t. overnight (65.5 %) [2915].

-Preparation by reaction of 3-carbomethoxypropanoyl chloride with veratrole in the presence of PPA at  $40^{\circ}$  for 2.5 h (79.6 %) [2915].

-Also refer to: [432, 722, 795, 984, 1246, 1247, 1635, 1667, 3485].

m.p. 95° [795], 94–95° [2915], 92° [984], 90.5–91° [3485], 90–91° [432], 89° [1246, 1247], 87° [1635, 1745]; ¹H NMR [984, 1635, 1745, 2915], ¹³C NMR [1635, 1745], IR [984, 1635, 1745, 2915], MS [1635, 1745].

Ethyl ester of the dimethyl ether [56872-60-7]  $C_{14}H_{18}O_5$  mol. wt. 266.29

-Ethyl succinyl chloride was added to a suspension of aluminium chloride in methylene chloride at  $0^{\circ}$ , and the mixture was stirred at  $0^{\circ}$  for 15 min. To this was added veratrole at  $0^{\circ}$ . The reaction mixture was stirred at r.t. for 15 h (81 %) [1436].

-Also refer to: [28, 795, 1286, 2499, 2879].

colourless oil [1436]; m.p. 70° [795], 62° [28], 57–58° [1286]; ¹H NMR [1436], IR [1436].

Diethyl ether	[63213-42-3]	$C_{14}H_{18}O_5$	mol. wt. 266.29
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3,4-Diethoxy-γ-oxobenzenebutanoic acid

-Obtained by condensation of pyrocatechol diethyl ether with succinic anhydride (80 %) [1451].

-Also refer to: [807, 2196].

m.p. 113° [1451], 108–109° [807]; ¹H NMR [807], IR [807], UV [807].

BIOLOGICAL ACTIVITY: Choleretic action [2196].

**Dipropyl ether** [568553-00-4] C₁₆H₂₂O₅ mol. wt. 294.35

-Obtained by condensation of pyrocatechol dipropyl ether with succinic anhydride (62 %) [1451].

m.p. 115° [1451].

## **Dibutyl ether** [856809-73-9] C₁₈H₂₆O₅ mol. wt. 322.40

-Obtained by condensation of pyrocatechol dibutyl ether with succinic anhydride (78 %) [1451].

m.p. 107° [1451].

# **Diamyl ether** [859059-36-2] C₂₀H₃₀O₅ mol. wt. 350.46

-Obtained by condensation of pyrocatechol diamyl ether with succinic anhydride (53 %) [1451].

m.p. 88° [1451].

Dihexyl ether	[856807-53-9]	$C_{22}H_{34}O_5$	mol. wt. 378.51
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-Obtained by condensation of pyrocatechol dihexyl ether with succinic anhydride (57 %) [1451].

m.p. 94–95° [1451].

#### **Methylenedioxy ether** [41764-07-2] $C_{11}H_{10}O_5$ mol. wt. 222.20

-Obtained by refluxing for 2.5 h a mixture of methyl piperonylate, acetone and sodium hydride in tetrahydrofuran (36 %) [1258]. -Also refer to: [2156].

m.p. 91–92° [2156], 89.5–91° [1258].

**Ethyl ester** [56872-61-8]  $C_{12}H_{14}O_5$  mol. wt. 238.24

-Obtained by treatment of ethyl 4-(3,4-dimethoxyphenyl)-4-oxobutanoate with boron tribromide in methylene chloride, first at  $0^{\circ}$ , then at r.t. for 15 h (45 %) [1436].

-Refer to: [1399, 1482, 2571, 2913].

colourless crystals [1436]; m.p. 118–119° [1436], 116–117° [2913], 111° [2571]; ¹H NMR [1436], IR [1436], MS [1399].

# 4-(2,3,4-Trihydroxyphenyl)-4-oxo-1-butanoic acid

HO HO HO

CO(CH₂)₂CO₂H -Obtained *via* initial Friedel-Crafts reaction of 1,2,3-trimethoxybenzene and monomethyl succinate in the presence of 70 % PPA; followed by alkaline hydrolysis of the product ester (95 %) [1671].

-The free acid is generated *in situ* during the strongly acidic conditions of the Clemmensen reduction, but no isolated [231].

# Ethyl ester $C_{12}H_{14}O_6$ mol. wt. 254.24

-Obtained by reaction of succinic anhydride with pyrogallol in the presence of boron trifluoride etherate in dioxan, first at  $-30^{\circ}$ , then at r.t. for 24 h (20 %) [231].

¹H NMR [58, 231], ¹³C NMR [231].

**Trimethyl ether** [63213-41-2] C₁₃H₁₆O₆ mol. wt. 268.27

2,3,4-Trimethoxy-γ-oxobenzenebutanoic acid

-Obtained by reaction of pyrogallol trimethyl ether with  $CH_3O_2C(CH_2)_2COCl$  in the presence of stannic chloride in benzene first at 5°, then at r.t. for 1 h. After, the methylester obtained was warmed at 50° with a 5 % methanolic potassium hydroxide for 1 h [2196].

-Also obtained by reaction of succinic anhydride with pyrogallol trimethyl ether [2196].

-Also obtained by hydrolysis of its ethyl ester ( $C_{15}H_{20}O_6$ ; m.p. 57°) with aqueous alkali [1981] or in refluxing 8 % alcoholic alkali for 3 h (35 %) [2440].

-Also obtained by basic hydrolysis of its methyl ester [535, 1093].

-Also refer to: [1671, 2463, 2487].

long needles [1981]; m.p. 89–90° [1093], 89° [1981], 88–89° [2440]; ¹H NMR [2440, 2487], IR [2440], MS [2440].

BIOLOGICAL ACTIVITY: Choleretic action [2196].

Methyl ester of the trimethyl ether [51686-50-1]  $C_{14}H_{18}O_6$  mol. wt. 282.29

-Obtained by Friedel-Crafts reaction of succinic acid monomethyl ester with pyrogallol trimethyl ether [535].

-Also obtained by reaction between 1,2,3-trimethoxybenzene and 3-carbomethoxypropanoic acid in the presence of PPA for 2.5 h at  $45^{\circ}$  (86 %) [2915], (78.8 %) [1093].

-Also refer to: [1302, 2487].

m.p. 48–49° [1093, 2915], 41–42° [1302]; ¹H NMR [2487, 2915], IR [2915].

Ethyl ester of the trimethyl ether [102222-55-9]  $C_{15}H_{20}O_6$  mol. wt. 296.32

-Obtained by reaction of ethyl succinoyl chloride with 1,2,3-trimethoxybenzene in the presence of stannic chloride in methylene chloride at  $0^{\circ}$  for 5 h (62 %) [2440]. -Also refer to: [1981].

m.p. 57° [1981, 2440]; ¹H NMR [2440], IR [2440], MS [2440].

# 4-(2,4,5-Trihydroxyphenyl)-4-oxo-1-butanoic acid

 $\begin{array}{cccc} [63213-28-5] & C_{10}H_{10}O_6 & \mbox{mol. wt. } 226.19 \\ & & \\ OH & \\ HO & \\ OH & \\ OH & \\ \end{array} \begin{array}{c} OH & \\ OH & \\ OH & \\ \end{array} \begin{array}{c} CO(CH_2)_2CO_2H & -Obtained by treatment of trimethyl ether with 57 \% \\ aqueous hydriodic acid at 130-140^\circ for 3 h \\ (36 \%) [2196]. \\ & \\ m.p. 234-235^\circ [2196]. \end{array} \right.$ 

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

**Trimethyl ether** [31914-19-9] C₁₃H₁₆O₆ mol. wt. 268.27

2,4,5-Trimethoxy- $\gamma$ -oxobenzenebutanoic acid

-Obtained by reaction of 1,2,4-benzenetriol trimethyl ether with  $CH_3O_2C$   $(CH_2)_2COCl$  in the presence of stannic chloride in benzene first at 5°, then at r.t. for 1 h. After, the methylester obtained was warmed at 50° with a 5 % methanolic potassium hydroxide for 1 h [2196].

-Also obtained by reaction of succinic anhydride with 1,2,4-benzenetriol trimethyl ether in the presence of aluminium chloride [257, 258, 2196].

-Also refer to: [99, 472, 1578, 2018, 2794, 3084].

m.p. 171.5–172° [472], 170–171° [2794], 168–169° [257, 258, 3084]; ¹H NMR [3084], IR [3084].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

Methyl ester of the trimethyl ether [82961-09-9]  $C_{14}H_{18}O_6$  mol. wt. 282.29

-Obtained by bubbling hydrogen chloride in 1-(2,4,5-trimethoxyphenyl)-4-oxo-1-butanoic acid in methanol for 1.5 h [258].

m.p. 110-111° [258].

**Triethyl ether** [41826-92-0] C₁₆H₂₂O₆ mol. wt. 310.35

2,4,5-Triethoxy- $\gamma$ -oxobenzenebutanoic acid

-Obtained by heating a mixture of 1,2,4-benzenetriol triethyl ether and succinic acid monomethyl ester in the presence of PPA at  $50-55^{\circ}$  for 40 min. Ice-water was added to the mixture, the resulting aqueous mixture was diluted with acetic acid and heated at  $80-90^{\circ}$  for 50 min (45 %) [2196].

-Preparation from 1,2,4-triethoxybenzene by reaction with succinic anhydride or succinic acid chloride ethyl ester [2195].

m.p. 150-151° [2196].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

Ethyl ester of the triethyl ether [63213-26-3] C₁₈H₂₆O₆ mol. wt. 338.40

2,4,5-Triethoxy-γ-oxobenzenebutanoic acid ethyl ester

-The 3-(2,4,5-triethoxybenzoyl)propionic acid was esterified with a hot ethanolsulfuric acid mixture (quantitative yield) [2196]. -Also refer to: [2192].

m.p. 97–98° [2192], 75° [2196].

N.B.: One of the reported melting point is obviously wrong.

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

**Tripropyl ether** [63213-39-8] C₁₉H₂₈O₆ mol. wt. 352.43

2,4,5-Tripropoxy-y-oxo-benzenebutanoic acid

-Obtained by reaction of phloroglucinol tripropyl ether with  $CH_3O_2C(CH_2)_2COCl$  in the presence of stannic chloride in benzene first at 5°, then at r.t. for 1 h. After, the methylester obtained was warmed at 50° with a 5 % methanolic potassium hydroxide for 1 h [2196].

m.p. 109–111° [2196].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

**Tributyl ether** [41827-09-2] C₂₂H₃₄O₆ mol. wt. 394.51

2,4,5-Tributoxy- $\gamma$ -oxobenzenebutanoic acid

-Obtained by reaction of phloroglucinol tributyl ether with  $CH_3O_2C(CH_2)_2COCl$  in the presence of stannic chloride in benzene first at 5°, then at r.t. for 1 h. After, the methylester obtained was warmed at 50° with a 5 % methanolic potassium hydroxide for 1 h [2196].

m.p. 117–118° [2196].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

# 4-(2,4,6-Trihydroxyphenyl)-4-oxo-1-butanoic acid

[4890-45-3]	$C_{10}H_{10}O_{6}$	mol. wt. 226.19
OH COCH ₂ CH ₂ CO ₂ H	Syntheses -Refer to: [1669, 2196, 235 m.p. 238° [2354, 2355], 219	4, 2355]. 9–220° [1669].

#### **Trimethyl ether** [63213-25-2] C₁₃H₁₆O₆ mol. wt. 268.27

2,4,6-Trimethoxy-y-oxobenzenebutanoic acid

-Obtained by reaction of phloroglucinol trimethyl ether with  $CH_3O_2C(CH_2)_2COCl$  in the presence of stannic chloride in benzene first at 5°, then at r.t. for 1 h. After, the methylester obtained was warmed at 50° with a 5 % methanolic potassium hydroxide for 1 h (72 %) [2196].

-Also obtained from succinic anhydride and phloroglucinol trimethyl ether [2196].

m.p. 142-143° [2196].

BIOLOGICAL ACTIVITY: Choleretic action [2196].

#### 4-(2-Hydroxyphenyl)-2-methyl-4-oxo-1-butanoic acid

[99865-80-2]	$C_{11}H_{12}O_4$	mol. wt. 208.21
OH CO-CH ₂ -CH-COOH	Syntheses -Refer to: [223, 2098]. m.p. 161° [2098], 158.5–159.5°	[223].

#### 4-(2-Hydroxyphenyl)-3-methyl-4-oxo-1-butanoic acid

[59010-62-7]	$C_{11}H_{12}O_4$	mol. wt. 208.21
OH CH ₃ CO-CH-CH ₂ CO ₂ H	Syntheses -Refer to: [687, 1032, 2913]. m.p. 95–97° [687, 2913].	

Methyl ether	[133101-50-5]	$C_{12}H_{14}O_{4}$	mol. wt. 222.24
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-Preparation: A solution of KHMDS in toluene was diluted in THF and cooled to  $-78^{\circ}$ . To this mixture was added dropwise a solution of 1-(2-methoxyphenyl)-1-propanone in THF and the solution was stirred at  $-78^{\circ}$  for 1 h. Then a solution of methyl bromoacetate in THF was added dropwise, and the solution was stirred at  $-78^{\circ}$  for 1 h. The mixture was quenched in 1 N HCl (80 %) [1032]. -Also refer to: [223].

m.p. 68–70° [1032], 55–57° [223]; ¹H NMR [1032], IR [1032].

#### **Methoxymethyl ether** [133101-52-7] C₁₃H₁₆O₅ mol. wt. 252.27

-Obtained from 1-(2-methoxymethoxyphenyl)-1-propanone (to see above) (66 %) [1032].

oil [1032]; ¹H NMR [1032], IR [1032].

#### 4-(4-Hydroxyphenyl)-2-methyl-4-oxo-1-butanoic acid

$$\begin{array}{cccc} & & & C_{11}H_{12}O_4 & & mol. \ wt. \ 208.21 \\ & & & \\ OH & & Synthesis \\ & & -Refer \ to: \ [2733]. \\ & & \\ & & \\ & & \\ COCH_2-CH-CO_2H & & \\ \end{array} \\ \begin{array}{c} & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\$$

-Refer to: [223, 262, 1074, 1170, 2039, 2098, 2427, 2537, 2733 (77 %), 2844, 2845].

m.p. 145–146° [2537], 144° [2098], 143–144° [223, 1170, 2733], 141° [2039]; ¹H NMR [1074, 2427, 2844, 2845], ¹³C NMR [1074, 2844, 2845], IR [2427, 2844, 2845].

Ethyl ester of the methyl ether [15118-68-0]  $C_{14}H_{18}O_4$  mol. wt. 250.29 -Refer to: [1055, 2039, 2098, 2733 (96 %)].

b.p. $_{0.1}$  110–112° [2733], b.p. $_5$  185–187° [2098], b.p. $_{30}$  190° [2039]; ¹H NMR [1055]; n_D^{35.7} = 1.5170 [2039].

# 4-(2,3-Dihydroxyphenyl)-2-methyl-4-oxo-1-butanoic acid

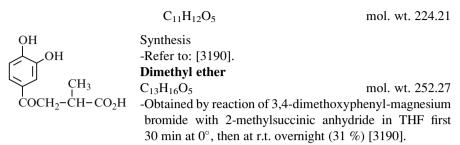
(Plumbagic acid)

$$\begin{array}{cccc} C_{11}H_{12}O_5 & \text{mol. wt. 224.21} \\ OH & Synthesis \\ HO & CO-CH_2-CH-COOH & -Refer to: [141]. \\ CH_3 & M.p. \ 109^{\circ} \ [141]; \ ^{1}H \ NMR \ [141], \ ^{13}C \ NMR \\ [141], \ IR \ [141], \ MS \ [141], \ UV \ [141]. \end{array}$$

**4-(2,3-Dihydroxyphenyl)-3-methyl-4-oxo-1-butanoic acid** (*Plumbagic acid*)

$C_{11}H_{12}O_5$		mol. wt. 224.21
HO HO CO-CH-CH ₂ COOH CH ₃	m.p. 112° [880];	¹ H NMR [880], ¹³ C NMR MS [880], UV [880];

#### 4-(3,4-Dihydroxyphenyl)-2-methyl-4-oxo-1-butanoic acid



**N.B.**: One obtained an inseparable mixture of the regioisomers 2-methyl and 3-methyl (ratio 4:6).

-Also obtained by reaction of methylsuccinic anhydride with veratrole in the presence of aluminium chloride in nitrobenzene at r.t. for 50 h [2632]. -Also refer to: [431, 2427].

m.p. 107–108° [431, 2632], 94–95° [3190]; ¹H NMR [2427, 3190], IR [2427].

Methyl ester of the dimethyl ether	$C_{14}H_{18}O_{6}$	mol. wt. 282.29
-Refer to: [431, 2632].		
m.p. 74–75° [431, 2632].		
2,4-Dinitrophenylhydrazone of the dimethyl ether and methyl ester	$C_{20}H_{22}N_4O_8$	mol. wt. 446.41
-Refer to: [431, 2632].		
m.p. 155–156° [431, 2632].		

#### 4-(3,4-Dihydroxyphenyl)-3-methyl-4-oxo-1-butanoic acid

	$C_{11}H_{12}O_5$	mol. wt. 224.21
но. Но. Ј	Synthesis -Refer to: [3190].	
	<b>Dimethyl ether</b> [358369-06-9]	
CO-CH-CH ₂ -CO ₂ H	$C_{13}H_{16}O_5$	mol. wt. 252.27

-Obtained by adding TFA to a solution of malonic acid di(tert-butyl) ester in methylene chloride. The reaction mixture was stirred at r.t. for 1.5 h and then refluxed for 3 h. After removal of the solvent in vacuo, acetic acid was added to the residue and the mixture was refluxed for 3 h (69 %) [3190]. -Also refer to: [265, 266, 431, 1282, 2632].

m.p. 129° [1282], 118–123° [3190]; ¹H NMR [3190].

Methyl ester of the dime	ethyl ether	$C_{14}H_{18}O_5$	mol. wt. 266.29
m.p. 74–75° [431, 263	2].		
4-(3,5-Dihydroxyphenyl) 4-oxo-1-butanoic acid	)-3-methyl-	$C_{11}H_{12}O_5$	mol. wt. 224.21
ОН	Synthesis		

-Refer to: [1030].  $\begin{array}{c} \textbf{Dimethyl ether} & [17103-73-0] \\ \textbf{CO-CH-CH}_2\textbf{CO}_2\textbf{H} & \textbf{C}_{13}\textbf{H}_{16}\textbf{O}_5 & \textbf{n} \\ \hline \textbf{L} \\ \textbf{CO-CH-CH}_2\textbf{CO}_2\textbf{H} & \textbf{C}_{13}\textbf{H}_{16}\textbf{O}_5 & \textbf{n} \end{array}$ mol. wt. 252.27

-Obtained from  $\alpha$ -bromo-3,5-dimethoxypropiophenone (40–45 %) [1030].

m.p. 105.5–106.5° [1030].

[101117-26-4] C₁₄H₁₉N₃O₅ mol. wt. 309.32 Semicarbazone of the dimethyl ether

m.p. 160–161° [1030].

## 4-(2,4,5-Trihydroxyphenyl)-2-methyl-4-oxo-1-butanoic acid

C11H12O6  $\begin{array}{c} CH_3 \\ COCH_2 - CH - CO_2H \\ COCH_2 - CH - CO_2H \end{array} \begin{array}{c} Synthesis \\ -Refer to: [2196]. \\ Trimethyl ether \\ C_{14}H_{18}O_6 \end{array}$ OH mol. wt. 282.29

-Obtained by reaction of methylsuccinic anhydride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride,

*in sym-tetrachloroethane at 10° for 2 days (30 %) [975]; *in carbon disulfide for 15 h at r.t. (27 %) [3303].

-Also refer to: [145].

m.p. 164–165° [145], 161° [975], 160–161° [3303]; UV [145].

**Methyl ester of the trimethyl ether** [1702-69-8] C₁₅H₂₀O₆ mol. wt. 296.32 -Obtained by treatment of above trimethyl ether with diazomethane in ethyl ether [3303].

m.p. 92-93° [3303].

mol. wt. 240.21

#### **Triethyl ether** [63213-32-1] C₁₇H₂₄O₆ mol. wt. 324.37

2,4,5-Triethoxy-α-methyl-γ-oxo-benzene-butanoic acid

-Preparation by reaction of 1,2,4-triethoxybenzene with 2-methylsuccinic anhydride in the presence of aluminium chloride in carbon tetrachloride at  $70^{\circ}$  for 2 h (40 %) [779, 2196].

m.p. 138–139° [2196]; ¹H NMR [2196].

BIOLOGICAL ACTIVITY: Choleretic action [2196].

## 4-(2,4,5-Trihydroxyphenyl)-3-methyl-4-oxo-1-butanoic acid

 $\begin{array}{cccc} & & & & & & & \\ & & & C_{11}H_{12}O_6 & & & & & \\ & & & & CH_3 & & Synthesis \\ & & & & CO-CH-CH_2-CO_2H & -Refer to: [3303]. \\ & & & & & Trimethyl \ ether & [1702-70-1] \\ & & & & C_{14}H_{18}O_6 & & & mol. \ wt. \ 282.29 \end{array}$ 

-Obtained by reaction of methylsuccinic anhydride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride in sym-tetrachloroethane,

- *at 40° for 8 h (20 %) [3303];
- *at 10° for 2 days (10 %) [975].

m.p. 130–131.5° [145], 128–129° [1250], 125–126° [975], 123–125° [3303]; UV [145].

Methyl ester of the trimethyl ether [2010-80-2] C₁₅H₂₀O₆ mol. wt. 296.32

m.p. 90-92° [1250].

#### 4-(4-Hydroxyphenyl)-2,2-dimethyl-4-oxo-1-butanoic acid

 $\begin{array}{cccc} C_{12}H_{14}O_4 & \text{mol. wt. } 222.24 \\ OH & Synthesis \\ & -Refer \text{ to: } [2358]. \\ & \mathbf{Methyl \ ether} \quad [15118-48-6] \\ & COCH_2-C-CO_2H \\ & CH_2 & -Obtained \ by \ reaction \ of \ dimethyl succinic \ anhydride \ with anisole \ in \ the \ presence \ of \ aluminium \ chloride \ [2359]. \end{array}$ 

-Also refer to: [2358, 2733 (51 %)].

m.p. 162–164° [2358, 2359, 2733]; pK [2358]; ¹H NMR [2733], IR [2733].

BIOLOGICAL ACTIVITY: Anorexigen [2358].

#### Methyl ester of the methyl ether [15118-69-1] $C_{14}H_{18}O_4$ mol. wt. 250.29

-Refer to: [2733 (70 %)].

m.p. 60–61° [2733].

#### 4-(3,4-Dihydroxyphenyl)-2,3-dimethyl-4-oxo-1-butanoic acid

	$C_{12}H_{14}O_5$		mol. wt. 238.24
ОН	Synthesis -Refer to: [1280].		
$\begin{array}{c} \begin{array}{c} \\ \\ \end{array} \\ \begin{array}{c} \\ \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ \begin{array}{c} \\ \end{array} \\ $	<b>Dimethyl ether</b> $C_{14}H_{18}O_5$	[358369-08-1]	mol. wt. 266.29
$CO-CH-CH-CO_2H$			

-Obtained by reaction of *cis* or *trans*  $\alpha$ , $\beta$ -dimethylsuccinic anhydride with veratrole in the presence of aluminium chloride in nitrobenzene at r.t. for 24 h (38 %) [1280]. -Also obtained by reaction of 3,4-dimethoxyphenylmagnesium bromide with 2,3-dimethylsuccinic anhydride (D,L and meso) in THF first 30 min at 0°, then at r.t. overnight (45 %) [3190].

m.p. 165–166° [1280], 163–166° [3190]; ¹H NMR [3190].

#### 2-Ethyl-4-(2,5-dihydroxyphenyl)-4-oxo-1-butanoic acid

$C_{12}H_{14}O_5$			mol. wt. 238.24
$\bigcup_{\bullet \\ \bullet \\$	Synthesis -Refer to: [472]. <b>Dimethyl ether</b> $C_{14}H_{18}O_5$	[132330-85-9]	mol. wt. 266.29

-Obtained by reaction of ethylsuccinic anhydride with hydroquinone dimethyl ether in the presence of aluminium chloride in nitropropane first at  $0-5^{\circ}$ , then 18 h at  $3-8^{\circ}$  (85 %) [472].

m.p. 121° [472].

# 15.2 Substituted Hydroxyketones

#### 4-(3,5-Dibromo-4-hydroxyphenyl)-4-oxo-1-butanoic acid

[392304-69-7]	$C_{10}H_8Br_2O_4$	mol. wt. 351.98
Br HO Br	Synthesis -Refer to: [1446]. ¹ H NMR [1446].	

## 4-(4-Chloro-3-fluoro-2-hydroxyphenyl)-4-oxo-1-butanoic acid

 $[1208265-79-5] C_{10}H_8CIFO_4 mol. wt. 246.62$  $\begin{array}{c} OH \\ F \\ Cl \end{array} CO(CH_2)_2COOH \\ -Refer to: [1008]. \\ ^{1}H NMR [1008]. \end{array}$ 

# 4-(3,5-Dichloro-2-hydroxyphenyl)-4-oxo-1-butanoic acid

 $[62903-22-4] C_{10}H_8Cl_2O_4 mol. wt. 263.08$   $Cl + CO(CH_2)_2COOH -Refer to: [2703, 2705].$   $m.p. 166-167^{\circ} [2703, 2705].$ 4-(3,5-Dichloro-4-hydroxyphenyl)-4-oxo-1-butanoic acid

#### 

# 4-(3-Bromo-2-hydroxyphenyl)-4-oxo-1-butanoic acid

$C_{10}H_9BrO_4$		mol. wt. 273.08
Br CO(CH ₂ ) ₂ CO ₂ H	Synthesis -Refer to: [2579]. <b>Methyl ether</b> C ₁₁ H ₁₁ BrO ₄	mol. wt. 287.14

-Obtained by Friedel-Crafts reaction of succinic anhydride with o-bromoanisole in the presence of aluminium chloride in nitrobenzene/tetrachloroethane mixture (1:4), first at  $0-5^{\circ}$  for 1-2 h, then at r.t. for 3 days (59 %) [2579].

b.p. 218° [2579]; m.p. 189.5–191.5° [2579].

# Ethyl ether $C_{12}H_{13}BrO_4$ mol. wt. 301.14

-Obtained by Friedel-Crafts reaction of succinic anhydride with o-bromophenetole in the presence of aluminium chloride in nitrobenzene/tetrachloroethane mixture (1:4), first at  $0-5^{\circ}$  for 1-2 h, then at r.t. for 3 days (86 %) [2579].

b.p. 224° [2579]; m.p. 201–202° [2579].

#### 4-(5-Bromo-2-hydroxyphenyl)-4-oxo-1-butanoic acid

$$\begin{array}{c} C_{10}H_9BrO_4 & \text{mol. wt. 273.08} \\ OH & Synthesis \\ \hline CO(CH_2)_2CO_2H & -Refer to: [2579]. \\ \hline Phenyl ether \\ C_{16}H_{13}BrO_4 & \text{mol. 349.18} \\ \end{array}$$

-Obtained by Friedel-Crafts reaction of succinic anhydride with p-bromophenyl phenyl ether in the presence of aluminium chloride in nitrobenzene/ tetrachloroethane mixture (1:4), first at  $0-5^{\circ}$  for 1-2 h, then at r.t. for 3 days (82 %) [2579].

-Also refer to: [1958].

b.p. 305° [2579]; m.p. 161.5–162.5° [2579].

#### 4-(5-Bromo-2,4-dihydroxyphenyl)-4-oxo-1-butanoic acid

[854676-84-9]	$C_{10}H_9BrO_5$	mol. wt. 289.08
OH	Syntheses	
$CO(CH_2)_2CO_2H$	-Refer to: [853, 2196].	
	m.p. 190° [853].	
HO	<b>Dimethyl ether</b> [63213-40-1]	
Br	$C_{12}H_{13}BrO_5$	mol. wt. 317.14

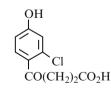
-Obtained by reaction of 4-bromoresorcinol dimethyl ether with CH₃O₂C (CH₂)₂COCl in the presence of stannic chloride in benzene first at 5°, then at r.t. for 1 h. After, the methylester obtained was warmed at  $50^\circ$  with a 5 % methanolic potassium hydroxide for 1 h [2196].

m.p. 188–189° [2196], 179° [853], 178° [795].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

#### 4-(2-Chloro-4-hydroxyphenyl)-4-oxo-1-butanoic acid

C10HoClO4 mol. wt. 228.63



Synthesis

-Obtained by Fries rearrangement of 3-chlorophenyl hydrogen succinate with aluminium chloride in tetrachloroethane or in toluene at 117° for 2.5 h [224].

#### Methyl ether [15572-03-9] $C_{11}H_{11}ClO_4$ mol. wt. 242.66

-Obtained by Friedel-Crafts reaction of succinic anhydride with m-chloroanisole in the presence of aluminium chloride in nitrobenzene/tetrachloroethane mixture (1:4), first at  $0-5^{\circ}$  for 1-2 h, then at r.t. for 3 days [224]. -Also refer to: [861].

m.p. 145° [861], 100–102° [224]; IR [224].

#### 4-(3-Chloro-2-hydroxyphenyl)-4-oxo-1-butanoic acid

#### [15572-06-2] $C_{10}H_{0}ClO_{4}$ mol. wt. 228.63



Syntheses CO(CH₂)₂CO₂H -Obtained by Fries rearrangement of 2-chlorophenyl hydrogen succinate with aluminium chloride in tetrachloroethane or in toluene at  $117^{\circ}$  for 2.5 h [224].

-Also obtained by Friedel-Crafts reaction of succinic anhydride with o-chlorophenol in the presence of aluminium chloride in tetrachloroethane, first at r.t. overnight, then the mixture was heated on a water bath for 4–5 h (20 %) [224].

m.p. 147–149° [224]; IR [224].

#### Methyl ether

C11H11ClO4 mol. wt. 242.66

-Obtained by treatment of the title ketone with diazomethane in ethyl ether [224].

#### C12H13ClO4 mol. wt. 256.69 Ethyl ether

-Obtained by Friedel-Crafts reaction of succinic anhydride with o-chlorophenetole in the presence of aluminium chloride in nitrobenzene/tetrachloroethane mixture (1:4), first at  $0-5^{\circ}$  for 1-2 h, then at r.t. for 3 days [224], (79 %) [2579].

b.p. 208° [2579]; m.p. 190–191.5° [2579], 184° [1333].

#### 4-(3-Chloro-4-hydroxyphenyl)-4-oxo-1-butanoic acid



Syntheses -Obtained by Fries rearrangement of 2-chlorophenyl hydrogen succinate with aluminium chloride in tetrachloroethane or in toluene at 117° for 2.5 h [224].  $CO(CH_2)_2CO_2H$  -Also refer to: [688, 1667, 1779, 2912, 2913, 3099].

m.p. 170° [1667], 160–162° [688, 2912, 2913], 159° [3099].

Methyl ether [39496-87-2]  $C_{11}H_{11}ClO_4$ mol. wt. 242.66

-Obtained by Friedel-Crafts reaction of succinic anhydride with o-chloroanisole in the presence of aluminium chloride in nitrobenzene/tetrachloroethane mixture (1:4), first at  $0-5^{\circ}$  for 1-2 h, then at r.t. for 3 days [224]. -Also refer to: [216, 1779, 2703, 2705].

m.p. 192–198° [2703, 2705], 189° [1333], 188–189° [224], 187–189° [208, 216], 186–188° [1779]; IR [224].

BIOLOGICAL ACTIVITY: Inhibition of aggregation of thrombocytes [1779]; Antiinflammatory efficacy [1779].

# 4-(4-Chloro-2-hydroxyphenyl)-4-oxo-1-butanoic acid

C10H9ClO4 [15572-01-7] mol. wt. 228.63 Syntheses OH

CO(CH₂)₂CO₂H -Obtained by Fries rearrangement of 3-chlorophenyl hydrogen succinate with aluminium chloride in tetrachloroethane or in toluene at 117° for 2.5 h [224].

-Also refer to: [2705].

m.p. 180–181° [2705], 176–178° [224]; IR [224].

[15572-02-8]  $C_{11}H_{11}ClO_4$ Methyl ether mol. wt. 242.66

-Obtained by Friedel-Crafts reaction of succinic anhydride with m-chloroanisole in the presence of aluminium chloride in nitrobenzene/tetrachloroethane mixture (1:4), first at  $0-5^{\circ}$  for 1-2 h, then at r.t. for 3 days [224].

m.p. 163–164° [224]; IR [224].

# 4-(5-Chloro-2-hydroxyphenyl)-4-oxo-1-butanoic acid

C10H9ClO4

mol. wt. 228.63

OH CO(CH₂)₂CO₂H

Syntheses -Obtained by Fries rearrangement of 4-chlorophenyl hydrogen succinate with aluminium chloride in tetrachloroethane or in toluene at 117° for 2.5 h [224]. -Also refer to: [676, 797, 997 (20 %), 2703, 2705, 3289].

m.p. 181-182° [2703, 2705], 180-181° [224], 178-182° [3289], 178–180° [997], 178° [797], 173–174° [676]; ¹H NMR [676, 997], IR [224, 676, 997].



**Methyl ether** [63213-94-5]  $C_{11}H_{11}ClO_4$  mol. wt. 242.66

-Obtained by Friedel-Crafts reaction of succinic anhydride with p-chloroanisole in the presence of aluminium chloride in nitrobenzene/tetrachloroethane mixture (1:4), first at 0–5° for 1–2 h, then at r.t. for 3 days [224], (25 %) [2579]. -Also refer to: [447, 797, 1403, 3289].

b.p. 200° [2579]; m.p. 120° [797], 119–121° [224, 2579], 118–119° [1403], 113–115° [3289]; IR [224].

Methyl ester of the methyl ether [15572-05-1] C₁₂H₁₃ClO₄ mol. wt. 256.69

-Obtained by treatment of 4-(5-chloro-2-hydroxyphenyl)-4-oxo-1-butanoic acid with dimethyl sulfate in the presence of potassium carbonate in acetone [224].

m.p. 66–67° [224]; IR [224].

**Ethyl ether** [105475-33-0]  $C_{12}H_{13}ClO_4$  mol. wt. 256.69

-Obtained by Friedel-Crafts reaction of succinic anhydride with p-chlorophenetole in the presence of aluminium chloride in nitrobenzene/tetrachloroethane mixture (1:4), first at 0–5° for 1–2 h, then at r.t. for 3 days [224], (20 %) [2579]. -Also refer to: [797].

b.p. 212° [2579]; m.p. 157° [797], 156–158° [2579].

**Methyl ester** [99854-21-4]  $C_{11}H_{11}CIO_4$  mol. wt. 242.66

-Refer to: [797, 997].

m.p. 50° [797], 44–49° [997]; ¹H NMR [997], IR [997].

#### 4-(4-Chloro-2,5-dihydroxyphenyl)-4-oxo-1-butanoic acid

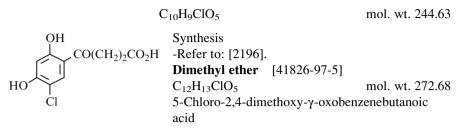
(	$C_{10}H_9ClO_5$	mol. wt. 244.63
OH	Synthesis -Refer to: [2196].	
	<b>Dimethyl ether</b> [41827-05-	-8]
CI	$C_{12}H_{13}ClO_5$	mol. wt. 272.68
ÓН	4-Chloro-2,5-dimethoxy-γ-ox	kobenzenebutanoic acid

-Obtained by reaction of 2-chlorohydroquinone dimethyl ether with  $CH_3O_2C$   $(CH_2)_2COCl$  in the presence of stannic chloride in benzene first at 5°, then at r.t. for 1 h. After, the methylester obtained was warmed at 50° with a 5 % methanolic potassium hydroxide for 1 h [2196].

m.p. 186-188° [2196].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

#### 4-(5-Chloro-2,4-dihydroxyphenyl)-4-oxo-1-butanoic acid



-Obtained by heating a mixture of 4-chlororesorcinol dimethyl ether and succinic acid monomethyl ester in the presence of PPA at  $50-55^{\circ}$  for 40 min. Ice-water was added to the mixture, the resulting aqueous mixture was diluted with acetic acid and heated at  $80-90^{\circ}$  for 50 min [2196].

m.p. 185-187° [2196].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

**Diethyl ether** [41827-02-5] C₁₄H₁₇ClO₅ mol. wt. 300.74

5-Chloro-2,4-diethoxy-γ-oxobenzenebutanoic acid.

-Obtained by reaction of 4-chlororesorcinol diethyl ether with  $CH_3O_2C$   $(CH_2)_2COCl$  in the presence of stannic chloride in benzene first at 5°, then at r.t. for 1 h. After, the methylester obtained was warmed at 50° with a 5 % methanolic potassium hydroxide for 1 h [2196].

m.p. 172–173° [2196].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

# 4-(4-Fluoro-2-hydroxyphenyl)-4-oxo-1-butanoic acid

[202715-97-7]	$C_{10}H_9FO_4$	mol. wt. 212.18
F CO(CH ₂ ) ₂ COOH	Synthesis -Refer to: [1901]. m.p. 134° [1901];	¹ H NMR [1008], IR [1901].

# 4-(3-Hydroxy-2-nitrophenyl)-4-oxo-1-butanoic acid

	$C_{10}H_9NO_6$		mol. wt. 239.18
ł	Synthesis		
$\sim NO_2$	-Refer to: [336	9].	
Ť -	Methyl ether	[103646-41-9]	
$\sim$ CO(CH ₂ ) ₂ CO ₂ H	$C_{11}H_{11}NO_6$		mol. wt. 253.21

-Refer to: [631, 1744, 2687, 3369].

m.p. 148.5–149° [1744, 2687], 139–139.5° (Unstable modification) [1744, 2687], 139° [631]; ¹H NMR [631], ¹³C NMR [631], IR [631, 3369].

# 4-(3-Hydroxy-4-nitrophenyl)-4-oxo-1-butanoic acid

[62893-18-9]	$C_{10}H_9NO_6$	mol. wt. 239.18
NO ₂ CO(CH ₂ ) ₂ CO ₂ H	Syntheses -Refer to: [1554, 2913]. m.p. 158–160.5° [1554, 2913].	

Methyl ether	[93534-71-5]	$C_{11}H_{11}NO_6$	mol. wt. 253.21
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-Refer to: [1554 (33 %)].

m.p. 137–138.5° [1554].

# 4-(4-Hydroxy-3-nitrophenyl)-4-oxo-1-butanoic acid

[56872-41-4]	$C_{10}H_9NO_6$	mol. wt. 239.18
$HO \longrightarrow CO(CH_2)_2CO_2H$	Syntheses -Refer to: [2494, 2912, 2913]. m.p. 172–174° [2912, 2913].	

Methyl ether	[26976-83-0]	$C_{11}H_{11}NO_6$	mol. wt. 253.21
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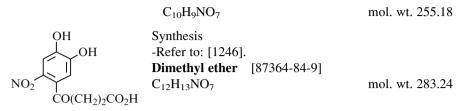
-Preparation by nitration of  $\beta$ -anisoylpropionic acid [2494]. -Also refer to: [953, 954, 2491].

pale yellow prisms [2494]; m.p. 158–159° [953, 954, 2491], 148° [652]; IR [652].

-Refer to: [2494].

colourless needles; m.p. 153° [2494].

#### 4-(4,5-Dihydroxy-2-nitrophenyl)-4-oxo-1-butanoic acid



-Obtained by adding nitric acid (d = 1.42) and sulfuric acid (d = 1.84) to a cold solution of  $\beta$ -3,4-dimethoxybenzoylpropionic acid in acetic acid [1246]. -Also obtained by treatment of  $\beta$ -veratroylpropionic acid with a mixture of nitric acid (d = 1.42) and acetic acid at 0° for 2 h (82 %) [2073].

-Also refer to: [596, 984, 2760].

m.p. 212° [1246, 2073], 210–212° [2760], 115° [596]; **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [984], IR [984], MS [984].

## **Methyl ester of the dimethyl ether** $C_{13}H_{15}NO_7$ mol. wt. 297.26

-Refer to: [1246].

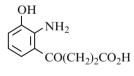
greenish-yellow needles [1246]; m.p. 118° [1246].

#### Ethyl ester of the dimethyl ether [87374-67-2] C₁₄H₁₇NO₇ mol. wt. 311.29

m.p. 84–85° [984]; ¹H NMR [984], IR [984], MS [984].

## 4-(2-Amino-3-hydroxyphenyl)-4-oxo-1-butanoic acid

C₁₀H₁₁NO₄ mol. wt. 209.20



Synthesis -Obtained by hydrogenation of 4-(3-hydroxy-2-nitrophenyl)-4-oxo-2-butenoic acid in solution in ethyl acetate/acetic acid with hydrogen gas at r.t. in the dark for 3 h (47 %) [2109].

m.p. 134–135° [2109]; ¹H NMR [2109], ¹³C NMR [2109], IR [2109], UV [2109], MS [2109]; Fluorescence spectroscopy [2109].

#### 4-(3-Amino-4-hydroxyphenyl)-4-oxo-1-butanoic acid

$$\begin{array}{c} C_{10}H_{11}NO_{4} & \text{mol. wt. 209.20} \\ NH_{2} & \\ HO \longrightarrow CO(CH_{2})_{2}CO_{2}H & \begin{array}{c} Synthesis \\ -\text{Refer to: [2494].} \\ \textbf{Methyl ether} & [39496-86-1] \\ C_{11}H_{13}NO_{4} & \text{mol. wt. 223.23} \end{array}$$

-Obtained by reduction of its nitro derivative [2494]. -Also refer to: [216].

colourless needles [2494]; m.p. 138° [2494], 136–137° [216].

-Obtained by reduction of its nitro derivative [2494].

colourless plates [2494]; m.p. 144–146° [2494].

## 1-(4-Amino-3-hydroxyphenyl)-4-oxo-1-butanoic acid

C	$_{0}H_{11}NO_{4}$	mol. wt. 209.20
OH NH ₂	Synthesis -Refer to: [1554].	
	Methyl ether [92422-41-8]	
CO(CH ₂ ) ₂ CO ₂ H	$C_{11}H_{13}NO_4$	mol. wt. 223.23

-Obtained by adding at r.t. a solution of 3-methoxy-4-nitrobenzoylpropionic acid in 2 N ammonia to an aqueous solution of ferrous sulfate. Then, boiling the mixture for few min (80 %) [1554].

m.p. 152-153° [1554].

#### 1-(5-Amino-2-hydroxyphenyl)-4-oxo-1-butanoic acid

[56872-54-9]	$C_{10}H_{11}NO_4$	mol. wt. 209.20
ŌН	Syntheses	

-Refer to: [687, 688, 2912, 2913].

m.p. 158–160° [687, 688, 2912, 2913].

OH CO(CH₂)₂COOH

#### 4-(2-Amino-4,5-dihydroxyphenyl)-4-oxo-1-butanoic acid

$$\begin{array}{cccc} C_{10}H_{11}NO_5 & \mbox{mol. wt. } 225.20 \\ OH & Synthesis \\ -Refer to: [1246]. \\ Dimethyl ether & [854677-84-2] \\ C_{12}H_{15}NO_5 & \mbox{mol. wt. } 253.25 \end{array}$$

-Obtained by treatment of 4,5-dimethoxy-2-nitrobenzoylpropionic acid in dilute ammonia with hydrated ferrous sulfate at 100° for 30 min [1246, 2073]. -Also refer to: [100, 1247, 2760].

m.p. 143–144° [2760], 141–142.5° [2073], 141° [1247], 139–141° [100], 118° [1246]; **N.B.**: One of the reported melting point is obviously wrong.

#### Acetylamino of the dimethyl ether $C_{14}H_{17}NO_6$ mol. wt. 295.29

-Refer to: [1246].

m.p. 187° [1246].

#### **Benzoylamino of the dimethyl ether** $C_{19}H_{19}NO_6$ mol. wt. 357.36

-Refer to: [1246].

m.p. 225° [1246].

Methyl ester of the dimethyl ether [4848-01-5] C₁₃H₁₇NO₅ mol. wt. 267.28

-Obtained by reaction of 4-(2-Amino-4,5-dimethoxyphenyl)-4-oxo-1-butanoic acid with boiling methanol in the presence of concentrated sulfuric acid for 4 h (59 %) [100].

-Also refer to: [1246, 1247].

yellow needles [100]; m.p. 127–128° [100], 127° [1246, 1247].

#### Ethyl ester of the dimethyl ether [87364-85-0] C₁₄H₁₉NO₅ mol. wt. 281.31

-Refer to: [984, 2760].

m.p. 110–111° [984], 103–104.5° [2760]; ¹H NMR [984], IR [984], MS [984].

# Monohydrate of the dimethyl ether $C_{12}H_{15}NO_5$ , $H_2O$ mol. wt. 271.26

-Refer to: [1246, 1247].

m.p. 118° [1246, 1247].

#### 1-(4-Cyano-3-hydroxyphenyl)-4-oxo-1-butanoic acid

$$C_{11}H_9NO_4 mtext{mol. wt. 219.20}$$

$$C_{11}H_9NO_4 mtext{mol. wt. 219.20}$$

$$C_{11}H_9NO_4 mtext{mol. wt. 219.20}$$

$$C_{11}H_9NO_4 mtext{mol. wt. 219.20}$$

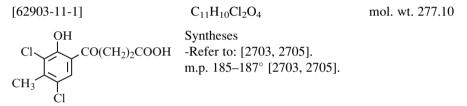
$$C_{11}H_9NO_4 mtext{mol. wt. 233.22}$$

$$C_{11}H_9NO_4 mtext{mol. wt. 233.22}$$

-Obtained by reaction of cuprous cyanide to a solution of diazonium salt of 4-amino-3-methoxy-benzoylpropionic acid, first at  $<4^{\circ}$ , then at 70° for 1 h (60 %) [1554].

m.p. 153-154° [1554].

### 4-(3,5-Dichloro-2-hydroxy-4-methylphenyl)-4-oxo-1-butanoic acid



#### 4-(2-Hydroxy-5-methylphenyl)-2,4-dioxo-1-butanoic acid

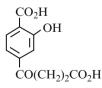
	$C_{11}H_{10}O_5$	mol. wt. 222.20
OH COCH2COCO2H	Synthesis -Refer to: [193].	
CH ₃	Ethyl ester C ₁₃ H ₁₄ O ₅	mol. wt. 250.25

-Refer to: [193].

needles [193]; m.p. 78–79° [193].

#### 1-(4-Carboxy-3-hydroxyphenyl)-4-oxo-1-butanoic acid

C₁₁H₁₀O₆ mol. wt. 238.20



Synthesis

-Obtained by refluxing its methyl ether below with 48 % hydrobromic acid in acetic acid overnight (7 %) [1554]. m.p. 211–214° [1554].

NΞ

Methyl ether [91964-06-6]  $C_{12}H_{12}O_{6}$ mol. wt. 252.22

-Obtained by refluxing a solution of 4-cyano-3-methoxybenzoylpropionic acid in 10 % aqueous sodium hydroxide and ethanol for 2.5 h under nitrogen (70 %) [1554].

m.p. 158–159.5° [1554].

#### 4-(2-Hydroxy-4-methoxyphenyl)-2,4-dioxo-1-butanoic acid

$C_{11}H_{10}O_6$		mol. wt. 238.20
OH COCH ₂ COCO ₂ H		
CH ₃ O	Ethyl ester C ₁₃ H ₁₄ O ₆	mol. wt. 266.25

-Refer to: [1739];

m.p. 107–108° [1739].

# 4-(6-Hydroxybenzodioxol-5-yl)-4-oxo-1-butanoic acid

	$C_{11}H_{10}O_6$	mol. wt. 238.20
.OH	Synthesis -Refer to: [2196].	
CO(CH ₂ ) ₂ CO ₂ H		

Methyl ether	[41827-06-9]	$C_{12}H_{12}O_{6}$	mol. wt. 252.22
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6-Methoxy-y-oxo-1,3-benzodioxole-5-butanoic acid

-Obtained by reaction of 5-methoxybenzodioxol with CH₃O₂C(CH₂)₂COCl in the presence of stannic chloride in benzene first at 5°, then at r.t. for 1 h. After, the methylester obtained was warmed at 50° with a 5 % methanolic potassium hydroxide for 1 h [2196].

-Also obtained by condensation of 5-hydroxybenzodioxol with succinic anhydride (70 %) [1451].

m.p. 140–141° [878], 140° [1451].

BIOLOGICAL ACTIVITY: Choleretic action [2196].

#### 4-(3-Bromo-2-hydroxy-5-methoxyphenyl)-4-oxo-1-butanoic acid

[87338-25-8]	$C_{11}H_{11}BrO_5$	mol. wt. 303.11
$ \begin{array}{c} OH \\ Br \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ $	5-methoxyphenyl)-4-oxo	promine with 4-(2-hydroxy- p-1-butanoic acid in acetic of sodium acetate at

## 4-(5-Chloro-2-hydroxy-4-methylphenyl)-4-oxo-1-butanoic acid

[62903-21-3]	$C_{11}H_{11}ClO_4$	mol. wt. 242.66
CH ₃ CI	Syntheses -Obtained by treatment hydrogen bromide in (89 %) [2652]. -Also refer to: [997 (7 %)	refluxing acetic acid

m.p. 181–182° [2703, 2705], 180–181° [2652], 178–182° [997]; ¹H NMR [997], IR [997].

Methyl ether	[857229-77-7]	$C_{12}H_{13}ClO_4$	mol. wt. 256.69

-Obtained by reaction of succinic anhydride (Bernstein anhydride) with 4-chloro-3-methylanisole in the presence of aluminium chloride in nitrobenzene for 24 h at r.t. (42.5 %) [2652].

m.p. 160–161° [2652].

Methyl ester	[127275-14-3]	$C_{12}H_{13}ClO_4$	mol. wt. 256.69
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-Refer to: [997].

m.p. 95–97° [997]; ¹H NMR [997], IR [997].

## 4-(2-Hydroxy-3-methylphenyl)-4-oxo-1-butanoic acid

[854677-51-3]	$C_{11}H_{12}O_4$	mol. wt. 208.21
CH ₃ CO(CH ₂ ) ₂ CO ₂ H	Syntheses -Obtained by reaction of succ o-cresol in the presence of all tetrachloroethane at 120–130 [197], (35–40 %) [2571].	uminium chloride in

m.p. 136–137° [2571].

Methyl ester  $C_{12}H_{14}O_4$  mol. wt. 222.24

 $C_{13}H_{16}O_{4}$ 

m.p. 78° [2571].

#### Ethyl ester

m.p. 78° [2571].

# Ethyl ether $C_{13}H_{16}O_4$ mol. wt. 236.27

-Obtained by Friedel-Crafts reaction of succinic anhydride and o-cresyl ethyl ether (79 %) [2579].

b.p. 192° [2579]; m.p. 172–173° [341, 2579].

#### 4-(2-Hydroxy-4-methylphenyl)-4-oxo-1-butanoic acid

[59010-46-7] 
$$C_{11}H_{12}O_4$$
 mol. wt. 208.21  
OH  
CO(CH₂)₂CO₂H Syntheses  
-Obtained by Fries rearrangement of m-tolyl hydro-  
gen succinate with aluminium chloride at 117° for  
2.5 h,  
*in tetrachloroethane (41 %) [197], (60–65 %)  
[2571];

*in nitrobenzene (42 %) [197];

*in chlorobenzene (44 %) [197];

*m-xylene (44 %) [197].

-Also obtained by reaction of succinic anhydride with m-cresol in the presence of aluminium chloride [685, 804].

-Also obtained by treatment of its methyl ether with hydrogen bromide in refluxing acetic acid (90 %) [2652].

-Also refer to: [397, 997 (4 %), 1524].

m.p. 155–156° [716], 154–157° [997], 154° [804, 2571], 153–154° [2652]; ¹H NMR [997], IR [997].

Methyl ether	[91497-61-9]	$C_{12}H_{14}O_4$	mol. wt. 222.24
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-Obtained by reaction of m-cresol methyl ether with succinic anhydride in the presence of aluminium chloride in a nitrobenzene/tetrachloroethane mixture (75 %) [2579], (23 %) [1530], (4 %) [930].

-Also refer to: [814, 1524, 1767, 2652 (95 %), 2804, 2832, 3139].

b.p. 177° [2579];
m.p. 138° [2649], 137–137.5° [2579], 127–128° [1524], 126–128° [814], 126–127° [1767], 126° [3139], 124–126° [930], 121–123.5° [1530];
¹H NMR [2804].

mol. wt. 236.27

Methyl ester [59010-47-8]

-Refer to: [687, 997].

m.p. 61–63° [687], 59–61° [997]; ¹H NMR [997], IR [997].

#### 4-(2-Hydroxy-5-methylphenyl)-4-oxo-1-butanoic acid

Syntheses

[103987-17-3]

$$C_{11}H_{12}O_4$$

mol. wt. 208.21

mol. wt. 222.24

OH CO(CH₂)₂CO₂H

H -Obtained by Fries rearrangement of p-tolyl hydrogen succinate with aluminium chloride at 117° for 2.5 h in tetrachloroethane (66 %), in nitrobenzene (56 %) or in chlorobenzene (38 %) [1987], (21 %) [197].

-Also obtained directly by Friedel-crafts reaction of succinic anhydride with p-cresol in the presence of aluminium chloride in tetrachloroethane first at  $55^{\circ}$ , then, at  $135^{\circ}$  for 2.5 h (52 %) [2263].

-Also obtained by treatment of its methyl ether with hydrogen bromide in refluxing acetic acid (94 %) [2652].

-Also refer to: [997 (18 %), 1667, 2263, 2571 (40-45 %), 2649].

m.p. 136–137° [2263, 2649], 136° [2652], 135–136° [3337], 132–134° [910], 131–134° [997], 122–124° [1667]; ¹H NMR [997], IR [997], UV [1827].

BIOLOGICAL ACTIVITY: Refer to: [1827, 2167].

Methyl ether[55007-22-2] $C_{12}H_{14}O_4$ mol. wt. 222.24

-Obtained by succinovlation of p-cresol methyl ether in a 4:1 mixture of tetrachloroethane and nitrobenzene at 0° for 24 h (84 %) [910], (65 %) [2579]. -Also refer to: [690, 858, 2649, 3065, 3273, 3289].

b.p. 176° [2579]; m.p. 115–117° [2649, 3337], 114–115.5° [690], 114–115° [3065], 107–109° [910], 107–108° [2579], 95.5–96.5° [690], 95° [588], 92° [3273], 89–94° [3289]; **N.B.**: The discrepancy between the found and the reported melting point is not explained at present [910].

Semicarbazone of the methyl ether	$C_{13}H_{17}N_3O_4$	mol. wt. 279.29
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m.p. 179° [3065].

Methyl ester of the methyl ether [20483-30-1]  $C_{13}H_{16}O_4$  mol. wt. 236.27

-Obtained by reaction of methanol with  $\beta$ -(2-methoxy-5-methylbenzoyl)propionic acid in the presence of concentrated sulfuric acid at reflux for 24 h (65 %) [910]. -Also refer to: [818].

m.p. 53–54.5° [910], 43.5–47.5° [818]; UV [910].  $C_{12}H_{14}O_4$ 

Ethyl ether [873396-80-6]  $C_{13}H_{16}O_4$  mol. wt. 236.27

-Obtained by Friedel-Crafts reaction of succinic anhydride and p-cresyl ethyl ether (52 %) [2579].

-Also refer to: [341].

b.p. 192° [2579]; m.p. 140° [341], 139–141° [2579].

Semicarbazone of the ethyl ether	$C_{14}H_{19}N_3O_4$	mol. wt. 293.32
1200 [241]		

m.p. 130° [341].

Methyl ester	[123471-86-3]	$C_{12}H_{14}O_4$	mol. wt. 222.24
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-Refer to: [997].

m.p. 52–53° [997]; ¹H NMR [997], IR [997].

#### 4-(3-Hydroxy-4-methylphenyl)-4-oxo-1-butanoic acid

[56872-34-5]

 $C_{11}H_{12}O_4$  mol. wt. 208.21

HO CO(CH₂)₂COOH Synthesis -Refer to: [1993]. m.p. 172–173° [1993].

# 4-(4-Hydroxy-2-methylphenyl)-4-oxo-1-butanoic acid

[319494-43-4]

$$C_{11}H_{12}O_4$$

mol. wt. 208.21

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Syntheses

-Obtained by Fries rearrangement of m-tolyl hydrogen succinate with aluminium chloride at  $117^{\circ}$  for 2.5 h in tetrachloroethane (1–2 %) [2571].

 $O(CH_2)_2CO_2H$  -Also obtained by heating its methyl ether with aluminium chloride at 150° [2649].

-Also obtained by treatment of its methyl ether with hydrogen bromide in refluxing acetic acid (22 %) [2652].

-Also refer to: [716, 1667, 2392, 2857].

m.p. 179.5–180.5° [2392], 172° [2571, 2857], 170° [716], 155° [2652], 108–110° [1667]. **N.B.**: One of the reported melting point is obviously wrong.

**Methyl ether** [67405-48-5] C₁₂H₁₄O₄ mol. wt. 222.24

-Obtained by reaction of m-cresol methyl ether with succinic anhydride [2649] in the presence of aluminium chloride in a nitrobenzene/tetrachloroethane mixture (47 %) [1530].

-Obtained by reaction of  $\beta$ -carbethoxypropionyl chloride with 3-methylanisole in the presence of aluminium chloride in tetrachloroethane at 0° for 3–4 h (90 %) [2392].

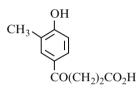
-Also refer to: [303, 340, 534, 858, 930, 1767, 2804, 2832, 2934, 3179].

m.p. 54° (monohydrate) [858];
m.p. 138–139° [1767], 138° [2649], 136.5–138° [2934], 136–137° [2392],
135–137.5° [2579], 135–136° [2804], 134–135° [534], 131–132° [303],
130–133° [1530];
¹H NMR [534, 2804, 2832], IR [534, 2804, 2832].

BIOLOGICAL ACTIVITY: Refer to: [3179].

# 4-(4-Hydroxy-3-methylphenyl)-4-oxo-1-butanoic acid

 $[91541-03-6] C_{11}H_{12}O_4 mol. wt. 208.21$ 



Syntheses -Obtained by reaction of succinic anhydride with o-cresol in the presence of aluminium chloride in tetrachloroethane at  $120-130^{\circ}$  for 2 h (27 %) [197], (15-20 %) [2571].

-Also obtained by treatment of its methyl ether with hydrogen bromide in refluxing acetic acid (97 %) [2652].

-Also refer to: [1619, 1667].

m.p. 198° [1667], 186° [2652], 184° [2571]; IR [1619].

Methyl ether	[33446-14-9]	$C_{12}H_{14}O_4$	mol. wt. 222.24
wieuryi euler	[55440-14-9]	$C_{12}\Pi_{14}O_4$	11101. Wt. 222.24

-Obtained by reaction of succinic anhydride with o-cresol methyl ether in the presence of aluminium chloride (80 %) [982],

*in nitrobenzene first at  $0^{\circ}$ , then at r.t. for 24 h (85 %) [3476] or at 40–45° (74 %) [2649];

*in nitrobenzene as usual (73 %) [2728], for 40 h (88 %) [288], for 60 h [858];

*in nitrobenzene at r.t. overnight (83.6 %) [1566].

-Also refer to: [261, 692, 857, 1033, 1972, 2645, 3233].

m.p. 166.4–169.2° [1972], 152° [3233], 151° [2645], 150.5–152° [288],
150° [692, 857, 858], 147° [3476], 146° [2649], 143° [261, 2728],
142–143° [1566];
¹H NMR [288, 1033, 1972, 3476], ¹³C NMR [288],
IR [692, 3476], UV [692], MS [1972, 3476].

# $\label{eq:constraint} \mbox{Ethyl ester of the methyl ether} \qquad [2954-68-9] \qquad C_{14}H_{18}O_4 \qquad \mbox{mol. wt. } 250.29$

-Obtained by reaction of  $\beta$ -(4-methoxy-3-methylbenzoyl)propionic acid with ethanol in the presence of sulfuric acid for 6 h at reflux (80 %) [692].

silvery plates [692]; m.p. 55° [692]; IR [692].

#### Semicarbazone of the methyl ether and ethyl ester

[4605-98-5]	$C_{15}H_{21}N_3O_4$		mol. wt. 307.35
m.p. 134° [692];	UV [692].		
Methyl ester	[115763-03-6]	$C_{12}H_{14}O_4$	mol. wt. 222.24
m.p. 108° [2571].			
Ethyl ester	$C_{13}H_{16}O_4$		mol. wt. 236.27
m.p. 106° [2571].			
4-(2,5-Dihydroxy-4-methylphenyl)-4-oxo-1-butanoic acid			

# $\begin{array}{cccc} C_{11}H_{12}O_5 & \text{mol. wt. } 224.21 \\ OH & Synthesis \\ CO(CH_2)_2CO_2H & -Refer to: [2196]. \\ Dimethyl ether & [856809-86-4] \\ C_{13}H_{16}O_5 & \text{mol. wt. } 252.27 \end{array}$

-Preparation from 2,5-dimethoxytoluene (90 %) [1937]. -Also refer to: [157, 2135].

m.p. 179° [2135], 177° [1937], 176–177° [157].

Methyl ester of the din	nethyl ether	$C_{14}H_{18}O_5$	mol. wt. 266.29
m.p. 89–90° [1755];	¹ H NMR [1755]	, IR [1755].	
Ethyl ester of the dime	ethyl ether	$C_{15}H_{20}O_5$	mol. wt. 280.32
m.p. 74° [157].			
Diethyl ether	[41827-11-6]	$C_{15}H_{20}O_5$	mol. wt. 280.32

2,5-Diethoxy-4-methyl-y-oxobenzenebutanoic acid

-Obtained by reaction of succinic anhydride with 2,5-diethoxytoluene in the presence of aluminium chloride in carbon tetrachloride first at r.t., then at  $70^{\circ}$  for 2 h [2196].

m.p. 128-129° [2196].

ÒН

BIOLOGICAL ACTIVITY: Choleretic action [2196].

#### 4-(2,6-Dihydroxy-4-methylphenyl)-4-oxo-1-butanoic acid

[854679-09-7]	$C_{11}H_{12}O_5$	mol. wt. 224.21
CO(CH ₂ ) ₂ COOH	Synthesis -Refer to: [856]. m.p. 207° [856].	
4-Nitrophenylhydrazone	$C_{17}H_{17}N_3O_6$	mol. wt. 359.33

m.p. 203–204° [856].

#### 4-(4,5-Dihydroxy-2-methylphenyl)-4-oxo-1-butanoic acid

	$C_{11}H_{12}O_5$		mol. wt. 224.21
ОН	Synthesis		
НО	-Refer to: [2196].		
	Dimethyl ether	[6575-51-5]	
CH ₃	$C_{13}H_{16}O_5$		mol. wt. 252.27
CO(CH ₂ ) ₂ CO ₂ H	4,5-Dimethoxy-2-	-methyl-γ-oxobenzene	butanoic acid

-Obtained by condensation of 1-methyl-3,4-dimethoxybenzene with succinic anhydride in the presence of aluminium chloride in carbon disulfide/nitrobenzene mixture. This one was left at r.t. overnight, then heated under reflux for 30 min (52 %) [1544].

-Also obtained by Friedel-Crafts succinovlation of 3,4-dimethoxytoluene [1837] according to [2835].

-Also obtained by treatment of homoveratrole with succinic anhydride in the presence of aluminium chloride in nitrobenzene for 24 h at r.t. (78.6 %) [2835]. -Also refer to: [2196].

m.p. 128–130° [1544], 108–112° [1837], 97° [2835]; ¹H NMR [1837], IR [1837].

BIOLOGICAL ACTIVITY: Choleretic action [2196].

**Diethyl ether** [41826-96-4] C₁₅H₂₀O₅ mol. wt. 280.32

4,5-Diethoxy-2-methyl-γ-oxobenzenebutanoic acid

-Obtained by reaction of succinic anhydride with 3,4-diethoxytoluene in the presence of aluminium chloride in carbon tetrachloride first at r.t., then at  $70^{\circ}$  for 2 h (44 %) [2196].

-Also refer to: [2191, 2192, 2194, 3037-3039].

m.p. 116–117° [2191, 2192, 2196, 3037, 3039]; ¹H NMR [3038].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

## **Ethyl ester of the ethyl ether** [41827-10-5] $C_{17}H_{24}O_5$ mol. wt. 308.38

-Refer to: [2191, 3037].

OН

CH₂O

m.p. 81-82° [2191, 3037].

#### 4-(2-Hydroxy-4-methoxyphenyl)-4-oxo-1-butanoic acid

γ-2-hydroxy-4-methoxyphenyl-γ-ketobutyric acid

CO(CH₂)₂CO₂H

[14617-02-8]  $C_{11}H_{12}O_5$  mol. wt. 224.21

Syntheses

-Obtained (by-product) by reaction of succinic anhydride with resorcinol dimethyl ether in the presence of aluminium chloride,

*in tetrachloroethane at 50–60° for 3 h, then at r.t. overnight (12 %) [2100];

*without solvent on the water bath for 3 h [2456].

-Also obtained by reaction of succinic anhydride with m-methoxyphenol in the presence of aluminium chloride in nitrobenzene at 49° for 1 h 30 min (32 %) [821]; -Also obtained by treatment of its methyl ester with boiling 2 % alcoholic potassium hydroxide for 24 h [615].

-Also obtained from anhydrobrazilic acid by its decomposition by baryta [2456]. -Also refer to: [660, 676, 795, 796, 2458, 3485].

m.p. 160–161° [615], 156° [660, 795, 796, 2456, 3485], 155–156° [676, 2458], 154° [2100], 148° [821]; ¹H NMR [676], IR [615, 676].

Methyl ester	[42907-96-0]	$C_{12}H_{14}O_5$	mol. wt. 238.24
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-Obtained by methylation of  $\beta$ -(2,4-dihydroxybenzoyl)propionic acid,

*with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 2 h (35 %) [1669];

*with diazomethane in ethyl ether at r.t. overnight (53 %) [1669].

-Also obtained by esterification of this acid with methanol/boron trifluoride [821]. -Also obtained from title acid in refluxing methanol for 2 h in the presence of 20 % sulfuric acid [2456].

-Also refer to: [615, 999, 1153, 1670].

m.p. 90–92° [1669], 87° [2456], 85.5–86° [821], 85° [1153], 73–76° [999]; ¹H NMR [999, 1669], IR [999], UV [999]; TLC [1669].

#### 4-(2-Hydroxy-5-methoxyphenyl)-4-oxo-1-butanoic acid

 $[75501-54-1] C_{11}H_{12}O_5 mtext{mol. wt. } 224.21$ OH CO(CH₂)₂CO₂H OCH₃ Syntheses -Obtained by partial demethylation of  $\beta$ -(2,5-dimethoxybenzoyl)propionic acid with aluminium chloride, *in chlorobenzene [2331]; *in acetonitrile at 65° (70 %) [3045].

-Also obtained by condensation of hydroquinone dimethyl ether with succinic anhydride in nitrobenzene in the presence of aluminium chloride. During this addition, which required 1 h, the reaction mixture was stirred and cooled in an ice bath. The temperature was maintained below  $35^{\circ}$ . The resulting solution was warmed slowly to  $60^{\circ}$  and stirred at this temperature for 3 h (51 %) [2262].

-Also obtained by photo-Fries rearrangement of 4-methoxyphenyl monosuccinate (60 %) [2004].

-Also refer to: [676, 747, 997 (60 %), 3320].

yellow crystals [2262]; m.p. 145–146° [2262], 145° [747, 3045], 142.5–143.5° [997, 2004], 140° [2331]; ¹H NMR [676, 997, 2004, 3320], IR [676, 997, 2004, 3045].

Methyl ester	[59701-66-5]	$C_{12}H_{14}O_5$	mol. wt. 238.24
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-Obtained by treatment of the acid above with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 2.5 h (48 %) [1349].

-Also obtained by photo-Fries rearrangement of 4-methoxyphenyl methyl succinate (58 %) [2004].

-Also refer to: [997].

oil [997, 2004]; m.p. 41–42° [1349]; ¹H NMR [997, 1349, 2004], IR [997, 1349, 2004].

#### 4-(5-Hydroxy-2-methoxyphenyl)-4-oxo-1-butanoic acid

C₁₁H₁₂O₅ mol. wt. 224.21

OCH₃ CO(CH₂)₂CO₂H

Synthesis

-Obtained (by-product) by reaction of succinic anhydride with hydroquinone dimethyl ether in the presence of aluminium chloride [696]. m.p. 135° [696].

#### 4-(2,3-Dihydroxy-4-methoxyphenyl)-4-oxo-1-butanoic acid

[74882-03-4]	$C_{11}H_{12}O_6$	mol. wt. 240.21
HO HO CH ₃ O COCH ₂ CH ₂ CO ₂ H	anhydride with in the presence of 1,1,2,2-tetrachloro-eth ture was allowed to wa	by reaction of succinic 1,2,3-trimethoxybenzene aluminium chloride in ane at $0^{\circ}$ . The red mix- arm to ambient tempera- t was heated on a steam

m.p. 174-176° [1209].

#### 4-(4-Hydroxy-7-benzo[b]thiophene)-4-oxo-1-butanoic acid

	$C_{12}H_{10}O_4S$	mol. wt. 250.28
CO(CH ₂ ) ₂ CO ₂ H	Synthesis -Refer to: [845].	
	<b>Methyl ether</b> [4878-81-3] C ₁₃ H ₁₂ O ₄ S	mol. wt. 264.30
ÓН		

-Obtained by treatment of its methyl ester below with sodium hydroxide in refluxing dilute ethanol for 1 h (99 %) [845]. -Also refer to: [639].

m.p. 223.5° [845]; IR [639, 845].

# Methyl ester of the methyl ether $C_{14}H_{14}O_4S$ mol. wt. 278.33

-Obtained by reaction of butanedioic acid, monochloride, methyl ester with 4-methoxy-benzo[*b*]thiophene (64.5 %) [845]. -Also refer to: [639].

b.p.₁₀ 255° [845]; m.p. 108° [845]; IR [639, 845].

#### 4-(2-Hydroxy-5-methylphenyl)-3-methyl-2,4-dioxo-1-butanoic acid

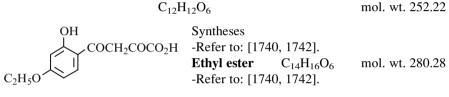
	$C_{12}H_{12}O_5$	mol. wt. 236.22
OH CO-CH-COCO ₂ H CH ₃	Synthesis -Refer to: [193]. Ethyl ester $C_{14}H_{16}O_5$	mol. wt. 264.28

-Obtained by treatment of 2-hydroxy-5-methylpropiophenone with diethyl oxalate in the presence of sodium [193].

# 4-[5-(Acetyloxy)-2-hydroxyphenyl]-4-oxo-1-butanoic acid

 $C_{12}H_{12}O_{6} \qquad \text{mol. wt. 252.22}$ OH Synthesis  $CO(CH_{2})_{2}CO_{2}H \quad -\text{Refer to: [997].}$ 

# 4-(4-Ethoxy-2-hydroxyphenyl)-2,4-dioxo-1-butanoic acid



m.p. 99–100° [1742].

# 4-(2-Hydroxy-3,4-dimethoxyphenyl)-2,4-dioxo-1-butanoic acid

$\begin{array}{c} OH & Synthesis \\ CH_3O & COCH_2COCO_2H & -Refer to: [810]. \\ CH_3O & CH_1O_7 & mol. wt. 296. \end{array}$	.28

-Obtained by reaction of diethyl oxalate with 2-hydroxy-3,4-dimethoxy-acetophenone in the presence of granulous sodium [810].

# 4-(3-Ethyl-4-hydroxyphenyl)-4-oxo-1-butanoic acid

OH	Synthesis		
C ₂ H ₅	-Refer to: [845	].	
	Methyl ether	[3728-79-8]	
$\mathbf{Y}$	$C_{13}H_{16}O_4$		mol. wt. 236.27
$CO(CH_2)_2CO_2H$			

-Obtained by treatment of its methyl ester below with sodium hydroxide in refluxing dilute ethanol for 1.5 h (86 %) [845].

-Also obtained by reaction of succinic anhydride with o-ethylanisole in the presence of aluminium chloride in nitrobenzene at r.t. for 24 h (80 %) [2727]. -Also refer to: [3038].

**ÓCOCH**₃

m.p. 151° [845], 149° [2727]; ¹H NMR [3038].

Methyl ester of the methyl ether [3728-78-7] C₁₄H₁₈O₄ mol. wt. 250.29 -Obtained by reaction of butanedioic acid, monochloride, methyl ester with 2-ethylanisole (81.5 %) [845].

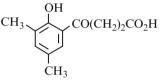
b.p.₁₇ 212–215° [845]; m.p. 45° [845].

#### 4-(2-Hydroxy-3,4-dimethylphenyl)-4-oxo-1-butanoic acid

mol. wt. 222.24 [83481-33-8] C12H14O4 **Svnthesis** OH CH₃  $CO(CH_2)_2COOH$  -Refer to: [693]. m.p. 188–189° [693]. CH

#### 4-(2-Hydroxy-3,5-dimethylphenyl)-4-oxo-1-butanoic acid

[7356-03-8] C12H14O4 mol. wt. 222.24



Syntheses CO(CH₂)₂CO₂H -Obtained by treatment of 2,4-xylyl hydrogen succinate (m.p. 78°) with aluminium chloride, *in nitrobenzene for 0.5 h at  $140^{\circ}$  (15 %) [694]; tetrachloroethane at  $130^{\circ}$  for 2.5 h *in (30 %) [1443].

-Also obtained (by-product) by Fries rearrangement of 2,4-xylyl succinate with aluminium chloride in tetrachloroethene at 125–135° [1443]. -Also refer to: [225, 269, 694, 997 (25 %)].

m.p. 149–151° [997], 147–148° [225, 269, 694], 147° [1443]. ¹H NMR [997], IR [997].

#### Methyl ether

C13H16O4

mol. wt. 236.27

-Obtained by reaction of succinic anhydride with 2,4-dimethylanisole in the presence of aluminium chloride in benzene for 3 h, then at r.t. overnight (25 %) [691]. -Also refer to: [3272].

long needles [691]; m.p. 129–130° [691]; ¹H NMR [3272].

S-Benzylthiuronium salt [102457-90-9] C₂₀H₂₄N₂O₄S mol. wt. 388.48

-Refer to: [269].

m.p. 151° [269].

#### 2,4-Dimethylphenyl ester $C_{20}H_{22}O_4$ mol. wt. 326.39

-Obtained by Fries rearrangement of 2,4-xylyl succinate with aluminium chloride in tetrachloroethane at 125–135° [1443].

m.p. 130–132° [1443].

#### [123471-91-0] $C_{13}H_{16}O_4$ mol. wt. 236.27 Methyl ester

-Refer to: [997].

m.p. 60–65° [997]; ¹H NMR [997], IR [997].

# 4-(2-Hydroxy-4,5-dimethylphenyl)-4-oxo-1-butanoic acid

$C_{12}H_{14}O_4$		mol. wt. 222.24
CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃	Synthesis -Refer to: [225]. m.p. 153–154° [225].	

# 4-(2-Hydroxy-4,6-dimethylphenyl)-4-oxo-1-butanoic acid

[96358-74-6]	$C_{12}H_{14}O_4$	mol. wt. 222.24
CH ₃ CO(CH ₂ ) ₂ COOH	Synthesis -Refer to: [397]. m.p. 117° [397];	¹ HNMR [397], IR [397].

# 4-(4-Hydroxy-3,5-dimethylphenyl)-4-oxo-1-butanoic acid

[52245-99-5]	$C_{12}H_{14}O_4$	mol. wt. 222.24
CH ₃ HO CO(CH ₂ ) ₂ COOH	Synthesis -Refer to: [669]. m.p. 168° [669].	

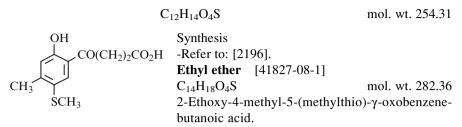
#### 4-(5-Hydroxy-2,4-dimethylphenyl)-4-oxo-1-butanoic acid

C	$_{12}H_{14}O_{4}$		mol. wt. 222.24
CH ₃	m.p. 139° [694		694, 695].
CO(CH ₂ ) ₂ CO ₂ H	Methyl ether	[78334-92-6]	
CH ₃	$C_{13}H_{16}O_4$		mol. wt. 236.27

-Refer to: [187, 694, 695, 3272].

m.p. 52° [695], 50–51° [187]; ¹H NMR [3272].

#### 4-(2-Hydroxy-4-methyl-5-methylthiophenyl)-4-oxo-1-butanoic acid



-Obtained by reaction of 3-methyl-4-methylthiophenetole with  $CH_3O_2C$   $(CH_2)_2COCl$  in the presence of stannic chloride in benzene first at 5°, then at r.t. for 1 h. After, the methylester obtained was warmed at 50° with a 5 % methanolic potassium hydroxide for 1 h [2196].

m.p. 117-118° [2196].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

#### 4-(2,4-Dihydroxy-5-ethylphenyl)-4-oxo-1-butanoic acid

	mol. wt. 238.24	
OH , $CO(CH_2)_2CO_2H$	Synthesis -Refer to: [2196].	
HO $C_2H_5$	<b>Dimethyl ether</b> [100972-91-6] $C_{14}H_{18}O_5$ -Refer to: [115, 1450].	mol. wt. 266.29

m.p. 141–142° [1450].

Diethyl ether	[41827-04-7]	$C_{16}H_{22}O_5$	mol. wt. 294.35
	[	~ 1022 ~ J	

2,4-Diethoxy-5-ethyl-\gamma-oxobenzenebutanoic acid

-Obtained by heating a mixture of 4-ethylresorcinol diethyl ether and succinic acid monomethyl ester in the presence of PPA at  $50-55^{\circ}$  for 40 min. Ice-water was added to the mixture, the resulting aqueous mixture was diluted with acetic acid and heated at  $80-90^{\circ}$  for 50 min [2196].

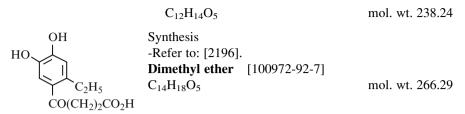
-Also refer to: [115, 3037, 3039].

m.p. 153–155° [2196, 3037, 3039].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

**2-Ethoxy-4-methoxy diether** [129201-57-6]  $C_{15}H_{20}O_5$  mol. wt. 280.32 -Refer to: [115].

#### 4-(4,5-Dihydroxy-2-ethylphenyl)-4-oxo-1-butanoic acid



-Obtained by treatment of ethylveratrole with succinic anhydride in the presence of aluminium chloride in nitrobenzene for 24 h at r.t. (77.5 %) [2835].

white flakes [2835]; m.p. 123° [2835].

#### 1-(2-Hydroxy-3,4-dimethoxyphenyl)-4-oxo-1-butanoic acid

 $\gamma$ -2-hydroxy-3,4-dimethoxyphenyl- $\gamma$ -ketobutyric acid

[68486-75-9]  $C_{12}H_{14}O_6$  mol. wt. 254.24 OH  $CH_3O$  OH  $CH_3O$  COCH₂CH₂CO₂H  $CH_3O$  COCH₂CH  $CH_3O$  COCH₂CH₂CH  $CH_3O$ 

-Also refer to: [798, 1093, 1302 (30–40 %), 2463].

pale yellow needles [2463]; m.p. 154–156° [1302], 154–155° [2463], 153° [798], 152° [257, 258, 2100], 148° [1981].

Methyl ester [74882-02-3] C₁₃H₁₆O₆ mol. wt. 268.27

-Obtained by bubbling hydrogen chloride in 1-(2-hydroxy-3,4-dimethoxyphenyl)-4-oxo-1-butanoic acid in methanol for 1.5 h [257, 258].

-Also obtained from the title acid in refluxing methanol in the presence of 1 % concentrated sulfuric acid [1302].

-Also refer to: [798].

m.p. 110° [798], 106° [257, 258], 99–101° [1302].

#### Ethyl ester

#### $C_{14}H_{18}O_{6}$

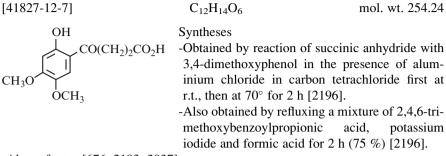
mol. wt. 282.29

-Refer to: [798].

m.p. 58° [798].

#### 4-(2-Hydroxy-4,5-dimethoxyphenyl)-4-oxo-1-butanoic acid

2-Hydroxy-4,5-dimethoxy-γ-oxubenzenebutanoic acid



-Also refer to: [676, 2193, 3037].

m.p. 162–163° [2193, 2196, 3037], 152–155° [676]; ¹H NMR [676], IR [676, 2196].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

# 1-(4-Hydroxy-3-propylphenyl)-4-oxo-1-butanoic acid

	$C_{13}H_{16}O_4$	mol. wt. 236.37
OH C ₃ H ₇	Synthesis -Refer to: [2728]. <b>Methyl ether</b> [100972-66-5] C ₁₄ H ₁₈ O ₄	mol. wt. 250.30
$CO(CH_2)_2CO_2H$		

-Obtained by reaction of succinic anhydride with 2-propylanisole in the presence of aluminium chloride in nitrobenzene (50 %) [2728].

white needles [2728]; m.p. 139° [2728].

Ethyl ether	[905590-12-7]	$C_{15}H_{20}O_4$	mol. wt. 264.32
¹ H NMR [3038].			

# 4-(2-Hydroxy-3,4,6-trimethylphenyl)-4-oxo-1-butanoic acid

[84978-12-1]	$C_{13}H_{16}O_4$	mol. wt. 236.27
CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃	Syntheses -Obtained (by-product) by Fries 2,3,5-trimethylphenyl succinate chloride in tetrachloroethane 140–150° (5 %) [2908].	with aluminium

-Also obtained by hydrolysis of its 2,3,5-trimethylphenyl ester below with aqueous methanolic potassium hydroxide (40 %) [2908]. -Also refer to: [2325].

m.p. 170–171° [2908], 168–171.5° [2325]; IR [2325].

#### C22H26O4 2,3,5-Trimethylphenyl ester mol. wt. 354.45

-Obtained (by-product) by Fries rearrangement of 2,3,5-trimethylphenyl succinate with aluminium chloride in tetrachloroethane for 1.5 h at  $140-150^{\circ}$  (6 %) [2908].

m.p. 134–135° [2908].

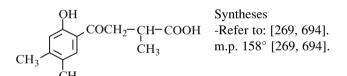
# 4-(2-Hydroxy-3,5-dimethylphenyl)-2-methyl-4-oxo-1-butanoic acid

 $\alpha$ 

$$\begin{array}{c} C_{13}H_{16}O_4 & \text{mol. wt. 236.27} \\ OH & CH_3 & \\ CH_3 & COCH_2 - CH - CO_2H \\ CH_3 & \\ CH_3$$

# 4-(2-Hydroxy-4,5-dimethylphenyl)-2-methyl-4-oxo-1-butanoic acid

C13H16O4

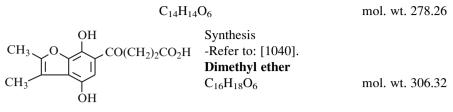


#### 4-(2-Hydroxy-3,5-dimethylphenyl)-3-methyl-4-oxo-1-butanoic acid

mol. wt. 236.27 C13H16O4 CH₃ CO-CH-CH₂CO₂H Synthesis -Obtained by treatment of 2,4-dimethylphenyl 3-methylsuccinate (m.p. 65°) with aluminium chloride for 40 min at 140° [694]. m.p. 133° [694].

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#### 4-(4,7-Dihydroxy-2,3-dimethyl-6-benzofuranyl)-4-oxo-1-butanoic acid



-Refer to: [1040].

#### Methyl ester of the dimethyl ether $C_{17}H_{20}O_6$ mol. wt. 320.34

-Refer to: [1040 (51 %)].

m.p. 92° [1040]; LD₅₀ [1040].

# 4-(2-Hydroxy-3,4,6-trimethylphenyl)-2-methyl-4-oxo-1-butanoic acid

[34927-48-5] C	$_{14}H_{18}O_4$	mol. wt. 250.29
CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃	Syntheses -Refer to: [2150, 3275]. m.p. 182–184° [3275], 17 ¹ H NMR [3275], IR [327	

# 4-(2-Hydroxy-3,4,6-trimethylphenyl)-2-methyl-4-oxo-1-butanoic acid

[34927-49-6]	$C_{14}H_{18}O_4$	mol. wt. 250.29
CH ₃ CH ₃ CH ₃ CH ₃ CH ₂ CH ₂ CO-CH-CH ₂ COOH CH ₃ CH ₃	Synthesis -Refer to: [3275]. m.p. 138–139° [3275]; ¹ H NMR [3275], IR [3275].	

#### 4-(2-Hydroxy-3,5-dimethylphenyl)-2-ethyl-4-oxo-1-butanoic acid

$$C_{14}H_{18}O_4$$
 mol. wt. 250.29

CH

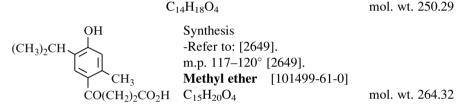
OH  $C_2H_5$  Synthesis COCH₂-CH-CO₂H -Obtained by Fries rearrangement of 2,4-dimethyl-phenyl 2-ethylsuccinate with aluminium chloride in nitrobenzene for 15 min at 140° (6 %) [694]. m.p. 140–141° [694].

#### 1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-4-oxo-1-butanoic acid

[106591-86-0]	$C_{14}H_{18}O_4$	mol. wt. 250.29
(CH ₃ ) ₂ CH CO(CH ₂ ) ₂ CO ₂ H	Synthesis -Obtained by reaction thymol in the presenc ride on heating at (75 %) [2960].	e of aluminium chlo-

m.p. 40° [2960].

# 4-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-4-oxo-1-butanoic acid



-Refer to: [2649, 2934, 3050].

m.p. 93–94° [3050], 92–93° [2934], 92° [2649].

**2,4-Dinitrophenylhydrazone of the methyl ether**  $C_{21}H_{24}N_4O_7$  mol. wt. 444.44 m.p. 144–145° [2934].

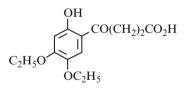
**Phenylhydrazone of the methyl ether** C₂₁H₂₆N₂O₃ mol. wt. 354.45

m.p. 144–145° [2934].

#### 4-(4,5-Diethoxy-2-hydroxyphenyl)-4-oxo-1-butanoic acid

[63213-45-6]	$C_{14}H_{18}O_{6}$	mol. wt. 282.29
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Synthesis



 $\begin{array}{c} \text{OH} & \text{Syntnesis} \\ \text{CO}(\text{CH}_2)_2\text{CO}_2\text{H} & \text{-Obtained by reaction of succinic anhydride with} \\ \text{3,4-diethoxyphenol in the presence of alum-inium chloride in carbon tetrachloride first at} \\ \text{OC}_2\text{H}_5 & \text{r.t., then at } 70^\circ \text{ for 2 h [2196].} \end{array}$ 

m.p. 142–144° [2196].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

#### 4-(5-Cyclopentyl-2-hydroxyphenyl)-4-oxo-1-butanoic acid

$$\begin{array}{c} C_{15}H_{18}O_4 & \text{mol. wt. } 262.31 \\ \hline \\ \hline \\ CO(CH_2)_2CO_2H & Synthesis \\ -Preparation by demethylation of its methyl \\ ether [496]. \\ m.p. \ 109^{\circ} [496]. \end{array}$$

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

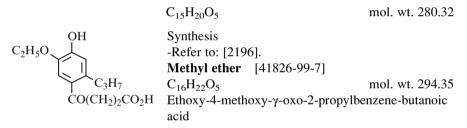
**Methyl ether** [874487-28-2] C₁₆H₂₀O₄ mol. wt. 276.33

-Preparation by reaction of succinic anhydride with p-cyclopentylanisole in the presence of aluminium chloride in nitrobenzene between 0 and 5° for 2 h [496].

m.p. 147° [496].

BIOLOGICAL ACTIVITY: Choleretic [496].

#### 4-(5-Ethoxy-4-hydroxy-2-propylphenyl)-4-oxo-1-butanoic acid



-Obtained by heating a mixture of 2-ethoxy-5-propylanisole and succinic acid monomethyl ester in the presence of PPA at  $50-55^{\circ}$  for 40 min. Ice-water was added to the mixture, the resulting aqueous mixture was diluted with acetic acid and heated at  $80-90^{\circ}$  for 50 min [2196].

m.p. 93° [2196].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

#### 4-(4'-Hydroxybiphenyl)-4-oxo-1-butanoic acid

$$[74277-78-4] C_{16}H_{14}O_4 mol. wt. 270.28$$

$$HO \longrightarrow CO(CH_2)_2CO_2H Syntheses -Obtained by demethylation of its methyl ether [496, 645, 986].$$

m.p. 218–220° [496]; UV [645]. BIOLOGICAL ACTIVITY: Choleretic [496]. Methyl ether [36330-87-7] C17H16O4 mol. wt. 284.31

-Obtained by condensation of 4-methoxydiphenyl with succinic anhydride in the presence of aluminium chloride in nitrobenzene in an ice bath. The mixture was stirred cold for 6 h at r.t. (24.5 %) [986], [645]. -Also refer to: [648, 649].

m.p. 201–202° [648, 649], 200–201° [986].

**BIOLOGICAL ACTIVITY:** Antiinflammatory [649].

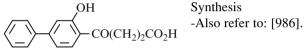
**Methyl ester of the methyl ether** [54011-26-6]  $C_{18}H_{18}O_4$  mol. wt. 298.34

Synthesis

m.p. 99–110° [986].

# 4-(5-Hydroxybiphenyl-4-yl)-4-oxo-1-butanoic acid

 $C_{16}H_{14}O_4$ mol. wt. 270.28



# Methyl ether

C17H16O4 mol. wt. 284.31

mol. wt. 298.34

-Obtained by condensation of 4-methoxydiphenyl with succinic anhydride in the presence of aluminium chloride in nitrobenzene in an ice bath. The mixture was stirred cold for 6 h at r.t. (60 %) [986].

C18H18O4

m.p. 155° [986].

# Methyl ester

m.p. 48-49° [986].

# 4-(6-Hydroxybiphenyl-3-yl)-4-oxo-1-butanoic acid

[408336-52-7] C₁₆H₁₄O₄ mol. wt. 270.28

**Synthesis** -Preparation by demethylation of its methyl ether with hydriodic acid in refluxing acetic anhydride for 20 min (84 %) [496]. m.p. 169–170° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

Methyl ether  $C_{17}H_{16}O_{4}$ mol. wt. 284.31 [408336-68-5]

-Preparation by reaction of succinic anhydride with 2-methoxydiphenylmethane in the presence of aluminium chloride in nitrobenzene between 0 and 5° for 2 h [496].

m.p. 131-132° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

# 4-(4-Hydroxy-3-phenoxyphenyl)-4-oxo-1-butanoic acid

$$C_{16}H_{14}O_5 \qquad \text{mol. wt. } 286.28$$
Synthesis
-Preparation by demethylation of its methyl
ether [496].
$$CO(CH_2)_2CO_2H \qquad \text{m.p. } 143^{\circ} [496].$$

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

# Methyl ether

-Preparation by reaction of succinic anhydride with o-phenoxyanisole in the presence of aluminium chloride in nitrobenzene between 0 and  $5^{\circ}$  for 2 h [496].

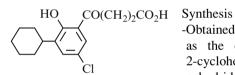
C17H16O5

m.p. 158° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

# 4-(5-Chloro-3-cyclohexyl-2-hydroxyphenyl)-4-oxo-1-butanoic acid

310.78



-Obtained by demethylation in situ in 5 % yield as the only product from the acylation of 2-cyclohexyl-4-chloro-anisole with succinic anhydride in the presence of aluminium chloride in nitrobenzene between 0 and  $5^{\circ}$  for 2 h [496].

m.p. 174° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

# 4-(3-Cyclohexyl-4-hydroxyphenyl)-4-oxo-1-butanoic acid

[702701-04-0]	$C_{16}H_{20}O_4$		mol. wt. 276.33
HO CO(CH ₂ ) ₂ CO ₂ H	Synthesis -Preparation by ether [496]. m.p. 194° [496].	demethylation	of its methyl
DIOLOCICAL ACTIVITY, CL	alamatic [406]		

BIOLOGICAL ACTIVITY: Choleretic [496].

Methyl ether [412022-96-9] C₁₇H₂₂O₄ mol. wt. 290.36

-Preparation by reaction of succinic anhydride with o-cyclohexylanisole in the presence of aluminium chloride in nitrobenzene between 0 and  $5^{\circ}$  for 2 h [496].

m.p. 161° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

mol. wt. 300.31

# 4-(5-Cyclohexyl-2-hydroxyphenyl)-4-oxo-1-butanoic acid

C16H20O4 mol. wt. 276.33 **Synthesis** -Preparation by demethylation of its methyl ether [496].  $CO(CH_2)_2CO_2H$ m.p. 126° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

Methyl ether [412033-83-1] C17H22O4 mol. wt. 290.36

-Preparation by reaction of succinic anhydride with p-cyclohexylanisole in the presence of aluminium chloride in nitrobenzene between 0 and 5° for 2 h [496].

m.p. 159–160° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

#### 1-(3-Isovaleryl-2,4,6-trihydroxyphenyl)-5-oxo-1-pentanoic acid

C16H20O7

mol. wt. 324.33

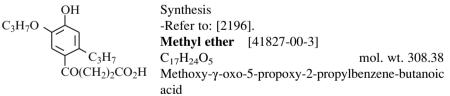
(CH₃)₂CHCH₂CO

Synthesis

 $\sim CO(CH_2)_3CO_2H$  -Obtained by reaction of glutaric anhydride with 2-isovalerylphloroglucinol in the presence of boron trifluoride etherate at r.t. for 2 h (40-45 %) [338].

# 4-(4-Hydroxy-5-propoxy-2-propylphenyl)-4-oxo-1-butanoic acid C₁₆H₂₂O₅

mol. wt. 294.35

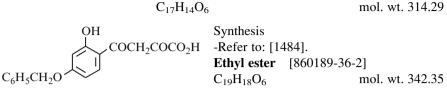


-Obtained by heating a mixture of 2-propoxy-5-propylanisole and succinic acid monomethyl ester in the presence of PPA at 50-55° for 40 min. Ice-water was added to the mixture, the resulting aqueous mixture was diluted with acetic acid and heated at 80-90° for 50 min [2196].

m.p. 70° [2196].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

# 4-(2-Hydroxy-4-(phenylmethoxy)phenyl)-2,4-dioxo-1-butanoic acid



-Refer to: [1484]; m.p. 117.5–118° [1484].

# 4-[4-(5-Chloro-2-hydroxyphenylmethyl)phenyl]-4-oxo-1-butanoic acid

 $C_{17}H_{15}ClO_4$ mol. wt. 318.76 OH Synthesis -Preparation by demethylation of its  $CO(CH_2)_2CO_2H$  dimethyl ether [496]. m.p. 200–201° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

#### Methyl ether C18H17ClO4 mol. wt. 332.78

-Preparation by reaction of succinic anhydride with 5-Chloro-2-methoxydiphenylmethane in the presence of aluminium chloride in nitrobenzene between 0 and 5° for 2 h [496].

m.p. 144–145° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

# 4-[3-(Phenylmethyl)-4-hydroxyphenyl]-4-oxo-1-butanoic acid

$C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{17}H_{16}C_{1$	D ₄ mol. wt. 284.31
HO $CH_2$ $CO(CH_2)_2CO_2H$	Synthesis -Preparation by demethylation of its methyl ether [496]. m.p. 185.5° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

# Methyl ether

mol. wt. 298.34

 $C_{18}H_{18}O_4$ -Preparation by reaction of succinic anhydride with 2-methoxydiphenylmethane in the presence of aluminium chloride in nitrobenzene between 0 and 5° for 2 h [496].

m.p. 133° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

mol. wt. 304.39

# 4-[5-(Phenylmethyl)-2-hydroxyphenyl]-4-oxo-1-butanoic acid

$$\begin{array}{c} C_{17}H_{16}O_4 & \text{mol. wt. 284.31} \\ \hline \\ \hline \\ CH_2 - CH_2 - OH & Synthesis \\ CO(CH_2)_2CO_2H & \text{ether [496].} \\ CO(CH_2)_2CO_2H & \text{m.p. 161}^{\circ} [496]. \end{array}$$

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

Methyl ether  $C_{19}H_{19}O_4$ mol. wt. 298.34

-Preparation by reaction of succinic anhydride with 4-methoxydiphenylmethane in the presence of aluminium chloride in nitrobenzene between 0 and 5° for 2 h [496].

m.p. 121° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

# 4-(4-Cyclohexyl-2-hydroxyphenyl)-2-methyl-4-oxo-1-butanoic acid

a 11 a

$C_{17}H_{22}O_4$	mol. wt. 290.36
Synthesis	

OH CH₃ -Preparation COCH₂-CH-CO₂H ether [496]. -Preparation by demethylation of its methyl m.p. 126° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

Methyl ether

-Preparation by reaction of methylsuccinic anhydride (also named pyrotartaric anhydride) with p-cyclohexylanisole in the presence of aluminium chloride in nitrobenzene between 0 and 5° for 2 h [496].

C18H24O4

m.p. 151° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

# 4-[3-(1-Phenylethyl)-4-hydroxyphenyl]-4-oxo-1-butanoic acid

$C_{18}H_{18}O_4$		mol. wt. 298.34
$ \begin{array}{c} HO \\ -CH \\ I \\ CH_3 \end{array} \begin{array}{c} CO(CH_2)_2CO_2H \end{array} $	Synthesis -Refer to: [496]. <b>Methyl ether</b> C ₁₉ H ₂₀ O ₄	mol. wt. 312.37

-Preparation by reaction of succinic anhydride with 2-(1-phenylethyl)anisole in the presence of aluminium chloride in nitrobenzene between 0 and 5° for 2 h [496].

m.p. 150° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

# 4-[4-Hydroxy-3-(2-hydroxyphenetyl)phenyl]-4-oxo-1-butanoic acid

mol. wt. 314.34 C18H18O5 OH Synthesis -Preparation by demethylation of its CH₂CH₂ dimethyl ether [496]. m.p. 147–148° [496]. CO(CH₂)₂CO₂H

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

#### **Dimethyl ether** C20H22O5 mol. wt. 342.39

-Preparation by reaction of succinic anhydride with 2',2''-dimethoxy-1,2-diphenylethane in the presence of aluminium chloride in nitrobenzene between 0 and 5° for 2 h [496].

m.p. 143° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

# 4-[5-(2-Cyclohexylethyl)-2-hydroxyphenyl]-4-oxo-1-butanoic acid

C18H24O4 mol. wt. 304.39 Synthesis CH₂CH₂ -Preparation by demethylation of its  $CO(CH_2)_2CO_2H$  methyl ether [496]. m.p. 105.5° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

# Methyl ether

-Preparation by reaction of succinic anhydride with p-( $\beta$ -cyclohexylethyl)anisole in the presence of aluminium chloride in nitrobenzene between 0 and  $5^{\circ}$  for 2 h [496].

C19H26O4

m.p. 103° [496].

**BIOLOGICAL ACTIVITY: Choleretic [496].** 

mol. wt. 318.41

# Chapter 3 Pentanones

# 1 Aromatic Hydroxyketones Derived from Pentanoic Acids

# 1.1 Unsubstituted Hydroxyketones

# 1-(2-Hydroxyphenyl)-1,3-pentanedione

Ethyl ether	$C_{13}H_{16}O_3$	mol. wt. 220.27
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-Obtained by reaction of 2-ethoxyacetophenone with ethyl propionate in the presence of sodium [3083]. -Also refer to: [2490].

m.p. 46° [2490], 44–45° [3083]; UV [3083].

#### mp. 10 [2190], 11 15 [5005], 01 [500

# Methyl ether

 $C_{12}H_{14}O_3$ 

mol. wt. 206.24

-Obtained by reaction of CH₃CO(CH₂)₂COSeCH₃ with anisole (20 %) [1750].

b.p._{0.8} 102° [1750]; ¹H NMR [1750], IR [1750], MS [1750].

# 1-(4-Hydroxyphenyl)-1,3-pentanedione

i (4 ilyuloxyphen	(ji) i,o pentaneurone		
[91143-26-9]	$C_{11}H_{12}O_{2}$	3	mol. wt. 192.21
OH COCH ₂ COC ₂ H ₅	Synthesis -Preparation from p-ace m.p. 43–44° [1538], 48°	• •	9 %) [1538].
Copper salt	C ₂₂ H ₂₂ O ₆ C	^C u	mol. wt. 447.53
m.p. 239–240° [	1538].		
Methyl ether	[54103-36-5]	$C_{12}H_{14}O_3$	mol. wt. 206.24
-Obtained by reacti -Also refer to: [120	on of CH ₃ CO(CH ₂ ) ₂ COS 7, 2898].	SeCH ₃ with anisole (4)	40 %) [1750].
b.p. _{0.8} 102° [175	50]; ¹ H NMR [1750], IF	R [1750], MS [1750].	
1-(2-Hydroxyphen	yl)-1,4-pentanedione		
[16850-80-9]	$C_{11}H_{12}O_{21}$	3	mol. wt. 192.21
OH CO(CH ₂ ) ₂ CO	Synthesis OCH ₃ -Refer to: [548]. m.p. $50^{\circ}$ [548],	¹ H NMR [548], IR [	548], UV [548].
1-(4-Hydroxyphen	yl)-1,4-pentanedione		
	$C_{11}H_{12}O_3$		mol. wt. 192.21
OH CO(CH ₂ ) ₂ COCH ₃	Synthesis -Refer to: [1749]. m.p. 99–100° [3311],	MS [2572].	
Methyl ether	[2108-54-5]	$C_{12}H_{14}O_3$	mol. wt. 206.24

-Obtained by Friedel-Crafts acylation of anisole with  $CH_3CO(CH_2)_2COSeMe$  during 3 min (60 %) [1749].

-Also obtained from acetaldehyde and 1-(4-methoxyphenyl)-2-propen-1-one (80 %) [2953].

-Also obtained by reaction of p-anisaldehyde with 3-buten-2-one in the presence of KOH and triethylamine in refluxing ethanol for 2 h (42.2 %) [2952].

-Also obtained by reaction of p-methoxyphenacyl bromide with ethyl acetoacetate in the presence of sodium metal in refluxing ethanol for 20 h (20.5 %) [1294]. -Refer to: [654, 1750, 1924, 2275, 2301, 2816 (46 %), 3358, 3392].

b.p._{0.6} 153–157° [2952], b.p._{0.8} 157–160° [2953], b.p.₄ 120–160° [1294];
m.p. 72° [2816], 58–59° [2952], 58° [2953], 55–57° [3358], 49–51° [3392], 46–48° [1294].
N.B.: One of the reported melting point is obviously wrong.
¹H NMR [654, 1749, 1750, 1924, 2275, 2301, 2816, 2952, 2953, 3358], ¹³C NMR [654, 3358, 3392],
IR [1294, 1749, 1750, 1924, 2275, 2301, 2816, 2952, 2953, 3358, 3392],
UV [1294, 2275], MS [2816].

#### 1-(2,4-Dihydroxyphenyl)-1,3-pentanedione

 $\begin{array}{c} C_{11}H_{12}O_4 & \text{mol. wt. } 208.21 \\ OH & Synthesis \\ \hline COCH_2COC_2H_5 & -Refer to: [3083]. \\ \hline \textbf{Diethyl ether} & C_{15}H_{20}O_4 & \text{mol. wt. } 264.32 \end{array}$ 

-Obtained by reaction of 2,4-diethoxyacetophenone with ethyl propionate in the presence of sodium [3083].

C13H16O4

-Refer to: [1741].

m.p. 74–75° [1741, 3083], UV [3083].

#### **Dimethyl ether**

m.p. 72° [1297].

# 1-(2,4-Dihydroxyphenyl)-1,4-pentanedione

 $\begin{array}{c} C_{11}H_{12}O_4 & \text{mol. wt. 208.21} \\ \\ OH & Synthesis \\ -Refer to: [2953]. \\ \textbf{Dimethyl ether} & [67756-19-8] \\ C_{13}H_{16}O_4 & \text{mol. wt. 236.27} \end{array}$ 

-Obtained from acetaldehyde and 1-(2,4-dimethoxyphenyl)-2-propen-1-one (73 %) [2953].

m.p. 63° [2953], ¹H NMR [2953], IR [2953].

mol. wt. 178.23

#### 1-(2,5-Dihydroxyphenyl)-1,3-pentanedione

$$\begin{array}{c} C_{11}H_{12}O_4 & \text{mol. wt. } 208.21 \\ \hline OH & Synthesis \\ \hline COCH_2COC_2H_5 & -Refer to: [3083]. \\ \hline Diethyl ether \\ C_{15}H_{20}O_4 & \text{mol. wt. } 264.32 \end{array}$$

-Obtained by reaction of 2,5-diethoxyacetophenone with ethyl propionate in the presence of sodium [3083].

-Refer to: [1741].

m.p. 49° [1741], 47–48° [3083], UV [3083].

#### 1-(2-Hydroxyphenyl)-1-pentanone

[18430-91-6]

C11H14O2

OH CO(CH₂)₃CH₃

Syntheses -Obtained by reaction of valeryl chloride with phenol in the presence of aluminium chloride at 125-130° for 1 h (56 %) [2700].

-Also obtained by reaction of valeric acid with phenol,

*in the presence of zinc chloride boiling for five min (by-product) [726];

*in the presence of zeolite [3266].

-Also obtained by treatment of 1-(2-hydroxyphenyl)-1-pentanol with manganese dioxide in methylene chloride for 7 h at r.t. (40 %) [77].

-Also obtained by photolysis of phenyl valerate in the presence of oxygen and  $\beta$ -cyclodextrin in aqueous solution for 1 h at 25° (20 %) [3216].

-Also obtained by Fries rearrangement of phenyl valerate with aluminium chloride, *at 140° for 45 min (37 %) [932];

*in refluxing carbon disulfide for 2 h, then at 130-140° for 3 h, after solvent elimination [2587];

*in refluxing petroleum ether under nitrogen for 3 h (36 %) [3255].

-Also obtained [2478] by the method [2074].

-Also refer to: [1331, 1681, 3156, 3464].

colourless oil [77]; b.p._{0.1} 74° [932]; b.p.₁₀ 130° [2700]; b.p.₁₅ 138–141° [2478]; ¹H NMR (Sadtler standard N° 38629M) [77, 3255], ¹³C NMR [77], IR (Sadtler standard N° 65678K) [77], UV [1996], MS [77, 3255]; TLC [1994].

#### 2,4-Dinitrophenylhydrazone [17744-53-5] C₁₇H₁₈N₄O₅ mol. wt. 358.35

m.p. 278–279° [524], 179° [2587], 178° [932].

**N.B.**: One of the reported melting point is obviously wrong.

# **Compound with \beta-cyclodextrin** (1:1) [264615-37-4]

-Refer to: [3215].

¹H NMR [3215], UV [3215].

Acetate	[97037-81-5]	$C_{13}H_{16}O_{3}$	mol. wt. 220.27
Acciaic	177057-01-51	$C_{311}_{6}C_{3}$	11101. Wt. 220.27

-Obtained by action of butylmagnesium bromide with acetylsalicoyl chloride in ether at  $0-5^{\circ}$  [2587].

-Also refer to: [1037].

b.p._{0.5} 113° [1037]; ¹H NMR [1037], IR [1037].

Methyl ether	[20359-54-0]	$C_{12}H_{16}O_2$	mol. wt. 192.26
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-Preparation by addition of the appropriate substituted phenyl Grignard to valeronitrile [3254].

-Also obtained by adding n-butyllithium in ether to 2-methoxybenzaldehyde o-methyloxime in THF at  $0^{\circ}$  for 1 h (80 %) [1476].

-Also obtained by hydrolysis of alkenyl sulfides (35 %) [3040].

-Also obtained from 2-methoxybenzaldehyde (4 steps) (91 %) [264].

-Also refer to: [1036, 1084, 1117, 1378, 1626, 2068, 2094, 2343, 2848, 3156].

yellow oil [264]; b.p._{0.1} 82° [1036]; b.p.₇ 142° [3254]; ¹H NMR [264, 1036, 1117, 1378, 1626, 2068, 2848], ¹³C NMR [1378, 1626], IR [264, 1036, 1378, 2068], UV [3254], MS [264, 1117, 1378, 3254]; ESR spectroscopy [3254], Phosphorescence spectroscopy [3254].

# 2,4-Dinitrophenylhydrazone of the methyl ether

 $\begin{array}{ll} \mbox{[64957-70-6]} & C_{18} H_{20} N_4 O_5 & \mbox{mol. wt. 372.38} \\ \mbox{m.p. 126.4-126.6}^\circ \mbox{[2068]}. \end{array}$ 

Benzyl ether	[127154-56-7]	$C_{18}H_{20}O_2$	mol. wt. 268.36
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-Obtained by treatment of the potassium salt with benzyl chloride in refluxing methanol for 2 h (53 %) [3255].

white platelets [3255]; m.p. 26–28° [3255]; ¹H NMR [3255], ¹³C NMR [3255], IR [3255], UV [3255], MS [3255]. 463

# 1-(3-Hydroxyphenyl)-1-pentanone

[62810-51-9]  $C_{11}H_{14}O_2$  mol. wt. 178.23 OH Syntheses -Synthesis of 3-hydroxyvalerophenone by means of organocadmium derivatives (75 %) [2586]. -Also obtained by treatment of its methyl ether with aluminium chloride in benzene by heating on a water bath for 2 h (80 %) [966].

-Also obtained in the same reaction that acetate below (25 %) [246]. -Also refer to: [1614, 1618].

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b.p.<sub>1.2</sub> 165° [2586]; m.p. 67° [966, 2586]; <sup>1</sup>H NMR [1614, 1618].
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-Also obtained (10) by reaction of 1-acetoxy-5-pentanoyl-1,3-cyclohexadiene tricarbonyl-iron complex 8 with triethylamine N-oxide in DMA at r.t. for 1 h (57 %) [246].

b.p.₁ 132–134° [2586].

Methyl ether	[20359-55-1]	$C_{12}H_{16}O_2$	mol. wt. 192.26
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-Obtained by condensation of butylmagnesium bromide with m-methoxybenzamide in ethyl ether. The reaction was carried out at  $60^{\circ}$  for 48 h under hydrogen atmosphere (60-70 %) [966].

-Also obtained by addition of n-butylmagnesium bromide to m-methoxybenzonitrile [3254].

-To lithium 3-methoxybenzoate, n-butyllithium in hexane was added and the mixture stirred for 2 h [273].

-Also obtained by introduction of 3-methoxybenzoyl chloride into n-butyllithium in ether at  $-78^{\circ}$ , previously added to cuprous iodide at  $0^{\circ}$ . After stirring 15 min at  $-78^{\circ}$  methanol was added and the mixture allowed to reach ambient temperature (79 %) [273].

-Also refer to: [1023, 1201, 1202, 1204, 1614, 1618, 2759, 3252].

b.p.₁ 105–108° [273]; b.p.₆ 134° [3254], b.p.₃₀ 190° [966, 967]; ¹H NMR [273, 1614, 1618], IR [273], UV [3254], MS [3254]; ESR spectroscopy [3254], Phosphorescence spectroscopy [3254].

USE: In preparation of platelet activating factor antagonists [1203].

# 2,4-Dinitrophenylhydrazone of the methyl ether $C_{18}H_{20}N_4O_5$ mol. wt. 372.38

m.p. 147° [967].

# 1-(4-Hydroxyphenyl)-1-pentanone

[2589-71-1]

 $C_{11}H_{14}O_2$ 

mol. wt. 178.23



Syntheses

-Phenol was energetically mixed with finely divided aluminium chloride (1 mol) and heated to  $100^{\circ}$  (1 mol of hydrochloric acid is evolved); 1 mol of n-valeroyl chloride was then added and heated to  $125-130^{\circ}$  for 1 h (29 %) [2700].

-Also obtained by reaction of valeroyl chloride with phenol in the presence of aluminium chloride,

*in methylene chloride for 14 h at r.t. (45 %) [1910];

*in nitrobenzene [2923], first at 5–10°, then at r.t. overnight (87 %) [686], (72 %) [2970].

-Also obtained by reaction of valeric acid with phenol,

*in the presence of zinc chloride (5 %) [726];

*in the presence of polyphosphoric acid for 15 min on a water bath (47 %) [2240] or for 10 min at  $100^{\circ}$  (58 %) [2238].

-Also obtained by Fries rearrangement of phenyl valerate,

*in the presence of polyphosphoric acid for 10 min at 100° (40 %) [2238];

*in the presence of aluminium chloride at  $140^{\circ}$  for 45 min (41 %) [932], in nitrobenzene [1116, 2923] or in refluxing carbon disulfide for 2 h, then at 130– $140^{\circ}$  for 3 h, after solvent elimination (22 %) [2587].

-Also obtained [2478] by the method [2074].

-Also obtained by photolysis of phenyl valerate in the presence of oxygen and  $\beta$ -cyclodextrin in aqueous solution for 1 h at 25° (6.4 %) [3216].

-Also obtained by reaction of 4-iodophenol with n-Bu₃In in the presence of Pd  $(PPh_3)_4$  in THF at 66° under atmospheric pressure of CO gas (64 %) [1851].

-Also refer to: [44, 62, 275, 556, 636, 868, 914, 1380, 1384, 1510, 1615–1617, 1936, 2043, 2732, 2861, 3165, 3254, 3379, 3454, 3471].

Isolation from natural sources

-Determination in coal extracts obtained from supercritical gas extraction by toluene [2330].

b.p._{0.6-0.8} 150-160° [686], b.p._{0.01} 156-158° [2970], b.p.₁ 160° [932];

b.p._{0.4} 167° [2587], b.p.₁₀ 197.5–198.5° [2700], b.p.₁₅ 210° [726]; b.p.₁₆ 213° [2478];

white solid [1910];

m.p.  $77^{\circ}$  [2240], 63–64° [2478], 63° (Sadtler standard  $N^{\circ}$  65672K) [2700],

62–64° [3254], 62–63° [726, 1910], 62° [932];

 1 H NMR [1910], (Sadtler standard N° 38623M),  13 C NMR [1910],

IR [1910], (Sadtler standard N° 65672K), UV [1995],

MS [1910, 2330]; X-ray data [1936]; GC-MS [2330]; TLC [1910, 1994]. Cryoscopic study [182].

ESR spectroscopy [3254], Phosphorescence spectroscopy [3254].

USE: Preparation of pyrovalerone analogs as monoamine uptake inhibitors [2043]. Synthesis of nonsteroidal antiinflamatory and analgesic drugs by Beckmann rearrangement of oximes with zeolites and mesoporus molecular sieve catalysts [684].

BIOLOGICAL ACTIVITY: Inhibition of  $17-\beta$ -hydroxysteroid dehydrogenase 3 [1910].

**Oxime** [864072-49-1] C₁₁H₁₅NO₂ mol. wt. 193.25

USE: Synthesis of nonsteroidal antiinflamatory and analgesic drugs by Beckmann rearrangement of oximes with zeolites and mesoporus molecular sieve catalysts [684].

2,4-Dinitrophenylhydrazone	$C_{17}H_{18}N_4O_5$	mol. wt. 358.35
m.p. 182° [932].		
Nicotinylhydrazone	$C_{17}H_{19}N_3O_2$	mol. wt. 297.36

m.p. 152° [520, 521].

USE: In chemotherapy of leprosy [520, 521].

isoNicotinylhydrazone	$C_{17}H_{19}N_3O_2$	mol. wt. 297.36	
m.p. 206° [520, 521].			
USE: In chemotherapy of leprosy [520, 521].			
<b>Compound with β-cyclodextrin</b> (1:	1) [264615-3	8-5]	

-Refer to: [3215].

¹H NMR [3215], UV [3215].

Benzyl ether	[35081-44-8]	$C_{18}H_{20}O_2$	mol. wt. 268.36
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-Preparation by reaction of benzyl chloride with p-hydroxyvalerophenone in the presence of potassium carbonate and potassium iodide in refluxing 90 % ethanol for 5 h (95 %) [556].

m.p. 65° [556].

Methyl ether	[1671-76-7]	$C_{12}H_{16}O_2$	mol. wt. 192.26
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-Obtained by direct acylation of 4-bromoanisole with valeraldehyde by palladium catalysis (91 %) [2668].

-Also obtained by reaction of valeryl chloride with anisole [1853],

*in the presence of aluminium chloride (100 %) [742] in 1,2-ethylene chloride [2942], first at 0° for 40 min, then at r.t. for 8–15 h (73 %) [2243];

*in the presence of Si-Fe catalyst at  $25^{\circ}$  (25 %) [427].

- -Also obtained by Et₂Zn-mediated rearrangement of a bromohydrin, the 2-bromo-1-(4-methoxy-phenyl)-1-pentanol (85 %) [1865].
- -Also obtained by reaction of valeric anhydride with anisole,
- *in the presence of aluminium chloride in boiling carbon disulfide for 30 min (85 %) [2297];
- *in the presence of microcrystalline beta zeolite-II for 3 h at  $130^{\circ}$  under argon (89 %) [1594];
- *in the presence of SbCl₅-LiClO₄ mixture in refluxing methylene chloride for 30 min (85 %) [2176].
- -Also obtained by reaction of methyl iodide with 4-hydroxyvalerophenone in the presence of potassium carbonate in refluxing acetone [2043].
- -Also obtained by reaction of dimethyl sulfate with 4-valerylphenol in the presence of aqueous sodium hydroxide for 4 h at reflux [2478].
- -Preparation from 4-bromoanisole and valeric anhydride using cobalt catalysis [1656].
- -Also obtained by reaction of 4-iodoanisole with n-Bu₃In in the presence of Pd  $(PPh_3)_4$  in THF at 66° under atmospheric pressure of CO gas (77 %) [1851].
- -Also obtained by oxidation of bis(4-methoxybenzyl)-1-butanol with bis (sym-collidine)bromine hexafluorophosphate (BBH) in methylene chloride at r.t. (83 %) [2659].
- -Also obtained from N-methoxy-N-methyl-2-pyridyl urethane and p-methoxyphenylmagnesium bromide (96 %) [1850].
- -Also obtained by reaction of 2-chloro-1-(trimethylsilyl)-1-pentanone with anisole in the presence of titanium tetrachloride (77 %) [1354].
- -Also obtained by adding n-butyllithium in ether to 4-methoxybenzaldehyde o-methyloxime in THF at  $0^{\circ}$  for 1 h (76 %) [1476].
- -Also obtained by reaction of n-butyllithium with 4-methoxybenzaldehyde in refluxing methylene chloride/toluene mixture for 16 h (56 %) [1329].
- **N.B.**: Toluene was added to the mixture which was kept at  $-78^{\circ}$  for 2 h after the addition of an aldehyde.
- -Preparation from organocopper reagent with n-butyryl chloride and 4-iodoanisole (63 %) [2622].
- -Also obtained by hydrolysis of alkenyl sulfides (55 %) [3040].
- -Also obtained by reaction of 4-methoxyphenyl n-pentyl dithiolane with  $P_2I_4$  in acetic anhydride for 9 h at r.t. (53 %) [2864].
- -Also refer to: [531, 619, 667, 698 (68 %), 944, 950, 970, 1023, 1200, 1202, 1249, 1266, 1849, 1941, 2106, 2240, 2279, 2322, 2357 (84 %), 2723, 2862, 2980, 3214, 3247, 3248, 3252, 3262, 3264, 3265, 3456, 3472].

yellow oil [2243]; oil [742];

b.p._{0.5} 113° [1354]; b.p.₂ 115° [1853]; b.p.₆ 143° [531, 3254]; b.p.₆ 150.5° [2297], b.p.₁₂ 162° [2240], b.p.₁₉ 175–178° [2478]; b.p.₇₃₉ 300° [2297]; m.p. 28° [2240], 27–28° [3214], 26° [2901], 22–23.5° [1103]; ¹H NMR [698, 1354, 1656, 1850, 1865, 2243, 2357, 2622], ¹³C NMR [698, 1354, 1656, 2357, 2622], IR [698, 1354, 1850, 2357, 2622], UV [2815, 3247, 3254], MS [698, 1656, 3254]; GC/MS [2357].

USE: Preparation of high purity 4-n-pentylphenol [3456]; Rhodium-catalyzed allylation of aldehydes with homoallylic alcs. by retroallylation and isomerization to satd. ketones with conventional or microwave heating [2980]; Preparation of pyrovalerone analogs as monoamine uptake inhibitors [2043].

#### 2,4-Dinitrophenylhydrazone of the methyl ether $C_{18}H_{20}N_4O_5$ mol. wt. 372.38

m.p. 154–155° [3214].

Propyl ether	$C_{14}H_{20}O_2$	mol. wt. 220.31
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-Obtained by reaction of valeryl chloride with phenyl propyl ether in the presence of zinc chloride at reflux for 30 min (44.5 %) [1237].

b.p.₁₅ 193–198° [1237]; m.p. 20–21° [1237].

Oxime of the propyl	ether	$C_{14}H_{21}NO_2$	mol. wt. 235.33
m.p. 58° [1237].			
Butyl ether	[101100-66-7]	$C_{15}H_{22}O_2$	mol. wt. 234.34
-Refer to: [3352].			
b.p.3 178° [1235];	m.p. 20° [1235].		
Pentyl ether	[93156-87-7]	$C_{16}H_{24}O_2$	mol. wt. 248.37
-Refer to: [3352].			
b.p.1 183° [1236];	m.p. 27° [1236].		
Hexyl ether	[93542-23-5]	$C_{17}H_{26}O_2$	mol. wt. 262.39
-Refer to: [3351, 335	2].		
b.p. ₈ 210° [1236];	m.p. 19° [1236].		

#### 4-Hydroxyphenylethyl ether

[109720-03-8], (CA **107**, 175432x); [113279-26-8] C₁₉H₂₂O₃ mol. wt. 298.38

-Obtained by reaction of 1-bromo-2-[4-(trimethylsiloxy)phenyl]ethane with 4-hydroxy-valerophenone in the presence of potassium carbonate in refluxing acetone for 17 h [2732].

-Also refer to: [1853].

m.p. 95–96° [2732]; ¹H NMR [2732], IR [2732].

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# 4-Methoxyphenylethyl ether

[109720-05-0], (CA **107**, 175432x); [113279-27-9]  $C_{20}H_{24}O_3$  mol. wt. 312.40 -Obtained by reaction of 4'-methoxy-2-phenethyl bromide with 4-hydroxyvalerophenone in the presence of potassium carbonate in refluxing acetone for 17 h [2732].

-Also refer to: [1853].

m.p. 49–50° [2732]; ¹H NMR [2732], IR [2732].

Trifluoromethyl ether[79619-25-3] $C_{12}H_{13}F_3O_2$ mol. wt. 246.23-Obtained by reaction of n-butylmagnesium bromide with p-(trifluoromethoxy)benzonitrile in ethyl ether at r.t. overnight [3251].

b.p._{4,5} 106–107° [3251]; ¹H NMR [3251], UV [3251], MS [3251].

N.B.: Electronic and phosphorescence spectrum and triplet energy [3251].

Ten various unsaturated ethers (photolysis of, triplet quenching in,) [3248].

<b>3-Butenyl ether</b>	[108919-69-3]	$C_{15}H_{20}O_2$	mol. wt. 232.32
5-Hexenyl ether	[108919-74-0]	$C_{17}H_{24}O_2$	mol. wt. 260.38
3-Methyl-3-butenyl ether	[108919-70-6]	$C_{16}H_{22}O_2$	mol. wt. 246.35
5-Methyl-4-hexenyl ether	[108919-68-2]	$C_{18}H_{26}O_2$	mol. wt. 274.40
4-Methyl-3-pentenyl ether	[108919-72-8]	$C_{17}H_{24}O_2$	mol. wt. 260.38
<b>3-Pentenyl ether</b>	[108919-67-1]	$C_{16}H_{22}O_2$	mol. wt. 246.35
<b>3-Pentenyl ether</b> (Z)	[108919-71-7]	$C_{16}H_{22}O_2$	mol. wt. 246.35
4-Pentenyl ether	[108919-73-9]	$C_{16}H_{22}O_2$	mol. wt. 246.35
2-Propenyl ether	[108919-66-0]	$\mathrm{C}_{14}\mathrm{H}_{18}\mathrm{O}_{2}$	mol. wt. 218.30
10-Undecenyl ether	[108919-75-1]	$C_{22}H_{34}O_2$	mol. wt. 330.51
Benzoate	$C_{18}H_{18}O_3$		mol. wt. 282.33
m n 92° [2700]			

m.p. 92° [2700].

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

[862666-33-9]  $C_{11}H_{14}O_3$  mol. wt. 194.23 OH HO CO(CH₂)₃CH₃ -Obtained by deprotection of its dimethyl ether with boron tribromide in methylene chloride at 0°. Then, the mixture was stirred overnight at r.t. (68 %) [82].

brown solid [82]; m.p. 50° [82]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82].

USE: Reactant for preparation of lithium gallium/titanium ketone complexes [82].

**Dimethyl ether** [15122-00-6] C₁₃H₁₈O₃ mol. wt. 222.28

-Obtained by treatment of 1-(2,3-dimethoxyphenyl)-1-pentanol in acetone with potassium dichromate in dilute sulfuric acid (Jones reagent) at r.t. for 3 h (23 %) [82]. -Obtained by oxidation of 1-(2,3-dimethoxyphenyl)-1-pentanol with sodium dichromate in dilute sulfuric acid (73–83 %) [2747]. -Also refer to: [82, 2747].

colourless oil [82]; pale yellow viscous oil [2747]; b.p._{0.08-0.1} 90.5–93.5° [2747]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82].

#### 2,4-Dinitrophenylhydrazone of the dimethyl ether

 $[15116-05-9] C_{19}H_{22}N_4O_6 mol. wt. 402.41$ 

m.p. 138-139° [2747].

#### 1-(2,4-Dihydroxyphenyl)-1-pentanone

[15116-13-9]	$C_{11}H_{14}O_3$	mol. wt. 194.23
HO CO(CH ₂ ) ₃ CH ₃	1 2	of valeric acid with resorcinol, n trifluoride [2382] for 2 h at
*in the presence of zinc chl (60 %) 2889].	oride [(51 %) 2294, (68–78	%) 2501, 2702, (82 %) 2747,

-Also refer to: [22, 893, 1628, 1629, 2114, 2842, 3127].

yellow viscous oil [2747]; b.p._{0.2} 111–114° [2747], b.p.₆₋₇ 190–192° [893, 2842]; m.p. 63° [2114, 2294, 2312], 62° [2889], 58.5–60° [893, 2842]; ¹H NMR [2889], IR [2382], UV [2382]; pK_a 5.39 [2889].

USE: As antioxidant for vitamin A [1628, 1629]; As fungicide for soy sauce [1053]; Antiseptic and germicidal product [2734]; Antifungal activity [2114]; In detn. of uranium by spectrophotometry [2891].

**Oxime** (*DHVOX*) [57991-55-6]  $C_{11}H_{15}NO_3$  mol. wt. 209.25

white solid [2890]; m.p. 170° [2890].

Metal complexes of 2,4-dihydroxyvalerophenone oxime [2890].

$Pd(C_{11}H_{14}NO_3)_2$	yellow	m.p. 250° (d)	IR, UV
$Cu(C_{11}H_{14}NO_3)_2$	buff	m.p. 245° (d)	IR, UV
$Ni(C_{11}H_{14}NO_3)_2$	light green	m.p. 230° (d)	IR, UV
$Co(C_{11}H_{14}NO_3)_2$	dark brown	m.p. 241° (d)	IR, UV
$Mn(C_{11}H_{14}NO_3)_2$	brownish black	m.p. 165° (d)	IR, UV
$VO(C_{11}H_{14}NO_3)_2$	black	m.p. 100° (d)	IR, UV
$UO_2(C_{11}H_{14}NO_3)_2$	orange	m.p. 180° (d)	IR, UV
$MoO_2(C_{11}H_{14}NO_3)_2$	orange yellow	m.p. 175° (d)	IR, UV
Semicarbazone	$C_{12}H_{17}N_3O_3$	mol	. wt. 251.29

m.p. 175° [2294].

Dimethyl ether	[854659-33-9]	$C_{13}H_{18}O_{3}$	mol. wt. 222.28
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-Obtained by treatment of the title ketone with methyl iodide [2294].

-Also obtained by hydrolysis of 4,6-dimethoxy-3-butylidenephthalide in acetone with concd. HCl, followed by the decarboxylation with Cu dust [2294].

m.p. 38.5° [2294].

# 1-(2,5-Dihydroxyphenyl)-1-pentanone

[4693-17-8]	$C_{11}H_{14}O_3$	mol. wt. 194.23
OH CO(CH ₂ ) ₃ CH ₃ OH	with valeryl chloride i chloride [1442]. -Also obtained by reactio	afts acylation of hydroquinone n the presence of aluminium n of valeric acid with hydroqui- zinc chloride (Nencki reaction)
-Also refer to: [1325, 2	2102, 2540, 2896, 2955, 32	04, 3394].

b.p.₃ 161–162° [1325], b.p.₄ 161–163° [150]; b.p.₂ 174–176° [2955];

b.p.₁₅ 180–185° [1442]; m.p. 62° [3204], 61–62.5° [2102].

Semicarbazone

 $C_{12}H_{17}N_3O_3$ 

mol. wt. 251.29

m.p. 189–190° [150].

mol. wt. 236.27

# 2-Acetate

# C₁₃H₁₆O₄

-Refer to: [1, 900].

Dimethyl ether	[38843-82-2]	$C_{13}H_{18}O_{3}$	mol. wt. 222.28
Dimentyl chici	[300+3-02-2]	$C_{13}I_{18}O_{3}$	11101. wt. 222.20

-Obtained by reaction of valeric acid with p-dimethoxybenzene, in the presence of polyphosphoric acid. The resulting deep was heated on the steam bath for 3 h (30 %) [2878].

-Also obtained by reaction of valeroyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride [3073],

*in methylene chloride at r.t. for 1 h (30 %) [2878]; *in carbon disulfide at 20–25° for 2.5 h (70 %) [2234]. -Also refer to: [2874, 2896].

pale amber oil [2878]; pale yellow liquid [2234]; b.p._{0.6} 142° [2234], b.p.₂₀ 185° [2874], b.p.₂₀ 188–192° [2878]; m.p. 63° [2874]; IR [2234, 2878].

C11H14O3

# 1-(2,6-Dihydroxyphenyl)-1-pentanone

[63411-80-3]	
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OH

Syntheses

CO(CH₂)₃CH₃ -Obtained from 2,6-dihydroxyacetophenone (54 %) [385]. -Also obtained by treatment of 4-methyl-7-hydroxy-8-nvalerylcoumarin with a 16 % aqueous sodium hydroxide solution at reflux for 4 h under nitrogen (88 %) [26].

-Also refer to: [32, 329].

m.p. 85–86° [26, 32], 85° [329], 78–79° [385].

**Dimethyl ether** 

b.p.₁₅ 172–175° [329].

# 1-(3,4-Dihydroxyphenyl)-1-pentanone

[2525-01-1]

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C_{11}H_{14}O_3
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C13H18O3

mol. wt. 194.23

mol. wt. 222.28

mol. wt. 194.23

OH OH CO(CH₂)₃CH₃ Syntheses

-Obtained by Fries rearrangement of guaiacol valerate with aluminium chloride in nitrobenzene (by-product) [726].

-Also obtained by Fries rearrangement of pyrocatechol divalerate with aluminium chloride in the presence of pyrocatechol for 4.5 h at  $135-140^{\circ}$  (50 %) [2075].

-Also obtained by Fries rearrangement of pyrocatechol valerate [3056]. -Also refer to: [27, 946, 1262]. b.p.₄ 210–220° [2075], b.p.₁₅ 230–240° [726], b.p.₁₀ 245–255° [3056]; m.p. 143–144° [726], 100–101° (dilute dioxane) or 93–94° (benzene) [2075], 99–100° [27], 97° [946, 1262]. **N.B.**: One of the reported melting point is obviously wrong.

**O-Methyloxime** [474668-86-5] C₁₂H₁₇NO₃ mol. wt. 223.27

-Refer to: [3177].

Dimethyl ether	[66053-97-2]	$C_{13}H_{18}O_{3}$	mol. wt. 222.28

-Obtained by Friedel-Crafts acylation of 3,4-dimethoxybenzene with pentanoic acid anhydride, Lewis acids and silver salts as catalysts [2180].

-Also obtained by reaction of valeryl chloride with veratrole in the presence of zinc chloride in refluxing carbon disulfide at  $60^{\circ}$  for 4 h (51.8 %) [2837].

-Also obtained from 3,4-dimethoxybenzaldehyde (4 steps) (92 %) [264].

-Also refer to: [1249, 1277, 1947, 2043, 2172, 2174, 2175, 2183, 2997, 2999].

viscous liquid [2837]; b.p._{0.05} 131° [1947]; b.p.₁ 160° [2837]; ¹H NMR [264, 1249, 1947, 2999], ¹³C NMR [1249, 2999], IR [264, 1249, 2999], MS [264].

USE: Preparation of pyrovalerone analogs as monoamine uptake inhibitors [2043].

Oxime of the dimethyl ether [100619-32-7]  $C_{13}H_{19}NO_3$  mol. wt. 237.30 -Refer to: [2183, 2837].

viscous oil [2837]; ¹H NMR [2182], ¹³C NMR [2182].

Syntheses

#### 

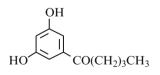
colourless needles [2837]; m.p. 179° [2837].

# 1-(3,5-Dihydroxyphenyl)-1-pentanone

[70627-61-1]

 $C_{11}H_{14}O_3$ 

mol. wt. 194.23



-Obtained by treatment of its diacetate with 5 % sodium hydroxide at reflux for 4.5 h (63 %) [1406]. -Also obtained by treatment of its dimethyl ether with

pyridinium chloride at 220° for 15 min (66 %) [2318]. -Also obtained by reaction of 3,5-diacetoxybenzoyl chloride with dibutylcadmium, followed by hydrolysis [227]. -Also obtained by reaction of n-butylmagnesium bromide with 3,5-bis-trimethylsilyloxy-benzamide in the presence of a crystal of iodine in refluxing ethyl ether for 48 h under nitrogen atmosphere (85 %) [259].

-The 3,5-dihydroxyphenyl *n*-butyl ketone was synthesized by the known method [1406] in four steps starting from 3,5-dihydroxybenzoic acid [3180].

-Also refer to: [1873 (Chinese paper)].

Isolation from natural sources

-From the roots and stems of Ardisia virens Kurz (Myrsinaceae) [607].

m.p. 119° [1406], 118° [3180], 116–117° [2318]; ¹H NMR [2318], IR [2318].

**2,4-Dinitrophenylhydrazone** [101593-70-8]  $C_{17}H_{18}N_4O_6$  mol. wt. 374.35

m.p. 247° (d) [1406].

**Diacetate** [101103-45-1] C₁₅H₁₈O₅ mol. wt. 278.30

-Preparation by reaction of dibutylcadmium with 3,5-diacetoxybenzoyl chloride in refluxing benzene for 1 h (82 %) [1406].

b.p. $_{0.1}$  146–153° [1406], b.p. $_{0.5}$  178–182° [1406]; n_D²⁰ = 1.5097 [1406].

#### 2,4-Dinitrophenylhydrazone of the diacetate

 $[112441-56-2] C_{21}H_{22}N_4O_8 mol. wt. 458.43$ 

m.p. 163° [1406].

Dimethyl ether	[5333-29-9]	$C_{13}H_{18}O_3$	mol. wt. 222.28
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-Provided by reductive alkylation (zinc amalgam) of 3,5-dimethoxybenzamide [1873 (Chinese paper)].

- -A reaction of 3,5-dimethoxybenzoic acid with lithium hydride gave a salt, which was acylated by butyllithium to synthesize 1-(3,5-dimethoxyphenyl)-1-pentanone [3357], in 83–85 % yield [227, 3356].
- -Also obtained by reaction of n-butylmagnesium bromide with N-trimethylsilyl 3,5-dimethoxy-benzamide in the presence of a crystal of iodine in refluxing ethyl ether for 72 h under nitrogen atmosphere (low yield) [259].
- -Also obtained by treatment of ethyl 3,5-dimethoxybenzoylpropylacetate with 6 % sulfuric acid for 1 h at 195° (82 %) [149].
- -Preparation by reaction of butylmagnesium bromide with 3,5-dimethoxybenzamide in refluxing ethyl ether for 5 h (80 %) [2990].
- -Also obtained by treatment of 3,5-dimethoxybenzoic acid with n-butyllithium at r.t. (95 %) [2900].
- -Also obtained by treatment of "methyläther-lobaritonsäure" with bronze in quinoline for 5 min at 215° [150].

-Preparation: n-butyl bromide was added to Li metal and a crystal of iodine in ether at 0°. Then, N,N-diethyl-3,5-dimethoxybenzamide in ether was added at  $-25^{\circ}$ , then maintained the temperature at  $-10^{\circ}$  for 2 h, and 5 h at 0° then at r.t. for 11 h (60.4 %) [1119].

-Oxidation potential of [33].

-Preparation from 1,3-dimethoxybenzene (92 %) [2792].

-Also refer to: [213 (19 %), 273 (84 %), 983, 1119, 1764, 1988 (11 %), 2209, 2575, 2793 (96 %), 2990, 3340, 3341].

pale yellow solid [2793]; b.p.₃ 140–142° [213], b.p._{0.3} 150° [273]; b.p.₁₁ 175–177° [2990]; m.p. 53° [2990], 42.5° [2990], 42–44° [227], 42–43° [33, 149, 150], 39–41° [1988], 38–39° [1119], 37–40° [259], 34–36° [273]; ¹H NMR [227, 259, 273, 983, 2209, 2793, 2900], ¹³C NMR [2209], IR [227, 983, 1119, 2209], MS [983].

USE: Preparation of cannabinoid derivatives as cannabinoid receptor agonists or antagonists [983].

# Dimethyl ether-1-¹⁴C

(Dimethoxy-3,5-phenyl)-1-pentanone-( 14 C-1) [54109-33-0]  $C_{12}^{(14)}$ CH₁₈O₃ mol. wt. 224.27 -Refer to: [2265]; ¹H NMR [2265], MS [2265].

# 1-(3,5-Dihydroxyphenyl)-1-pentanone-1-¹³C

	$C_{10}^{(13)}CH_{14}O_3$	mol. wt. 195.22
ОН	Synthesis	
$\wedge$	-Refer to: [2507].	
	<b>Dimethyl ether</b> [98631-68-6]	
HO ¹³ COC ₄ H ₉	$C_{12}^{(13)}CH_{16}O_3$	mol. wt. 222.28

-Obtained by reaction of [carboxy-¹³C]-3,5-dimethoxybenzoic acid with n-butyllithium (98 %) [2507]. -Also refer to: [149].

m.p. 42–43° [149], 39–41° [2507].

# 1-(2,3,4-Trihydroxyphenyl)-1-pentanone

[43043-25-0]	$\mathrm{C}_{11}\mathrm{H}_{14}\mathrm{O}_{4}$	mol. wt. 210.23
HO HO HO	Syntheses -Obtained by reaction of valeric aci in the presence, *of zinc chloride [214, 2678], at 1 (70 %) [506];	

*of boron trifluoride in ethyl ether at  $0^{\circ}$  for 1 h (88 %) [540]. -Also refer to: [1260].

yellow needles [1260]; b.p.₁₄ 202–204° [506]; m.p. 108° [214, 2678], 84–84.5° [1260], 82° [540]; UV [540].

# 1-(2,4,5-Trihydroxyphenyl)-1-pentanone

$$[62060-62-2] C_{11}H_{14}O_4$$
mol. wt. 210.23  
OH CO(CH₂)₃CH₃ Syntheses  
-Obtained by reaction of valeryl chloride with  
1,2,4-trihydroxybenzene in the presence of aluminium  
chloride in nitrobenzene at r.t. for several hrs. and  
heated 0.5 h at 65° [290].

-Also refer to: [771, 1508].

m.p. 151–153° [290]; ¹³C NMR [1508].

USE: Antioxidant in fats and oils [290].

	Trimethyl ether	[90834-05-2]	$C_{14}H_{20}O_{4}$	mol. wt. 252.31
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-Obtained by reaction of pentanoyl chloride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride (60–74 %) [771].

-Also obtained by reaction of valeric acid with 1,2,4-trimethoxybenzene in the presence of polyphosphoric acid for 4 h at  $45-50^{\circ}$  [2695].

-Also obtained by reaction of valeric anhydride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride in methylene chloride at  $60^{\circ}$  for 1 h (60 %) [772].

-Also refer to: [2696].

white powder [772]; m.p. 62–64° [772], 62–63° [2695]; ¹H NMR [772, 2695], ¹³C NMR [772], IR [772, 2695], MS [772, 2695].

 $C_{11}H_{14}O_4$ 

# 1-(2,4,6-Trihydroxyphenyl)-1-pentanone

(Phlorovalerophenone)

[2999-18-0]

. . . . .

mol. wt. 210.23

Syntheses

HO CO(C

OH

CO(CH₂)₃CH₃ -Obtained by reaction of pentanoyl chloride with phloroglucinol in the presence of aluminium chloride, *in nitrobenzene for 3 days at r.t. (60–70 %) [421];

*in nitrobenzene and carbon disulfide mixture (58 %) [2113];

*in nitrobenzene and methylene chloride mixture [2580].

-Also obtained by adding valeronitrile to a mixture of phloroglucinol and zinc chloride in ether at  $0^{\circ}$ , hydrogen chloride fed 7–8 h, and the mixture kept overnight at r.t. [2110, 2111, 2531].

-Also obtained by reaction of valeric anhydride with phloroglucinol in the presence of boron trifluoride etherate (70–80 %) [2014].

-Also obtained by acid hydrolysis of its 2- $\beta$ -D-glucopyranoside in methanol in the presence of 1 N HCl at reflux for 4 h [209].

-Also refer to: [56, 708, 1026, 1375, 1439, 1917, 1942, 1992, 3202, 3297].

# Isolation from natural sources

-In bud exudate of *Populus cathayana* (Salicaceae) [1163]. -In bud exudate of *Populus koreana*, *Populus maximowiczii* and *Populus suaveolens* (Salicaceae) [1164].

m.p. 155–157° [209], 152° [2113], 149–150° [1439], 149° [421, 3297]; ¹H NMR [209, 421], IR [421], MS [209, 421]; GLC [2531]; GC-MS [1163, 1164].

BIOLOGICAL ACTIVITY: Antifungal [2113].

# 1-(3,4,5-Trihydroxyphenyl)-1-pentanone

$$\begin{array}{cccc} & C_{11}H_{14}O_4 & & \mbox{mol. wt. 210.23} \\ & & OH & Synthesis \\ & HO & OH & -Refer to: [151]. \\ & & Trimethyl \ ether & [114085-80-2] \\ & & C_{14}H_{20}O_4 & & \mbox{mol. wt. 252.31} \end{array}$$

-Obtained by oxidation of 1-(3,4,5-trimethoxyphenyl)-1-pentanol (81 %) [917].

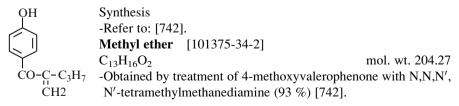
-Also obtained by reaction of butylmagnesium bromide with 3,4,5-trimethoxybenzoyl chloride in the presence of zinc chloride in benzene at  $20^{\circ}$  for 12 h (90 %) [211, 212].

-Also refer to: [149, 151, 1426].

yellow oil [917]; b.p._{0.4} 152° [211], b.p.₄ 174–175° [151], b.p.₄ 174° [149]; m.p. 27° [151]; ¹H NMR [211, 917], ¹³C NMR [917], MS [917].

# 1-(4-Hydroxyphenyl)-2-methylene-1-pentanone

 $C_{12}H_{14}O_2$  mol. wt. 190.24



-Also obtained from 2-bromo-1-(4-methoxyphenyl)-2-methyl-1-propanone [742].

liquid (93 %) [742]; ¹H NMR [742].

#### 1.2 Substituted Hydroxyketones

#### 1-(3-Bromo-5-fluoro-2-hydroxyphenyl)-1-pentanone

[1813-21-4]  $C_{11}H_{12}BrFO_2$ mol. wt. 275.12 **Synthesis** OH CO(CH₂)₃CH₃ -Obtained by Fries rearrangement of 2-bromo-4-fluoro-phenyl valerate with aluminium chloride at 130–140° for 3 h (70 %) [1550]. b.p.4.5 145-150° [1550].

**2,4-Dinitrophenylhydrazone** [1644-98-0] C₁₇H₁₆BrFN₄O₅ mol. wt. 455.24

-Refer to: [1550].

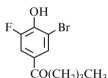
m.p. 183–184° [1550].

#### 1-(3-Bromo-5-fluoro-4-hydroxyphenyl)-1-pentanone

[455-53-8]

 $C_{11}H_{12}BrFO_2$ 

mol. wt. 275.12



Synthesis -Obtained by reaction of bromine with 3-fluoro-4-hydroxyvalerophenone in acetic acid [516]. m.p. 87° [516].

# 1-(3,5-Dibromo-2-hydroxyphenyl)-1-pentanone

[22362-69-2]	$C_{11}H_{12}Br_2O_2$	mol. wt. 336.02
Br Br Br	Syntheses -Refer to: [647, 2002]. m.p. 76° (Sadtler standard N° 650 ¹ H NMR (Sadtler standard N° 38 IR (Sadtler standard N° 65680K)	631M),

# 1-(3,5-Dibromo-4-hydroxyphenyl)-1-pentanone

[5408-44-6]	$C_{11}H_{12}Br_2O_2$	mol. wt. 336.02
Br Br	Syntheses -Obtained by reaction of bromine valerophenone [1995].	with 4-hydroxy-
Ý	-Also refer to: [516].	
CO(CH ₂ ) ₃ CH ₃	m.p. 75° [516], 70° (Sadtler standard N°	65676K);
1		

¹H NMR (Sadtler standard N° 38627M), IR (Sadtler standard N° 65676K).

# 1-(3-Chloro-5-fluoro-2-hydroxyphenyl)-1-pentanone

[1813-22-5]	$C_{11}H_{12}ClFO_2$	mol. wt. 230.67
$\begin{array}{c} OH\\ CI \\ F\\ F\end{array} CO(CH_2)_3CH_3\\ F\end{array}$	Synthesis -Obtained by Fries rearrangement of phenyl valerate with aluminium of for 3 h (78 %) [1550]. b.p. ₁ 140–141° [1550].	

**2,4-Dinitrophenylhydrazone** [2193-04-6] C₁₇H₁₆ClFN₄O₅ mol. wt. 410.79

-Refer to: [1550].

m.p. 159° [1550].

# 1-(2,3-Dichloro-4-hydroxyphenyl)-1-pentanone

[55507-79-4]	$C_{11}H_{12}Cl_2O_2$	mol. wt. 247.12
ОН	Syntheses	
Cl	-Refer to: [7, 8, 342, 739].	
	m.p. 107–110° [739].	
¥∕C1	Methyl ether [101375-31-9]	
CO(CH ₂ ) ₃ CH ₃	$C_{12}H_{14}Cl_2O_2$	mol. wt. 261.15

-Preparation by reaction of valeroyl chloride with 2,3-dichloroanisole in the presence of aluminium chloride (84 %) [742]. -Also refer to: [342, 343].

m.p. 61–63° [742]; ¹H NMR [742].

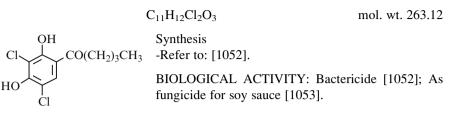
# 1-(3,4-Dichloro-2-hydroxyphenyl)-1-pentanone

[196307-75-2]	$C_{11}H_{12}Cl_2O_2$	mol. wt. 247.12
Cl CO(CH ₂ ) ₃ CH ₃	Synthesis -Refer to: [949]. ¹³ C NMR [949].	

# 1-(3,5-Dichloro-2-hydroxyphenyl)-1-pentanone

	$C_{11}H_{12}Cl_2O_2$			mol. wt. 24	7.12
Cl Cl Cl Cl Cl Cl	Synthesis -Obtained 2,4-dichloro at 170° for 4 m.p. 46–47°	phenyl 40 min	valerate v	vith aluminium chlo	of oride

#### 1-(3,5-Dichloro-2,4-dihydroxyphenyl)-1-pentanone



# 1-(2,3-Difluoro-4-hydroxyphenyl)-1-pentanone

	$C_{11}H_{12}F_2O_2$	mol. wt. 214.21
ŌН	Synthesis	
F	-Refer to: [1161].	
	Methyl ether [134364-70-8]	
Ϋ́F	$C_{12}H_{14}F_2O_2$	mol. wt. 228.24
CO(CH ₂ ) ₃ CH ₃	-Refer to: [1161].	

#### 1-(4-Bromo-2-hydroxyphenyl)-1-pentanone

[189875-21-6]	$\mathrm{C}_{11}\mathrm{H}_{13}\mathrm{BrO}_2$	mol. wt. 257.13
OH CO(CH ₂ ) ₃ CH ₃	Synthesis -Refer to: [2340].	
Br		

#### 1-(3-Bromo-4-hydroxyphenyl)-1-pentanone

 $C_{11}H_{13}BrO_2$ [67548-61-2] mol. wt. 257.13

ОН	Syntheses
Br	-Obtained (by-product) by reaction of valeryl chloride with
Br	2-bromoanisole in the presence of aluminium chloride
$\mathbf{i}$	( <b>XX</b> ) [1334].
CO(CH ₂ ) ₃ CH ₃	-Also refer to: [1673, 1798].

m.p. 101° [1334], 100–101° [1673].

**BIOLOGICAL ACTIVITY:** Nematocide [1798].

Methyl ether [859968-25-5]  $C_{12}H_{15}BrO_2$ mol. wt. 271.15

-Preparation by reaction of valeryl chloride with 2-bromoanisole in the presence of aluminium chloride (XI) [1334].

-Also refer to: [1160].

m.p. 56–57° [1160], 49° [1334].

#### **Phenylhydrazone of the methyl ether** $C_{18}H_{21}BrN_2O$

# $C_{18}H_{21}BrN_2O$ mol. wt. 361.28

m.p. 124° [1334].

# 1-(5-Bromo-2-hydroxyphenyl)-1-pentanone

$$[67548-62-3] C_{11}H_{13}BrO_2 mol. wt. 257.13$$

OH CO(CH₂)₃CH₃ Br

Syntheses -Obtained by Fries rearrangement of 4-bromophenyl valerate in the presence of aluminium chloride [1701, 2797], in tetrachloroethane at 120° for 30 min (45 %) [2026]. -Also refer to: [1798, 2202].

needles [2026]; m.p. 30° [2026]; b.p.₁ 120–121° [2026], b.p.₂ 127–136° [1701]; TLC [2026].

BIOLOGICAL ACTIVITY: Nematocide [1798].

**2,4-Dinitrophenylhydrazone** [101876-10-2]  $C_{17}H_{17}BrN_4O_5$  mol. wt. 437.25

m.p. 231° [2026].

# 1-(4-Bromo-2,5-dihydroxyphenyl)-1-pentanone

Semicarbazone [52376-26-8] C₁₂H₁₆BrN₃O₃ mol. wt. 330.18

-Refer to: [1105].

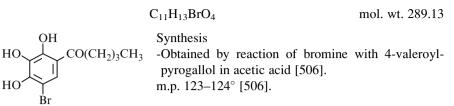
m.p. 208-209° [1105].

#### 1-(5-Bromo-2,4-dihydroxyphenyl)-1-pentanone

	$C_{11}H_{13}BrO_3$	mol. wt. 273.13
OH ↓ ,CO(CH₂)₃CH₃	Syntheses -Refer to: [2416, 2417].	
	-Refer to: [2416, 2417]. Oxime [235103-27-2]	1 4 200.00
Br	$C_{11}H_{14}BrNO_3$	mol. wt. 288.08

USE: Gravimetric reagent for Ni(II) and Cu(II) and spectrophotometric study of the complexes [2416]; Gravimetric reagent for Pd(II) and Mn(II) and spectrophotometric study of the complexes [2417].

#### 1-(5-Bromo-2,3,4-trihydroxyphenyl)-1-pentanone



# 1-(2-Chloro-4-hydroxyphenyl)-1-pentanone

	$C_{11}H_{13}ClO_2$	mol. wt. 212.68
Н	Synthesis	
∠C1	-Refer to: [3335].	
J CI	b.p. ₂ 190–192° [3335].	
O(CH ₂ ) ₃ CH ₃		

#### 1-(3-Chloro-4-hydroxyphenyl)-1-pentanone

[34190-36-8]	$C_{11}H_{13}ClO_2$	mol. wt. 212.68
$\bigcup_{CO(CH_2)_3CH_3}^{OH}$	Syntheses -Obtained (by-product) by reaction 2-chloroanisole in the presence ( <b>XVII</b> ) [1334]. -Also refer to: [1673, 3335].	-

b.p.₄ 180–184° [3335]; m.p. 97–98° [3335], 97° [1334], 96–97° [1673].

# Methyl ether

-Refer to: [1160].

m.p. 58–59° [1160].

# 1-(4-Chloro-2-hydroxyphenyl)-1-pentanone

[27581-18-6] OH

 $C_{11}H_{13}ClO_2$ 

mol. wt. 212.68

mol. wt. 226.70

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Syntheses
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CO(CH₂)₃CH₃ -Preparation by Fries rearrangement of m-chlorophenyl valerate with aluminium chloride, *for 3 h at 140–150° (90 %) [2432]; *for 1 h at 165° (50 %) [3335].

-Also refer to: [2428, 2429].

b.p.12 160-164° [3335], b.p.20 165° [2432]; ¹H NMR [2432], IR [2432].

 $C_{12}H_{15}ClO_2$ 

# 1-(5-Chloro-2-hydroxyphenyl)-1-pentanone

[209462-25-9]	$C_{11}H_{13}ClO_2$	mol. wt. 212.68
OH CO(CH ₂ ) ₃ CH ₃	Syntheses -Obtained by Fries rearrangement of a ate with aluminium chloride [3170]. -Also refer to: [1702, 1945, 3283]. b.p. _{0.9} 107° [3170], b.p. ₂ 120–125° [1 m.p. 28° [3170]; UV [3170].	

# 1-(5-Chloro-2,4-dihydroxyphenyl)-1-pentanone

[101043-59-8]	$C_{11}H_{13}ClO_3$	mol. wt. 228.68
OH CO(CH ₂ ) ₃ CH ₃	Synthesis -Refer to: [1479].	
HOCI	USE: Recording material contg. enca donating dye and, as colour develop	-

# 1-(2-Fluoro-4-hydroxyphenyl)-1-pentanone

	$C_{11}H_{13}FO_2$	mol. wt. 196.22
ОН	Synthesis	
$\checkmark$	-Refer to: [2437].	
	Methyl ether [80222-35-1]	
$\searrow$ _F	$C_{12}H_{15}FO_2$	mol. wt. 210.25
CO(CH ₂ ) ₃ CH ₃	-Refer to: [2437].	

# 1-(3-Fluoro-4-hydroxyphenyl)-1-pentanone

[350-26-5]	$C_{11}H_{13}FO_2$	mol. wt. 196.22
OH F CO(CH ₂ ) ₃ CH ₃	Syntheses -Obtained by refluxing 3-fluoro-4-methoxyva pyridinium chloride for 15 min [516]. -Also refer to: [3150]. m.p. 49° [516].	lerophenone with

isoNicotinylhydrazone [449-31-0] C₁₇H₁₈FN₃O₂ mol. wt. 315.35

m.p. 228° [520, 521].

USE: In chemotherapy of leprosy [520, 521].

Methyl ether [586-20-9]  $C_{12}H_{15}FO_2$ mol. wt. 210.25

-Obtained by Friedel-Crafts acylation of 2-fluoroanisole with valeroyl chloride in the presence of aluminium chloride in carbon disulfide (75-85 %) [516]. -Also refer to: [2437, 3260].

liquid [516]; b.p.₃₂ 198–201° [516];  $n_D^{28} = 1.5350$  [516].

# 1-(4-Fluoro-2-hydroxyphenyl)-1-pentanone

	$C_{11}H_{13}FO_2$	mol. wt. 196.22
OH CO(CH ₂ ) ₃ CH ₃	Synthesis -Refer to: [2437].	
F	<b>Methyl ether</b> [80222-34-0] C ₁₂ H ₁₅ FO ₂	mol. wt. 210.25

-Refer to: [2437].

# 1-(5-Fluoro-2-hydroxyphenyl)-1-pentanone

[319-30-2]	$C_{11}H_{13}FO_2$	mol. wt. 196.22
$\bigcup_{F}^{OH} CO(CH_2)_3CH_3$	Synthesis -Obtained by Fries rearrangemer ate with aluminium chloride at b.p. ₁₄ 131–135° [2991]; m.p. 7	150° [2991].

# 1-(4-Hydroxy-2-iodophenyl)-1-pentanone

	$C_{11}H_{13}IO_2$	mol. wt. 304.12
ОН	Synthesis	
$\checkmark$	-Refer to: [608].	
	Methyl ether [447439-58-9]	
×_I	$C_{12}H_{15}IO_2$	mol. wt. 318.15
CO(CH ₂ ) ₃ CH ₃	-Refer to: [608, 2573].	

# 1-(4-Hydroxy-3-nitrophenyl)-1-pentanone



Synthesis

NO₂ -Obtained by reaction of valeryl chloride with 2-nitrophenol in the presence of aluminium chloride in nitrobenzene, first at 55–60° for 2.5 h, then at r.t. overnight (34 %) [465]. CO(CH₂)₃CH₃ m.p. 27.4–28.2° [465].

2,4-Dinitrophenylhydrazone	C ₁₇ H ₁₇ N ₅ O ₇	mol. wt. 403.35
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-Refer to: [465].

m.p. 167.8–168.4° [465].

**Methyl ether** [1032174-12-1] C₁₂H₁₅NO₄ mol. wt. 237.25

-Obtained by adding potassium nitrate to an ice-cold solution of 4-methoxyvalerophenone in sulfuric acid, then the reaction was run at r.t. for 8–15 h (63 %) [2243].

-Also refer to: [3451].

white crystalline solid [2243]; m.p. 81–82° [2243]; ¹H NMR [2243].

#### 1-(3,4-Dihydroxy-2-nitrophenyl)-1-pentanone

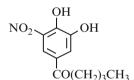
 $\begin{array}{cccc} [383383-01-5] & C_{11}H_{13}NO_5 & \text{mol. wt. } 239.23 \\ OH & Synthesis \\ -Refer to: [1842]. \\ USE: As COMT inhibitor for treatment of central and peripheral \\ nervous system disorders [1842]. \end{array}$ 

# 1-(3,4-Dihydroxy-5-nitrophenyl)-1-pentanone

[125628-93-5]

 $C_{11}H_{13}NO_5$ 

mol. wt. 239.23



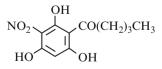
[119691-95-1]

OH Syntheses -Refer to: [322, 323, 429]. pK_a [429].

Synthesis

# 1-(2,4,6-Trihydroxy-3-nitrophenyl)-1-pentanone

 $C_{11}H_{13}NO_6$  mol. wt. 255.23



 $\begin{array}{rl} \text{CO}(\text{CH}_2)_3\text{CH}_3 & \text{-Obtained by adding hexane, then a mixture of concentrated sulfuric acid and fuming nitric acid at 0° to a solution of 1-(2,4,6-trihydroxyphenyl)-1-pentanone in concentrated sulfuric acid below 0° (70–80 %) [3114]. \end{array}$ 

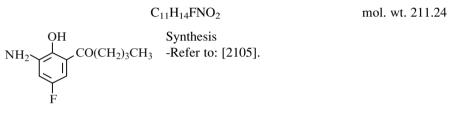
bright yellow needles [3114]; m.p. 81–84° [3114]; ¹H NMR [3114], IR [3114], MS [3114].

BIOLOGICAL ACTIVITY: Germination inhibition [3114]; PET inhibition [3114].

# 1-(3-Amino-5-chloro-2-hydroxyphenyl)-1-pentanone

$$C_{11}H_{14}CINO_{2}$$
mol. wt. 227.69  
NH₂  $\downarrow$   $\downarrow$  CO(CH₂)₃CH₃ -Refer to: [2105].

# 1-(3-Amino-5-fluoro-2-hydroxyphenyl)-1-pentanone



# 1-(2-Amino-5-hydroxyphenyl)-1-pentanone

	$C_{11}H_{15}NO_2$	mol. wt. 193.25
ŌН	Synthesis	
$\checkmark$	-Refer to: [710].	
	Methyl ether [90033-66-2]	
CO(CH ₂ ) ₃ CH ₃	$C_{12}H_{17}NO_2$	mol. wt. 207.27
NH ₂	-Refer to: [710].	

# 1-(5-Amino-2-hydroxyphenyl)-1-pentanone

[497934-63-1]	$C_{11}H_{15}NO_2$	mol. wt. 193.25
ОН	Syntheses	
CO(CH ₂ ) ₃ CH ₃	-Obtained by treatment of paracetamol	valerate with Lewis
	acid catalyst in organic solvent, the	en deacetylating the
Ŷ	4-hydroxy-3-valeroylacetamide obta	ined with an inor-
NH ₂	ganic acid at refluxing temperature [3	6464].

-Also refer to: [3281].

# 1-(3,4-Methylenedioxyphenyl)-1,4-pentanedione

# 1-(3,5-Dichloro-2,4-dihydroxy-6-methoxyphenyl)-1-pentanone

[118191-27-8]	$C_{12}H_{14}Cl_2O_4$	mol. wt. 293.15
$\begin{array}{c} OH \\ Cl \\ HO \\ Cl \\ Cl \\ Cl \\ \end{array} CO(CH_2)_3CH_3 \\ CO(CH_3)_3CH_3 \\ Cl \\ C$	Synthesis -Obtained by reaction of chlorine 6-methoxyvalerophenone in wat ¹ H NMR [2012], MS [2012].	

BIOLOGICAL ACTIVITY: Differentiation-inducing factor from the slime mould *Dictyostelium discoideum* [2012].

# 1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-1-pentanone (*DIF-1*) [-1]

[113411-16-8]

 $C_{12}H_{14}Cl_2O_4$  mol. wt. 293.15

Syntheses

 $CI \rightarrow CO(CH_2)_3CH_3$   $CH_3O \rightarrow CI \rightarrow OH$ 

CO(CH₂)₃CH₃ -Preparation by adding a solution of sulfuryl chloride (1.5 equiv.) in ethanol to a solution of 2,6-dihydroxy-4-methoxyvalerophenone in chloroform at r.t.; then, the solution was stirred for 1 h at r.t. [1129].

-Also obtained by reaction of chlorine with 2,6-dihydroxy-4-methoxyvalerophenone in water [2012].

-Also refer to: [1653, 1772, 1773, 1804, 2011, 3153].

yellow amorphous solid [1129]; ¹H NMR [2012], MS [1129, 2012]; GC [2154].

BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773]; Differentiation-inducing factor-1 and -2 function also as modulators for Dictyostelium chemotaxis [1804]; Structural requirements of Dictyostelium differentiationinducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129]; Differentiationinducing factor from the slime mould *Dictyostelium discoideum* [2012]; Cell differentiation regulation by, in *Dictyostelium discoideum* [1653]; Differentiation-inducing factor, from *Dictyostelium discoideum*, characterization of, [2011].

#### 1-(2-Hydroxy-5-methylphenyl)-1,3-pentanedione

[104516-35-0] mol. wt. 206.24 C12H14O3 Syntheses OH COCH₂COCH₂CH₃ -Obtained by heating a mixture of 2-acetyl-4-methylphenol, ethyl propionate (b.p. 98.5–100°) and powdered sodium on the steam bath for 30 min CH₃ (57 %) [235]. -Also refer to: [1011, 2745, 2746].

colourless, thick, rectangular prisms [235]; m.p. 75–76° [235].

# 1-(2,4-Dihydroxy-6-methylphenyl)-1,3-pentanedione

	$C_{12}H_{14}O_4$	mol. wt. 222.24
HO COCH ₂ COC ₂ H ₅	Synthesis -Obtained by refluxing phenone, propionic anhy nate at 180–190° for 9 h	a mixture of orcaceto- ydride and sodium propio- [2814].

dark brown viscous oil [2814].

<b>Dimethyl ether</b> [62036-47-9] $C_{14}H_{18}O_4$ mol. wt. 250	0.29
-------------------------------------------------------------------	------

-Obtained by Claisen condensation of 2,4-dimethoxy-6-methylacetophenone with ethyl propionate (11 %) [49].

-Refer to: [2003, 2814].

oil [49]; b.p.₂₋₄ 185–190° [2814].

# 2,4-Dihydroxy-6-(1-oxopentyl)benzaldehyde

	$C_{12}H_{14}O_4$	mol. wt. 222.24
он Сно	Synthesis -Refer to: [1986].	
HO CO(CH ₂ ) ₃ CH ₃	Dimethyl ether	mol. wt. 250.29

-Refer to: [1986].

¹H NMR [1986], MS [1986].

#### 3,5-Dihydroxy-2-(1-oxopentyl)benzaldehyde

$$\begin{array}{c} C_{12}H_{14}O_4 \\ OH \\ OH \\ CO(CH_2)_3CH_3 \\ HO \\ CHO \\ CHO$$

-Refer to: [1986].

¹H NMR [1986].

# 4-Hydroxy-3-valeroylbenzoic acid

[20031-96-3]	$C_{12}H_{14}$	$O_4$	mol. wt. 222.24
OH CO(CH ₂ ) ₃ CH ₃		olysis of its ethyl ester	[967].
	m.p. 190° [1], 184 Acetate	$C_{14}H_{16}O_5$	mol. wt. 264.28
СООН			

-Obtained by acetylation of 4-hydroxy-3-valeroylbenzoic acid [967].

m.p. 101° [967].

# Methyl ether $C_{13}H_{16}O_4$ mol. wt. 236.27

-Obtained by methylation of 4-hydroxy-3-valeroylbenzoic acid [967].

m.p. 154° [967].

Methyl ester	[20035-78-3]	$C_{13}H_{16}O_4$	mol. wt. 236.27

m.p. 86–87° [1], 78–80° [900].

#### Ethyl ester

C₁₄H₁₈O₄ mol. wt. 250.29

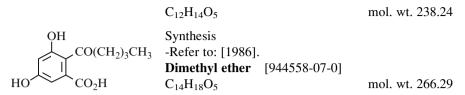
-Obtained by reaction of valeroyl chloride with ethyl 4-hydroxybenzoate in the presence of aluminium chloride in tetrachloroethane, then the reaction mixture was heated at  $120^{\circ}$  for 3–4 h [967].

b.p.₃₀ 190° [967];  $n_D^{34} = 1.5220$  [967];  $d_{28} = 1.108$  [967].

mol. wt. 238.24

mol. wt. 226.70

#### 3,5-Dihydroxy-2-(1-oxopentyl)benzoic acid



-Refer to: [1986, 2294].

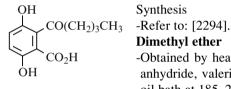
m.p. 99–100° [1986], 98° [2294]; ¹H NMR [1986], MS [1986].

**BIOLOGICAL ACTIVITY: Ecotoxicology** [1986].

#### 3,6-Dihydroxy-2-(1-oxopentyl)benzoic acid

C12H14O5 mol. wt. 238.24

Synthesis



**Dimethyl ether**  $C_{14}H_{18}O_5$ mol. wt. 266.29 -Obtained by heating a mixture of 3,5-dimethoxyphthalic anhydride, valeric anhydride and sodium valerate on the oil bath at 185–210° for 2 h [2294].

cooling in an ice bath for 30 min (70 %) [421].

m.p. 134° [2294].

# 2,4,6-Trihydroxy-3-(1-oxopentyl)benzaldehyde

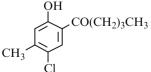
[96573-31-8]	$C_{12}H_{14}O_5$	mol. wt. 238.24
ОН	Synthesis	
CHO CO(CH ₂ ) ₃ CH ₃	-Obtained by reaction of eth	yl orthoformate with
	2,4,6-trihydroxyvalerophenor	ne in the presence of
ностон	aluminium chloride in met	hylene chloride with

m.p. 135–137° [421]; ¹H NMR [421], IR [421], MS [421].

BIOLOGICAL ACTIVITY: As photosynthetic electron transport (PET) [3405].

#### 1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-pentanone

C₁₂H₁₅ClO₂ Synthesis



CO(CH₂)₃CH₃ -Refer to: [3138]. Fluorescence [3138].

# **1-(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)-1-pentanone** (*DIF-3*) (-1)

 $[120529-47-7] C_{12}H_{15}ClO_4 mol. wt. 258.70$  OH Syntheses  $Cl + CO(CH_2)_3CH_3 -Preparation by adding a solution of sulfuryl$ 

chloride (1.5 equiv.) in ethanol to a solution of 2,6-dihydroxy-4-methoxyvalerophenone in chloroform at r.t.; then, the solution was stirred for 1 h at r.t. [1129].

-Also refer to: [1772, 2153].

OН

CH₃O

colourless amorphous solid [1129]; MS [1129].

BIOLOGICAL ACTIVITY: Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

# 1-(4-Hydroxy-3-methoxy-2-nitrophenyl)-1-pentanone

 $\begin{array}{cccc} [383383-10-6] & C_{12}H_{15}NO_5 & \text{mol. wt. } 253.25 \\ OH & Synthesis \\ & & -\text{Refer to: } [1842]. \\ & & & \text{USE: As COMT inhibitor for treatment of central and peripheral} \\ & & & \text{nervous system disorders } [1842]. \end{array}$ 

# 1-[3-(Aminomethyl)-5-bromo-4-hydroxyphenyl]-1-pentanone

	$C_{12}H_{16}BrNO_2$		mol. wt. 286.16
Br CO(CH ₂ ) ₃ CH ₃	Synthesis -Refer to: [3373]. <b>O-Methyloxime</b> C ₁₃ H ₁₉ BrN ₂ O ₂	[104129-16-0]	mol. wt. 315.21

-As diuretic and antihypertensive [3373].

<b>Oxime</b> [104145-15-5]	$C_{12}H_{17}BrN_2O_2$	mol. wt. 301.22
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-As diuretic and antihypertensive [3373].

#### 1-(2-Hydroxy-3-methylphenyl)-1-pentanone

[189875-29-4]	$C_{12}H_{16}O_2$	mol. wt. 192.26
CH ₃ CO(CH ₂ ) ₃ CH ₃	Syntheses -Obtained by Fries rearrange valerate in the presence of 160–180° for 30 min (46 % -Also obtained by acylat reagents (94 %) [1627].	(5) [726].
Also refer to: $[2340]$		

-Also refer to: [2340].

colourless oil [1627]; b.p.₁₅ 143–145° [726]; m.p. 18° [726]; ¹H NMR [1627], ¹³C NMR [1627].

Phenylhydrazone

 $C_{18}H_{22}N_2O$ 

#### mol. wt. 282.38

m.p. 116–118° [726].

#### 1-(2-Hydroxy-4-methylphenyl)-1-pentanone

[173851-66-6]  $C_{12}H_{16}O_2$  mol. wt. 192.26

Syntheses
H₃ -Preparation by Fries rearrangement of 3-methylphenyl valerate in the presence of aluminium chloride,
*without solvent at 160° for 2 h (85 %) [726] or at 120–140° for 10–20 min (80 %) [243];

*in nitrobenzene at 25° for 24 h (67 %) [243] or at 25–30° for 24 h (70 %) [244]. -Also obtained by Fries rearrangement of 3-methylphenyl valerate in the presence of boron trifluoride etherate at 110° under nitrogen for 4 h (85 %) [1118].

b.p.₃ 121–125° [243, 244], b.p.₃ 122–123° [1118], b.p.₁₅ 152–154° [726]; m.p. 16° [726, 1118]; ¹H NMR [244, 1118].

Methyl ether	[173851-69-9]	$C_{13}H_{18}O_2$	mol. wt. 206.28
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-Obtained by reaction of pentanoic acid with 3-methylanisole in the presence of trifluoroacetic anhydride under nitrogen at r.t. for 3.5 h (22 %) [656].

-Also obtained by reaction of dimethyl sulfate with 2-hydroxy-4-methylvalerophenone in the presence of potassium carbonate in refluxing acetone for 2 h (97 %) [1118].

oil [1118]; ¹H NMR [1118], IR [1118].

# Phenylhydrazone

 $e C_{18}H_{22}N_2O$ 

mol. wt. 282.38

492

m.p. 138–139° [243].

# 2,4-Dinitrophenylhydrazone

-Refer to: [243].

m.p. 138-139° [243].

# 1-(2-Hydroxy-5-methylphenyl)-1-pentanone

Syntheses -Obtained by Fries rearrangement of 4-methylphenyl valerate with aluminium chloride at 160° for 2 h (65 %) [726]. -Also obtained by reaction of valeric acid (0.95 mmol) with p-cresol (0.95 mmol) in the presence of ferric chloride (0.32 mmol) under microwave irradiation (600 W) (85 %) [2213].

-Also obtained by reaction of valeric acid with p-cresol in the presence of,

*zinc chloride under microwave irradiation with 500 W for 3.3 min (98 %) [2211]; *stannic chloride under microwave irradiation with 700 W for 2 min at 50° and atmospheric pressure conditions (95 %) [2212];

 $C_{18}H_{20}N_4O_5$ 

*boron trifluoride etherate under microwave irradiation for 2 min at r.t. (98 %) [2210]. -Also refer to: [2340].

oil [2210–2212]; b.p.₁₅ 144–145° [726]; m.p. 32–33° [726]; ¹H NMR [2210–2213], IR [2210–2213]; TLC [2210, 2211, 2213].

# Methyl ether

# $C_{13}H_{18}O_2$

mol. wt. 206.28

mol. wt. 192.26

-Preparation by reaction of valeroyl chloride with 4-methylanisole in the presence of aluminium chloride in carbon disulfide (52 %) [515].

C12H16O2

colourless oil [515]; b.p.  $_{13}$  161–162° [515];  $n_D^{21}=1.5240$  [515].

# 1-(4-Hydroxy-2-methylphenyl)-1-pentanone

OH

Syntheses

-Obtained by Fries rearrangement of 3-methylphenyl valerate in the presence of aluminium chloride in nitrobenzene at  $25-30^{\circ}$  for 24 h (14 %) [244].

CO(CH₂)₃CH₃ -Also obtained by Fries rearrangement of 3-methylphenyl valerate in the presence of boron trifluoride etherate at 110° under nitrogen for 4 h (2 %) [1118].

colourless needle prisms [244]; m.p. 88-89° [244, 1118].

mol. wt. 372.38

wt. 192.26

-Obtained by reaction of pentanoic acid with 3-methylanisole in the presence of trifluoroacetic anhydride under nitrogen at r.t. for 3.5 h (77 %) [656], (51 %) [1118].

colourless crystals [1118]; m.p. 52–53° [1118]; ¹H NMR [1118], IR [1118], MS [1118].

# 1-(4-Hydroxy-3-methylphenyl)-1-pentanone

C₁₂H₁₆O₂ mol. wt. 192.26

 $\begin{array}{ccc} OH & Synthesis \\ -Obtained by Fries rearrangement of 2-methylphenyl valerate in the presence of aluminium chloride at 160–180° for 30 min (30 %) [726]. \\ CO(CH_2)_3CH_3 & b.p._{15} 205° [726]; m.p. 103–104° [726]. \end{array}$ 

Methyl ether	[5394-88-7]	$C_{13}H_{18}O_2$	mol. wt. 206.28
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-Obtained by reaction of valeric anhydride with 2-methylanisole in the presence of aluminium chloride in boiling carbon disulfide for 30 min (83 %) [2297]. -Also refer to: [660, 1249, 2172, 2174, 2175, 2997, 2999].

b.p.₄ 151.5° [2297], b.p.₅ 151–155° [660], b.p.₇₄₄ 308.7° [2297]; m.p. 31–33° [2297]; ¹H NMR [1249, 2999], ¹³C NMR [1249, 2999], IR [1249, 2999].

 $C_{19}H_{20}O_3$ 

# Phenylhydrazone

# $C_{18}H_{22}N_2O$

mol. wt. 282.38

mol. wt. 296.36

m.p. 120-121° [726].

# Benzoate

m.p. 72–73° [726].

# 1-(5-Hydroxy-2-methylphenyl)-1-pentanone

C₁₂H₁₆O₂ mol. wt. 192.26

OH Synthesis  
-Refer to: [574].  
Methyl ether [74571-50-9]  

$$CH_3$$
 CO(CH₂)₃CH₃  $C_{13}H_{18}O_2$  mol. wt. 206.28

-Refer to: [574 (45 %)].

b.p._{0.002} 110–120° [574]; ¹H NMR [574], IR [574], UV [574], MS [574].

#### 2,4-Dinitrophenylhydrazone of the methyl ether

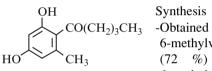
[74571-52-1]  $C_{19}H_{22}N_4O_5$  mol. wt. 386.41 m.p. 75–76° [574].

#### 1-(2,3-Dihydroxy-5-methylphenyl)-1-pentanone

	$C_{12}H_{16}O_3$	mol. wt. 208.26
CO(CH ₂ ) ₃ CH ₃	Synthesis -Obtained from creosol (66 %) [784] yellow crystals [784]; m.p. 85–86° [784].	].

#### 1-(2,4-Dihydroxy-6-methylphenyl)-1-pentanone

 $[154921-41-2] C_{12}H_{16}O_3 mtext{mol. wt. } 208.26$ 



Synthesis -Obtained from 2-difluoroboryloxy-4-hydroxy-6-methylvalerophenone by refluxing in dilute ethanol (72 %) [2938]. 2-Difluoroboryloxy-4-hydroxy-6-methyl-valerophenone, still named:

mol. wt. 236.31

Difluoro[1-(2,4-dihydroxy-6-methylphenyl)-1-valeronato-O,O'] boron (72 %, m.p.  $134^{\circ}$ , IR).

m.p. 132° [2938]; IR [2938], UV [2938].

#### **Dimethyl ether**

¹H NMR [554].

Dimethyl ether (D)	[92120-60-0]	$C_{14}H_{18}D_2O_3$	mol. wt. 238.29
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C14H20O3

1-[2,4-Dihydroxy-6-(methyl-d)phenyl]-1-pentanone

-Obtained by adding n-butyllithium in hexane to a solution of ethyl 2,4-dimethoxy-6-methyl-benzoate in THF under nitrogen and cooled at  $-78^{\circ}$ . Then, D₂O in THF was added (71 %) [554].

N.B.: The compound was found to be deuterated on the methylene next to the ketone.

oil [554]; b.p._{0.01} 100° [554]; ¹H NMR [554], IR [554], UV [554], MS [554].

# 1-(2,5-Dihydroxy-4-methylphenyl)-1-pentanone

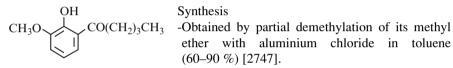
-Prepared by Friedel-Crafts acylation (95 %) [1755].

b.p. ₃ 140–143° [1755]; m.p. 33.5–34° [1755]; ¹H NMR [1755], IR [1755].

# 1-(2-Hydroxy-3-methoxyphenyl)-1-pentanone

[15116-08-2]

C12H16O3 mol. wt. 208.26



oil [2747]; b.p._{0.08-0.1} 87–89.5° [2747].

# 1-(2-Hydroxy-4-methoxyphenyl)-1-pentanone

[372486-18-5]	$C_{12}H_{16}O_{3}$		mol. wt. 20	8.26
CH ₃ O ^{OH} CO(CH ₂ ) ₃ CH ₃	2,4-dihydroxy-	valerophenone	methylation with dimethyl ssium carbonate	sul-

C12H16O3

(60-90 %) [2747].

Synthesis

2,4-dihydroxy-valerophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 4-6 h (85-90 %) [2501].

mol. wt. 208.26

# 1-(2-Hydroxy-5-methoxyphenyl)-1-pentanone

[57314-80-4]

OCH₃

Syntheses CO(CH₂)₃CH₃ -Obtained by reaction of valeroyl chloride with hydroqui-none dimethyl ether in the presence of aluminium chloride [156], (51 %) [770],

*in methylene chloride at r.t. for 1 h (9 %) [2878];

*in refluxing carbon disulfide for 4 h, and the mixture left overnight (77 %) [1113]. -Also obtained by Fries rearrangement of 4-methoxyphenyl valerate in the presence of aluminium chloride [156].

-Also obtained by heating 4-methoxyphenyl valerate with aluminium chloride on the steam bath for 6 h (49 %) [770].

pale yellow needles [770]; b.p.₀₁ 135–145° [1113], b.p.₀₂ 146–154° [770]; m.p. 62–62.5° [2878], 62° [770, 1113]; UV [2878].

Oxime	[140943-12-0]	$C_{12}H_{17}NO_3$	mol. wt. 223.27

-Refer to: [285].

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

 $C_{18}H_{20}N_4O_6$ 2,4-Dinitrophenylhydrazone mol. wt. 388.38

m.p. 186° [770], 185° [1113].

#### C14H18O4 mol. wt. 250.29 Acetate

-Acetylation of 2-hydroxy-5-methoxyvalerophenone by means of acetic anhydride in pyridine solution [1113].

m.p. 72–73° [1113].

# 1-(3-Hydroxy-5-methoxyphenyl)-1-pentanone

(	$C_{12}H_{16}O_3$	mol. wt. 208.26
CH ₃ O ^{OH} CO(CH ₂ ) ₃ CH ₃	Synthesis -Refer to: [607]. Isolation from natural sources -From the roots and stems of (Myrsinaceae) [607].	Ardisia virens Kurz

colourless needles [607], m.p. 66–67° [607], ¹H NMR [607], ¹³C NMR [607], IR [607], UV [607], MS [607].

# 1-(4-Hydroxy-3-methoxyphenyl)-1-pentanone

[114515-51-4]	$C_{12}H_{16}O_3$	mol. wt. 208.26
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OH	OCH ₃
CO(C	CH ₂ ) ₃ CH ₃

Syntheses

-Obtained by Fries rearrangement of 2-methoxyphenyl valerate in the presence of aluminium chloride in nitrobenzene (50%) [726]. -Also obtained by hydrolysis of its benzoate (90 %) [1377].

-Also obtained by adding DDQ to 1-(4-hydroxy-3-methoxyphenyl)-1-pentanol in benzene and the reaction mixture stirred for 4 h at r.t. [2989].

-Also obtained by hydrolysis and conversion of fractionated glycoside aroma precursors in neutral grapes by Saccharomyces cerevisiae [981].

b.p.₁₅ 195–197° [726]; m.p. 62.9° [1377], 61° [2989], 60–62° [726]; ¹H NMR [2989].

BIOLOGICAL ACTIVITY: Choleretic [2989].

Benzoate C	$C_{19}H_{20}O_4$	mol. wt. 312.36
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-Obtained by oxidation of  $\alpha$ -(4-benzoyloxy-3-methoxyphenyl)-n-butyl carbinol with potassium dichromate in acetic acid and dilute sulfuric acid, first 10 min at r.t., then at 85° (92 %) [1377].

m.p. 88.1° [1377], 85–87° [726].

C25H26N2O3 Phenylhydrazone of the benzoate mol. wt. 402.49

m.p. 163° [1377].

# 1-(2,6-Dihydroxy-4-methoxyphenyl)-1-pentanone

(Desaspidinol Y)

[69480-05-3]

CH₂O

C12H16O4 mol. wt. 224.26

**Syntheses** 

CO(CH₂)₃CH₃ -Preparation by reaction of pentanoyl chloride with phloroglucinol monomethyl ether in the presence of aluminium chloride in methylene chloride at r.t. [1129].

-Also refer to: [3301].

OH

Isolation from natural sources

OH

-In bud exudate of Populus nigra L [1162]. -Of Populus tritis bud exudate [955]. -In bud exudate of Populus cathayana (Salicaceae) [1163]. -In bud exudate of Populus koreana, Populus maximowiczii and Populus suaveolens (Salicaceae) [1164]. -From Ctenitis apiciflora [3295].

-From Ctenitis nidus [3295].

colourless amorphous solid [1129]; m.p. 96–98° [3301]; MS [1129]; GLC [2531]; GC-MS [1162–1164].

BIOLOGICAL ACTIVITY: Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

#### 1-(2,4,6-Trihydroxy-3-methylphenyl)-1-pentanone

mol. wt. 224.26 [49583-26-8] C12H16O4 Syntheses .CO(CH₂)₃CH₃ -Refer to: [1917, 2531, 3297, 3301]. CH₃. Isolation from natural sources HO -From Dryopteris bissetiana [3299].

m.p. 166–167° [3301], 149–151° [1917, 3297]; MS [1917]; GC-MS [2531]; GLC [2531].

#### 1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1,3-pentanedione

[62036-46-8] C13H16O4 mol. wt. 236.27

> **Syntheses** COCH₂COCH₂CH₃ -Obtained by reaction of ethyl propionate with 2-hydroxy-4-methoxy-6-methylacetophenone in the presence of pulverized sodium. Then, the reaction mixture was heated for 1 h on a

-Also refer to: [1097].

CH₂

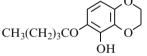
colourless needles [49]; m.p. 124–125° [49, 1097].

# 5-Hydroxy-6-(1-oxopentyl)-1,4-benzodioxa

$$C_{13}H_{16}O_4$$
 mol. wt. 236.27

-Obtained by Fries rearrangement of 5-valeryloxy-

1.4-benzodioxane in the presence of aluminium chlo-



ride in nitrobenzene at  $20^{\circ}$  (73 %) [801].

b.p.₀₁ 165–167° [801]; m.p. 98–99° [801]; UV [801].

#### 1-(3-Chloro-6-hydroxy-2,4-dimethylphenyl)-1-pentanone

[100607-74-7]

Synthesis

[100794-83-0]

3

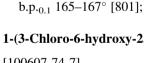
CO(CH₂)₃CH₃ -Preparation by Fries rearrangement of 4-chloro-3,5-dimethylphenyl valerate with aluminium chloride in carbon disulfide at  $80^{\circ}$  for 2 h, then at  $110^{\circ}$ for 2 h after solvent elimination (73 %) [3114].

m.p. 63° [3114].

#### Semicarbazone

 $C_{14}H_{20}CIN_{3}O_{2}$ mol. wt. 297.78

m.p. 200° [3114].



boiling water bath (38 %) [49].

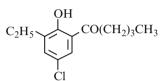
C Synthesis

-Obtained by reaction of allyl bromide with the title ketone in the presence of potassium carbonate in refluxing acetone for 6 h (98 %) [3114].

b.p.₃ 160° [3114].

USE: Insecticide [3114].

# 1-(5-Chloro-3-ethyl-2-hydroxyphenyl)-1-pentanone

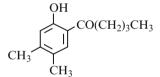


OH CO(CH₂)₃CH₃ Synthesis -Obtained by Fries rearrangement of 4-chloro-2-ethylphenyl valerate in the presence of aluminium chloride at 120° for 1.5 h (80.6 %) [2763].

b.p._{0.2} 111–112° [2763];  $n_D^{20} = 1.5416$  [2763].

# 1-(2-Hydroxy-4,5-dimethylphenyl)-1-pentanone

[149696-17-3] C13H18O2 mol. wt. 206.28



Synthesis  $CO(CH_2)_3CH_3$  -Obtained by reaction of valeric acid with 3,4-dimethyl-phenol in the presence of boron trifluoride etherate (78 %) [2939]. m.p. 41–42° [2939];

¹H NMR [2939], IR [2939], UV [2939].

[159847-59-3]  $C_{14}H_{20}O_2$ Methyl ether mol. wt. 220.31

¹H NMR [533], ¹³C NMR [533], IR [533], MS [533].

-Formation in allylation of benzamide deriv. [533].

# 1-(4-Hydroxy-2,3-dimethylphenyl)-1-pentanone

C13H18O2 mol. wt. 206.28

ОН	Synthesis	
CH ₃	-Refer to: [742].	
	Methyl ether [101375-32-0]	
CH ₃	$C_{14}H_{20}O_2$	mol. wt. 220.31
CO(CH ₂ ) ₃ CH ₃		

-Preparation by reaction of valeroyl chloride with 2,3-dimethylanisole in the presence of aluminium chloride (96 %) [742].

m.p. 68–69° [742].

# 1-(4-Ethoxy-2-hydroxyphenyl)-1-pentanone

$$\begin{array}{cccc} & & C_{13}H_{18}O_3 & & \text{mol. wt. } 222.28 \\ & & OH & & Synthesis \\ & & CO(CH_2)_3CH_3 & -Refer to: [2221]. \\ & & Oxime & [928769-73-7] \\ & & C_{13}H_{19}NO_3 & & \text{mol. wt. } 237.30 \end{array}$$

USE: As an analytical reagent for molybdenum (VI) determination by spectrophotometry [2221].

# 1-(5-Ethoxy-2-hydroxyphenyl)-1-pentanone

$$\begin{array}{cccc} [140943-31-3] & C_{13}H_{18}O_3 & \mbox{mol. wt. } 222.28 \\ OH & Synthesis \\ - CO(CH_2)_3CH_3 & -Refer to: [285]. \\ Oxime & [140943-18-6] \\ C_{13}H_{19}NO_3 & \mbox{mol. wt. } 237.30 \\ \end{array}$$

-Refer to: [285].

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

# 1-(5-Ethyl-2,4-dihydroxyphenyl)-1-pentanone

	$C_{13}H_{18}O_3$	mol. wt. 222.28
HO CH ₂ CH ₂ CH ₃	Synthesis -Obtained by Fries rearrangement of divalerate with aluminium chloride 50–60° or without solvent at 40–50 b.p. ₉ 220° [2651]; m.p. 92° [2651]	in nitrobenzene at ° [2651].

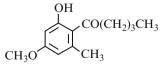
2,4-Dinitrophenylhydrazone C	$C_{19}H_{22}N_4O_6$	mol. wt. 402.41
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-Refer to: [2651]; m.p. 125–126° [2651].

1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1-pentanone

(*Evernipentanone*)

mol. wt. 222.28  $C_{13}H_{18}O_3$ 



 $\begin{array}{rl} \text{Synthesis} \\ \text{CO}(\text{CH}_2)_3\text{CH}_3 \\ \text{CH}_3 \end{array} \begin{array}{rl} \text{Synthesis} \\ \text{-To a solution of ethyl everninate (1 mol) in THF} \\ \text{at } -78^\circ \text{ was added a cold n-BuLi (7 mol) in} \\ \text{hexane. The reaction mixture was kept for 1 h} \end{array}$ at  $-78^{\circ}$  and then for 18 h at r.t. (80 %) [871].

m.p. 59–63° [871]; ¹H NMR [871], UV [871], MS [871]; TLC [871].

#### 1-(3-Hydroxy-5-methoxy-4-methylphenyl)-1-pentanone

	$C_{13}H_{18}O_3$	mol. wt. 222.28
CH ₃ O CH ₃ O CO(CH ₂ ) ₃ CH ₃	Isolation from natural sources -From the roots of <i>Ardisia</i> (Myrsinaceae) [604]. colourless needles [604]; m.p. 84–86° [604];	cornudentata Mez.

¹H NMR [604], ¹³C NMR [604], IR [604], UV [604], MS [604].

1-(2,6-Dihydroxy-4-methoxy-3-methylphenyl)-1-pentanone (Aspidinol V)

[57765-52-3]	$C_{13}H_{18}O_4$	mol. wt. 238.28
CH ₃ CO(CH ₂ ) ₃ CH ₃	Syntheses -Refer to: [2531, 3297]. Isolation from natural sources	

-From Ctenitis apiciflora [3295].

-From Ctenitis nidus [3295].

m.p. 138–140° [3297]; GLC [2531].

#### 1-(2-Hydroxy-4,6-dimethoxyphenyl)-1-pentanone

[80986-13-6]

СН30

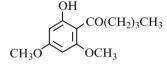
 $C_{13}H_{18}O_{4}$ 

Syntheses

C

Syntheses

mol. wt. 238.28



CO(CH₂)₃CH₃ -Obtained by reaction of dimethyl sulfate with phlorovalerophenone in the presence of potassium carbonate in refluxing acetone for 30-40 min (80-90 %) [2014].

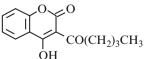
-Also obtained by methylation of 2,4,6-trihydroxyvalerophenone [56] according to [1375].

-Also refer to: [56, 1986].

¹H NMR [1986].

# 4-Hydroxy-3-(1-oxopentyl)-2H-1-benzopyran-2-one

$$_{14}H_{14}O_4$$
 mol. wt. 246.26



-Obtained by reaction of valeryl chloride with CO(CH₂)₃CH₃ 4-hydroxy-coumarin in pyridine containing one drop of piperidine for 3 h on a water bath (39 %) [3174].

-Also refer to: [2306, 3144].

m.p. 99° [3174].

# 1-(8-Hydroxy-5-quinolinyl)-1-pentanone

$$\begin{array}{ccc} C_{14}H_{15}NO_2 & \text{mol. wt. 229.28} \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\$$

-Refer to: [1335 (62 %)].

[194792-59-1]

m.p. 105–107° [1335].

BIOLOGICAL ACTIVITY: Fungicide [1335].

# 1-[2,4-Dihydroxy-3-(2-propenyl)phenyl]-1-pentanone

[194792-60-4]  $C_{14}H_{18}O_3$  mol. wt. 234.30  $CH_2=CHCH_2$  OH Synthesis  $CH_2=CHCH_2$   $CO(CH_2)_3CH_3$  -Refer to: [22].

C14H18O3

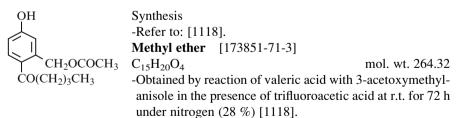
# 1-[2-Hydroxy-4-(2-propenyloxy)phenyl]-1-pentanone

mol. wt. 234.30

OH Synthesis  $CH_2=CHCH_2O$   $CO(CH_2)_3CH_3$  -Refer to: [22].

# 1-[2-(Acetoxymethyl)-4-hydroxyphenyl]-1-pentanone

C₁₄H₁₈O₄ mol. wt. 250.29



white crystals [1118]; m.p. 42.5–43° [1118]; ¹H NMR [1118], IR [1118], MS [1118].

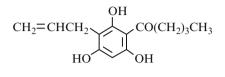
# 1-[2,4,6-Trihydroxy-3-(2-propenyl)phenyl]-1-pentanone

2-Valeryl-4-(propen-2-yl)phloroglucinol (19) [1026].

[74477-99-9]

 $C_{14}H_{18}O_4$ 

mol. wt. 250.29



Syntheses

CO(CH₂)₃CH₃ -Obtained by adding cuprous chloride, a saturated aqueous solution of sodium carbonate and allyl chloride to a solution of phlorovalerophenone in ethyl ether at r.t., then to keep the mixture for 12 h at this temperature [1026].

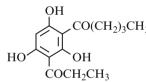
-Also obtained by reaction of allyl chloride with phlorovalerophenone in an alkaline two-phase (aqueous-ether) system and catalyzed by CuCl [838, 3193].

m.p. 123–126° [3193]; ¹³C NMR [839, 1026, 3193], IR [1026].

BIOLOGICAL ACTIVITY: Bactericidal and fungicidal [1026, 3193].

# 1-(3-Propionyl-2,4,6-trihydroxyphenyl)-1-pentanone

$C_{14}H_{18}O_5$	mol. wt. 266.29
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Synthesis CO(CH₂)₃CH₃ -Preparation [3405] according to the method [421].

BIOLOGICAL ACTIVITY: As photosynthetic electron transport (PET) [3405]; This compound was assayed for its inhibition of the Hill reaction using chloroplasts isolated from the leaves of *Spinacia oleracea*; pl 50 = 4.9 [3405].

# 1-[4-(3-Bromopropoxy)-3-hydroxyphenyl]-1-pentanone

 $C_{14}H_{19}BrO_3$ 

mol. wt. 315.20

4-hydroxy-

with

ŎН	Synthesis	
Br(CH ₂ ) ₃ O	-Refer to: [296	5].
	Methyl ether	[133455-19-3]
CO(CH ₂ ) ₃ CH ₃	$C_{15}H_{21}BrO_3$	mol. wt. 329.23

1,3-dibromopropane

-Obtained by reaction of 3-methoxyvalerophenone [2962]. -Also refer to: [2963–2967].

# 1-(2,4,6-Trihydroxy-3-nitro-5-propylphenyl)-1-pentanone

[119692-01-2]	$C_{14}H_{19}NO_6$	mol. wt. 297.31
$\begin{array}{c} OH\\ NO_2 \\ HO \\ C_3H_7 \end{array} CO(CH_2)_3CH_3 \\ OH \\ C_3H_7 \end{array}$	Synthesis -Obtained by adding a mixtu and acetic acid to the s hydroxy-3-propyl-phenyl)- acid at 60° for 30 min (30–	olution of 1-(2,4,6-tri- 1-pentanone in acetic

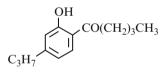
m.p. 51–52° [3114]; ¹H NMR [3114], IR [3114], MS [3114].

BIOLOGICAL ACTIVITY: Germination inhibition [3114]; PET inhibition [3114].

# 1-[4-Hydroxy-3-(1-methylethyl)phenyl]-1-pentanone

[95102-37-7]  $C_{14}H_{20}O_2$ mol. wt. 220.31 Syntheses .CH(CH₃)₂ -Refer to: [1595, 2704]. USE: Colour developer, for thermal recording materials [1595]. CO(CH₂)₃CH₃

# 1-(2-Hydroxy-4-propylphenyl)-1-pentanone



H Synthesis CO(CH₂)₃CH₃ -Obtained by reaction of valeric acid with 3-propylphenol in the presence of zinc chloride (Nencki reaction) (30–40 %) [728].

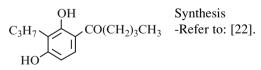
b.p._{0.7} 84–85° [728], b.p.₁₈ 127–129° [728].

# 1-(2,4-Dihydroxy-3-propylphenyl)-1-pentanone

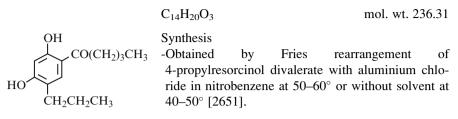
[194792-61-5]

 $C_{14}H_{20}O_3$ 

mol. wt. 236.31

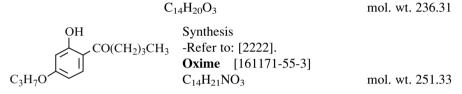


#### 1-(2,4-Dihydroxy-5-propylphenyl)-1-pentanone



b.p._{9.5} 205° [2651]; m.p. 85° [2651].

# 1-(2-Hydroxy-4-propoxyphenyl)-1-pentanone



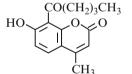
-Iron complex; stability const. and free energy of formation and molar absorptivity [2222].

-As a spectrophotometric reagent for iron (III) [2222].

#### 7-Hydroxy-4-methyl-8-(1-oxopentyl)-2H-1-benzopyran-2-one

$$C_{15}H_{16}O_{4}$$

mol. wt. 260.29



Syntheses -Obtained by treatment of 4-methyl-7-n-valeroxycoumarin with aluminium chloride at  $150^{\circ}$  for 1 h (68 %) [1275]. -Also refer to: [329]. pale yellow needles [1275];

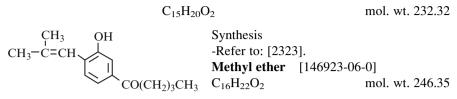
m.p. 106° [329], 98–103° [1275].

# 1-[2-Hydroxy-4-(2-methyl-1-propenyl)phenyl]-1-pentanone

$$\begin{array}{c} C_{15}H_{20}O_{2} & \text{mol. wt. } 232.32 \\ OH & Synthesis \\ -Refer to: [2323]. \\ Methyl ether & [146923-05-9] \\ CH_{3}-C=CH & C_{16}H_{22}O_{2} & \text{mol. wt. } 246.35 \end{array}$$

-Refer to: [2323].

# 1-[3-Hydroxy-4-(2-methyl-1-propenyl)phenyl]-1-pentanone



-Refer to: [2323].

# 1-(6,7-Dihydroxy-5-propyl-1,3-benzodioxol-4-yl)-1-pentanone

 $\begin{array}{ccc} C_{15}H_{20}O_5 & \text{mol. wt. } 280.32 \\ OH & Synthesis \\ OH & -Refer to: [2179]. \\ \hline Dimethyl ether & [82652-25-3] \\ C_3H_7 & (Valeryl \ dihydrodillapiole) \\ CO(CH_2)_3CH_3 & C_{17}H_{24}O_5 & \text{mol. wt. } 308.37 \end{array}$ 

-Obtained by reaction of valeryl chloride with dihydrodillapiole in the presence of fused zinc chloride in methylene chloride for 3–4 h in an ice bath (70 %) [2179].

colourless solid [2179]; ¹H NMR [2179], IR [2179].

USE: Synergistic insecticidal activity with pyrethrum [2179].

# 1-[3-Chloro-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-pentanone

[102003-73-6]	$C_{15}H_{21}ClO_2$	mol. wt. 268.78
$\begin{array}{c} OH\\CI \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ $	Synthesis -Obtained by Fries rearrangement of butyl-phenyl valerate with aluminium (75 %) [3119]. b.p. ₂₀ 154° [3119].	

**2,4-Dinitrophenylhydrazone** [102596-47-4]  $C_{21}H_{25}ClN_4O_5$  mol. wt. 448.91 -Refer to: [3119]; m.p. 185° [3119].

# 1-[4-(Chloromethyl)-2-hydroxy-3-propylphenyl]-1-pentanone

$$[97582-39-3] C_{15}H_{21}ClO_2 mol. wt. 268.78$$

$$OH Synthesis$$

$$C_3H_7 OH CO(CH_2)_3CH_3 -Refer to: [877].$$

$$ClCH_2 OH CO(CH_2)_3CH_3 -Refer to: [877].$$

mol. wt. 414.46

# 1-[3-(1,1-Dimethylethyl)-4-hydroxyphenyl]-1-pentanone

[95185-63-0]  $C_{15}H_{22}O_2$  mol. wt. 234.34 OH Synthesis -Refer to: [2704].

# 1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-pentanone

[75060-52-5]	$C_{15}H_{22}O_2$		mol. wt. 234.34
$\bigcup_{C(CH_3)_3}^{OH} CO(CH_2)_3CH_3$	Synthesis -Obtained by treatme phenone with 47 % acid mixture in reflux	hydrobromic acid	/57 % hydriodic
¹ H NMR [1475], IF	R [1475]; TLC [1475]		
Methyl ether	[75060-45-6]	$C_{16}H_{24}O_2$	mol. wt. 248.37

-Obtained by reaction of valeryl chloride with 4-tert-butylanisole in the presence of aluminium chloride in methylene chloride under nitrogen, first at  $0^{\circ}$ , then at  $20^{\circ}$  for 30 min (100 %) [1475].

TLC [1475].

CH₃

CO(CH₂)₃CH₃

# 1-[2-Hydroxy-3-methyl-6-(1-methylethyl)phenyl]-1-pentanone

	$C_{15}H_{22}O_2$	mol. wt. 234.34
	Synthesis	
$CO(CH_2)_3CH_3$	-Obtained by Fries rearrangement o	•
CH(CH ₃ ) ₂	ate with aluminium chloride at 120 b.p. ₃ 154° [2798].	$J^{\circ}(66\%)[2/98].$
011(0113)2	0.0.3 13 1 [2790]:	

 $C_{21}H_{26}N_4O_5$ 

**2,4-Dinitrophenylhydrazone** -Refer to: [2798]; m.p. 136° [2798].

# 1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-pentanone

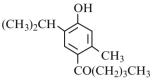
[859786-47-3]	$C_{15}H_{22}O_2$	mol. wt. 234.34
(CH ₃ ) ₂ CH (CH ₃ ) ₂ CH (CH ₃ ) ₂ CH ₃	Synthesis -Obtained by Fries rearran valerate with aluminium (77 %) [2803]. b.p. ₂ 164° [2803].	gement of thymyl chloride at 120°

# 2,4-Dinitrophenylhydrazone

-Refer to: [2803]; m.p. 170° [2803].

# 1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-pentanone

 $C_{21}H_{26}N_4O_5$ 



-Obtained by photo-Fries rearrangement of thymyl pentanoate in methanol for 6 h at 25° under nitrogen (32 %) (2a) [2421].

Also obtained from its methyl ether by boiling in pyridinium chloride (205–215°) for 105 min (XXIV) (19 %) [2660].

-Also refer to: [2704].

b.p.₁₆ 200° [2660]; m.p. 86° [2421], 84° [2660]; ¹H NMR [2421], IR [2421].

Methyl ether (V)  $C_{16}H_{24}O_2$  mol. wt. 248.37

-Obtained by reaction of valeryl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (62 %) [2660].

b.p.15 185-187° [2660].

# 1-[2,4-Dihydroxy-5-(1,1-dimethylethyl)phenyl]-1-pentanone

	$C_{15}H_{22}O_3$	mol. wt. 250.34
OH	Synthesis	
CO(CH ₂ ) ₃ CH ₃	-Refer to: [2704]. m.p. 153° [2704].	
HO		
C(CH ₃ ) ₃	USE: As colour developer [2704].	

# 1-[2,4-Dihydroxy-5-(2-methylpropyl)phenyl]-1-pentanone

 $[798559-94-1] C_{15}H_{22}O_3 mtext{mol. wt. 250.34} \\ OH \\ OH \\ CO(CH_2)_3CH_3 \\ OB \\ OB \\ CH_2CH(CH_3)_2 \\ CH_2CH(CH_3)_$ 

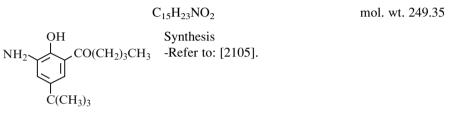
mol. wt. 414.45

# **Dimethyl ether** [798559-82-7] C₁₇H₂₆O₃ mol. wt. 278.39

-Preparation by reaction of pentanoyl chloride with 4-isobutylresorcinol dimethyl ether in the presence of titanium tetrachloride in methylene chloride at  $0^{\circ}$  (94 %) [816].

m.p. 78–80° [816]; ¹H NMR [816], ¹³C NMR [816], IR [816].

# 1-[3-Amino-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-pentanone



# 1-[2,4-Dihydroxy-6-[(trimethylsilyl)methyl]phenyl]-1-pentanone

 $\begin{array}{cccc} C_{15}H_{24}O_{3}Si & \text{mol. wt. } 280.44 \\ OH & Synthesis \\ + & CO(CH_{2})_{3}CH_{3} & -Refer to: [554]. \\ Dimethyl ether & [92120-78-0] \\ HO & CH_{2}Si(CH_{3})_{3} & C_{17}H_{28}O_{3}Si & \text{mol. wt. } 308.49 \end{array}$ 

-Obtained by adding n-butyllithium in hexane to a solution of ethyl 2,4-dimethoxy-6-trimethyl-silyl-methylbenzoate in THF under argon and cooled at  $-78^{\circ}$ . Then, D₂O in THF was added (58 %) [554].

**N.B.**: The compound was found to be deuteriated on the methylene next to the ketone  $C_{17}H_{27}DO_3Si$  mol. wt. 309.19

oil [554]; ¹H NMR [554], IR [554], UV [554], MS [554].

# 1,1'-(5-Bromo-2,4,6-trihydroxy-1,3-phenylene)bis-1-pentanone

[3136-48-9]	C ₁₆ H	$_{21}$ BrO ₅	mol. wt. 373.24
CH ₃ (CH ₂ ) ₃ CO	OH CO(CH ₂ ) ₃ CH ₃ OH Br	Synthesis -Refer to: [933]. m.p. 89–91° [457, 2911].	

**4-Trichloromethane** [30509-74-1]  $C_{17}H_{20}BrCl_3O_5S$  mol. wt. 522.67 sulfenate

-Refer to: [933].

# 1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl)-1-pentanone

[35049-65-1]

C₁₆H₂₂O₄ mol. wt. 278.35

но	CO(CH ₂ ) ₃ CH ₃	
	CH ₃	
	ОН	

Syntheses -Obtained from 2-pentanoylcyclohexane-1,3,5-trione [709]. -Also obtained (by-product) by reaction of 2-methyl-2-buten-1-ol with phlorovalerophenone in the presence of Amberlite IR-120 resin (H⁺ form) in dioxane at r.t. for 0.5 h. Then, the mixture was refluxed for 24 h (4–5 %) [708]. (Alkenylation of 2-valeroyl-cyclohexane-1,3,5-trione).

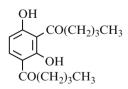
m.p. 102° [708, 709]; ¹H NMR [709], IR [709], UV [709].

# 2,6-Dihydroxy-3-(2-methylpropyl)-5-(1-oxopentyl)benzaldehyde

¹H NMR [816], ¹³C NMR [816], IR [816].

BIOLOGICAL ACTIVITY: Antiangiogenic [816].

# 1,1'-(2,4-Dihydroxy-1,3-phenylene)bis-1-pentanone



Synthesis .CO(CH₂)₃CH₃ -Obtained by Fries rearrangement of resorcinol divalerate with aluminium chloride (>2 mol) at 130–135° [2651]. b.p.₂₀ 196° [2651].

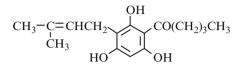
# 1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-pentanone

(2-Valeryl-4-(3-methylbuten-2-yl)phloroglucinol) (4) [1026].

[69916-09-2]

 $C_{16}H_{22}O_4$ 

mol. wt. 278.35



Syntheses

-Obtained by adding cuprous chloride, a saturated aqueous solution of sodium carbonate and prenyl bromide to a solution of phlorovalerophenone in ethyl ether at r.t., then to keep the mixture for 12 h at this temperature [1026].

-Also prepared by adding prenyl bromide to a suspension of the sodium salt of phlorovalero-phenone in benzene, then the mixture obtained was refluxed for 3 h [1026, 2111].

-Also obtained by reaction of prenyl chloride with phlorovalerophenone in an alkaline two-phase (aqueous-ether) system and catalyzed by CuCl [838, 3193].

-Also prepared by adding at  $5^{\circ}$  a solution of sodium methoxide in methanol, followed by isopentenyl bromide in ethyl ether to a solution of phlorovalerophenone in benzene and ethyl ether; then the mixture was stirred at r.t. for 6 h (22 %) [2110, 2113].

m.p. 145° [2113], 126–127° [3193];

N.B.: One of the reported melting point is obviously wrong.

¹³C NMR [839, 1026, 3193], IR [1026].

BIOLOGICAL ACTIVITY: Antifungal against *Trichophyton* species [2113]; Antifungal [2113]; Min. inhibitory *Trichophyton interdigitale, Candida albicans, Staphylococcus aureus*, etc. [2110]; Bactericidal and fungicidal [1026, 3193].

# 1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-pentanone

(Divaleryl phloroglucinol)

[3118-32-9]	$C_{16}H_{22}O_5$	mol. wt. 294.35
HO CO(CH ₂ ) ₃ CH ₃ HO CO(CH ₂ ) ₃ CH ₃	Syntheses -Refer to: [457, 644, 2190, 2 m.p. 104–106° [457, 2911].	2911, 3405].
BIOLOGICAL ACTIVITY	: As photosynthetic electron	transport (PET) [3405];

BIOLOGICAL ACTIVITY: As photosynthetic electron transport (PET) [3405]; Antiviral towards the replication of vesicular stomatitis virus in HEL cell cultures [644]; Inhibited aldose reductase in swines [2190]; Inhibition of the Hill reaction using chloroplasts isolated from the leaves of *Spinacia oleracea* [3405].

Na salt m.p. 140–180° [2911]. Tri-Na salt [5862-26-0]  $C_{16}H_{19}O_5Na_3$ mol. wt. 361.32 -Refer to: [457]. m.p. 180° (d) [457]. Mg salt [24070-03-9]. -Refer to: [457, 2911]. m.p. >200° [457, 2911], 200° (d) [457]. [4963-67-1]  $C_{16}H_{22}O_5*C_4H_{10}N_2$  mol. wt. 380.48 **Piperazine salt** m.p. 80–85° [457, 2911]. **1,3,5-Tris-trichloromethane sulfenate** [30509-76-3].

-Refer to: [933].

#### 1-(2-Hydroxy-5-pentylphenyl)-1-pentanone

[63494-45-1] C₁₆H₂₄O₂ mol. wt. 248.37 **Synthesis** OH CO(CH₂)₃CH₃ -Obtained by reaction of pentanoic acid with 4-pentylphenol in the presence of boron trifluoride at 25-30° for 45 min, then at  $140^{\circ}$  for 12 min and at  $140\text{--}150^{\circ}$  for 1 h  $C_{5}H_{11}$ (77 %) [142].

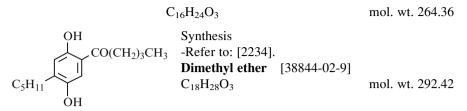
b.p.₁ 122–127° [142].

H

# 1-(2,4-Dihydroxy-5-pentylphenyl)-1-pentanone

C₁₆H₂₄O₃ mol. wt. 264.36 Syntheses OH CO(CH₂)₃CH₃ -Refer to: [1628, 1629]. USE: As antioxidant for vitamin A [1628, 1629].  $5H_{11}$ 

#### 1-(2,5-Dihydroxy-4-pentylphenyl)-1-pentanone



-Obtained by reaction of valeryl chloride with 1,4-dimethoxy-2-pentylbenzene in the presence of aluminium chloride in carbon disulfide (71 %) [2234].

colourless liquid [2234]; b.p._{0.6} 155–160° [2234].

# 1-[2,4-Dihydroxy-3-(hydroxymethyl)-5-(2-methylpropyl)phenyl]-1-pentanone

[798559-83-8] 
$$C_{16}H_{24}O_4$$
 mol. wt. 280.36  
 $HOCH_2$  OH  $CO(CH_2)_3CH_3$  Synthesis  
 $HOCH_2$  CO(CH_2)_3CH_3 -Obtained by reaction of formaldehyde with  
 $1-[2,4-dihydroxy-5-(2-methylpropyl)phenyl]-1$ -pentanone in the presence of KOH and CaCl₂  
in methanol (62 %) [816].  
m.p. 90–92° [816];  
¹H NMR [816], ¹³C NMR [816], IR [816].

 $C_{18}H_{28}O_4$ **Dimethyl ether** [798559-84-9] mol. wt. 308.42

-Obtained by treatment of the title ketone with methyl iodide in the presence of potassium carbonate in propanone (72 %) [816].

¹H NMR [816], ¹³C NMR [816], IR [816].

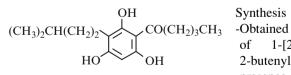
#### 1-[2,4,6-Trihydroxy-3-(3-methylbutyl)phenyl]-1-pentanone

[74477-98-8]

C₁₆H₂₄O₄

Synthesis

mol. wt. 280.36



hydrogenation by of 1-[2,4,6-tri-hydroxy-3-(3-methyl-2-butenyl)phenyl]-1-pentanone in the presence of PtO₂ in methanol under a hydrogen atmosphere at r.t. for 1 h (88 %) [2113].

m.p. 169° [2113].

**BIOLOGICAL ACTIVITY:** Antifungal [2113].

#### 1-(2,4,6-Trihydroxy-3-pentylphenyl)-1-pentanone

[69916-12-7]	$C_{16}H_{24}O_4$	mol. wt. 280.36
OH C ₅ H ₁₁ HO OH OH	chloride in nitrobenz 2,4,6-trihydroxypentyl-	a solution of pentanoyl ene to a suspension, of benzene and aluminium sulfide at r.t., then stirring $30-35^{\circ}$ (40 %) [2113].

m.p. 134° [2113].

# BIOLOGICAL ACTIVITY: Antifungal [2113].

#### 1-[(3-Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-pentanone

[75060-95-6]	$C_{16}H_{25}NO_2$	mol. wt. 263.38
NH ₂ CH ₂ CO(CH ₂ ) ₃ CH ₃	Synthesis -Refer to: [1475]. <b>Hydrochloride</b> [75060-70-7] $C_{16}H_{25}NO_2$ , HCl	mol. wt. 299.84

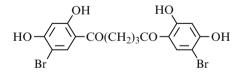
-Obtained by treatment of 1-[3-(N-chloroacetylaminomethyl)-5-(1,1-dimethylethyl)-2-hydroxy-phenyl]-1-pentanone with conc. hydrochloric acid in refluxing ethanol for 20 h (68 %) [1475].

white amorphous crystals [1475]; m.p. 187–190° [1289, 1475]; ¹H NMR [1475], IR [1475]; TLC [1475].

BIOLOGICAL ACTIVITY: As inflammation inhibitor [1475].

1,5-Bis(5-bromo-2,4-dihydroxyphenyl)-1,5-pentanedione

[16093-32-6]  $C_{17}H_{14}Br_2O_6$  mol. wt. 474.10



Br Syntheses -Obtained by treatment of its tetramethyl ether below with 40 % hydrobromic acid in refluxing acetic acid for 15 h (67 %) [592].

-Also obtained by condensation of 4-bromoresorcinol with glutaryl chloride in the presence of aluminium chloride in nitrobenzene [592].

m.p. 214-215° [592].

# **Di-2,4-dinitrophenylhydrazon**e [16093-33-7] $C_{29}H_{22}Br_2N_8O_{12}$ mol. wt. 834.35

m.p. 262° [592].

# **Tetramethyl ether** [16093-34-8] $C_{21}H_{22}Br_2O_6$ mol. wt. 530.21

-Obtained by reaction of glutaryl chloride with 4-bromoresorcinol dimethyl ether in the presence of aluminium chloride in nitrobenzene at  $0^{\circ}$  for 13 h (21 %) [592].

m.p. 193° [592].

m.p. 205° [592].

#### 1,5-Bis(5-chloro-2-hydroxyphenyl)-1,5-pentanedione

 $[52016-63-4] \qquad C_{17}H_{14}Cl_2O_4 \qquad \text{mol. wt. 353.20}$   $(C_1) \qquad (C_1) \qquad (C$ 

# 1,5-Bis(5-chloro-2,4-dihydroxyphenyl)-1,5-pentanedione

[16093-26-8] C17H14Cl2O6 mol. wt. 385.20 Syntheses -Also obtained by condensation of -OH CO(CH₂)₃CO HO 4-chloro-resorcinol with glutaryl chloride in the presence of aluminium chloride in nitrobenzene [592]. -Also refer to: [586]. m.p. 284° [586], 223° [592]. Dioxime [16093-27-9] C₁₇H₁₆Cl₂N₂O₆ mol. wt. 415.23 m.p. 321° [592].

 $\label{eq:2.1} \textbf{Di-2,4-dinitrophenylhydrazone} ~~ [96809-09-5] ~~ C_{29}H_{22}Cl_2N_8O_{12} ~~ mol. ~~ wt. ~~ 745.45$ 

m.p. 290° [586].

# **Tetramethyl ether** [16093-28-0] C₂₁H₂₂Cl₂O₆ mol. wt. 441.31

-Obtained by reaction of glutaryl chloride with 4-chlororesorcinol dimethyl ether in the presence of aluminium chloride in nitrobenzene [592].

m.p. 198° [592].

Dioxime of the	[16093-29-1]	$C_{21}H_{24}Cl_2N_2O_6$	mol. wt. 471.34
tetramethyl ether			

m.p. 208° [592].

#### 1,5-Bis(2-hydroxyphenyl)-1,5-pentanedione

(1,3-Disalicycloylpropane)

[4945-79-3] C17H16O4 mol wt 284 31

$$OH$$
 HO  
-CO(CH₂)₃CO-

Syntheses -Obtained by Fries rearrangement of phenyl glutarate with aluminium chloride (15-20 %)[1576], (11%) [1400],

 $C_{10}H_{20}O_4$ 

-in the presence of solvents:

*in the tetrachloroethane/nitrobenzene mixture (500 ml/125 ml) for 4 h at  $50-60^{\circ}$ [902]:

*in nitrobenzene for 4 h at  $50^{\circ}$ , then 30 min at  $60^{\circ}$  [902];

*in tetrachloroethane at  $80^{\circ}$  [902].

-without solvent at 120° [902].

-Also obtained by alkaline hydrolysis of dicoumarol [723, 941 (66 %), 2324, 3293 (98-99 %)].

-Also refer to: [1575, 2118, 2936].

m.p. 102° [902, 941, 1575, 1576], 101–102° [2936], 100–102° [1400]; ¹H NMR [2324], UV [2118]; pK_a [2118].

[190248-05-6]

**Dimethyl ether** 

-Obtained by reaction of methyl iodide with the title ketone in the presence of silver oxide at reflux for 1 h (50 %) [1400].

-Also obtained by reaction of dimethyl sulfate with the title bisphenol in the presence of potassium hydroxide in ethanol (87 %) [3293]. -Also refer to: [2936].

m.p. 86–88° [1400, 2936]; UV [1400].

#### Dimethanesulfonate

-Obtained by reaction of MsCl with 1,5-bis(2-hydroxyphenyl)-1,5-pentanedione in the presence of triethylamine in methylene chloride at r.t. for 1.5 h under nitrogen

 $C_{19}H_{20}O_8S_2$ 

atmosphere (87.7 %) [2324].

m.p. 68–69° [2324]; ¹H NMR [2324], IR [2324]; TLC [2324].

#### 1,5-Bis(3-hydroxyphenyl)-1,5-pentanedione

[10365-53-4]	$C_{17}H_{16}O_4$		mol. wt. 284.31		
HO CO(CH ₂ ) ₃ CO			by diazotization of 1,5-bis nenyl)-1,5-pentanedione (40–50 %) to: [1575].		

m.p. 152° [1575, 1576].

mol. wt. 312.36

mol. wt. 440.50

#### 1,5-Bis(4-hydroxyphenyl)-1,5-pentanedione

 $\label{eq:constraint} [20837-35-8] \qquad \qquad C_{17}H_{16}O_4 \qquad \qquad \mbox{mol. wt. } 284.31$ 

-Obtained by Fries rearrangement of phenyl glutarate with aluminium chloride [1400],

-in the presence of solvents:

*in the tetrachloroethane/nitrobenzene mixture (500 ml/125 ml) for 4 h at 50–60 $^{\circ}$  [902];

Syntheses

*in nitrobenzene for 4 h at  $50^{\circ}$ , then 30 min at  $60^{\circ}$  [902];

*in tetrachloroethane at  $80^{\circ}$  [902].

-without solvent at  $120^{\circ}$  [902].

-Also obtained by reaction of hydrobromic acid with its dimethyl ether in acetic acid for 2 h at  $120^{\circ}$  [902].

m.p. 222–225° [1148], 220–221° [902]; IR [588].

**Diacetate** [102474-24-8]  $C_{21}H_{20}O_6$  mol. wt. 368.39

-Obtained by reaction of acetic anhydride with 1,5-bis(4-hydroxyphenyl)-1,5-pentanedione [902].

m.p. 122° [902].

Syntheses

-Obtained by reaction of dimethyl sulfate with 1,5-bis(4-hydroxyphenyl)-1,5-pentanedione in the presence of 2 N NaOH [902].

-Also obtained by reaction of glutaryl dichloride with anisole in the presence of aluminium chloride [2644], (80–90 %) [1576],

*in carbon disulfide (78 %) [2495];

*in nitrobenzene/tetrachloroethane mixture (1:4) [588];

*in methylene chloride [2329].

-Also obtained by reaction of glutaric anhydride with anisole in the presence of aluminium chloride in tetrachloroethane/nitrobenzene (1:2) at  $0-5^{\circ}$  [588].

-Also obtained by oxidation (ozonization) of 1,2-bis-(p-methoxyphenyl) cyclopentene [2169].

-Also refer to: [1890 (40 %)].

Isolation from natural sources

-In the New Zealand manuka honey [790].

m.p. 101° [588, 2694], 99.8–100.2° [2169], 99.5–100.5° [2644], 99–101° [2329], 99–100° [1148], 99° [902, 1575, 1576, 1890, 3288], 97–98° [1155], 95–96° [1867];

¹H NMR [1867], ¹³C NMR [1867], IR [588, 1867]; GC-MS [790].

Di-2,4-dinitrophenyl of the dimethyl ethe		$C_{31}H_{28}N_8O_{10}\\$	mol. wt. 672.61
m.p. 245–246° [21	69].		
Diethyl ether	[101684-67-7]	$C_{21}H_{24}O_4$	mol. wt. 340.42
-Refer to: [1890 (35 9	%), 2466, 2469, 328	8].	
m.p. 133° [3288],	112.5° [1890].		
Dipropyl ether	[101684-68-8]	$C_{23}H_{28}O_4$	mol. wt. 368.47
-Refer to: [1890 (30 9	%), 2466, 2469].		
m.p. 88° [1890].			
Dibutyl ether	[101684-69-9]	$C_{25}H_{32}O_4$	mol. wt. 396.53
-Obtained by reaction of aluminium chloric -Also refer to: [1890,	le in methylene chlo		ether in the presence
m.p. 95–97° [2329	], 85° [1890].		
Dipentyl ether	[101684-71-3]	$C_{27}H_{36}O_4$	mol. wt. 424.58
-Refer to: [1890 (37 9	%), 2466].		
m.p. 75° [1890].			
Dihexyl ether	[104192-31-6]	$C_{29}H_{40}O_4$	mol. wt. 452.63
-Refer to: [1890 (33 9	%), 2468].		
m.p. 93–95° [2468], 85° [1890].			
Diphenyl ether	C ₂₉ H	I ₂₄ O ₄	mol. wt. 436.51
-Obtained by reaction of glutaryl dichloride with diphenyl oxide in the presence of aluminium chloride in methylene chloride [2329].			
m.p. 99–101° [232	9].		

### 1-(2-Hydroxyphenyl)-5-(4-hydroxyphenyl)-1,5-pentanedione

[101597-59-5]	(	$C_{17}H_{16}O_4$	mol. wt. 284.31
	НQ	Syntheses	
		-Obtained by Fries	rearrangement of phenyl

HO  $\leftarrow$  CO(CH₂)₃CO  $\leftarrow$  glutarate with aluminium chloride, -in the presence of solvents,

*in the tetrachloroethane/nitrobenzene mixture (500 ml/125 ml) for 4 h at  $50-60^{\circ}$ [902]:

*in nitrobenzene for 4 h at  $50^{\circ}$ , then 30 min at  $60^{\circ}$  [902]; *in tetrachloroethane at  $80^{\circ}$  [902].

-without solvent at 120° [902].

m.p. 141° [902].

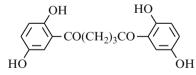
#### 1,5-Bis(2,4-dihydroxyphenyl)-1,5-pentanedione

[16093-22-4]	(	$C_{17}H_{16}O_6$		mol. wt. 316.31
НО-СО(С	HO CH ₂ ) ₃ CO	-OH cinol preser nitrob	ned by conden with glutaryl	sation of resor- chloride in the ium chloride in
m.p. 225° [592]	, 201–202° [586]			
Dioxime	[16093-23-5]	$C_{17}H_{1}$	₈ N ₂ O ₆	mol. wt. 346.34
m.p. 238° [592]				
Di-2,4-dinitropher	nylhydrazone [	[96273-02-8]	$C_{29}H_{24}N_8O_{12}\\$	mol. wt. 676.56
m.p. 290° [586]				

**Tetramethyl ether** [95002-59-8] C21H24O6 mol. wt. 372.42 -Refer to: [586]; m.p. 115° [586].

### 1,5-Bis(2,5-dihydroxyphenyl)-1,5-pentanedione

[91453-24-6] C₁₇H₁₆O₆ mol. wt. 316.31



Synthesis -Obtained by treatment of 1,5-bis(2-hydroxy-5-methoxyphenyl)-1,5-pentanedione with boron tribromide in methylene chloride (80 %) [2103].

yellow solid [2103]; m.p. 211–213° [2103].

 $C_{21}H_{24}O_6$ **Tetramethyl ether** [10365-22-7] mol. wt. 372.42

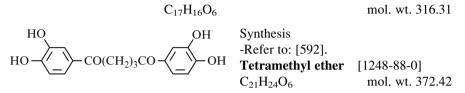
-Obtained by reaction of glutaric acid dichloride (1 mol) with hydroquinone dimethyl ether (2.5 mol) in the presence of aluminium chloride (1 mol) (64 %) [1575].

-Also obtained by reaction of glutaric acid dichloride with hydroquinone dimethyl ether in the presence of aluminium chloride in carbon disulfide or methylene chloride (53–56 %) [1402]; in carbon disulfide for 3 h at 0°, then 13 h at r.t. (67 %) [2159].

-Also refer to: [1575].

m.p. 66° [1575], 64–65° [2159]; IR [2159].

### 1,5-Bis(3,4-dihydroxyphenyl)-1,5-pentanedione



-Obtained by condensation of veratrole with glutaryl chloride in the presence of aluminium chloride in tetrachloroethane [592] or in carbon disulfide (12 %) [2376]. -Also obtained by condensation of veratrole with glutaric acid in the presence of boron trifluoride at 80–90° for 2 h (15 %) [2376].

-Also obtained by condensation of veratrole with glutaric anhydride [2524]. -Also refer to: [1014 (85 %)].

m.p. 144-146° [2524], 124° [592], 121° [2376], 120-121° [1014].

**Dioxime of the tetramethyl ether**  $C_{21}H_{26}N_2O_6$  mol. wt. 402.44

m.p. 146° [2376], 112–114° [1014].

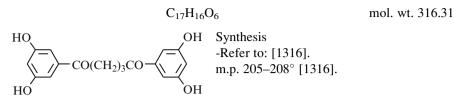
#### Di-methyloxime of the tetramethyl ether

[50766-27-3]	$C_{23}H_{30}N_2O_6$	mol. wt. 430.50
m.p. 112–114° [1014].		

#### Di-2,4-dinitrophenylhydrazone of the tetramethyl ether

[16148-93-9]	$C_{33}H_{32}N_8O_{12}$	2	mol. wt. 732.66
m.p. 200–202° [2524	4], 150° [592].		
Tetrabutyl ether	[123387-95-1]	C33H48O6	mol. wt. 540.74
-Refer to: [97].			
¹ H NMR [97].			
Tetradecyl ether	[123387-96-2]	C ₅₇ H ₉₆ O ₆	mol. wt. 877.38
-Refer to: [97].			
¹ H NMR [97].			

#### 1,5-Bis(3,5-dihydroxyphenyl)-1,5-pentanedione



#### 1,5-Bis(2,3,4-trihydroxyphenyl)-1,5-pentanedione

C₁₇H₁₂O₂

$$\begin{array}{c} C_{17}H_{16}O_8 & \text{mol. wt. 348.31} \\ HO & OH & HO & OH \\ HO & CO(CH_2)_3CO & OH & -Refer to: [1574]. \\ Hexamethyl ether \\ C_{23}H_{28}O_8 & \text{mol. wt. 432.47} \end{array}$$

-Obtained reaction of dimethyl sulfate with 1,5-bis(2-hydroxyby 3,4-dimethoxyphenyl)-1,5-pentanedione the presence of 30 % sodium hydroxide (65-90 %) [1574].

-Also refer to: [1575].

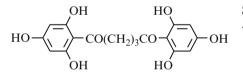
m.p. 84° [1574, 1575].

#### 1,5-Bis(2,4,6-trihydroxyphenyl)-1,5-pentanedione

[16093-48-4]

C17H16O8

mol. wt. 348.31



**Syntheses** 

-Obtained by reaction of glutaryl chloride with phloroglucinol in the presence of aluminium chloride in a nitrobenzene/ tetrachloroethane mixture [592].

-Also refer to: [3374].

m.p. 276° [592].

m.p. 288° [592], 286–286.5° (d) [3374].

#### Di-2,4-dinitrophenylhydrazone

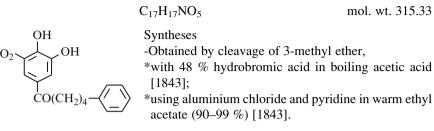
[16093-15-5]

C29H24N8O14

mol. wt. 708.56

522

#### 1-(3,4-Dihydroxy-5-nitrophenyl)-5-phenyl-1-pentanone

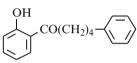


m.p. 109–111° [1843]; ¹H NMR [1843], ¹³C NMR [1843], IR [1843]; HPLC [1843].

#### 1-(2-Hydroxyphenyl)-5-phenyl-1-pentanone

[37765-93-8]

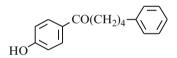
C₁₇H₁₈O₂ mol. wt. 254.33



### 1-(4-Hydroxyphenyl)-5-phenyl-1-pentanone

(Daphnolon)

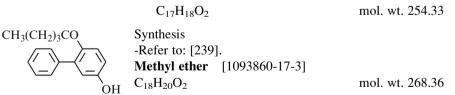
$C_{17}H_{18}O_2$	mol. wt. 254.33
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Isolation from natural product -From Callus cells of *Daphne Giraldii* [3263].

BIOLOGICAL ACTIVITY: Cytotoxic [3263].

#### 1-[1,1'-Biphenyl]-2-yl-5-hydroxy-1-pentanone



-Refer to: [239].

USE: Preparation of aminobiphenylcyclopentanecarboxamide derivs. for use as renin inhibitors [239].

#### 1-[1,1'-Biphenyl]-3-yl-2-hydroxy-1-pentanone

HO 
$$CO(CH_2)_3CH_3$$
 Syntheses  
-Obtained by Fries rearrangement of 2-(valeroyloxy)-biphenyl with aluminium chloride at 160° for 30–45 min (20 %) [1257].

-Also refer to: [661].

b.p.₅ 200–210° [661, 1257].

#### Methyl ether

 $C_{18}H_{20}O_2$ 

mol. wt. 268.36

-Refer to: [504, 661, 1257].

b.p.₄ 202–204° [661, 1257], b.p.₂₇ 275–280° [504];  $n_D^{27}=1.5857$  [504].

#### 1-(1,1'-Biphenyl)-4-yl-3-hydroxy-1-pentanone

[792706-16-2]  $C_{17}H_{18}O_2$  mol. wt. 254.33  $\swarrow -CO(CH_2)_3CH_3$  Synthesis -Refer to: [2553].

#### 1-[1,1'-Biphenyl]-5-yl-2-hydroxy-1-pentanone

-Also obtained by treatment of its methyl ether wit pyridinium chloride for 1 h at reflux [504].

-Also refer to: [661, 1595].

Fine colourless needles [504]; m.p. 104° [661, 1257], 75° [504].

USE: Colour developer, for thermal recording materials [2704].

Methyl ether [854659-36-2] C₁₈H₂₀O₂ mol. wt. 268.36

-Obtained by reaction of valeroyl chloride (or valeroyl anhydride [1334]) with 2-methoxydiphenyl in the presence of aluminium chloride in carbon disulfide for 4 h at r.t. (85 %) or for 2 h at reflux (40 %) [1257].

pale yellow oil [504]; b.p.₄ 202–204° [1257], b.p.₂₇ 275–280° [504];  $n_D^{27} = 1.5857$  [504].

#### Semicarbazone of the methyl ether

$$C_{19}H_{23}N_3O_2$$
 mol. wt. 325.41

m.p. 141° [504].

### 1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-1-pentanone

[219513-03-8] 
$$C_{17}H_{18}O_2$$
 mol. wt. 254.33  
HO  $-$  CO(CH₂)₃CH₃ Synthesis  
-Refer to: [1518].

USE: For preparation of biphenylyl sulfamates as steroid sulfatase inhibitors for estrogen-dependent diseases [1518].

Acetate	[214534-24-4]	$C_{19}H_{20}O_3$	mol. wt. 296.36
---------	---------------	-------------------	-----------------

-Obtained by reaction of pentanoyl chloride with 4-acetoxybiphenyl in the presence of aluminium chloride in methylene chloride at  $0-5^{\circ}$  for 6 h (63.1 %) [2344].

Methyl ether	[56116-77-9]	$C_{18}H_{20}O_2$	mol. wt. 268.36
-Refer to: [847]			

Refer to: [84/].

Octyl ether	[56117-37-4]	$C_{25}H_{34}O_2$	mol. wt. 366.54
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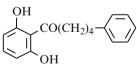
USE: Lamellar liquid crystals comprising [920].

### 1-(2,4-Dihydroxyphenyl)-5-phenyl-1-pentanone

[20038-59-9]	$C_{17}H_{18}O_3$	mol. wt. 270.33
HO CO(CH ₂ ) ₄	Synthesis -Refer to: [1010]. m.p. 80–83° [1010].	

#### 1-(2,6-Dihydroxyphenyl)-5-phenyl-1-pentanone

	$C_{17}H_{18}O_3$	mol. wt. 270.33
/=\	Synthesis Refer to: [1389]	



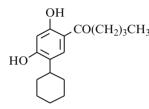
 $CO(CH_2)_4$  - Refer to: [1389]. ¹H NMR [1389], IR [1389].

### 1-(2-Hydroxy-4-phenoxyphenyl)-1-pentanone

[307000-44-8]	$C_{17}H_{18}O_3$	mol. wt. 270.32
OH CO(CH	Synthesis $H_2$ ) ₃ CH ₃ -Refer to: [1345].	
C ₆ H ₅ O	USE: Preparation of hyoantimicrobials [1345].	droxydiphenyl ethers as
1 (5 Couldbarred 2.4 )	191	

### 1-(5-Cyclohexyl-2,4-dihydroxyphenyl)-1-pentanone

 $[159977-39-6] C_{17}H_{24}O_3 mol. wt. 276.38$ 

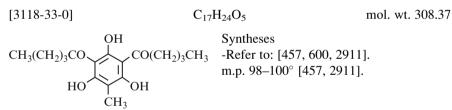


CO(CH₂)₃CH₃ -Obtained (**XIX**) by reaction of valeric acid with 4-cyclohexylresorcinol in the presence of boron trifluoride etherate at 105–108° for 15 min, the hydrolysis of the BF₂-chelate (**IX**) obtained [2382]. m.p. 134–135° [2382]; IR [2382], UV [2382].

**BF2-chelate** (**IX**) C₁₇H₂₃BF₂O₃ mol. wt. 324.17 (61 %) [2382].

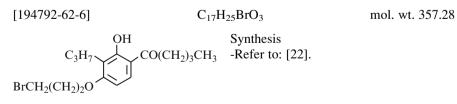
m.p. 106–107° [2382]; IR [2382].

### 1,1'-(2,4,6-Trihydroxy-5-methyl-1,3-phenylene)bis-1-pentanone



BIOLOGICAL ACTIVITY: For treatment of immune dysfunction associated with human immunodeficiency virus infection [600]; Anthelmintic [457].

### 1-[4-(3-Bromopropoxy)-2-hydroxy-3-propylphenyl]-1-pentanone



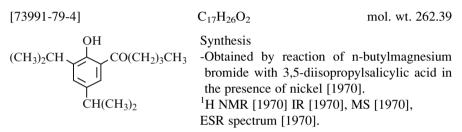
### 1-[2-(β-D-Glucopyranosyloxy)-4,6-dihydroxyphenyl]-1-pentanone

[853913-75-4]	C ₁₇ H ₂₅ O ₉	mol. wt. 373.15
ОН CO(CH ₂ ) ₃ CH ₃ HO	Isolation from natural sources -From the whole plant of (Leguminosae) [209]. colourless gummy solid [209]	Indigofera heterantha

¹H NMR [209], ¹³C NMR [209], IR [209], UV [209], MS [209],

BIOLOGICAL ACTIVITY: Lipoxygenase enzyme inhibitor [209].

### 1-[2-Hydroxy-3,5-di(1-methylethyl)phenyl]-1-pentanone



### ion(1-), radical ion (1-)

[57133-44-5].

-Formation of, in reaction of diisopropylsalicylic acid with butylmagnesium bromide, ESR spectrum of [1970].

### 1-[4-Hydroxy-3-(hydroxymethyl)-2-methoxy-5-(2-methylpropyl)phenyl]-1-pentanone

[798559-89-4] C17H26O4 mol. wt. 294.39

 $(CH_3)_2CHCH_2 + CH_2OH + CH_2OH + OCH_3 + O$ in propanone. The intermediate obtained (93 %) was methylated by reaction with methyl iodide in the presence of potassium carbonate in propanone at 57°, followed by deprotection of the acetal (60 %) [816].

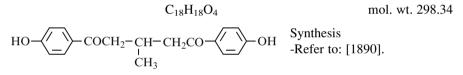
-The synthesis of this ketone was accomplished in eight steps and 33 % overall yield [2310] in accordance to [816].

m.p. 60-61° [816]; ¹H NMR [816], ¹³C NMR [816], IR [816].

#### 1-[4-[(Dimethylamino)methyl]-2-hydroxy-3-propylphenyl]-1-pentanone

¹H NMR [2925], ¹³C NMR [2925], IR [2925], UV [2118].

#### 1,5-Bis(4-hydroxyphenyl)-3-methyl-1,5-pentanedione



**Dimethyl ether** 

 $C_{20}H_{22}O_4$ 

mol. wt. 326.39

-Refer to: [1890, 3288].

### 1-(3'-Fluoro-4'-hydroxy[1,1'-biphenyl]-4-yl)-3-methyl-1-pentanone

 $C_{18}H_{19}FO_{2} \qquad \text{mol. wt. } 286.35$   $F \qquad CH_{3} \qquad Fefer to: [2107].$ 

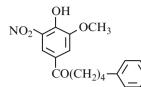
Heptyl ether (S)	[112780-60-6]	$C_{25}H_{33}FO_2$	mol. wt. 384.53

USE: Liq.-crystal compns. contg., for display devices [2107].

 $\label{eq:constraint} \mbox{Octyl ether (S)} \qquad [112780\mbox{-}61\mbox{-}7] \qquad C_{26}H_{35}FO_2 \qquad \mbox{mol. wt. } 398.56$ 

USE: Liq.-crystal compns. contg., for display devices [2107].

#### 1-(4-Hydroxy-3-methoxy-5-nitrophenyl)-5-phenyl-1-pentanone



Synthesis -Obtained by regioselective nitration under mild conditions of 1-(4-hydroxy-3-methoxyphenyl)-5-phenyl-1-pentanone with 70 % nitric acid in acetic acid (67–77 %) [1843].

### 1-(6-Hydroxy-3'-methoxy[1,1'-biphenyl]-3-yl)-1-pentanone

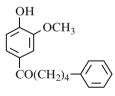
 $[565203-88-5] C1_{18}H_{20}O_3 mol. wt. 284.35$   $CH_3O CO(CH_2)_3CH_3 Synthesis -Obtained by treatment of its methyl ether with boron tribromide in methylene chloride [1885].$ 

1-(3',6-Dimethoxy[1,1'-biphenyl]-3-yl)-1-pentanone

-Obtained treatment of the mixture of 3-iodo-4-methoxyvalerophenone and 3-methoxyphenyl-boronic acid with Pd(PPh₃)₄ [1885].

#### 1-(4-Hydroxy-3-methoxyphenyl)-5-phenyl-1-pentanone

$$C_{18}H_{20}O_3$$
 mol. wt. 284.35



Synthesis

-Selective deprotection of the benzyl-protecting group proceeded smoothly under acidic conditions (excess 30 % HBr in acetic acid and methylene chloride) at r.t. for 2 h (75–87 %) or more conveniently by catalytic hydrogen transfer using ammonium formate as hydrogen donor and palladium catalysis to provide the phenol (84–92 %) [1843].

#### **Benzyl ether**

-Obtained from 1-(4-benzyloxy-3-methoxyphenyl)-5-phenyl-1-pentanol by Oppenauer oxidation using sodium tert-butoxide as base and cyclohexanone as hydrogen acceptor in warm toluene (87–94 %) [1843].

### 1-[3-(N-Chloroacetylaminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-pentanone

C₁₈H₂₆ClNO₃ mol. wt. 339.86 Synthesis OH CO(CH₂)₃CH₃ -Obtained by adding N-hydroxyme-CICH2CONHCH2 thylchloro-acetamide

to 2-valervl-4-tert-butylphenol dissolved in acetic acid and conc. sulfuric acid mixture at r.t. and stirred at  $60^{\circ}$  for 2 h (60 %) [1475].

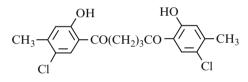
¹H NMR [1475], IR [1475]; TLC [1475].

 $C(CH_3)_3$ 

### 1,5-Bis(5-chloro-2-hydroxy-4-methylphenyl)-1,5-pentanedione

[4609-08-9]

C19H18Cl2O4



m.p. 160° [588]; IR [588].

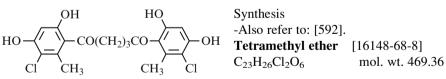
**Dioxime** [4609-09-0] C19H20Cl2N2O4 mol. wt. 411.28

m.p. 210° [588].

### 1,5-Bis(5-chloro-2,4-dihydroxy-6-methylpheny)l-1,5-pentanedione



mol. wt. 413.25



-Obtained by condensation of 4-chloroorcinol dimethyl ether with glutaryl chloride in the presence of aluminium chloride [592].

m.p. 145° [592].

### Di-2,4-dinitrophenylhydrazone of the tetramethyl ether

$$[16093-49-5] C_{35}H_{34}Cl_2N_8O_{12} mol. wt. 829.61$$

m.p. 176° [592].

mol. wt. 381.26

Synthesis

-Obtained by reaction of glutaric anhydride with 4-chloro-3-methylphenol in the presence of aluminium chloride in tetrachloro-ethane/ nitrobenzene mixture (1:1) [588].

#### 1,5-Bis(2-hydroxy-3-methylphenyl)-1,5-pentanedione

[10365-65-8]		$C_{19}H_{20}O_4$		mol. v	wt. 312.36
CH ₃ OH	HO CH ₃		with	rearrangement c aluminium	of o-cresol chloride

-Also refer to: [1575].

m.p. 105° [1575, 1576].

#### 1,5-Bis(2-hydroxy-4-methylphenyl)-1,5-pentanedione

[10571-10-5]

 $C_{19}H_{20}O_4$ 

mol. wt. 312.36



Syntheses

-Obtained by Fries rearrangement of m-cresol glutarate with aluminium chloride (70–80 %) [1576].

-Also refer to: [1575].

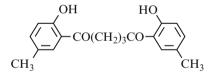
m.p. 180° [1575, 1576].

#### 1,5-Bis(2-hydroxy-5-methylphenyl)-1,5-pentanedione

[4649-03-0]

 $C_{19}H_{20}O_4$ 

mol. wt. 312.36



Syntheses

-Obtained by Fries rearrangement of di (4-methyl-phenyl) glutarate with aluminium chloride,

*in refluxing chlorobenzene for 6 h (66 %) [3107];

*in tetrachloroethane at  $80^{\circ}$  for 1.5 h (61 %) [1603].

-Also obtained by reaction of glutaric anhydride with p-cresol in the presence of aluminium chloride in tetrachloroethane/nitrobenzene mixture (1:1), first at  $50-60^{\circ}$  for 2 h, then at  $100^{\circ}$  for 30 min [588].

-Also obtained by reaction of glutaryl chloride with p-methylanisole in the presence of aluminium chloride in refluxing methylene chloride for 1 h (86 %) [889]. -Also refer to: [889, 890, 941 (64 %), 1575, 2250].

m.p. 143° [1603], 141° [941], 140–141° [3107], 140° [588], 139–141° [889], 69° [1575].

N.B.: One of the reported melting point is obviously wrong.

¹H NMR [889], IR [588, 3107].

BIOLOGICAL ACTIVITY: Inhibition against HIV-1 integrase [2250].

#### Dioxime [4649-04-1]

m.p. 175° [588].

Dimethyl ether	10400-49-4]	$C_{21}H_{24}O_4$	mol. wt. 340.42

-Obtained by reaction of glutaryl chloride with p-cresol methyl ether in the presence of aluminium chloride (50-70 %) [1576], in methylene chloride [2329]. -Also obtained by reaction of glutaric anhydride with p-cresol methyl ether [3064]. obtained treatment of 1,5-bis(2-hydroxy-5-methylphenyl)--Also bv 1,5-pentanedione with dimethyl sulfate in the presence of sodium hydroxide in dilute ethanol (93 %) [1603].

m.p. 71–72° [1603], 69° [1576], 68.5–69.5° [3064], 68–70° [2329]; ¹H NMR [3064], IR [3064].

#### 1,5-Bis(4-hydroxy-2-methylphenyl)-1,5-pentanedione

[4642-34-6]	$C_{19}H_{20}$	$O_4$	mol. wt. 312.36
но — СН3	CH ₂ ) ₃ CO-CH ₃ -OH	Synthesis -Obtained by reaction ride with m-cresol in aluminium chloride [588].	n the presence of
m.p. 178° [588	3]; IR [588].		
Dioxime	[4592-84-1]	$C_{19}H_{22}N_2O_4$	mol. wt. 342.39
m.p. 225° [588	5].		

Dimethyl ether	[4642-38-0]	$C_{21}H_{24}O_4$	mol. wt. 340.42
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-Obtained by reaction of glutaryl chloride with m-cresol methyl ether in the presence of aluminium chloride in tetrachloroethane [588].

b.p._{0.2} 150° [588]; IR [588].

#### 1,5-Bis(4-hydroxy-3-methylphenyl)-1,5-pentanedione

$$[4592-83-0] C_{19}H_{20}O_4$$
mol. wt. 312.36  

$$CH_3 CH_3 CH_3 - CO(CH_2)_3CO - OH CH_3 - Obtained by reaction of glutaryl chloride in nitrobenzene of aluminium chloride in nitrobenze$$

[588].

m.p. 175° [588]; IR [588].

 $C_{19}H_{22}N_2O_4$ 

mol. wt. 342.39

**Dioxime** [4642-26-6]  $C_{19}H_{22}N_2O_4$  mol. wt. 342.39

m.p. 220° [588].

Dimethyl ether	[4642-31-3]	$C_{21}H_{24}O_4$	mol. wt. 340.42
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-Obtained by reaction of glutaryl chloride with o-cresol methyl ether in the presence of aluminium chloride,

*in tetrachloroethane [588];

*in methylene chloride [2329].

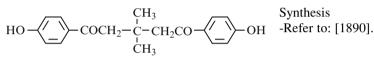
m.p. 135–137° [2329], 130° [588]; IR [588].

#### 1,5-Bis(4-hydroxyphenyl)-3,3-dimethyl-1,5-pentanedione



 $C_{21}H_{24}O_4$ 

ride (80 %) [2103].



**Dimethyl ether** [102447-83-6]

-Refer to: [1328, 1890].

¹H NMR [1328], IR [1328].

### 1,5-Bis(2,5-dihydroxy-4-methylphenyl)-1,5-pentanedione

 $[91453-25-7] C_{19}H_{20}O_6 mtext{mol. wt. 344.36} \\ CH_3 \longrightarrow CO(CH_2)_3CO \longrightarrow CH_3 \\ HO OH OH OH Synthesis -Obtained by treatment of 1,5-bis (2-hydroxy-5-methoxy-4-methyl-phenyl)-1,5-pentane-dione with boron tribromide in methylene chlo-$ 

yellow solid [2103]; m.p. 202–203° [2103].

#### 

-Obtained by reaction of glutaric acid dichloride with 2,5-dimethoxytoluene in the presence of aluminium chloride,

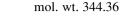
*in methylene chloride first at  $0^{\circ}$  under an argon atmosphere, then at r.t. for 16 h (67 %) [2570];

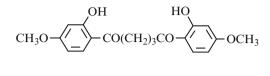
*in carbon disulfide first at  $0^{\circ}$ , then at r.t. for 16 h (50 %) [1402].

colourless needles [1402]; m.p. 156° [1402], 152–153° [2570]; ¹H NMR [2570], ¹³C NMR [2570], IR [2570]. mol. wt. 340.42

### 1,5-Bis(2-hydroxy-4-methoxyphenyl)-1,5-pentanedione

[4642-40-4]  $C_{19}H_{20}O_{6}$ 





Syntheses

-Obtained by reaction of glutaryl chloride with resorcinol monomethyl ether in the presence of aluminium chloride in nitrobenzene [588].

-Also obtained by reaction of glutaric anhydride with resorcinol monomethyl ether in the presence of aluminium chloride in nitrobenzene, first at 10-15° for 2 h, then at 30° for 1 h [588].

-Also obtained by reaction of resorcinol dimethyl ether with glutaryl chloride in the presence of aluminium chloride in nitrobenzene [592].

m.p. 130° [588, 592]; IR [588].

<b>Dioxime</b> [4714-77-6]	$C_{19}H_{22}N_2O_6$	mol. wt. 374.39
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m.p. 190° [588, 592].

#### 1,5-Bis(2-hydroxy-5-methoxyphenyl)-1,5-pentanedione

[10365-74-9]	C ₁₉ H	₂₀ O ₆ mol. wt. 3	44.36
CH ₃ O	(CH ₂ ) ₃ CO-CH ₃	Syntheses -Obtained by reaction of gl dichloride with p-dimethoxybenze the presence of aluminium ch [1575] in refluxing 1,2-ethylene ch for 8 h [2103].	ene in

m.p. 122° [1575].

 $C_{23}H_{24}O_8$ Diacetate [10365-32-9] mol. wt. 428.44

-Obtained by reaction of acetic anhydride (4 vol) with the title ketone in the presence of 1-2 drops concentrated sulfuric acid, the mixture warmed 5 min [1575].

m.p. 119° [1575].

### 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-pentanone

[14035-37-1]	$C_{19}H_{30}O_2$	mol. wt. 290.45
$(CH_3)_3C$ $C(CH_3)_3$ $C(CH_3)_3$ $CO(CH_2)_3CH_3$	Syntheses -Preparation by reaction of 2,6-di-tert-butylphenol in the chloride [2145] at -10° for -Also obtained by reaction of 2,6-di-tert-butylphenol in the 70 % perchloric acid, first overnight (91 %) [2136].	e presence of aluminium 1–13 min (91 %) [2506]. of valeric anhydride with the presence of 5 drops

-Also refer to: [655, 951, 2139].

m.p. 89–90° [951], 85.5–87° [2139], 76–77° [2136], 75–78° [2506]; ¹H NMR [2136], IR [2136].

BIOLOGICAL ACTIVITY: Inflammation inhibitor [2139].

Acetate	[903883-85-2]	$C_{21}H_{32}O_3$	mol. wt. 332.48
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-Obtained by acetylation of 3,5-di-tert-butyl-4-hydroxyvalerophenone [2145].

¹H NMR [2145], IR [2145].

### 1-(2,5-Dihydroxy-4-octylphenyl)-1-pentanone

[103798-50-1]  $C_{19}H_{30}O_3$  mol. wt. 306.45 OH Syntheses  $\downarrow$  CO(CH₂)₂CH₂ -Obtained by reaction of valeric acid with

 $(CO(CH_2)_3CH_3)$  -Obtained by reaction of valeric acid with 2-octyl-hydroquinone in the presence of boron trifluoride in *sym*-tetrachloroethane at 40–50°. The mixture was allowed to stand overnight, then heated on a steam bath for 6 h (65 %) [142].

-Also refer to: [1907].

CH₃(CH₂

m.p. 59-60° [142].

### 1-(4-Hydroxy-3-methyl-6-phenyl-2-benzofuranyl)-1-pentanone

[678184-56-0]	$C_{20}H_{20}O_3$	mol. wt. 308.78

$C_6H_5$ $O$ $CO(CH_2)_3CH_3$	
	-
CH ₃	
ОН	

Synthesis -Obtained by treatment of its methoxymethyl ether with hydrogen chloride in a tetrahydrofuran/methanol solution (1:1) [1288].

mol. wt. 352.43

mol. wt. 340.42

mol. wt. 372.42

mol. wt. 404.41

#### Methoxymethyl ether [678184-70-8]

-Refer to: [1288].

[175731-80-3]

OH

#### 1,5-Bis(2-hydroxyphenyl)-3-tert-butyl-1,5-pentanedione

HO

**Dimethyl ether** 
$$C_{23}H_{28}O_4$$
 mol. wt. 368.47  
-Preparation by refluxing trimethylacetaldehyde with 2-methoxyacetophenone in

the presence of methanolic sodium methoxide for 18 h (38 %) [890].

m.p. 82–83° [890]; ¹H NMR [890].

Ċ(CH₃)₃

m.p. 88–89° [890]; ¹H NMR [890].

### 1,5-Bis(2-hydroxy-5-methoxy-4-methylphenyl)-1,5-pentanedione

[344578-29-6] C21H24O6 OH HC CO(CH₂)₃C CH₃ CH₃O OCH₃

**Synthesis** -Obtained by reaction of glutaryl dichloride with 2,5-dimethoxytoluene in the presence of aluminium chloride in refluxing 1,2-dichloroethane for 8 h [2103].

### 1,5-Bis(2-hydroxy-3,4-dimethoxylphenyl)-1,5-pentanedione

C₂₁H₂₄O₈

CH₃O OH HO OCH₂ OCH₃

Syntheses -Obtained by reaction of glutaric acid dichloride with pyrogallol trimethyl ether in the presence of aluminium chloride,

*in tetrachloroethane [1574]; *in nitrobenzene [592]. -Also refer to: [1575, 1890].

[10483-67-7]

Dioxime

m.p. 125° [592], 123° [1574, 1575].

[16148-67-7]

C21H26N2O8

mol. wt. 434.45

m.p. 167–168° [592].

CH₃

Synthesis -Obtained by treatment of its dimethyl ether with excess of trimethylsilyl iodide at 85° for 7 h (80 %) [890].

 $C_{22}H_{24}O_{4}$ 

 $C_{21}H_{24}O_4$ 

#### 1-[2,4,6-Trihydroxy-3,5-bis(3-methyl-2-buten-1-yl)phenyl]-1-pentanone

$$[50874-43-6] C_{21}H_{30}O_{4}$$

$$(CH_{3})_{2}C = CHCH_{2} \qquad OH \qquad S$$

$$HO \qquad OH \qquad CO(CH_{2})_{3}CH_{3} \qquad OH$$

$$HO \qquad OH \qquad CH_{2}CH = C(CH_{3})_{2} \qquad HO$$

Syntheses -Obtained by reaction of resvalerophenone with 1-bromo-3-methyl-2-buten (3 mol) in the presence of the weakly basic resin DeAcidite H-IP ( $OH^{-}$  form) in boiling benzene (13.8 %) [708].

**N.B.**: The yield was similar at this obtained by using 2-methyl-3-buten-2-ol but the isolation was simpler [708].

-Also refer to: [707].

m.p. 86° [708].

#### 1,1'-[Sulfonylbis(6-chloro-4-hydroxy-3,1-phenylene)]bis-1-pentanone

[50444-96-7] mol. wt. 471.40 C22H24Cl2O5S CO(CH₂)₃CH₃ Synthesis CH₃(CH₂)₃CC -Obtained by treatment of 4-chloro-2-hydroxyvalerophenone with thionyl chloride in the presence of aluminium chloride in carbon disulfide (58 %) [2430].

USE: Antifungal [2430].

#### 1,1'-(4,4'-Dihydroxy[1,1'-biphenyl]-3,3'-diyl)-1-pentanone

[103650-06-2]

C22H26O4

mol. wt. 354.45

CH₃(CH₂)₃CO

CO(CH₂)₃CH₃ Syntheses

-Preparation by Fries rearrangement of 4,4'-biphenyl dipentanoate with aluminium chloride in refluxing chlorobenzene for 24 h (92 %) [2377].

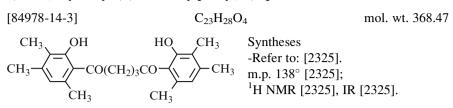
-Also refer to: [226].

m.p. 70–80.5° [2377]; IR [2377].

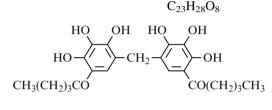
BIOLOGICAL ACTIVITY: Antibacterial against a cariogenic bacterium, Streptococcus mutans OMZ 176 [226].

mol. wt. 346.47

#### 1,5-Bis(2-hydroxy-3,4,6-trimethylphenyl)-1,5-pentanedione



#### 1,1'-[Methylenebis(2,3,4-trihydroxy-5,1-phenylene)]bis-1-pentanone

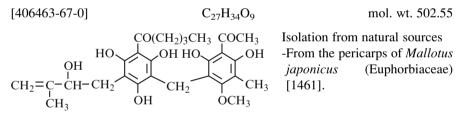


mol. wt. 432.47

Synthesis -Obtained by treatment of 4-valeroyl-pyrogallol with 35 % aqueous solution of formaldehyde in refluxing ethanol for 10 min [506].

m.p. 173° [506].

1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl) methyl]-2,4,6-trihydroxy-5-(2-hydroxy-3-methyl-3-butenyl) phenyl]-1-pentanone

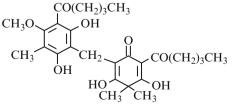


3'-[(5-Valeroyl-2,4-dihydroxy-3,3-dimethyl-6-oxo-1,4-cyclohexadien-1-yl) methyl]-2',6'-dihydroxy-4'-methoxy-5'-methylvalerophenone (*Aspidin*)

 $C_{27}H_{36}O_8$ 

mol. wt. 488.58

Isolation from natural sources -From *Hypericum uliginosum* HBK (**XIX**) [2408]. UV [2408].



#### 1,5-Bis(5-bromo-2,4-dihydroxyphenyl)-2,4-diphenyl-1,5-pentanedione



-Obtained by condensation 5-bromo-2,4-dihydroxy-phenyl benzyl ketone with methylene iodide in the presence of sodium ethoxide [587].

silky needles [587]; m.p. 225° [587]; IR [587].

Tetraacetate [96710-34-8] C37H30Br2O10 mol. wt. 794.45 rectangular plates [587]; m.p. 140° [587].

#### 1,5-Bis(2,4-dihydroxyphenyl)-2,4-di(4-bromophenyl)-1,5-pentanedione

[96271-44-2]

 $C_{20}H_{22}Br_2O_6$ 

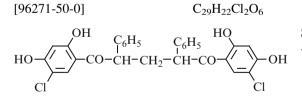
HO OH Synthesis -Obtained by condensation

mol. wt. 626.30

of 2,4-dihydroxyphenyl 4'-bromobenzyl ketone with methylene iodide in the presence of sodium ethoxide [587].

prismatic rods [587]; m.p. 265° [587].

#### 1,5-Bis(5-chloro-2,4-dihydroxyphenyl)-2,4-diphenyl-1,5-pentanedione



mol. wt. 537.40

Synthesis -Obtained by condensation of 5-chloro-2,4-dihydroxyphenyl benzyl ketone with methylene iodide in the presence of sodium ethoxide [587].

silky needles [587]; m.p. 229–230° [587]; IR [587], UV [587].

C37H30Cl2O10 mol. wt. 705.54 Tetraacetate [96710-35-9] rectangular plates [587]; m.p. 125° [587].

#### 1,5-Bis(2,4-dihydroxyphenyl)-2,4-diphenyl-1,5-pentanedione

$$[96273-22-2] \qquad C_{29}H_{24}O_6 \qquad \text{mol. wt. 468.51}$$

-Obtained by condensation of 2,4-dihydroxyphenyl benzyl ketone with methylene iodide in the presence of sodium ethoxide [587].

colourless rods [587]; m.p. 192° [587]; IR [587], UV [587], MS [587].

усо-с́н-сн₂-с́н-со-√ у

**Tetramethyl ether** [96676-42-5]  $C_{33}H_{32}O_6$  mol. wt. 524.61 prismatic rods; m.p. 136–137° [587].

#### 1,5-Bis(2,4-dihydroxyphenyl)-2,4-di(4-methylphenyl)-1,5-pentanedione

 $C_{31}H_{28}O_6$   $CH_3$   $CH_3$  HO OH HO HO CO CH  $CH_3$   $CH_3$  HO HO HO CO CO CH  $CH_3$   $CH_3$ CH

rods [587]; m.p. 218° [587]; IR [587].

#### Tetraacetate

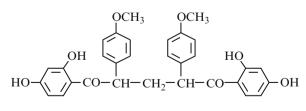
rectangular plates [587]; m.p. 132° [587].

#### 1,5-Bis(2,4-dihydroxyphenyl)-2,4-di(4-methoxyphenyl)-1,5-pentanedione

[96590-58-8]



C₃₉H₃₆O₁₀



Synthesis

mol. wt. 528.56

-Obtained by condensation of 2,4-dihydroxyphenyl 4'-methoxybenzyl ketone with methylene iodide in the presence of sodium ethoxide [587].

prismatic rods [587]; m.p. 195° [587].

### Tetraacetate

[97924-31-7]

 $C_{39}H_{36}O_{12}$ 

mol. wt. 696.71

rectangular plates [587]; m.p. 130° [587].

mol. wt. 496.56

Synthesis

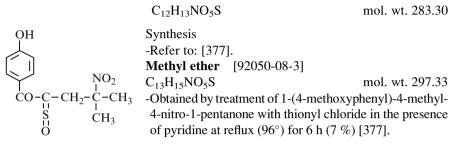
-Obtained by condensation of 2,4-dihydroxyphenyl 4'-methylbenzyl ketone with methylene iodide in the presence of sodium ethoxide [587].

mol. wt. 664.71

# 2 Aromatic Hydroxyketones Derived from 4-Methylpentanoic Acid

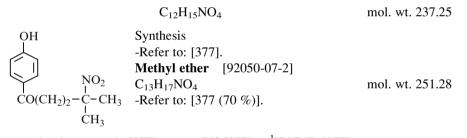
### 2.1 Unsubstituted Hydroxyketones

#### 1-(4-Hydroxyphenyl)-4-methyl-4-nitro-2-sulfinyl-1-pentanone



yellow powder [377]; m.p. 83° [377]; ¹H NMR [377], IR [377].

#### 1-(4-Hydroxyphenyl)-4-methyl-4-nitro-1-pentanone



colourless crystals [377]; m.p. 70° [377]; ¹H NMR [377].

#### 1-(2-Hydroxyphenyl)-4-methyl-1-pentanone

[22526-26-7]	$C_{12}H_{16}O_2$	mol. wt. 192.26
OH CO(CH ₂ ) ₂ CH(CH ₃ ) ₂	Syntheses -Refer to: [1674, 3338]. b.p. _{0.5} 115–120° [3338];	pK _a =4.884 [192].

Methyl ether	[22526-27-8]	$C_{13}H_{18}O_2$	mol. wt. 206.28
D 6 ( [2220]			

-Refer to: [3338].

#### 1-(3-Hydroxyphenyl)-4-methyl-1-pentanone

	$C_{12}H_{16}O_2$	mol. wt. 192.26
OH CO(CH ₂ ) ₂ CH(CH ₃ ) ₂	Synthesis -Preparation by reaction of m-aride with diisohexylcadmium for 30 min. Then, treatment of the by refluxing with 10 % sodium (70 %) [2586].	in refluxing benzene the ketoester obtained

b.p.₂ 158° [2586]; m.p. 70° [2586].

Acetate	$C_{14}H_{18}O_3$	mol. wt. 234.30	b.p. ₁ 119° [2586].
2,4-Dinitrophen	ylhydrazone	$C_{18}H_{20}N_{4}O_{5}\\$	mol. wt. 372.38

m.p. 178° [2586].

#### 1-(4-Hydroxyphenyl)-4-methyl-1-pentanone

 $\begin{array}{ccc} [286439-54-1] & C_{12}H_{16}O_2 & \text{mol. wt. } 192.26 \\ OH & Syntheses \\ -Obtained by treatment of its methyl ether with pyridinium \end{array}$ 

-Obtained by treatment of its methyl ether with pyridinium chloride at  $190^{\circ}$  [627].

-Preparation by Fries rearrangement of phenyl isocaproate with aluminium chloride in nitrobenzene at  $70^{\circ}$  for 6 h (57 %) [2163].

-Also refer to: [2941, 3090].

b.p._{0.5} 190° [2163]; m.p. 71–72° [2163].

Methyl ether	[21550-01-6]	$C_{13}H_{18}O_2$	mol. wt. 206.28

-Obtained by reaction of 4-methylpentanoic acid with anisole in the presence of PPA at 80° (76–100 %) [627] or at 90–100° for 1.5 h (75 %) [2942].

-Also obtained by reaction of 4-methylpentanoyl chloride with anisole [2236] in the presence of aluminium chloride in methylene chloride at  $0^{\circ}$  (76–100 %) [627].

-Preparation by addition of the Grignard from 1-bromo-3-methylbutane to p-methoxybenzonitrile [3254].

-Also refer to: [950, 1114, 2302 (65 %), 3090, 3232, 3253].

pale yellow oil [2942];

b.p._{0.5} 107–108° [2302]; b.p.₁ 131–133° [2236]; b.p.₅ 136–137° [950], b.p.₃ 138° [3254];

b.p.₁₅ 169–170° [3232];

¹H NMR [2942, 3090], ¹³C NMR [2942], IR [3232], UV [3254], MS [2942, 3254]; ESR spectroscopy [3254]; Phosphorescence spectroscopy [3254].

USE: Photoelimination and photocyclisation of, biradical formation in, [3253].

CO(CH₂)₂CH(CH₃)₂

Semicarbazone of the methyl ether [30299-35-5] C₁₄H₂₁N₃O₂ m.p. wt. 263.34 m.p. 124–125° [2236], 133–133.5° [2302].

 Ethyl ether
 [30299-36-6]
  $C_{14}H_{20}O_2$  mol. wt. 220.31

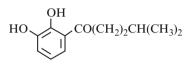
 -Refer to: [2302 (82 %)].
 .

b.p.₁ 132–135° [2302]; m.p. 31–32° [2302].

Semicarbazone of the ethyl ether [30299-37-7]  $C_{15}H_{23}N_3O_2$  mol. wt. 277.37 -Refer to: [2302]; m.p. 161.5–162° [2302].

1-(2,3-Dihydroxyphenyl)-4-methyl-1-pentanone

$$C_{12}H_{16}O_3$$
 mol. wt. 208.26



#### Synthesis

 $CO(CH_2)_2CH(CH_3)_2$  -Obtained (by-product) by Fries rearrangement of pyrocatechol diisocaproate with aluminium chloride in the presence of pyrocatechol for 4.5 h at 135–140° [2075].

b.p.₅ 195–205° [2075].

#### 1-(2,4-Dihydroxyphenyl)-4-methyl-1-pentanone

(Resisocapronophenone)

[116529-92-1]	$C_{12}H_{16}O_3$	mol. wt. 208.26
HO HO HO	Syntheses -Obtained by reaction of i resorcinol in the presence for 2 h at 70° (72 %) [2312 -Also obtained by reaction with resorcinol (Hoesch re	of boron trifluoride 2]. of isocaproic nitrile

-Also obtained by reaction of iso-hexylyl chloride with resorcinol at  $85-90^{\circ}$  for 20-30 min (82.7 %) [731].

-Also refer to: [893, 1588, 2842].

b.p.₆₋₇ 192–194° [893, 2842]; m.p. 84.5° [1588], 83–84° [1608, 2312], 76–77.5° [893, 2842].

#### Hydrate

 $C_{12}H_{16}O_3$ , x H₂O m.p. 47° [1608].

#### 1-(2,5-Dihydroxyphenyl)-4-methyl-1-pentanone

[18787-32-1] C12H16O3 mol. wt. 208.26 Syntheses OH CO(CH₂)₂CH(CH₃)₂ -Preparation by reaction of isocaproic acid with hydroquinone in the presence of boron trifluoride for 2 h at 125° [2063]. -Also obtained by Fries rearrangement of 4-methoxyphenyl isocaproate with aluminium chloride (5 part)/sodium chloride (2 part) mixture at 180-200° (30 %) [1796].

-Also refer to: [458, 1795].

m.p. 68° [1795, 1796], 65–66° [2063].

#### **Dimethyl ether** $C_{14}H_{20}O_3$ mol. wt. 236.31

-Obtained by reaction of isocaproyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride in boiling carbon disulfide for 2 h (40-50 %) [458].

pale yellow oil [458]; b.p.₇ 172–173° [458].

#### 1-(3,4-Dihydroxyphenyl)-4-methyl-1-pentanone

[26115-81-1]	$C_{12}H_{16}O_3$	mol. wt. 208.26
OH OH CO(CH ₂ ) ₂ CH(CH ₃ ) ₂	Syntheses -Obtained by Fries rearrang diisocaproate with aluminium of pyrocatechol for 4.5 h at 135 -Also obtained by reaction of 4 with pyrocatechol in the present in chlorobenzene at 110° for 3	chloride in the presence 5–140° (60 %) [2075]. 4-methylvaleroyl chloride ace of aluminium chloride

m.p. 127–128° [3226], 73–73.5° [2075]. One of the reported melting points is obviously wrong.

Dimethyl ether	[4101-16-0]	$C_{14}H_{20}O_3$	mol. wt. 236.31
b.p. _{0.02} 129–133° [	[1525]; $n_D^{25} = 1.533$	7 [1525].	

### 1-(3,5-Dihydroxyphenyl)-4-methyl-1-pentanone

1-(3,5-Dillyul oxyphenyi)-4-meth	y1-1-pentanoin		
[100257-43-0]	$C_{12}H_{16}O_3$		mol. wt. 208.26
HO COCH ₂ CH ₂ CH(CH ₃ ) ₂	•	n hydroxide at 6].	ts diacetate with reflux for 4.5 h
m.p. 102° [1406].			
BIOLOGICAL ACTIVITY: Anthe	elmintic [2781]		
<b>2,4-Dinitrophenylhydrazone</b> [ m.p. 245° [1406].	101741-97-3]	$C_{18}H_{20}N_4O_6$	mol. wt. 388.38
<b>Diacetate</b> [101430-33-5	] C ₁₆	$H_{20}O_{5}$	mol. wt. 292.33
-Preparation by reaction of diisopentylcadmium with 3,5-diacetoxybenzoyl chloride in refluxing benzene for 1 h (77 %) [1406].			
b.p. _{0.7} 184–188° [1406]; m.p. 51° [1406].			
2,4-Dinitrophenylhydrazone of t	he diacetate		
[102810-70-8]	C ₂₂ H ₂₄ N ₄ O ₈		mol. wt. 472.45
m.p. 185° [1406].			
Dimethyl ether	$C_{14}H_{20}O_3$		mol. wt. 236.31
-Preparation in the usual way [25 (81 %), 2990].			
b.p. _{0.5} 135° [25].			
4-Methyl-1-(2,4,5-trihydroxyphe	nyl)-1-pentano	one	

C ₁₂ H	I ₁₆ O ₄	mol. wt. 224.26
OH CO(CH ₂ ) ₂ CH(CH ₃ ) ₂	Synthesis -Refer to: [1708].	
но Он	USE: Antioxidant [1708]. Toxicity [1708].	

#### 4-Methyl-1-(2,4,6-trihydroxyphenyl)-1-pentanone

(Phloroisocaprophenone)

[2999-14-6]	$C_{12}H_{16}O_4$	mol. wt. 224.26
HO OH CO(CH ₂ ) ₂ CH(CH ₃ ) ₂	Syntheses -Obtained by reaction of iso with phloroglucinol in the p inium chloride, *in nitrobenzene (67 %) [2618	presence of alum-
	*in nitrobenzene (67 %) [2017 *in nitrobenzene and carbon (67 %) [2620], (64 %) [2113	disulfide mixture

-Also obtained by reaction of isocapronitrile with phloroglucinol (Hoesch reaction) [1608].

-Also refer to: [1026, 1375, 1607, 2617].

m.p. 122.5° [1375], 122° [1607, 1608, 2113, 2617, 2618, 2620], 120° [2624].

Monohydrate	$C_{12}H_{16}O_4, H_2O$	mol. wt. 242.28
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m.p. 104° [1607, 1608], 103–104° [1375].

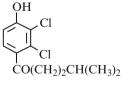
BIOLOGICAL ACTIVITY: Antifungal [2113].

Monosodium salt	[85602-44-4]	$C_{12}H_{15}O_4Na$	mol. wt. 246.24
-Refer to: [1026].			

## 2.2 Substituted Hydroxyketones

#### 1-(2,3-Dichloro-4-hydroxyphenyl)-4-methyl-1-pentanone

 $[1210-21-5] C_{12}H_{14}Cl_2O_2 mol. wt. 261.15$ 



Syntheses -To 4-methylpentanoyl chloride and 2,3-dichloroanisole in carbon disulfide was added aluminium chloride in small portions at 25°. The mixture stirred 1 h at r.t. and 45 min at 55°; pentane and aluminium chloride added, heated 3 h at 80° [2056].

-Also refer to: [2047, 2048, 2767 (84 %)].

m.p.  $107.5-108.5^{\circ}$  [2767],  $86-87^{\circ}$  [2047, 2048, 2056]. One of the reported melting points is obviously wrong.

#### 5-Isocaproyl-2-hydroxybenzoic acid

**Synthesis** -Obtained by hydrolysis of methyl 5-isocaproyl-2-hydroxy-COOH benzoate with boiling 20 % solution of potassium hydroxide [730]. CO(CH₂)₂CH(CH₃)₂ m.p. 132–133.5° [730].

#### Methyl ester C14H18O4 mol. wt. 250.29

-Obtained by Fries rearrangement of methyl 2-(isocaproyloxy)benzoate with aluminium chloride in boiling carbon disulfide for 2 h, then the reaction mixture heated at 90–110° for a few min after solvent elimination [730].

b.p.15 195-198° [730].

 $CH_2$ 

#### 1-(2-Hydroxy-4-methylphenyl)-4-methyl-1-pentanone

C ₁₃ H	₁₈ O ₂ mol. wt. 206.28
CH ₃ CO(CH ₂ ) ₂ CH(CH ₃ ) ₂	Synthesis -Preparation by Fries rearrangement of 3-methylphenyl isocaproate with aluminium chloride without solvent at 140–150° [906]. b.p. ₁ 106–107° [906].

#### 1-(2-Hydroxy-5-methylphenyl)-4-methyl-1-pentanone

[93429-81-3]	$C_{13}H_{18}O_2$	mol. wt. 206.28
OH CO(CH ₂ ) ₂ CH(CH ₃ ) ₂	1 *	rearrangement of 4-methyl- th aluminium chloride [423].

#### 1-(4-Hydroxy-3-methylphenyl)-4-methyl-1-pentanone

[101267-59-8]	$C_{13}H_{18}O_2$	mol. wt. 206.28
CH ₃ CO(CH ₂ ) ₂ CH(CH ₃ ) ₂ HO	-Fleparation by f	Fries rearrangement of caproate with aluminium

### 1-(3-Hydroxy-4-methoxyphenyl)-4-methyl-1-pentanone

	$C_{13}H_{18}O_3$	mol. wt. 222.28
OCH ₃	Synthesis	
ОН	-Refer to: [1803].	
	Benzyl ether [159211-07-1]	
Ŷ	$C_{20}H_{24}O_3$	mol. wt. 312.40
$CO(CH_2)_2CH(CH_3)_2$	-Refer to: [1803].	

### 1-(4-Hydroxy-3-methoxyphenyl)-4-methyl-1-pentanone

	$C_{13}H_{18}O_3$		mol. wt. 222.28
OH OCH ₃ CO(CH ₂ ) ₂ CH(CH ₃ ) ₂	Synthesis -Obtained by Fries isocaproate with a first at 80° for 45 m	luminium chloride	in nitrobenzene,
b.p. ₂₁ 205° [319];	m.p. 35–36° [319].		
Benzyl ether	[159211-02-6]	$C_{20}H_{24}O_3$	mol. wt. 312.40

-Refer to: [1803].

# 4-Methyl-1-(2,4,6-trihydroxy-3-methylphenyl)-1-pentanone

[131868-27-4]	$C_{13}H_{18}O_4$	mol. wt. 238.28
CH ₃ HO OH CO(CH ₂ ) ₂ CH(CH ₃ ) ₂	Syntheses -Obtained by reaction of with 3-methylphlorog reaction) [1608]. -Also refer to: [1441].	of isocaproic nitrile glucinol (Hoesch

m.p. 156° [1608], 155–156° [1441].

### 2-[2,3-Dichloro-4-(4-methylvaleryl)phenoxy]acetic acid

[1154-71-8]	$C_{14}H_{16}Cl_2O_4$	mol. wt. 319.19
$\begin{array}{c} OCH_2CO_2H \\ \hline \\ Cl \\ CO(CH_2)_2CH(CH_3)_2 \end{array}$	Syntheses -Refer to: [2047, 2048, 2056, 2766]. m.p. 108.5–109.5° [2047, 2048, 2766],	, 106–108.5° [2056].

#### 4-Methyl-1-(2,4,6-trihydroxy-3,5-dimethylphenyl)-1-pentanone

mol. wt. 252.31  $C_{14}H_{20}O_4$ Synthesis OH CO(CH₂)₂CH(CH₃)₂ -Refer to: [2781]. CH₃ m.p. 76° [2781].

#### 4-Hydroxy-3-(4-methyl-1-oxopentyl)-2H-1-benzopyran-2-one

C15H16O4

Synthesis

-Obtained by reaction of isocaproyl chloride  $CO(CH_2)_2CH(CH_3)_2$  with 4-hydroxycoumarin in pyridine containing one drop of piperidine for 3 h on a water bath [3174].

m.p. 79-80° [3174].

#### 4-Methyl-1-[2,4,6-trihydroxy-3-(2-propenyl)phenyl]-1-pentanone

2-isocaproyl-4-(propen-2-yl)phloroglucinol (15) [1026].

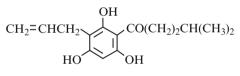
[85602-25-1]

HO



mol. wt. 264.32

mol. wt. 260.29



Syntheses

CO(CH₂)₂CH(CH₃)₂ -Obtained by adding cuprous chlo-ride, a saturated aqueous solution of sodium carbonate and allyl chloride to a solution of phloroisocaprophenone in ethyl ether at r.t., then to keep the mixture for 12 h at this temperature [1026].

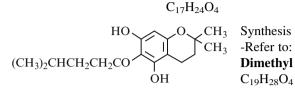
-Also obtained by reaction of allyl chloride with phloroisocaprophenone in an alkaline two-phase (aqueous-ether) system and catalyzed by CuCl [838, 3193].

m.p. 140–143° [3193]; ¹H NMR [1026], ¹³C NMR [1026, 3193], IR [1026].

BIOLOGICAL ACTIVITY: Bactericidal and fungicidal [1026, 3193].

mol. wt. 292.37

### 1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2*H*-1-benzopyran-6-yl)-4-methyl-1-pentanone



Synthesis -Refer to: [3193]. **Dimethyl ether** [105334-08-5] C₁₉H₂₈O₄ mol. wt. 320.43

-Refer to: [3193].

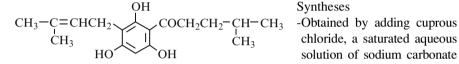
# **4-Methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-buten-1-yl)phenyl]-1-pentanone** (*Caespitin*)

2-isocaproyl-4-(3-methylbuten-2-yl)phloroglucinol (7) [1026].

[74478-09-4]

$$C_{17}H_{24}O_{4}$$

mol. wt. 292.37



Syntheses -Obtained by adding cuprous chloride, a saturated aqueous solution of sodium carbonate and prenyl bromide to a solution of phloroisocaprophenone in ethyl ether at r.t., then to keep the mixture for 12 h at this temperature [1026].

-Also prepared by adding prenyl bromide to a suspension of the sodium salt of phloroisocaprophenone in benzene, then the mixture obtained was refluxed for 3 h [1026].

-Also prepared by adding at  $5^{\circ}$  a solution of sodium methoxide in methanol, followed by isopentenyl bromide in ethyl ether to a solution of 3-methyl-1-(2,4,6-trihydroxyphenyl)-1-pentanone in benzene and ethyl ether; then the mixture was stirred at r.t. for 6 h (14 %) [2113].

-Also obtained by adding prenyl chloride to a two-phase mixture consisting of 2-(4-methyl-pentanoyl)phloroglucinol in diethyl ether and saturated aqueous sodium carbonate. A catalytic amount of CuCl was added and the mixture was stirred or shaken vigorously for 3 h at r.t. (41 %) [838].

-Also refer to: [839, 3193].

Isolation from natural sources

-Obtained from the entire plant material of *Helichrysum caespititium* [337, 1026, 2066].

straw coloured crystals [1026]; light yellow crystals [838]; m.p. 147° [2113], 132–133° [1026], 131–134° [838]; ¹H NMR [1026], ¹³C NMR [838, 1026], IR [838, 1026], MS [838].

BIOLOGICAL ACTIVITY: Antiprotozoal and antimicrobial [337]; Antimycobacterial [2066]; Antifungal [2113]; Bactericidal and fungicidal [1026]. **4,6-Dimethyl ether** [85602-35-3] C₁₉H₂₈O₄ mol. wt. 320.43

-Obtained by reaction of dimethyl sulfate with 2-isocaproyl-4-(3-methylbuten-2-yl) phloroglucinol (7) in the presence of potassium carbonate in refluxing acetone for 4 h [1026].

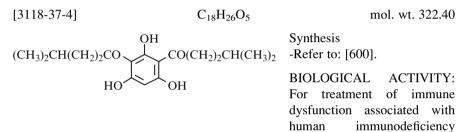
¹H NMR [1026], IR [1026].

**4,6-Dibenzoate** (**28**), [1026] C₃₁H₃₀O₆ mol. wt. 498.58

-Obtained by reaction of p-bromobenzoyl chloride with 2-isocaproyl-4-(3-methylbuten-2-yl)phloroglucinol (7) in refluxing benzene for 2 h [1026].

¹H NMR [1026], IR [1026]; X-ray crystallographic data [1026].

### 1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-4-methyl-1-pentanone



virus infection [600].

12 h at this temperature [1026].

### 4-Methyl-1-[2,4,6-trihydroxy-3-(phenylmethyl)phenyl]-1-pentanone

2-isocaproyl-4-benzylphloroglucinol (23) [1026].

$[85602-31-9]  C_{19}H_{22}O_4$	mol. wt. 314.38
HO OH carbona tion of	d by adding cuprous chloride, a d aqueous solution of sodium te and benzyl chloride to a solu- phloroiso-caprophenone in ethyl r.t., then to keep the mixture for

-Also obtained by reaction of benzyl chloride with phloroheptanophenone in an alkaline two-phase (aqueous-ether) system and catalyzed by CuCl [838, 3193].

m.p. 121–122° [3193]; ¹H NMR [1026], ¹³C NMR [1026, 3193], IR [1026].

BIOLOGICAL ACTIVITY: Bactericidal and fungicidal [1026, 3193].

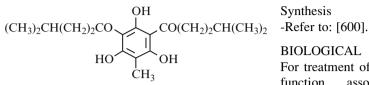
### 1,1'-(2,4,6-Trihydroxy-5-methyl-1,3-phenylene)bis-4-methyl-1-pentanone

[2999-17-9]

C19H28O5

mol. wt. 336.43

mol. wt. 486.57



Synthesis

BIOLOGICAL **ACTIVITY:** For treatment of immune dysfunction associated with immunodeficiency human virus infection [600].

### 1-(4-Heptyl-2,5-dihydroxyphenyl)-4-methyl-1-pentanone

C ₁₉ H ₃₀ O	3 mol. wt. 306.45
$C_{7}H_{15} \xrightarrow{OH} OH COCH_{2}CH_{2}CH(CH_{3})_{2}$	Synthesis -Obtained by treatment of 4-heptyl- 2-hydroxy-5-methoxyisocaprophenone with aluminium bromide [161]. m.p. 74–75° [161].

### 2,4-Dinitrophenylhydrazone

m.p. 142–143° [161].

### 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-4-methyl-1-pentanone

$$\begin{array}{ccc} C_{20}H_{32}O_2 & \text{mol. wt. 304.47} \\ OH & Synthesis \\ (CH_3)_3C & C(CH_3)_3 & -Obtained by reaction of isocaproyl chloride with 2,6-di-tert-butylphenol in the presence of titanium tetrachloride [1468]. \end{array}$$

 $C_{25}H_{34}N_4O_6$ 

### 1-(4-Heptyl-2-hydroxy-5-methoxyphenyl)-4-methyl-1-pentanone

[857973-71-	8] C	$_{20}H_{32}O_{3}$	mol. wt. 320.47
C ₇ H ₁₅ OC	COCH ₂ CH ₂ CH(CH ₃ ) ₂	Synthesis -Obtained by reaction of ride with 2-heptylhydroo ether in the presence of ride in carbon disulfide [	uinone dimethyl aluminium chlo-
1			

b.p._{0.5} 171° [161].

#### Aromatic Hydroxyketones Derived from Various 3 **Alkylpentanoic Acids**

# 3.1 Unsubstituted Hydroxyketones

### 1-(4-Hydroxyphenyl)-2-methyl-1-pentanone-2-d

	$C_{12}H_{15}DO_2$	mol. wt. 192.26
CO-CD-C ₃ H ₇	Synthesis -Refer to: [2106]. <b>Methyl ether</b> [733016-52-9] $C_{13}H_{17}DO_2$	mol. wt. 193.12
D		

-Preparation (complex synthesis) **28a** (80 %, D = 95 %) [2106].

colourless oil [2106]; ¹H NMR [2106], IR [2106], MS [2106].

### 1-(2-Hydroxyphenyl)-3-methyl-1-pentanone

[39575-43-4]  $C_{12}H_{16}O_2$ mol. wt. 192.26 COCH₂-CH-CH₂CH₃ Synthesis .COCH₂-CH-CH₂CH₃ -Refer to: [2093]. b.p.₁₅ 160–165° [2093];  $n_D^{30} = 1.473$  [2093]. OH

### 1-(4-Hydroxyphenyl)-2-methyl-1-pentanone

	$C_{12}H_{16}O_2$	mol. wt. 192.26
ŌН	Synthesis	
$\checkmark$	-Refer to: [2106].	
	Methyl ether [115975-32-1]	
CH ₃	$C_{13}H_{18}O_2$	mol. wt. 206.28
ĊO-ĊH-C ₃ H ₇		

-Preparation by treatment of adduct 26a with methyl iodide (99 %) (complex synthesis) [2106].

colourless oil [2106]; ¹H NMR [2106], IR [2106], MS [2106].

#### 1-(4-Hydroxyphenyl)-3-methyl-1-pentanone (+)

[62439-32-1]	$C_{12}H_{16}O_2$	mol. wt.	192.26
OH COCH ₂ -CH ₃ COCH ₂ -CH-C ₂ H ₅	Synthesis -Obtained by Fries rearrangement 3-methylvalerate (58.6 %) [2093]. b.p. ₁₅ 160–165° [2093]; ( $\alpha$ ) ³⁰ _D = + 2.61 [2093].	of (+)	phenyl

-Obtained by reaction of  $\beta$ -methylvaleroyl chloride with anisole in the presence of stannic chloride in benzene for 24 h at 50° (20 %) [2906].

b.p.₁₄ 155–160° [2906]; UV [2906];  $n_D^{25} = 1.5263$  [2906].

### 2,4-Dinitrophenylhydrazone of the methyl ether $C_{19}H_{22}N_4O_5$ mol. wt. 386.41

m.p. 138.6-139.4° [2906].

### 1-(2,4-Dihydroxyphenyl)-3-methyl-1-pentanone

$C_{12}H_{16}O_3$		mol. wt. 208.26
OH CH ₃ COCH ₂ -CH-CH ₂ CH ₃	Synthesis -Refer to: [2605]. m.p. 64–66° [2605].	

#### 1-(3,5-Dihydroxyphenyl)-2-methyl-1-pentanone

	$C_{12}H_{16}O_3$	mol. wt. 208.26
HO CO-CH-C ₃ H ₇	Synthesis -Refer to: [25]. <b>Dimethyl ether</b> $C_{14}H_{20}O_3$	mol. wt. 236.31

-Preparation in the usual way [25 (61 %), 2990].

b.p.1 150° [25].

### **3-Methyl-1-(2,4,6-trihydroxyphenyl)-1-pentanone** (S)

$$[108991-24-8] C_{12}H_{16}O_4 mtext{mol. wt. } 224.26$$

 $\begin{array}{c} H & CH_3 \\ COCH_2 - CH - C_2H_5 \\ OH \end{array}$  Syntheses -Obtained by reaction of S-3-methylvaleryl chloride with phloroglucinol in the presence of aluminium chloride in nitrobenzene for 60 h at r.t. (65 %) [3405].

-Also refer to: [2003].

m.p. 145° [3405], 129–130° [2003]; ¹H NMR [3405], IR [3405], MS [3405],

### 1-(2-Hydroxyphenyl)-4,4-dimethyl-1-pentanone

[935277-48-8]

C13H18O2

Synthesis

mol. wt. 206.28

OH	CH ₃
CO(C	$H_2)_2 - C - CH_3$
	CH ₃
~	

-Obtained by stirring a solution of salicylaldehyde, 3,3-dimethyl-1-butene, RhCl(PPh₃)₃, acetonitrile and sodium acetate in methylene chloride at r.t. for 7 h under an argon atmosphere (82 %) [1434].

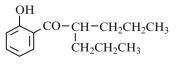
¹H NMR [1434], ¹³C NMR [1434], IR [1434].

# 1-(4-Hydroxyphenyl)-4,4-dimethyl-1-pentanone

	$C_{13}H_{18}O_2$	mol. wt. 206.28
H	Synthesis	
	-Refer to: [2404].	
l]	Methyl ether [152269-19-7]	
CH ₃	$C_{14}H_{20}O_2$	mol. wt. 220.31
$O(CH_2)_2 - C - CH_3$	-Refer to: [1557, 2404].	
CH3		

### 1-(2-Hydroxyphenyl)-2-propyl-1-pentanone

$C_{14}H_{20}O_2$	mol.	wt.	220.31
-------------------	------	-----	--------



CO-CH-CH₂CH₂CH₃ L CO-CH-CH₂CH₂CH₃ CH₂CH₂CH₃ Syntheses -Preparation by Fries rearrangement of phenyl valproate in the presence of aluminium chloride in chlorobenzene for 4 h at  $140^{\circ}$  (74 %) [2003]. -Also refer to: [2002].

b.p._{0.2} 89–91° (Sadtler standard N° 84927K) [2002, 2003]; ¹H NMR (Sadtler standard N° 57879M) [2002, 2003];

IR (Sadtler standard N° 84927K) [2002, 2003], UV [2002, 2003], MS [2002, 2003].

## 1-(4-Hydroxyphenyl)-2-propyl-1-pentanone

[137937-44-1]	$C_{14}H_{20}O_2$	mol. wt. 220.31
OH CO-CH-CH ₂ CH ₂ CH ₃ CH ₂ CH ₂ CH ₃		d N° 84928K) [2003];

IR (Sadtler standard N° 84928K) [2003], UV [2003], MS [2003].

Methyl ether	[119748-12-8]	$C_{15}H_{22}O_2$	mol. wt. 234.34
--------------	---------------	-------------------	-----------------

-Obtained by reaction of dipropylacetyl chloride with anisole in the presence of aluminium chloride in methylene chloride first at 0°, then at r.t. for 4 h [1290].

b.p.13 183-186° [3178].

# 1-(3,4-Dihydroxyphenyl)-2-propyl-1-pentanone

	$C_{14}H_{20}O_3$	mol. wt. 236.31
OH OH COCH(C ₃ H ₇ ) ₂	Synthesis -Refer to: [2183]. <b>Dimethyl ether</b> [180698-61-7] $C_{16}H_{24}O_3$	mol. wt. 264.36

-Refer to: [2183].

**Oxime of the dimethyl ether** [180698-62-8]  $C_{16}H_{25}NO_3$  mol. wt. 279.38

-Refer to: [2183].

¹H NMR [2182], ¹³C NMR [2182].

# 3.2 Substituted Hydroxyketones

# 1-(2,3-Dichloro-4-hydroxyphenyl)-2-methylene-1-pentanone

 $\begin{array}{cccc} C_{12}H_{12}Cl_2O_2 & \text{mol. wt. 259.13} \\ & \\ OH & Synthesis \\ & \\ Cl & -Refer \text{ to: [742].} \\ & \\ & \\ Cl & C_{13}H_{14}Cl_2O_2 \\ & \\ & \\ CO-C-C_3H_7 \\ & \\ & \\ & \\ CH_2 \end{array} \end{array} \qquad \text{mol. wt. 273.16}$ 

-Obtained by treatment of 2,3-dichloro-4-methoxy-valeophenone with N,N,N', N'-tetramethyl-methanediamine (92 %) [742].

-Also obtained from 2-bromo-1-(2,3-dichloro-4-methoxyphenyl)-2-methyl-1-propanone [742].

m.p. 58–60° [742]; ¹H NMR [742].

[165538-94-3]

# 1-(2-Chloro-4-hydroxyphenyl)-3-methyl-1-pentanone

Synthesis OH -Refer to: [1717]. C1 COCH₂-CH-C₂H₅

# 1-(3-Chloro-2-hydroxyphenyl)-3-methyl-1-pentanone (+)

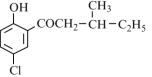
[39575-47-8]  $C_{12}H_{15}ClO_2$ mol. wt. 226.70 OH  $CH_3$  Synthesis Cl  $COCH_2 - CH - C_2H_5$  -Obtained by Fries rearrangement of (+) o-chlorophenyl 3-methylvalerate (53.2 %) [2093]. b.p.₁₅ 200–205° [2093];  $(\alpha)_D^{30} = +1.95$  [2093].

# 1-(4-Chloro-2-hydroxyphenyl)-3-methyl-1-pentanone (+)

[39575-48-9] mol. wt. 226.70 C₁₂H₁₅ClO₂ H  $CH_3$  Synthesis COCH₂-CH- $C_2H_5$  -Obtained by Fries rearrangement of (+) m-chlorophenyl 3-methylvalerate (58.6 %)

b.p.₁₅ 208–212° [2093];  $(\alpha)_D^{30} = +2.66$  [2093].

# 1-(5-Chloro-2-hydroxyphenyl)-3-methyl-1-pentanone (+)



OCH₂- $\dot{C}H$ - $C_2H_5$  -Obtained by Fries rearrangement of p-chlorophenyl 3-methylvalerate (64 %) [2093]. b.p.₁₅ 215–220° [2093]; ( $\alpha$ )³⁰_D = + 2.91 [2093]. (+)

C₁₂H₁₅ClO₂

mol. wt. 226.70

### 2,4,6-Trihydroxy-3-(3-methyl-1-oxopentyl)benzaldehyde (S)

[120716-97-4]	$C_{13}H_{16}$	₆ O ₅ mol. wt. 252.27	7
CHO HO OH COCH ₂ -	with 1-pen	neses ined by reaction of ethyl orthoformate 3-methyl-1-(2,4,6-trihydroxyphenyl) ntanone (S) in the presence of aluminium ride for 30 min (53 %) [3405].	)-

-Also refer to: [3415].

m.p. 74° [3405]; ¹H NMR [3405], IR [3405], MS [3405]; M+, 238 (compound **6**, p. 473).

BIOLOGICAL ACTIVITY: As photosynthetic electron transport (PET) [3405].

# 1-(2-Hydroxy-3-methylphenyl)-3-methyl-1-pentanone (+)

$$[39575-44-5] \qquad C_{13}H_{18}O_2 \qquad \text{mol. wt. } 206.28$$

$$CH_3 \qquad COCH_2 - CH - C_2H_5 \qquad Obtained by Fries rearrangement of (+) o-cresyl 3-methylvalerate (45.8 \%) [2093].$$

b.p.₁₅ 195–200° [2093]; ( $\alpha$ )³⁰_D = +2.03 [2093].

### 1-(2-Hydroxy-4-methylphenyl)-3-methyl-1-pentanone (+)

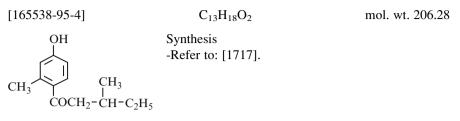
$$[39575-45-6] C_{13}H_{18}O_2 mol. wt. 206.28$$

$$OH CH_2 - CH - C_2H_5 OB Fries rearrangement of (+) OB Fries rearrangement of (+) OB Fries (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-0.28) (-$$

b.p.₁₅ 208–210° [2093];  $(\alpha)_D^{30} = +2.46$  [2093].

# 1-(2-Hydroxy-5-methylphenyl)-3-methyl-1-pentanone (+)

## 1-(4-Hydroxy-2-methylphenyl)-3-methyl-1-pentanone



## 1-(2,4-Dihydroxy-3-methylphenyl)-2-methyl-1-pentanone

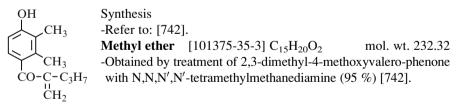
 $\begin{array}{c} C_{13}H_{18}O_{3} \\ OH \\ CH_{3} \\ HO \end{array} \begin{array}{c} CH_{3} \\ CH_{-}CO - CH - (CH_{2})_{2}CH_{3} \\ HO \end{array} \begin{array}{c} Synthesis \\ -Refer to: [3147]. \\ H NMR [3147], {}^{13}C NMR [3147]. \end{array}$ 

# **3-Methyl-1-(2,4,6-trihydroxy-3-methylphenyl)-1-pentanone** (S)

BIOLOGICAL ACTIVITY: As photosynthetic electron transport (PET) [3405].

# 1-(4-Hydroxy-2,3-dimethylphenyl)-2-methylene-1-pentanone

C₁₄H₁₈O₂ mol. wt. 218.30



-Also obtained from 2-bromo-1-(2,3-dimethyl-4-methoxyphenyl)-2-methyl-1-propanone [742].

liquid [742]; ¹H NMR [742].

# 2,4,6-Trihydroxy-3-methyl-5-(3-methyl-1-oxopentyl)benzaldehyde

(Homograndinol)

[132341-31-2] racemic	$C_{14}H_{18}O_5$	mol. wt. 266.29
CH ₃ CH ₃ HO CHO CH ₃ CH ₃ CH ₃ CH ₃ COCH ₂ -CH-CH ₂ CH ₃ CH-CH ₂ CH ₃ CH-CH ₂ CH ₃ CH-CH ₂ CH-CH ₂ CH ₃	Synthesis -Refer to: [3033]. Isolation from natural sour -From fresh leaves of <i>Euc</i>	

(Myrtaceae) [1106, 1346, 3114, 3405, 3406, 3413].

amorphous solid [3413]; m.p. 145–146° [3405]; ¹H NMR [3413], IR [3413], UV [3413], MS [3413]; HPLC [3413].

USE: G-inhibitor (plant growth regulator) [3413].

BIOLOGICAL ACTIVITY: Inhibitory effects of treatment with this acylphenol on TPA Induced EBV-EA Activation [3033]; Strongly inhibit photosynthesis as well as germination of cress seeds [1346]; As photosynthetic electron transport (PET) [3405].

**S-isomer** (*S-Homograndinol*) [116425-03-7] C₁₄H₁₈O₅ mol. wt. 266.29

-Obtained by reaction of methyl iodide with 3'-formyl-3-methylvalerophenone in the presence of potassium carbonate in 30 % aqueous acetone at 50° for 3 h (45 %) [3405].

-Also refer to: [3033, 3413].

m.p. 153–155° [3405, 3413];  $(\alpha)_D^{20} = +14.1^{\circ}$  [3413]; ¹H NMR [3405, 3413], IR [3405, 3413], UV [3413], MS [3405].

BIOLOGICAL ACTIVITY: As photosynthetic electron transport (PET) [3405].

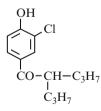
-Also refer to: [3033, 3413].

# 1-(3-Chloro-4-hydroxyphenyl)-2-propyl-1-pentanone

[4798-12-3]

C14H19ClO2

mol. wt. 254.76



Syntheses

-To dipropylacetyl chloride, 2-chloroanisole and carbon disul--Also refer to: [2767].

b.p._{0.5} 140° [2056, 2767].

#### 1-(3,5-Dimethyl-4-hydroxyphenyl)-2-methyl-1-pentanone

### 1-(2-Hydroxy-3-methylphenyl)-2-propyl-1-pentanone

 $[137937-40-7] C_{15}H_{22}O_2 mol. wt. 234.34$   $CH_3 \qquad OH \\ CH_2CH_2CH_2CH_3 \\ I \\ CH_2CH_2CH_3 \\ I \\ CH_2$ 

 $b.p._{0.6}$  124° (Sadtler standard N° 84999K) [2003]; ¹H NMR (Sadtler standard N° 57945M) [2003],

IR (Sadtler standard N° 84999K) [2003], UV [2003], MS [2003].

## 1-(2-Hydroxy-4-methylphenyl)-2-propyl-1-pentanone

170 h at 20° (63 %) [2003].

-Also refer to: [2002].

Methyl ether	[137937-53-2]	$C_{16}H_{24}O_2$	mol. wt. 248.37
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-Obtained by treatment of 3-methylanisole with valproyl chloride in the presence of aluminium chloride in carbon disulfide at +5° for 6 h (46 %) [2003]. -Also obtained by direct methylation of its above ketone (95 %) [2003].

b.p._{0.7} 128–130° (Sadtler standard N° 57883M) [2003]; ¹H NMR (Sadtler standard N° 57883M) [2003]; IR (Sadtler standard N° 84931K), [2003], UV [2003], MS [2003].

### 1-(4-Hydroxy-2-methylphenyl)-2-propyl-1-pentanone

[137937-46-3]	$C_{15}H_{22}O_2$	mol. wt. 234.34
OH CH ₃ CO-CH-CH ₂ CH ₂ CH ₃	Syntheses -Obtained by treatment of its me aluminium bromide in refluxi (40 %) [2003]. -Also refer to: [2002]. m.p. 80° (Sadtler standard N° 84 ¹ H NMR (Sadtler standard N° 57)	ng benzene for 4 h 930K) [2003];

IR (Sadtler standard N° 84930K) [2003], UV [2003], MS [2003].

Methyl ether	[137937-54-3]	$C_{16}H_{24}O_2$	mol. wt. 248.37
--------------	---------------	-------------------	-----------------

-Obtained by direct methylation of its above ketone (80 %) [2003].

b.p._{0.7} 128–130° (Sadtler standard N° 57884M) [2002, 2003]; ¹H NMR (Sadtler standard N° 57884M) [2002, 2003], IR (Sadtler standard N° 84932K) [2002, 2003], UV [2002, 2003], MS [2002, 2003].

### 1-(4-Hydroxy-3-methylphenyl)-2-propyl-1-pentanone

	$C_{15}H_{22}O_2$	mol. wt. 234.34
ОН	Syntheses	
CH ₃	-Preparation by Fries rearrange	ement of 2-methylphenyl
	valproate in the presence of	aluminium chloride in
$\mathbf{i}$	nitromethane for 170 h at 20°	(78 %) [2003].
CO-CH-CH ₂ CH ₂ CH ₃	-Also refer to: [2002].	

 1  m.p. 86° (Sadtler standard N° 84929K) [2003];

¹H NMR (Sadtler standard N $^{\circ}$  57881M) [2003];

IR (Sadtler standard N° 84929K) [2003], UV [2003], MS [2003].

## 5,7-Dihydroxy-2,2-dimethyl-2H-1-(benzopyran-8-yl)-3-methyl-1-pentanone

$$\begin{array}{c} CH_3 \\ COCH_2-CH-CH_2CH_3 \\ HO \\ HO \\ HO \\ CH_3 \\ OH \end{array} \begin{array}{c} Synthesis \\ -Refer to: \\ Dimethyl \\ C_{19}H_{26}O_4 \\ -Obtained \\ diethyl et \\ how in b \end{array}$$

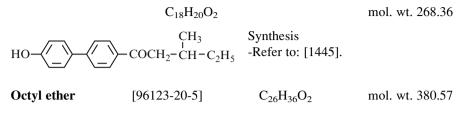
 $C_{17}H_{22}O_4$ 

mol. wt. 290.36

-Refer to: [3203]. **Dimethyl ether** [924889-50-9]  $C_{19}H_{26}O_4$  mol. wt. 318.41 -Obtained by adding a solution of malloapelta B in diethyl ether to the solution of the ethylmagnesium bromide in diethyl ether under a nitrogen atmosphere at  $-10^\circ$  for 30 min (85 %) [3203].

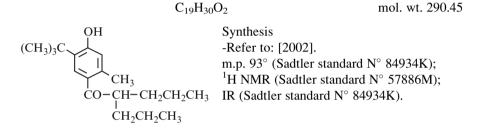
white solid [3203]; m.p. 62.2° [3203]; ¹H NMR [3203], IR [3203]; TLC [3203].

### 1-(4'-Hydroxy[1,1-biphenyl]-4-yl)-3-methyl-1-pentanone



-Refer to: [1445].

# 1-[5-(1,1-Dimethylethyl)-4-hydroxy-2-methylphenyl]-2-propyl-1-pentanone



# 4 Aromatic Hydroxyketones Derived from Various Halogenopentanoic Acids

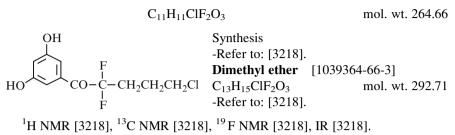
# 4.1 Unsubstituted Hydroxyketones

### 5-Chloro-1-(2,5-dihydroxyphenyl)-2,2-difluoro-1-pentanone

 $\begin{array}{cccc} C_{11}H_{11}ClF_2O_3 & \mbox{mol. wt. 264.66} \\ & & & \\ OH & & F & \\ CO-C-CH_2CH_2CH_2Cl & -Refer to: [3218]. \\ & & & \\ F & & \\ OH & & & \\ OH & & & \\ \end{array} \begin{array}{c} Synthesis & \\ -Refer to: [3218]. \\ & & \\ Dimethyl \ ether & \\ C_{13}H_{15}ClF_2O_3 & \\ -Refer to: [3218]. \\ & & \\ Refer to: [3218]. \end{array}$ 

¹H NMR [3218], ¹³C NMR [3218], ¹⁹F NMR [3218], IR [3218], MS [3218].

### 5-Chloro-1-(3,5-dihydroxyphenyl)-2,2-difluoro-1-pentanone



¹H NMR [3218], ¹³C NMR [3218], ¹⁹F NMR [3218], IR [3218].

### 5,5,5-Trifluoro-1-(3-hydroxyphenyl)-1-pentanone

[104325-67-9] C₁₁H₁₁F₃O₂ mol. wt. 232.20 **Synthesis** OH -Refer to: [2198]. USE: In preparation of antiinflammatory and antiallergic CO(CH₂)₃CF₃ agents [2198].

# 2-Bromo-5-chloro-1-(4-hydroxyphenyl)-1-pentanone

	$C_{11}H_{12}BrClO_2$	mol. wt. 291.57
OH CO-CH-(CH ₂ ) ₂ CH ₂ Cl	Synthesis -Refer to: [122]. <b>Methyl ether</b> [1104634-92-5] $C_{12}H_{14}BrClO_2$ -Refer to: [122].	mol. wt. 305.60

BIOLOGICAL ACTIVITY: Preparation of phenyl pentalen-1-ones as therapeutic and diagnostic estrogen receptor ligands [122].

## 5-Bromo-1-(2-hydroxyphenyl)-1-pentanone

[173055-13-5]	$C_{11}H_{13}BrO_2$	mol. wt. 257.13
OH CO(CH ₂ ) ₃ CH ₂ Br	Synthesis -Refer to: [2623].	

### 2-Bromo-1-(2-hydroxyphenyl)-1-pentanone

$$\begin{array}{cccc} C_{11}H_{13}BrO_2 & \text{mol. wt. } 257.13 \\ OH & Synthesis \\ CO-CH-(CH_2)_2CH_3 & -Refer to: [2848]. \\ & & & & & \\ Br & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & &$$

# 2-Bromo-1-(4-hydroxyphenyl)-1-pentanone

USE: Preparation of 4-acetamido-3-(4-aryl-2-thiazolylamino)benzoate as fungicide and insecticide [1379]; Preparation of 2-methyl-1-(2-thiazolyl)-1*H*-benzimidazole-6-carboxylic acid ethyl ester derivatives and determination of their activity as agrochemical fungicides [1383].

**Methyl ether** [36412-64-3] C₁₂H₁₅BrO₂ mol. wt. 271.15

-Obtained by bromination of 4-methoxyvalerophenone in ethyl ether or methylene chloride with bromine in an ice bath at  $0^{\circ}$  for 10 min; then the mixture was warmed to r.t. [2043].

-Also obtained by treatment of 1-(4-methoxyphenyl)-1-pentanone with bromine in ethyl ether and glacial acetic acid at r.t. (74 %) [1114].

-Also obtained by treatment of 1-(4-methoxyphenyl)-1-pentanone with bromine in the presence of a catalytic amount of aluminium chloride (nearly quantitative yield) [2157].

-Also refer to: [1114, 1630, 1947, 2157, 2158, 3231 (60 %), 3383, 3472].

oil [3231]; b.p.₁₅ 150–165° [3231]; m.p. 48–49° [1114], 39–41° [2157]; ¹H NMR [1114, 1947, 2043, 2157, 3231], ¹³C NMR [2157], IR [3231], MS [1114].

USE: Preparation of pyrovalerone analogs as monoamine uptake inhibitors [2043].

**Benzyl ether** [35081-50-6] C₁₈H₁₉BrO₂ mol. wt. 347.25

-Preparation by bromination of 4-benzyloxyvalerophenone with cupric bromide, *in an ethyl acetate/chloroform mixture (82.6-97.5 %) [1384]; *in refluxing ethanol [546] according to the process [1382]. -Also obtained by irradiation with UV lamp of a solution of 4-benzyloxyvalerophenone and bromine in methylene chloride for 15 h between  $15-18^{\circ}$  (67 %) [556].

m.p. 84° [556].

## 5-Bromo-1-(2,6-dihydroxyphenyl)-1-pentanone

[1111652-02-8]	$C_{11}H_{13}BrO_3$	mol. wt. 273.13
OH CO(CH ₂ ) ₃ CH ₂ Br	Synthesis -Refer to: [1219 (Chinese patent)].	
ОН	USE: Preparation of low swelling sul proton exchange membrane for fuel c	1 .

# 2-Bromo-1-(3,4-dihydroxyphenyl)-1-pentanone

	$C_{11}H_{13}BrO_3$		mol. wt. 273.13
ОН	Syntheses		
ОН	-Preparation [194	[8] using the general j	procedure [1738].
	-Also refer to: [1	947, 2043].	
Br	Dimethyl ether	[850352-39-5]	
$\dot{CO}$ - $\dot{CH}$ - $(CH_2)_2CH_3$	$C_{13}H_{17}BrO_3$		mol. wt. 301.18

-Obtained by bromination of 3,4-dimethoxyvalerophenone in ethyl ether or methylene chloride with bromine in an ice bath at  $0^{\circ}$  for 10 min; then the mixture was warmed to r.t. [2043].

¹H NMR [2043].

USE: Preparation of pyrovalerone analogs as monoamine uptake inhibitors [2043]; Preparation of pyrovalerone analogs as selective dopamine transporter inhibitors [1947].

# 2-Bromo-1-(2,4,5-trihydroxyphenyl)-1-pentanone

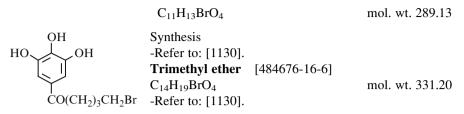
	$C_{11}H_{13}BrO_4$		mol. wt. 289.13
OH COCHBrC ₃ H ₇	Synthesis -Refer to: [2696].		
но он	<b>Trimethyl ether</b> C ₁₄ H ₁₉ BrO ₄	[90834-08-5]	mol. wt. 331.20

-Obtained by reaction of bromine with 2,4,5-trimethoxyvalerophenone in acetic acid at  $35-40^\circ$ , then at  $25^\circ$  for 40 min [2695].

-Also refer to: [2696].

m.p. 67–68° [2695, 2696]; ¹H NMR [2695, 2696], IR [2695, 2696], MS [2695, 2696].

## 1-(5-Bromo-3,4,5-trihydroxyphenyl)-1-pentanone



# 2-Chloro-1-(4-hydroxyphenyl)-1-pentanone

[1108128-60-4]	$C_{11}H_{13}ClO_2$	mol. wt. 212.68
OH	Synthesis -Refer to: [1379 (Chinese patent)].	
CO-CH-(CH ₂ ) ₂ CH ₂	USE: Preparation of 4-acetamido-3-(4 amino)benzoate as fungicide and insec	• •

# 5-Chloro-1-(2-hydroxyphenyl)-1-pentanone

[501083-61-0]	$C_{11}H$	$_{13}ClO_2$	mol. wt. 212.68
OH CO(CH ₂ ) ₃ CH ₂ (		59]. 9], MS [2459].	
Methyl ether	[43228-96-2]	$C_{12}H_{15}ClO_2$	mol. wt. 226.70

-Obtained by treatment of  $\delta$ -lactone of 2-(o-methoxybenzoyl)-5-hydroxyvaleric acid with concentrated HCl (90 %) [371]. -Also refer to: [2459].

colourless oil [371]; b.p._{0.5} 100° [371]; ¹H NMR [2459], MS [2459].

# 5-Chloro-1-(3-hydroxyphenyl)-1-pentanone

[501083-63-2]	$C_{11}H_{13}ClO_2$	mol. wt. 212.68
OH CO(CH ₂ ) ₃ CH ₂ Cl	Synthesis -Refer to: [2459]. ¹ H NMR [2459], MS [2459].	

**Methyl ether** [258882-49-4] C₁₂H₁₅ClO₂ mol. wt. 226.70

-Preparation by treatment of 5-chloro-1-(3-methoxyphenyl)-1-pentanol with chromium trioxide in dilute sulfuric acid (Jones' Reagent) in acetone first at 0°, then at r.t. for 6 h (89 %) [2460]. -Also refer to: [2459].

colourless oil [2460]; ¹H NMR [2460], MS [2460].

## 5-Chloro-1-(4-hydroxyphenyl)-1-pentanone

[83882-87-5]	$C_{11}H_{13}$	ClO ₂	mol. wt. 212.68
OH CO(CH ₂ ) ₃ CH ₂ Cl	•	of aluminium chlo (60 %) [1581]. ), 2371, 2372].	royl chloride with phe- ride [2459] in nitroben-
Methyl ether	[949-06-4]	$C_{12}H_{15}ClO_2$	mol. wt. 226.70

-Preparation: To an ethereal solution of p-methoxyphenylmagnesium bromide was added 5-chlorovaleronitrile in ether. The reaction mixture was stirred at r.t. for 10 h and hydrolyzed by 15 % hydrochloric acid (17.7 %) [2413].

-Also obtained [1589] from the method [555].

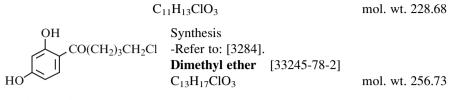
-Also refer to: [122, 1125, 1126, 1171, 1367, 1459, 1460, 1956, 3076, 3286, 3287].

m.p. 82–84° [2413], 67–68° [1460]; **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [1171, 1367, 1460], IR [1171].

USE: Preparation of phenyl pentalen-1-ones as therapeutic and diagnostic estrogen receptor ligands [122]; Preparation of imidazolyl inhibitors of 15-lipoxygenase [3287].

Phenyl ether	[820968-19-2]	$C_{17}H_{17}ClO_2$	mol. wt. 288.77
-Refer to: [2905].			
Difluoromethyl ethe	r [83882-88-6]	$C_{12}H_{13}ClF_2O_2$	mol. wt. 262.68
-Refer to: [2371].			

### 5-Chloro-1-(2,4-dihydroxyphenyl)-1-pentanone



-Refer to: [677, 3284].

m.p. 56–57° [677].

# 5-Chloro-1-(3,4-dihydroxyphenyl)-1-pentanone

	$C_{11}H_{13}ClO_3$	mol. wt. 228.68
он ,он	Synthesis -Refer to: [677, 1171].	
	<b>Dimethyl ether</b> [33245-77-1] C ₁₃ H ₁₇ ClO ₃	mol. wt. 256.73
CO(CH ₂ ) ₃ CH ₂ Cl	-15 17 - 5	

-Refer to: [677, 1171, 2561].

m.p. 46–47° [677], 42–45° [1171]; IR [1171].

### 5-Chloro-1-(2,4,6-trihydroxyphenyl)-1-pentanone

C	$_{11}H_{13}ClO_4$		mol. wt. 244.67
HO OH CO(CH ₂ ) ₃ CH ₂ Cl	Synthesis -Refer to: [3284]. <b>Trimethyl ether</b> C ₁₄ H ₁₉ ClO ₄	[187396-81-2]	mol. wt. 286.76

-Refer to: [308, 926, 927, 3284].

m.p. 53–55° [3284], 50° [926]; ¹H NMR [926].

## 1-(2-Hydroxyphenyl)-5-iodo-1-pentanone

	$C_{11}H_{13}IO_2$	mol. wt. 304.12
ОН	Synthesis	
CO(CH ₂ ) ₃ CH ₂ I	-Refer to: [371].	
	<b>Methyl ether</b> [43228-97-3]	
$\sim$	$C_{12}H_{15}IO_2$	mol. wt. 318.15

-Obtained by treatment of 5-chloro-1-(2-hydroxyphenyl)-1-pentanone with sodium iodide in boiling acetone for 30 h under nitrogen atmosphere (86 %) [371].

b.p._{0.005} 95° [371].

# 4.2 Substituted Hydroxyketones

### 5-Chloro-1-(2,3-dichloro-4-hydroxyphenyl)-1-pentanone

	$C_{11}H_{11}Cl_{3}O_{2}$	mol. wt. 281.57
ОН	Synthesis	
CI	-Refer to: [741].	
	Methyl ether [115595-92-1]	
SY CI	$C_{12}H_{13}Cl_{3}O_{2}$	mol. wt. 295.59
CO(CH ₂ ) ₃ CH ₂ Cl	-Refer to: [741].	

### 2-Bromo-1-(4-chloro-2-hydroxyphenyl)-1-pentanone

$C_{11}H_{12}$	BrClO ₂	mol. wt. 291.57
Cl CO-CH-(CH ₂ ) ₂ CH ₃	Synthesis -Refer to: [1199]. Methyl ether [1001441-59- $C_{12}H_{14}BrClO_2$	3] mol. wt. 305.60

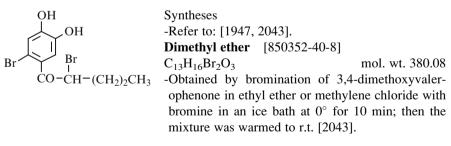
-Refer to: [1199].

¹H NMR [1199], IR [1199], MS [1199].

USE: For preparation of triphenyl-substituted 5-membered heterocycles as anticancer and antiinflammatory agents [1199].

### 2-Bromo-1-(2-bromo-4,5-dihydroxyphenyl)-1-pentanone

 $C_{11}H_{12}Br_2O_3$  mol. wt. 352.03



-Also refer to: [1947].

¹H NMR [1947, 2043].

USE: Preparation of pyrovalerone analogs as selective dopamine transporter inhibitors [1947].

# 5-Chloro-1-(4-fluoro-2-hydroxyphenyl)-1-pentanone

[149412-46-4]	$C_{11}H_{12}ClFO_2$	mol. wt. 230.67
F CO(CH ₂ ) ₃ CH ₂ Cl	Synthesis -Refer to: [2065]. m.p. 45–47° [2065]; [2065], IR [2065].	¹ H NMR [2065], ¹³ C NMR

# 5-Chloro-1-(2-hydroxy-3-nitrophenyl)-1-pentanone

$$\begin{array}{cccc} C_{11}H_{12}ClNO_4 & mol. \ wt. \ 257.67 \\ OH & Synthesis \\ NO_2 & CO(CH_2)_3CH_2Cl & -Refer \ to: \ [308]. \\ & \textbf{Methyl ether} & [187396-94-7] \\ & C_{12}H_{14}ClNO_4 & mol. \ wt. \ 271.70 \end{array}$$

-Refer to: [308].

# 5-Chloro-1-(2-hydroxy-5-nitrophenyl)-1-pentanone

	$C_{11}H_{12}CINO_4$	mol. wt. 257.67
OH CO(CH ₂ ) ₃ CH ₂ Cl	Synthesis -Refer to: [3284]	
	Methyl ether [1 $C_{12}H_{14}CINO_4$	mol. wt. 271.70
NO ₂		

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-Refer to: [308, 3284].
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m.p. 57–58° [3284].

# 5-Chloro-1-(2,4-dihydroxy-5-nitrophenyl)-1-pentanone

$C_{11}H_{12}CINO_5$			mol. wt. 273.67
HO NO ₂ HO	Synthesis -Refer to: [308]. <b>Dimethyl ether</b> $C_{13}H_{16}CINO_5$	[187396-92-5]	mol. wt. 301.73

-Refer to: [308].

### 5-Chloro-1-(2,4,6-trihydroxy-3-nitrophenyl)-1-pentanone

$C_{11}I$	$H_{12}CINO_6$	mol. wt. 289.67
OH NO ₂ CO(CH ₂ ) ₃ CH ₂ Cl HO OH	Synthesis -Refer to: [3284]. <b>Trimethyl ether</b> $C_{14}H_{18}CINO_6$	[187396-96-9] mol. wt. 331.75

-Refer to: [308, 3284].

# 5-Chloro-1-(5-chloro-2-hydroxyphenyl)-1-pentanone

$$\begin{array}{cccc} C_{11}H_{12}Cl_2O_2 & \mbox{mol. wt. } 247.12 \\ OH & Synthesis \\ \hline CO(CH_2)_3CH_2Cl & -Refer to: [3284]. \\ \hline Methyl \ ether & [187396-80-1] \\ C_{12}H_{14}Cl_2O_2 & \mbox{mol. wt. } 261.15 \\ \end{array}$$

-Refer to: [3284].

m.p. 52–54° [3284].

Benzyl ether	[1146443-98-2]	$C_{18}H_{18}Cl_2O_2$	mol. wt. 337.25
Defer to: [229/]			

-Refer to: [3284].

# 5-Chloro-1-(3-chloro-4,5-dihydroxyphenyl)-1-pentanone

	$C_{11}H_{12}Cl_2O_3$	mol. wt. 263.12
OH HO Cl	Synthesis -Refer to: [3087].	
	<b>Dimethyl ether</b> [817630-32-3] C ₁₃ H ₁₆ Cl ₂ O ₃	mol. wt. 291.17
CO(CH ₂ ) ₃ CH ₂ Cl	C13H16C12O3	11101. wt. 291.17

-Refer to: [3087].

USE: Preparation of N-substituted phenylpiperazines as dual function compounds [3087].

# 1-(4-Amino-5-chloro-2-hydroxyphenyl)-5-chloro-1-pentanone

$C_{11}H_{13}Cl_2NO_2$		mol. wt. 262.14
OH CO(CH ₂ ) ₃ CH ₂ Cl	Synthesis -Refer to: [680].	
	Methyl ether [166816-20-2]	
NH ₂	$C_{12}H_{15}Cl_2NO_2$	mol. wt. 276.16
Ċl	-Refer to: [680].	

### 1-(5-Amino-2,4-dihydroxyphenyl)-5-chloro-1-pentanone

$C_{11}H_{14}CINO_3$		mol. wt. 243.69
HO NH ₂ HO	Synthesis -Refer to: [3284]. <b>Dimethyl ether</b> [187396-97-0] C ₁₃ H ₁₈ ClNO ₃ -Refer to: [308, 3284].	mol. wt. 271.74

m.p. 87-88° [3284].

## 1-(3-Amino-2,4,6-trihydroxyphenyl)-5-chloro-1-pentanone

$$\begin{array}{ccc} C_{11}H_{14}CINO_4 & \text{mol. wt. 259.69} \\ OH & Synthesis \\ NH_2 & CO(CH_2)_3CH_2Cl & -Refer to: [3284]. \\ HO & OH & C1_{14}H_{20}CINO_4 & \text{mol. wt. 301.77} \\ \end{array}$$

-Refer to: [308, 3284].

# 5-Bromo-1-(6-hydroxy-1,3-benzodioxol-5-yl)-1-pentanone

 $[173055-10-2] C_{12}H_{13}BrO_4 mol. wt. 301.14$   $HO \qquad O \qquad Synthesis \\ BrCH_2(CH_2)_3CO \qquad O \qquad -Refer to: [2623].$ 

# 5-Chloro-1-(4-hydroxy-3-methoxy-5-nitrophenyl)-1-pentanone

[817630-35-6]	C ₁₂ H ₁₄ ClNO ₅	mol. wt. 287.70
OH NO ₂ OCH ₃	Synthesis -Refer to: [3087].	
CO(CH ₂ ) ₃ CH ₂ Cl	USE: Preparation of N-substituted ph dual function compounds [3087].	enylpiperazines as

# 2-Bromo-1-(2-hydroxy-5-methylphenyl)-1-pentanone

C	$_{12}H_{15}BrO_2$	mol. wt. 271.15
$\bigcup_{\substack{I \\ CH_3}}^{OH} CO-CH-(CH_2)_2CH_3$	Synthesis -Refer to: [181]. m.p. 51–52° [181].	

# 5-Bromo-1-(2-hydroxy-5-methylphenyl)-1-pentanone

[173055-00-0] 
$$C_{12}H_{15}BrO_2$$
 mol. wt. 271.15  
OH Synthesis  
CO(CH₂)₃CH₂Br -Refer to: [2623].  
CH₃

### 2-Bromo-1-(4-hydroxyphenyl)-4-methyl-1-pentanone

	$C_{12}H_{15}BrO_2$		mol. wt. 271.15
он Д	Synthesis -Refer to: [323	2].	
$\square$	Methyl ether C ₁₃ H ₁₇ BrO ₂	-	mol. wt. 285.18
COCHBrCH ₂ CH(CH ₃ ) ₂	-15 17 -2		

-Obtained by treatment of 1-(4-methoxyphenyl)-4-methyl-1-pentanone with bromine in a mixture of ethyl ether/dioxane at r.t. (90 %) [3232]. -Also refer to: [1114].

b.p._{0.1} 124–125° [3232]; ¹H NMR [1114, 3232], IR [3232], MS [1114].

# $\label{eq:constraint} {\mbox{Trimethylsilyl ether}} \ \ [719311-19-0] \ \ \ C_{21}H_{35}BrO_2Si \ \ \ mol. \ wt. \ 427.50$

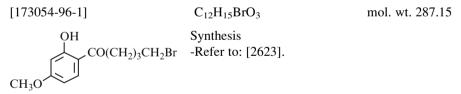
-Refer to: [627].

OCH3

# 5-Bromo-1-(2-hydroxy-3-methoxyphenyl)-1-pentanone

[173055-16-8]	$C_{12}H_{15}BrO_3$	mol. wt. 287.15
CH ₃ O CO(CH ₂ ) ₃ CH ₂ Br	Synthesis -Refer to: [2623].	

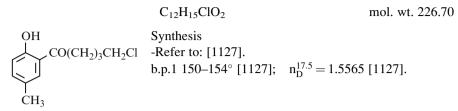
### 5-Bromo-1-(2-hydroxy-4-methoxyphenyl)-1-pentanone



### 5-Bromo-1-(2-hydroxy-5-methoxyphenyl)-1-pentanone

[173054-85-8] 
$$C_{12}H_{15}BrO_3$$
 mol. wt. 287.15  
OH Synthesis  
CO(CH₂)₃CH₂Br -Refer to: [2623].

### 5-Chloro-1-(2-hydroxy-5-methylphenyl)-1-pentanone



### 5-Chloro-1-(2-hydroxy-4-methoxyphenyl)-1-pentanone

[66832-66-4]	C ₁₂ H ₁₅ ClO ₃	mol. wt. 242.70
CH ₃ O ^{OH} CO(CH ₂ ) ₃ CH ₂ Cl	Synthesis -Obtained by reaction of 5-chlor with m-methoxyphenol in the p trifluoride etherate at 90° for 1	presence of boron

yellow needles [820]; m.p. 25.5–26.5° [820]; ¹H NMR [820], IR [820].

# 5-Chloro-1-(4-hydroxy-3-methoxyphenyl)-1-pentanone

[817630-37-8]	$C_{12}H_1$	₅ ClO ₃	mol. wt. 242.70
OH OCH ₃	Synthesis -Refer to: [3087].		
CO(CH ₂ ) ₃ CH ₂ Cl	USE: Preparation of function compounds	-	nylpiperazines as dual
Benzyl ether	[817630-36-7]	C ₁₉ H ₂₁ ClO ₃	mol. wt. 332.82

-Refer to: [3087].

USE: Preparation of N-substituted phenylpiperazines as dual function compounds [3087].

# 5-Bromo-1-(2-hydroxy-3,4-dimethoxyphenyl)-1-pentanone

[173055-06-6]	$C_{13}H_{17}BrO_4$	mol. wt. 317.18
CH ₃ O CH ₃ O CH ₃ O CH ₂ O CH ₂ D CH ₂ Br	Synthesis -Refer to: [2623].	

### 5-Bromo-1-(2-hydroxy-4,5-dimethoxyphenyl)-1-pentanone

[173054-91-6] 
$$C_{13}H_{17}BrO_4$$
 mol. wt. 317.18  
OH Synthesis  
CH₃O  $CO(CH_2)_3CH_2Br$  -Refer to: [2623].

## 5-Bromo-1-(2-hydroxy-3,4,6-trimethoxyphenyl)-1-pentanone

[173055-08-8]  $C_{14}H_{19}BrO_5$  mol. wt. 347.20 OH Synthesis  $CH_3O + CO(CH_2)_3CH_2Br$  -Refer to: [2623].  $CH_3O + OCH_3$ 

## 5-Bromo-1-[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-pentanone

C ₁₅	$H_{21}BrO_2$	mol. wt. 313.23
(CH ₃ ) ₂ CH	Synthesis -Refer to: [220].	
	Methyl ether [72236-93-2]	
CH ₃	$C_{16}H_{23}BrO_2$	mol. wt. 327.26
CO(CH ₂ ) ₃ CH ₂ Br		

-Obtained by reaction of 5-bromopentanoyl chloride with thymol methyl ether in the presence of aluminium chloride in methylene chloride at r.t. (90 %) [220].

m. p. 40° [220].

BIOLOGICAL ACTIVITY: Amebicidal and bactericidal and molluscicidal [220].

### 5-Chloro-1-(4-hydroxy[1,1'-biphenyl]-3-yl)-1-pentanone

C ₁₇	$H_{17}ClO_2$	mol. wt. 288.77
CO(CH ₂ ) ₃ CH ₂ Cl	Synthesis -Refer to: [308]. <b>Methyl ether</b> [187396-84-5] C ₁₈ H ₁₉ ClO ₂	mol. wt. 302.80

-Refer to: [308].

# 2-Bromo-1-[3,5-bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-pentanone

[17055-15-1]	$C_{19}H_{29}BrO_2$	mol. wt. 369.34
$(CH_3)_3C$ $(CH_3)_3C$ $(CH_3)_3$ $(CCH_3)_3$ $(COCHBr(CH_2)_2CH_3)$	Syntheses -Obtained by reaction of 4-hydroxy-3,5-di-tert-butylval octane for 30 min at 70° (84 9 m.p. 78–80° [3238, 3239].	erophenone in

# 2-Bromo-1-[3,5-bis(1,1-dimethylethyl)-4-hydroxyphenyl]-4-methyl-1-pentanone

C II Dro

$C_{20}H_{31}B_{1}$	O ₂ mol. wt. 383.37
$(CH_3)_3C$ $(CH_3)_3C$ $(CH_3)_3$ $(COCHBrCH_2CH(CH_3)_2)$	Synthesis -Obtained by reaction of 2-bromoisocaproyl chloride with 2,6-di-tert-butylphenol in the presence of titanium tetrachloride [1468].

# 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-3-chloro-3-ethyl-1-pentanone

[174635-33-7]	$C_{21}H_{33}ClO_2$	mol. wt. 352.94
(CH ₃ ) ₃ C C(CH ₃ ) ₃ C C	Synthesis -Refer to: [2482]. m.p. 72° [1499].	
$\begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \end{array} \\ COCH_2 - \begin{array}{c} C - C_2H_5 \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \end{array}$	BIOLOGICAL ACTIVITY: and analgesic agent [2482].	Antiinflammatory

#### Aromatic Hydroxyketones Derived from 5 **5-Oxopentanoic Acid**

# 5.1 Unsubstituted Hydroxyketones

# 5-(2-Hydroxyphenyl)-5-oxo-1-pentanoic acid

[4648-97-9]	$C_{11}H_{12}O_4$	mol. wt. 208.21
OH CO(CH ₂ ) ₃ CO ₂ H	Syntheses -Obtained by Fries rearrangement with aluminium chloride,	of phenyl glutarate

-in the presence of solvents,

*in the tetrachloroethane/nitrobenzene mixture (500 ml/125 ml) for 4 h at 50-60° [902];

*in nitrobenzene for 4 h at 50°, then 30 min at  $60^{\circ}$  [902];

*in tetrachloroethane at  $80^{\circ}$  [902].

-without solvent at  $120^{\circ}$  [902].

-Also obtained by reaction of glutaric anhydride with phenol in the presence of aluminium chloride in tetrachloroethane/nitrobenzene mixture (1:1) at  $130-140^{\circ}$ for 3 h (50 %) [588].

-Also obtained by dehydrogenation of 2-(4-carbomethoxybutyroyl)cyclohexanone in the presence of 10 % Pt/C in refluxing p-cymene for 24 h [331], (37 %) [332]. -Refer to: [588].

pale yellow heavy prisms [332]; m.p. 112° [902], 110° [331, 332], 100° [588]; IR [331, 332], UV [331, 332].

### **2,4-Dinitrophenylhydrazone** [4648-98-0] C₁₇H₁₆N₄O₇ mol. wt. 388.34

-Refer to: [588].

m.p. 185° [588].

### 5-(4-Hydroxyphenyl)-5-oxo-1-pentanoic acid

C₁₁H₁₂O₄ [4648-94-6] mol. wt. 208.21

HO-

 $\sim$  -CO(CH₂)₃CO₂H Syntheses -Obtained by Fries rearrangement of phenyl glutarate with aluminium chloride,

-in the presence of solvents:

*in the tetrachloroethane/nitrobenzene mixture (500 ml/125 ml) for 4 h at  $50-60^{\circ}$ [902];

*in nitrobenzene for 4 h at  $50^{\circ}$ , then 30 min at  $60^{\circ}$  [902];

*in tetrachloroethane at 80° [902].

-without solvent at  $120^{\circ}$  [902].

-Also obtained by reaction of glutaryl chloride with phenol in the presence of aluminium chloride in nitrobenzene, first at 0–5°, then at r.t. for 18 h (38 %) [588]. -Also obtained by reaction of glutaric anhydride with phenol in the presence of aluminium chloride in tetrachloroethane/nitrobenzene mixture (1:1) at  $130-140^{\circ}$ for 3 h (50 %) [588].

-Also obtained by treatment of methylphenyl glutarate ( $C_{12}H_{14}O_4$ ; b.p. 12 178–179°) with aluminium chloride in nitrobenzene at 50° for 20 min (60-80 %) [3135]. -Refer to: [3099].

colourless needles [588]; m.p. 202–203° [3135], 200–202° [902], 199–200° [588], 190–195° [3099];

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IR [588].
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**2,4-Dinitrophenylhydrazone** [4648-95-7] C₁₇H₁₆N₄O₇ mol. wt. 388.34

m.p. 175° [588].

-Obtained by reaction of acetic anhydride with the title ketone in refluxing pyridine for 6 h [588].

m.p. 120° [588].

Methyl ether	[4609-10-3]	$C_{12}H_{14}O_4$	mol. wt. 222.24
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-Obtained by reaction of glutaryl chloride with anisole in the presence of aluminium chloride in nitrobenzene/tetrachloroethane mixture (1:4) [588].

- -Also obtained by reaction of glutaric anhydride with anisole in the presence of aluminium chloride [2359, 3401], (90 %) [2132] in tetrachloroethane/nitrobenzene (1:2) at  $0-5^{\circ}$  [588].
- -Also obtained by hydrolysis of 2-anisoylglutarimide ([19450-23-8], m.p. 169.5– $170.5^{\circ}$ ) with refluxing 6 N HCl for 24 h (87 %) [3327].

-Also obtained by treatment of its ethyl ester with boiling 10 % aqueous potassium hydroxide for 6 h (87.5 %) [1422].

-Also refer to: [435, 878 (32 %), 989 (85 %), 1131, 1370, 1394, 1529 (90 %), 1567 (61 %), 2025, 2358, 2392 (93.5 %), 2519 (82 %), 2843, 3152, 3288, 3367].

m.p. 141–142° [939, 1131], 140–141° [1422], 140° [2820], 139.5–141° [3327], 139.5–140.5° [989], 138.5–141° [1529, 2358, 2359], 138–140° [1688, 2519], 138–139° [2392], 138° [588, 1567], 137–138° [3401], 137° [3288], 136–138° [1370], 133° [435], 132–135° [878], 126–128° [2132], 115–125° [874]; ¹H NMR [435, 939, 1131, 1370, 3152], ¹³C NMR [435], IR [588, 939, 1370], MS [435, 1567];  $pK_a$  [2358].

BIOLOGICAL ACTIVITY: Human zinc insulin, delivery of [1131]; Anorexigen [2358].

# 2,4-Dinitrophenylhydrazone of the methyl ether

[4626-83-9]	$C_{18}H_{18}N_4O_7$	mol. wt. 402.36
m.p. 145° [588].		

# Methyl ester of the methyl ether [1847-68-3] $C_{13}H_{16}O_4$ mol. wt. 236.27

-Refer to: [219, 939, 1274, 1512, 1528, 1567, 1693, 1734, 2843, 3157, 3254];

b.p._{0.6} 152–153° [1274]; m.p. 54–55° [3254], 52–54° [1512, 1528], 52–53° [1274], 52° [939], 49–51° [3157]; ¹H NMR [939, 1512, 1734], ¹³C NMR [1512], IR [939, 1512], UV [3254], MS [1512, 1567, 3254]; ESR spectroscopy [3254]; Phosphorescence Spectroscopy [3254].

## Ethyl ester of the methyl ether [25305-58-2] $C_{14}H_{18}O_4$ mol. wt. 250.29

-Preparation by reaction of ethanol with  $\gamma$ -anisoylbutyric acid in the presence of concentrated sulfuric acid in refluxing benzene for 7 h (93 %) [1529].

-Preparation by refluxing a mixture of ethanol and  $\gamma$ -anisoylbutyric acid in the presence of concentrated sulfuric acid for 6 h [247].

-Also obtained (90 %) [2241] according to the procedure [3121].

-Also refer to: [2241, 2426, 2689, 2805 (24 %), 3254].

pale yellow crystalline solid [1529]; white needles [247]; colourless crystals [2241]; b.p._{0.15-0.20} 170–185° [1529]; b.p.₅₋₆ 193–195° [391]; m.p. 60° [2820], 59–60° [247], 58.5–59.5° [1529], 58.5–59° [2689], 56.5–56.6° [2241], 56–58° [2805], 54° [3254]. ¹H NMR [2241], ¹³C NMR [2241], IR [2241], MS [3254].

**Ethyl ether** [34670-10-5] C₁₃H₁₆O₄ mol. wt. 236.27

-Obtained by reaction of glutaric anhydride with phenetole in the presence of aluminium chloride in refluxing carbon disulfide for 2 h (64 %) [3288].

-Also obtained by treatment of its ethyl ester with boiling 10 % aqueous potassium hydroxide for 6 h (70.1 %) [1422].

-Also obtained by oxidation of 5-ethoxyphenylvaleric acid (m.p. 105°) with chromic acid in dilute acetic acid (25 %) [3194].

m.p. 118° [3194], 116.5–117° [3194], 114–116° [1422], 114° [3288].

**Ethyl ester** [66123-78-2]  $C_{13}H_{16}O_4$  mol. wt. 236.27

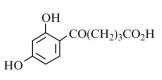
-Obtained by treatment of ethyl 5-(4-methoxyphenyl)-5-oxo-1-pentanoate with pyridinium chloride at 185° for 20 h (37 %) [878]. -Also refer to: [3099].

m.p. 75–78° [3099], 74–75° [878].

### 5-(2,4-Dihydroxyphenyl)-5-oxo-1-pentanoic acid

C₁₁H₁₂O₅ mo

mol. wt. 224.21



 $CO(CH_2)_3CO_2H$  -Obtained by reaction of glutaric anhydride with resorcinol in the presence of aluminium chloride in nitrobenzene, first at 10–15° for 2 h, then at  $30-32^\circ$  for 2 h (78 %) [588].

-Also refer to: [346 (30 %), 3370].

m.p. 181° [445], 180° [588], 175–178° [3374]; IR [588].

Syntheses

### **2,4-Dinitrophenylhydrazone** [4642-44-8] C₁₇H₁₆N₄O₈ mol. wt. 404.34

m.p. 285° [588].

Dimethyl ether	[4654-07-3]	$C_{13}H_{16}O_5$	mol. wt. 252.27
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-Obtained by reaction of glutaric anhydride with resorcinol dimethyl ether in the presence of aluminium chloride in nitrobenzene between -5 and  $0^{\circ}$  for 2 h (80 %) [588].

-Also refer to: [878, 1293, 1567, 1971, 2820].

m.p. 110–118° [878], 110° [588], 106–107° [1293], 98–100° [2820]; ¹H NMR [1971], IR [588, 1971], MS [1567].

### 5-(2,5-Dihydroxyphenyl)-5-oxo-1-pentanoic acid

 $\begin{array}{ccc} C_{11}H_{12}O_5 & \text{mol. wt. } 224.21 \\ OH & Synthesis \\ CO(CH_2)_3CO_2H & -Refer to: [1567]. \\ \hline \textbf{Dimethyl ether} & [63467-20-9] \\ C_{13}H_{16}O_5 & \text{mol. wt. } 252.27 \end{array}$ 

-Obtained by reaction of glutaric anhydride with hydroquinone dimethyl ether in the presence of aluminium chloride in a nitrobenzene/tetrachloroethane mixture first at  $0-5^{\circ}$ , then at r.t. for 24 h [1567], (75 %) [2998].

-Also obtained by acylation of 1,4-dimethoxybenzene with  $\gamma$ -carbethoxybutyryl chloride as described [1365], (80 %) [112]. The acylation in polyphosphoric acid at 71° for 2.5 h gave only 28 % [112].

-Also refer to: [1210].

m.p. 98–100° [2820], 98–99° [2998], 78–98.5 (?) [112], 96–99° [1210]; N.B.: Probably 98–98.5° [112]. MS [1567].

# **Semicarbazone of the dimethyl ether** C₁₄H₁₉N₃O₅ mol. wt. 309.32

m.p. 123° [2820], 122–123° [2998].

### Methyl ester of the dimethyl ether [855153-59-2] C₁₄H₁₈O₅ mol. wt. 266.29

-Refer to: [112, 1734].

m.p. 50.5° [1734], 48–50° [112]; ¹H NMR [1734], ¹³C NMR [1734], IR [1734].

Ethyl ester of the dimethyl ether	$C_{15}H_{20}O_5$	mol. wt. 280.32
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b.p.75 260-263° [2820].

**Diethyl ether** [94119-33-2]

 $C_{15}H_{20}O_5$ 

-Refer to: [768].

m.p. 105–106° [768]; ¹H NMR [768].

### 5-(3,4-Dihydroxyphenyl)-5-oxo-1-pentanoic acid

	$C_{11}H_{12}O_5$	mol. wt. 224.21
ŌН	Syntheses	
ОН	-Refer to: [477, 1293].	
	m.p. 210–215° [477].	
Ý	<b>Dimethyl ether</b> [4378-55-6]	
CO(CH ₂ ) ₃ CO ₂ H	$C_{13}H_{16}O_5$	mol. wt. 252.27

-Obtained by reaction of glutaric anhydride with veratrole in the presence of aluminium chloride,

*in a nitrobenzene/tetrachloroethane mixture first at  $0-5^{\circ}$ , then at r.t. for 24 h (41 %) [1567];

*in nitrobenzene at 0° for 12 h (45 %) [1278], (68 %) [2892]. -Also refer to: [569, 1293, 2071].

Yellowish solid [2071]; colourless prisms [2892]; m.p. 152° [2820], 145.7–146° [2071], 142° [1567], 140–142° [569, 1278, 2892]; ¹H NMR [2071, 2892], ¹³C NMR [2071], IR [2071, 2892], MS [1567, 2071].

Methyl ester of the dimethyl ether [131699-22-4] C₁₄H₁₈O₅ mol. wt. 266.29

-Refer to: [569, 1567, 1734].

m.p. 58–59° [569]; ¹H NMR [1734], MS [1567].

### Ethyl ester of the dimethyl ether [101499-71-2] $C_{15}H_{20}O_5$ mol. wt. 280.32

b.p._{0.05} 179–180° [1358]; m.p. 63° [1358], 58.5–60.5° [1363].

Methylenedioxy[87961-41-9] $C_{12}H_{12}O_5$ mol. wt. 236.22

-Refer to: [1132 (71 %), 2497].

¹H NMR [1132], IR [1132], MS [2497].

### 5-(2,3,4-Trihydroxyphenyl)-5-oxo-1-pentanoic acid

$C_{11}H_{12}O_6$			mol. wt. 240.21
OH	Synthesis		
HO CO(CH ₂ ) ₃ CO ₂ H	-Refer to: [592].		
	Trimethyl ether	[16093-16-6]	
HO	$C_{14}H_{18}O_{6}$		mol. wt. 282.29

-Obtained by reaction of glutaryl chloride with pyrogallol trimethyl ether or in the presence of aluminium chloride in nitrobenzene [592]. -Also refer to: [1285, 1358, 1902, 2487, 2933].

m.p. 76–77° [1358], 74–76° [1902], 73–75° [1285], 72–73° [592]. ¹H NMR [2487, 2933], IR [2933].

BIOLOGICAL ACTIVITY: Antibacterial [2933].

# 2,4-Dinitrophenylhydrazone of the trimethyl ether

m.p. 155° [592].

# 5-(2,4,5-Trihydroxyphenyl)-5-oxo-1-pentanoic acid

$C_{11}H_{12}O_6$			mol. wt. 240.21
OH CO(CH ₂ ) ₃ CO ₂ H	Synthesis -Refer to: [2196].		
но ОН	Trimethyl ether $C_{14}H_{18}O_6$	[92865-60-6]	mol. wt. 282.29

-Obtained by reaction of glutaric anhydride with 1,2,4-trimethoxybenzene in the presence of boron trifluoride at  $0^{\circ}$  for 3 days (95 %) [2274]. -Also refer to: [112].

m.p. 164–165° [2274], 162–164° [112].

Methyl ester of trimethyl ether	$C_{15}H_{20}O_{6}$	mol. wt. 296.32
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m.p. 90.5-92.5° [112].

Ethyl ester of trimethyl ether	$C_{16}H_{22}O_{6}$	mol. wt. 310.35
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-Obtained by acylation of 1,2,4-trimethoxybenzene with  $\gamma$ -carbethoxybutyric acid in the presence of polyphosphoric acid for 2.5 h at 45° (62.6 %) [112].

colourless broad prismatic plates [112]; m.p. 80-82° [112].

**Triethyl ether** [63213-33-2] C₁₇H₂₄O₆ mol. wt. 324.37

-Obtained by reaction of glutaric anhydride with 1,2,4-triethoxybenzene in the presence of aluminium chloride in carbon tetrachloride first at r.t., then at  $70^{\circ}$  for 2 h (90 %) [2196].

m.p. 121° [2196].

BIOLOGICAL ACTIVITY: Spasmolytic action [2196]; Choleretic action [2196].

### 5-(2,4,6-Trihydroxyphenyl)-5-oxo-1-pentanoic acid

 $\begin{array}{c} C_{11}H_{12}O_6 & \text{mol. wt. } 240.21 \\ OH & Synthesis \\ + CO(CH_2)_3COOH & -Refer to: [3374]. \\ HO & OH \end{array}$ 

# 4-Ethyl-5-(2-hydroxyphenyl)-5-oxo-1-pentanoic acid

C ₁₃ I	$H_{16}O_4$	mol. wt. 236.27
OH CO-CH-CH ₂ CH ₂ CO ₂ H	Synthesis -Refer to: [2300]. <b>Methyl ether</b> C ₁₄ H ₁₈ O ₄	mol. wt. 250.29

-Refer to: [2299, 2300 (72 %)].

b.p.1 190° [2299, 2300].

## 5-(4-Hydroxyphenyl)-2,2-dimethyl-5-oxo-1-pentanoic acid

	$C_{13}H_{16}O_4$	mol. wt. 236.27
OH CH	Synthesis -Refer to: [2358]. <b>Methyl ether</b> [61468-98-2]	
CH ₃	$C_{14}H_{18}O_4$	mol. wt. 250.29
COCH ₂ CH ₂ -C-CO ₂ H CH ₃	-Obtained by reaction of anhydride with anisole in the p chloride [2359],	

*first at 0°, then at r.t. (10 %) [2358]; *at 0–5° (80–85 %) [1097]. -Also refer to: [2479].

m.p.  $102^{\circ}$  [2358, 2359], 92–94° [2479], 90° [1097]; ¹H NMR [1097, 2358, 2479], ¹³C NMR [2479], IR [2479], UV [1097], MS [2479]; pK_a [2358].

BIOLOGICAL ACTIVITY: Anorexigen [2358].

### 5-(4-Hydroxyphenyl)-3,3-dimethyl-5-oxo-1-pentanoic acid

[135312-40-2]	$C_{13}H_{16}O_4$	mol. wt. 236.27
$\bigcup_{\substack{I \in I \\ COCH_2 - C - CH_2CO_2H \\ CH_3}}^{OH}$	Synthesis -Obtained by treatment 3,3-dimethyl-5-oxo-1-penta chloride at 185° for 20 h (7 m.p. 112–114° [878].	anoic acid with pyridinium

**Methyl ether** [31526-44-0] C₁₄H₁₈O₄ mol. wt. 250.29

-Obtained by reaction of 3,3-dimethylglutaric anhydride with anisole in the presence of aluminium chloride,

*in methylene chloride with ice-ethanol bath cooling and stirring for 3 h (96 %) [878];

*in nitrobenzene for 3 weeks at  $0^{\circ}$  (95 %) [172].

oil [172, 878]; ¹H NMR [172], IR [172].

# 2,4-Dinitrophenylhydrazone of the methyl ether $C_{20}H_{22}N_4O_7$ mol. wt. 430.42

m.p. 177.5–180° [172].

Ethyl ester of the methyl ether [31526-46-2]  $C_{16}H_{22}O_4$  mol. wt. 278.35

-Obtained by treatment of 5-(4-methoxyphenyl)-3,3-dimethyl-5-oxo-1-pentanoic acid with ethanol in the presence of hydrogen chloride gas (quantitative yield) [172].

¹H NMR [172].

Ethyl ester	[119348-66-2]	$C_{15}H_{20}O_4$	mol. wt. 264.32
L'unyi cotei		015112004	mon. wt. 201.32

-Obtained by treatment of 5-(4-hydroxyphenyl)-3,3-dimethyl-5-oxo-1-pentanoic acid in ethanol with methanesulfonic acid at 25° for 25 h (79 %) [878].

m.p. 98-100° [878].

### 4-Ethyl-5-(2,3-dihydroxyphenyl)-5-oxo-1-pentanoic acid

Dimethyl ether $C_{15}H_{20}O_5$ mol. wt. 280.32-Refer to: [2300 (67 %)];b.p._{0.4} 190° [2300].

# 5.2 Substituted Hydroxyketones

### 5-(5-Bromo-2,4-dihydroxyphenyl)-5-oxo-1-pentanoic acid

[16093-14-4]	$C_{11}H_{11}BrO_5$	mol. wt. 303.11
HO Br	Synthesis -Obtained by reaction of 4-bromo-resorcinol in the chloride in nitrobenzene [59 m.p. 123° [592].	presence of aluminium

**2,4-Dinitrophenylhydrazone** [16093-59-7]  $C_{17}H_{15}BrN_4O_8$  mol. wt. 483.23

m.p. 225° [592].

Dimethyl ether	[16093-36-0]	$C_{13}H_{15}BrO_5$	mol. wt. 331.16
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-Obtained by reaction of glutaryl chloride with 4-bromoresorcinol dimethyl ether in the presence of aluminium chloride in nitrobenzene at  $0^{\circ}$  for 13 h (35 %) [592].

m.p. 134–135° [592].

### 2,4-Dinitrophenylhydrazone of the dimethyl ether

[16093-37-1]	$C_{19}H_{19}BrN_4O_8$	mol. wt. 511.29
m.p. 181° [592].		

### 5-(2-Chloro-4-hydroxyphenyl)-5-oxo-1-pentanoic acid

	$C_{11}H_{11}ClO_4$	mol. wt. 242.66
ОН	Synthesis -Refer to: [1567].	
Cl CO(CH ₂ ) ₃ CO ₂ H	Methyl ether [71354-35-3] $C_{12}H_{13}CIO_4$	mol. wt. 256.69

-Obtained by reaction of glutaric anhydride with 3-chloroanisole in the presence of aluminium chloride in a nitrobenzene/tetrachloroethane mixture first at  $0-5^{\circ}$ , then at r.t. for 24 h (48 %) [1567].

m.p. 114–116° [1567]; IR [1567], MS [1567].

Methyl ester of the methyl ether [71354-36-4] C₁₃H₁₅ClO₄ mol. wt. 270.71

-Refer to: [1567].

b.p.₁ 200° [1567]; MS [1567].

#### 5-(2-Chloro-5-hydroxyphenyl)-5-oxo-1-pentanoic acid

$$\begin{array}{cccc} C_{11}H_{11}ClO_4 & \text{mol. wt. } 242.66 \\ OH & Synthesis & \\ & -Refer \text{ to: } [1567]. \\ & \mathbf{Methyl \ ether} & [71354-33-1] \\ CO(CH_2)_3CO_2H & C_{12}H_{13}ClO_4 & \text{mol. wt. } 256.69 \end{array}$$

-Obtained by reaction of glutaric anhydride with 4-chloroanisole in the presence of aluminium chloride in a nitrobenzene/tetrachloroethane mixture first at  $0-5^{\circ}$ , then at r.t. for 24 h (61 %) [1567].

m.p. 105° [1567]; IR [1567], MS [1567].

# Methyl ester of the methyl ether [71354-34-2] C₁₃H₁₅ClO₄ mol. wt. 270.71

-Refer to: [1567].

b.p.₁ 190° [1567]; MS [1567].

### 5-(3-Chloro-4-hydroxyphenyl)-5-oxo-1-pentanoic acid

	$C_{11}H_{11}ClO_4$	mol. wt. 242.66
ОН	Synthesis	
CI	-Refer to: [1567].	
	Methyl ether [71354-31-9]	
$\mathbf{i}$	$C_{12}H_{13}ClO_4$	mol. wt. 256.69
CO(CH ₂ ) ₃ CO ₂ H		

-Obtained by reaction of glutaric anhydride with anisole in the presence of aluminium chloride in a nitrobenzene/tetrachloroethane mixture first at 0–5°, then at r.t. for 24 h (80.3 %) [1567].

-Also refer to: [1779, 3228].

m.p. 160–162° [1779], 156° [1567]; IR [1567], MS [1567].

BIOLOGICAL ACTIVITY: Refer to: [1779, 3228].

Methyl ester of the methyl ether [71354-32-0]  $C_{13}H_{15}ClO_4$  mol. wt. 270.71

-Refer to: [1567].

m.p. 76° [1567]; MS [1567].

### 5-(5-Chloro-2,4-dihydroxyphenyl)-5-oxo-1-pentanoic acid

[16093-18-8]	$C_{11}H_{11}ClO_5$	mol. wt. 258.66
HO CO(CH ₂ ) ₃ CO ₂ H	Synthesis -Obtained by reaction of 4-chloro-resorcinol in the chloride in nitrobenzene [59 m.p. 140° [592].	presence of aluminium

### **2,4-Dinitrophenylhydrazone** [16093-19-9] C₁₇H₁₅ClN₄O₈ mol. wt. 438.78

m.p. 171° [592].

Dimethyl ether	[16093-30-4]	$C_{13}H_{15}ClO_5$	mol. wt. 286.71
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-Obtained by reaction of glutaryl chloride with 4-chlororesorcinol dimethyl ether in the presence of aluminium chloride in nitrobenzene [592].

m.p. 160° [592].

### 2,4-Dinitrophenylhydrazone of the dimethyl ether

[16093-31-5] C₁₉H₁₉ClN₄O₈ mol. wt. 466.84 m.p. 171° [592].

# 5-(4-Hydroxy-3-nitrophenyl)-5-oxo-1-pentanoic acid

$$C_{11}H_{11}NO_6$$
 mol. wt. 253.21

$NO_2$	Synthesis		
$\rightarrow \sim$	-Refer to: [249	4].	
HO-CO(CH ₂ ) ₃ CO ₂ H	Methyl ether	$C_{12}H_{13}NO_6$	mol. wt. 267.24

-Preparation from  $\gamma$ -anisoylbutyric acid [2494].

colourless prisms [2494]; m.p. 145° [2494].

Ethyl ether	$C_{13}H_{15}NO_6$	mol. wt. 281.26
-Preparation from γ-p	henetoylbutyric acid [2494].	

colourless plates [2494]; m.p. 127° [2494].

#### 5-(3-Amino-4-hydroxyphenyl)-5-oxo-1-pentanoic acid

$$C_{11}H_{13}NO_4 \qquad \text{mol. wt. } 223.23$$

$$NH_2 \qquad Synthesis \\ -Refer to: [2494]. \\ Methyl ether C_{12}H_{15}NO_4 \qquad \text{mol. wt. } 237.26$$

$$Pafor to: [2404]$$

 $C_{13}H_{17}NO_{4}$ 

-Refer to: [2494].

pale brown needles [2494]; m.p. 162–164 $^{\circ}$  [2494].

### Ethyl ether

-Refer to: [2494].

almost colourless plates [2494]; m.p. 176–178° [2494].

### 5-(2,4-Dihydroxy-6-methylphenyl)-3,5-dioxo-1-pentanoic acid

$$\begin{array}{cccc} C_{12}H_{12}O_6 & \text{mol. wt. 252.22} \\ OH & Syntheses \\ + & COCH_2COCH_2CO_2H & -Refer to: [454, 1330]. \\ Dimethyl ether & [66757-68-4] \\ CH_3 & C_{14}H_{16}O_6 & \text{mol. wt. 280.28} \end{array}$$

-Obtained by treatment of 2,4-dimethoxy-6-methylacetophenone with sodium amide in liquid ammonia, followed by carbon dioxide [2974]. -Also refer to: [1168, 1330 (54 %)].

liquid [1330]; ¹H NMR [1168, 1330], IR [1168, 1330].

 Dibenzyl ether
 [97271-30-2]
 C₂₆H₂₄O₆
 mol. wt. 432.47

 -Obtained
 from
 5-(2,4-dibenzyloxy-6-methylphenyl)-1,3-butanedione

 (78 %) [454].
 -Also refer to: [455].

colourless oil [454].

# 5-(5-Bromo-2,4-dihydroxy-6-methylphenyl)-5-oxo-1-pentanoic acid

[16093-45-1]	$C_{12}H_{13}BrO_5$	mol. wt. 317.14
HO Br CO(CH ₂ ) ₃ CO ₂ H CH ₃	Synthesis -Obtained by reaction of glutar 4-bromo-orcinol in the presence chloride in nitrobenzene [592]. m.p. 176° [592].	yl chloride with e of aluminium

mol. wt. 251.28

### **2,4-Dinitrophenylhydrazone** [16093-46-2] C₁₈H₁₇BrN₄O₈ mol. wt. 497.26

m.p. 179° [592].

Dimethyl ether	[16093-47-3]	$C_{14}H_{17}BrO_5$	mol. wt. 345.19

-Obtained by reaction of glutaryl chloride with 4-bromoorcinol dimethyl ether in the presence of aluminium chloride in nitrobenzene [592].

m.p. 117° [592].

## 5-(5-Chloro-2-hydroxy-4-methylphenyl)-5-oxo-1-pentanoic acid

```
mol. wt. 256.69
[4609-06-7]
                                   C12H13ClO4
                                 Synthesis
             \mathrm{CO}(\mathrm{CH}_2)_3\mathrm{CO}_2\mathrm{H} -Obtained by reaction of glutaric anhydride with
                                  4-chloro-3-methylphenol in the presence of alu-
CH
                                  minium chloride in tetrachloroethane/nitrobenzene
                                  mixture (1:1) [588].
   m.p. 140° [588]; IR [588].
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2,4-Dinitrophenylhydrazone

m.p. 210° [588].

### 5-(5-Chloro-2,4-dihydroxy-6-methylphenyl)-5-oxo-1-pentanoic acid

[16093-43-9] C12H13ClO5 mol. wt. 272.68 Synthesis

.CO(CH₂)₃CO₂H -Obtained by reaction of glutaryl chloride with 4-chloro-orcinol in the presence of aluminium chloride in nitrobenzene [592]. m.p. 172° [592].

### **2,4-Dinitrophenylhydrazone** [16093-44-0] C₁₈H₁₇ClN₄O₈ mol. wt. 452.81

C12H14O4

m.p. 191° [592].

[4642-27-7]

CH₃

HO

### 5-(2-Hydroxy-3-methylphenyl)-5-oxo-1-pentanoic acid

**Synthesis** H -Obtained by reaction of glutaric anhydride with o-cresol in the presence of aluminium chloride in tetrachloro-ethane/nitrobenzene mixture (1:1) at 120–130° for 3 h (60 %) [588].

mol. wt. 222.24

m.p. 125° [588]; IR [588].

 $C_{18}H_{17}Cl_2N_4O_7$ mol. wt. 436.81 2,4-Dinitrophenylhydrazone C₁₈H₁₈N₄O₇ mol. wt. 402.36 [4642-28-8]

m.p. 180° [588].

#### 5-(2-Hydroxy-4-methylphenyl)-5-oxo-1-pentanoic acid

$$[4642-35-7] C_{12}H_{14}O_4 mol. wt. 222.24$$

CO(CH₂)₃CO₂H -Obtained by reaction of glutaric anhydride with m-cresol in the presence of aluminium chloride in tetrachloroethane (80 %) [588], (36 %) [2724]. -Also refer to: [3224, 3366, 3398, 3399].

m.p. 140–141.5° [3398], 140° [588], 135° [3224], 134–135° [2724]; IR [588, 2724], UV [3398, 3399].

Syntheses

**2,4-Dinitrophenylhydrazone** [4642-36-8] C₁₈H₁₈N₄O₇ mol. wt. 402.36

m.p. 200° [588].

Methyl ester [63023-50-7]  $C_{13}H_{16}O_{4}$ mol. wt. 236.27

-Obtained by refluxing for 10 h a mixture of the title ketone, methanol and concentrated sulfuric acid (90 %) [2724]. -Also refer to: [3224].

b.p.₁₀ 169–170° [2724]; m.p. 50° [3224];

¹H NMR [2724, 3224], IR [2724, 3224].

**Methyl ester of the methyl ether** [29207-19-0]  $C_{14}H_{18}O_4$  mol. wt. 250.29

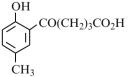
-Obtained by reaction of dimethyl sulfate with the methyl ester above in the presence of aqueous sodium hydroxide at r.t. for 1 h and then heated on a water bath for 3 h (70 %) [2724].

-Also refer to: [3398, 3399].

m.p. 38.5–39.2° [3399], 38.5–39° [3398]; ¹H NMR [2724], IR [2724, 3398], UV [3398, 3399]; TLC [2724].

#### 5-(2-Hydroxy-5-methylphenyl)-5-oxo-1-pentanoic acid

[4649-01-8]	$C_{12}H_{14}O_4$	mol. wt. 222.24



Synthesis -Obtained by reaction of glutaric anhydride with p-cresol in the presence of aluminium chloride in tetrachloroethane/nitrobenzene mixture (1:1), first at  $50-60^{\circ}$  for 2 h, then at  $100^{\circ}$  for 30 min [588].

m.p. 95° [588]; IR [588].

**2,4-Dinitrophenylhydrazone** [4649-02-9] C₁₈H₁₈N₄O₇ mol. wt. 402.36

m.p. 150° [588].

## 5-(4-Hydroxy-2-methylphenyl)-5-oxo-1-pentanoic acid

$$[4642-32-4]$$
 $C_{12}H_{14}O_4$ mol. wt. 222.24OHSynthesis-Obtained by reaction of glutaryl chloride with m-cresol in the

presence of aluminium chloride in nitrobenzene [588].

 $H_3$  m.p. 200° [588]; IR [588].

 $CO(CH_2)_3CO_2H$ 

**2,4-Dinitrophenylhydrazone** [4642-33-5]  $C_{18}H_{18}N_4O_7$  mol. wt. 402.36

m.p. 190° [588].

**Methyl ether** [4642-37-9] C₁₃H₁₆O₄ mol. wt. 236.27

-Obtained by reaction of glutaryl chloride with m-cresol methyl ether in the presence of aluminium chloride in tetrachloroethane [588].

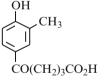
-Also obtained by reaction of glutaric anhydride with m-cresol methyl ether in the presence of aluminium chloride in tetrachloroethane/nitrobenzene mixture (1:1) at  $0-5^{\circ}$  (70 %) [588, 1567].

-Also refer to: [2820, 3366].

m.p. 138° [2820], 121° [1567], 120° [588]; IR [588], MS [1567].

## 5-(4-Hydroxy-3-methylphenyl)-5-oxo-1-pentanoic acid

[4592-82-9]	$C_{12}H_{14}O_4$	mol. wt. 222.24



Syntheses -Obtained by reaction of glutaryl chloride with o-cresol in the presence of aluminium chloride, in nitrobenzene [588].

-Also obtained by reaction of glutaric anhydride with o-cresol in the presence of aluminium chloride in tetrachloroethane/ nitrobenzene mixture (1:1) at 120–130° for 3 h (40 %) [588].

m.p. 184° [588]; IR [588].

## **2,4-Dinitrophenylhydrazone** [4680-89-1] C₁₈H₁₈N₄O₇ mol. wt. 402.36

m.p. 180° [588].

Methyl ether	[4642-30-2]	$C_{13}H_{16}O_4$	mol. wt. 236.27
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-Obtained by reaction of glutaryl chloride with o-cresol methyl ether in the presence of aluminium chloride in tetrachloroethane [588]. -Also obtained by reaction of glutaric anhydride with o-cresol methyl ether in the presence of aluminium chloride in tetrachloroethane/nitrobenzene mixture (1:1) at  $0-5^{\circ}$  (75 %) [588].

-Also refer to: [1567, 2820, 2838].

m.p. 141° [2820], 140° [588], 139° [1567], 126° [2838]; IR [588].

## 5-(2,4-Dihydroxy-6-methylphenyl)-5-oxo-1-pentanoic acid

-Obtained by reaction of glutaryl chloride with orcinol dimethyl ether in the presence of aluminium chloride in nitrobenzene [592].

m.p. 130° [592].

## 2,4-Dinitrophenylhydrazone of the dimethyl ether

[16093-42-8]	$C_{20}H_{22}N_4O_8$	mol. wt. 446.42
m.p. 155° [592].		

## 5-(2-Hydroxy-4-methoxyphenyl)-5-oxo-1-pentanoic acid

[4642-41-5]	$C_{12}H_{14}O_5$
CH ₃ O ^H CO(CH ₂ ) ₃ CO ₂ H	Syntheses -Obtained by resorcinol r aluminium

-Obtained by reaction of glutaric anhydride with resorcinol monomethyl ether in the presence of aluminium chloride in nitrobenzene, first at  $10-15^{\circ}$  for 2 h, then at  $30-32^{\circ}$  for 1 h (75 %) [588].

mol. wt. 238.24

-Also refer to: [1971, 2820].

m.p. 159° [588], 157° [2820]; ¹H NMR [1971], IR [588, 1971].

#### **2,4-Dinitrophenylhydrazone** [4642-42-6] C₁₈H₁₈N₄O₈ mol. wt. 418.36

m.p. 138° [588].

#### **Methyl ester** [66832-64-2] C₁₃H₁₆O₅ mol. wt. 252.27

-Obtained by reaction of dimethyl sulfate with  $\gamma$ -(2,4-dihydroxybenzoyl)butyric acid in the presence of potassium carbonate in refluxing acetone for 6 h (74 %) [346].

-Preparation from 5-(2-hydroxy-4-methoxyphenyl)-5-oxopentanoic acid by esterification with boron trifluoride/methanol [820].

m.p. 60–60.5° [820], 58° [346]; ¹H NMR [820], MS [820].

#### **Ethyl ester** $C_{14}H_{18}O_5$ mol. wt. 266.29

-Preparation from 5-(2-hydroxy-4-methoxyphenyl)-5-oxopentanoic acid by esterification with boron trifluoride/ethanol [820].

b.p._{0.07} 160–164° [820]; ¹H NMR [820], MS [820].

#### Methyl 5-(2-hydroxy-4-carbomethoxyphenyl)-5-oxo-1-pentanoate

 $\begin{array}{cccc} C_{14}H_{16}O_6 & \text{mol. wt. } 280.28 \\ OH & Synthesis \\ -Refer to: [3398]. \\ Methyl \ ether & [29207-22-5] \\ C_{15}H_{18}O_6 & \text{mol. wt. } 294.30 \end{array}$ 

-Preparation: Methyl 4-(2-methoxy-p-toluoyl)-butyrate (m.p.  $38.5-39^{\circ}$ ) was converted without purification of intermediates by the following sequence of reagents: N-bromosuccinimide in CCl₄ with ABIN, silver nitrate in aqueous acetonitrile, chromic acid in acetic acid, methanolic HCl [3398]. -Also refer to: [3399].

m.p. 78–79° [3398, 3399]; IR [3398], UV [3398, 3399].

#### 5-(2-Hydroxy-3,4,6-trimethylphenyl)-5-oxo-1-pentanoic acid

[84978-13-2]	$C_{14}H_{18}O_4$	mol. wt. 250.30
CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃	Synthesis -Refer to: [2325]. m.p. 155–157° [2325]; ¹ H IR [2325].	NMR [2325],

## Chapter 4 Hexanones

## 1 Aromatic Hydroxyketones Derived from Hexanoic Acids

## 1.1 Unsubstituted Hydroxyketones

#### 1-(4-Hydroxyphenyl)-1,3,5-hexanetrione

	$C_{12}H_{12}O_4$		mol. wt. 220.22
OH	Synthesis -Refer to: [116	8].	
	Methyl ether $C_{13}H_{14}O_4$	[4808-89-3]	mol. wt. 234.25
COCH ₂ COCH ₂ COCH ₃			

-Preparation: Trityl-lithium in THF was injected into pentane-2,4-dione under nitrogen at  $-15^{\circ}$ . When the red colour persisted methyl p-methoxybenzoate in THF was injected. The solution was stirred at  $-15^{\circ}$  and trityl-lithium solution added to just maintain the red colour [1168].

-Also obtained by reaction of acetylacetone with methyl p-methoxybenzoate in the presence of sodium hydride in 1,2-dimethoxyethane (72 %) [1218, 2944].

pale yellow crystals [1168]; m.p. 85–86° [1218], 81° [2944], 80–81° [1168]; ¹H NMR [1168], IR [1168], UV [1168], MS [1168]. **N.B.**: Several tautomers [1168].

#### 1-(2,4,6-Trihydroxyphenyl)-1,3,5-hexanetrione

mol. wt. 252.22 C12H12O6 **Synthesis** COCH₂COCH₂COCH₃ -Refer to: [2699]. **Trimethyl ether** [76631-01-1]  $C_{15}H_{18}O_{6}$ mol. wt. 294.30

-Preparation by acylation of dianion of 2,4-pentanedione with methyl 2,4,6-trimethoxybenzoate (61 %) [2699].

yellow crystals [2699]; m.p. 68.5–71.5° [2699]; ¹H NMR [2699], IR [2699], MS [2699].

Tribenzyl ether	[76631-02-2]	$C_{33}H_{30}O_6$	mol. wt. 522.60
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-Preparation by acylation of dianion of 2,4-pentanedione with methyl 2,4,6-tribenzyloxybenzoate (100 %) [2699].

yellow oil [2699]; ¹H NMR [2699], MS [2699].

#### 1-(3,4-Dihydroxyphenyl)-6-nitro-1,3-hexanedione

	$C_{12}H_{13}NO_6$	mol. wt. 267.24
OH OH	Synthesis -Refer to: [2780].	
	<b>Dimethyl ether</b> $C_{14}H_{17}NO_6$	mol. wt. 295.29
COCH ₂ CO(CH ₂ ) ₂ CO	$CH_2NO_2$	

-Obtained by reaction of 1-(3,4-dimethoxyphenyl)-1-trimethylsilyloxyethene with 4-nitrobutyryl chloride in tetrahydrofuran in the presence of methyllithium in ethyl ether at  $-100^{\circ}$  (55 %) [2780].

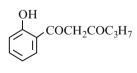
m.p. 95–96° [2780]; ¹H NMR [2780], IR [2780], MS [2780].

#### 1-(2-Hydroxyphenyl)-1,3-hexanedione

[80856-35-5]

C12H14O3

mol. wt. 206.24



Syntheses COCH₂COC₃H₇ -Obtained by reaction of 2-hydroxyacetophenone with methyl butyrate in the presence of sodium hydride (51 %) [2807].

-Also refer to: [3400].

pale yellow oil [2807]; b.p.₂ 135° [2807]; ¹H NMR [2807], IR [2807], UV [2807].

**Methyl ether** [1001024-93-6] C₁₃H₁₆O₃ mol. wt. 220.27

-Obtained by reaction of 2-methoxybenzoyl chloride with 2-pentanone in the presence of LDA (lithium diisopropylamide; 1.5 equiv.) in tetrahydrofuran (72 %) [3400].

## 1-(4-Hydroxyphenyl)-1,4-hexanedione

	$C_{12}H_{14}O_3$	mol. wt. 206.24
OH	Synthesis -Refer to: [2953].	
CO(CH ₂ ) ₂ COC ₂ H ₅	Methyl ether [62596-41-2] $C_{13}H_{16}O_3$	mol. wt. 220.27

-Obtained from propanal and 1-(4-methoxyphenyl)-2-propen-1-one (64 %) [2953]. -Also refer to: [162, 2856].

b.p._{0.65} 168° [2953]; m.p. 77° [162], 75° [2953]; ¹H NMR [2856, 2953], ¹³C NMR [2856], IR [2856, 2953], MS [2856].

## 1-(2,4-Dihydroxyphenyl)-1,4-hexanedione

$C_{12}H$	$I_{14}O_4$	mol. wt. 222.24
HO COCH ₂ CH ₂ COC ₂ H ₅	Synthesis -Refer to: [2953]. <b>Dimethyl ether</b> C ₁₄ H ₁₈ O ₄	[67756-20-1] mol. wt. 250.29

-Obtained from propanal and 1-(2,4-dimethoxyphenyl)-2-propen-1-one (60 %) [2953].

b.p._{0.1} 155° [2953]; m.p. 55–57° [2953], ¹H NMR [2953], IR [2953].

## 1-(3,4-Dihydroxyphenyl)-1,4-hexanedione

	$C_{12}H_{14}O_4$	mol. wt. 222.24
OH ↓ ,OH	Synthesis -Refer to: [2953].	
	<b>Dimethyl ether</b> [67756-23-4] C ₁₄ H ₁₈ O ₄	mol. wt. 250.29
COCH ₂ CH ₂ COC ₂ H ₅	- 17 10 - 7	

-Obtained from propanal and 1-(3,4-dimethoxyphenyl)-2-propen-1-one (60 %) [2953].

b.p._{0.2} 155° [2953]; m.p. 79° [2953], ¹H NMR [2953], IR [2953].

## 1-(2-Hydroxyphenyl)-1-hexanone

[3226-15-1]

 $C_{12}H_{16}O_2$ 

mol. wt. 192.26

Syntheses

- CH₂)₄CH₃ -Phenol was energetically mixed with finely divided aluminium chloride (1 mol) and heated to 100° (1 mol of hydrochloric acid is evolved); 1 mol of n-caproyl chloride was then added and heated to 125–130° for 1 h (56 %) [2700].
- -Also obtained by Fries rearrangement of phenyl caproate with aluminium chloride for 2 h at  $160-180^{\circ}$  (50 %) [726] or in tetrachloroethane [3169].
- -Also obtained [2478] by the method [2074].
- -Also obtained by adding TMSOTf (0.96 mmol) to a solution of 2-(1-hexynyl) phenol (0.48 mmol) and isobutyraldehyde (0.57 mmol) in ethyl ether under nitrogen at  $-78^{\circ}$ . After the reaction mixture had been stirred at  $-78^{\circ}$  for 3 h, it was warmed slowly to r.t. and stirred for 14 h until the reaction was complete (**4a**) (20 %) (Table 2, entry 2 in [2403]).
- -Also obtained by treatment of 2-(1-hexynyl)phenol with TMSOTf (2 equiv.) in ethyl ether at r.t. for 22 h (4) (94 %) (Table 6, entry 5 in [2403]).
- -Also obtained by treatment of 1-(2-hydroxyphenyl)-1-hexanol with manganese dioxide in methylene chloride for 7 h at r.t. (46 %) [77].
- -Also obtained by Fries rearrangement of phenyl caproate with aluminium chloride at  $140^{\circ}$  for 45 min (45 %) [932].
- -Also obtained by zeolite-catalyzed Friedel-Crafts reaction of anisole with caproic acid [3266].
- -Also obtained by treatment of 2-(1-hexynyl)phenol with TMSOTf in ether for 22 h at r.t. (94 %) [2403].
- -Also obtained by reaction of 2-(1-hexynyl)phenol with isopropyl aldehyde in the presence of TMSOTf in ether at  $-78^{\circ}$ , then at r.t. for 14 h (20 %) [2403].
- -Also obtained by reaction of 2-(1-hexynyl)phenol with 4-nitrophenylbenzaldehyde in the presence of TMSOTf in ether at  $-78^{\circ}$ , then at r.t. for 14 h (37 %) [2403]. -Also refer to: [260, 2134, 2946].

colourless oil [77]; light-yellow oil [2403]; b.p._{0.1} 83° [932], b.p._{0.6} 96–98° [3169], b.p._{1.2} 106–107° [2478], b.p.₁₀ 142–143°, b.p.₁₅ 145–147° [726]; m.p. 22° [726], 17.2–17.4° [2700]; ¹H NMR [77, 2403], ¹³C NMR [77, 2403], IR [77], UV [1996], MS [77]; TLC [1994]; pKa [2590]; dipole moment [2590];  $n_D^{25} = 1.5262$  [932].

USE: Preparation of chromanones and related chalcones *via* trimethylsilyl triflatepromoted electrophilic addition of (o-hydroxyphenyl)alkynes to aldehydes [2403]; Preparation and reduction of, [260].

## **2,4-Dinitrophenylhydrazone** [18405-71-5] C₁₈H₂₀N₄O₅ mol. wt. 372.38

m.p. 153° [932, 3169], 84–85° [524].

N.B.: One of the reported melting point is obviously wrong.

## **Methyl ether** [35031-70-0] C₁₃H₁₈O₂ mol. wt. 206.28

-Obtained by treatment of 2-(1-hexynyl)anisole with TMSOTf (2 equiv.) in ethyl ether at r.t. for 90 h (4) (51 %) (Table 6, entry 6 in [2403]).

-Also obtained by direct acylation of 2-bromoanisole with caproaldehyde by palladium catalysis (60 %) [2668].

-Also obtained treatment of 1-(2-methoxyphenyl)-1-hexyne with p-toluenesulfonic acid (PTSA) in ethanol at  $78^{\circ}$  for 144 h (80 %) [2337].

-Also obtained by treatment of 2-(1-hexynyl)anisole with TMSOTf in ether for 90 h at r.t. (51 %) [2403].

-Also obtained by action of hexanoyl chloride with anisole,

*in the presence of ferric chloride at reflux for 5 h (4 %) [3435];

*in the presence of aluminium chloride at reflux for 5 h (6 %) [3435].

-Also refer to: [1485, 1840, 1999, 3227 (67 %)].

light-yellow oil [2403]; m.p. 111–112° [3227]; ¹H NMR [1485, 1840, 2337, 2403, 2668, 3227], ¹³C NMR [1485, 1840, 2337, 2403, 2668], IR [1485, 1840, 2668], MS [1485, 2668].

USE: Preparation of chromanones and related chalcones *via* trimethylsilyl triflatepromoted electrophilic addition of (o-hydroxyphenyl)alkynes to aldehydes [2403].

## Ethyl ether $C_{14}H_{20}O_2$ mol. wt. 220.31

-Obtained by action of hexanoyl chloride with phenetole,

*in the presence of ferric chloride at reflux for 5 h (1 %) [3435];

*in the presence of aluminium chloride at reflux for 5 h (6 %) [3435].

Phenylhydrazone	$C_{18}H_{22}N_2O$	mol. wt. 282.38
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m.p. 102-103° [726].

Oxime	[204859-45-0]	$\mathrm{C}_{12}\mathrm{H}_{17}\mathrm{NO}_{2}$	mol. wt. 207.27
		ID [0555]	

m.p. 112° [2555]; ¹H NMR [2555], IR [2555].

USE: For preparation of copper/lead/zinc salicylaldoxime complexes [2555].

#### 1-(3-Hydroxyphenyl)-1-hexanone

[103119-13-7]	$C_{12}H_{16}O_2$	mol. wt. 192.26
OH	Syntheses -Preparation by reaction of m	n-acetoxybenzoyl chloride
CO(CH ₂ ) ₄ CH ₃	with dipentylcadmium in reflu Then, treatment of keto-ester of 10 % sodium hydroxide for 2–3	obtained by refluxing with

-Also obtained (13) by treatment of *trans*-3-(3-hex-2-enoyl)phenol (11) with hydrogen over Pd/C in ethyl acetate (58 %) [3292].

-Also obtained by treatment of its methyl ether with aluminium chloride in benzene by heating on a water bath for 2 h (80 %) [966].

-Also refer to: [583].

b.p.₁ 147–150° [2586]; m.p. 69° [2586], 63° [966].

#### Acetate

$$C_{14}H_{18}O_3$$
 mol. wt. 234.30

b.p._{0.5} 127–128° [2586].

#### **2,4-Dinitrophenylhydrazone of the acetate** C₂₀H₂₂N₄O₆ mol. wt. 414.42

m.p. 158° [2586].

Methyl ether	[342423-70-5]	$C_{13}H_{18}O_2$	mol. wt. 206.28
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-Obtained by condensation of pentylmagnesium bromide with m-methoxybenzamide in ethyl ether. The reaction was carried out at  $60^{\circ}$  for 48 h under hydrogen atmosphere (60–70 %) [966].

-Also obtained by direct acylation of 3-bromoanisole with caproaldehyde by palladium catalysis (88 %) [2668].

-Also refer to: [967, 1485, 1840, 2868, 3440].

b.p.₂₀ 220° [966, 967]; ¹H NMR [1485, 1840, 2668, 2868], ¹³C NMR [1485, 1840, 2668, 2868], IR [1485, 1840, 2668, 2868], MS [2668];  $n_D^{35} = 1.5189$  [967].

USE: Structures and catalytic activity in C-C coupling reactions of dinuclear and trinuclear triazolyldiylidene palladium complexes [3440]; Aquapalladium complex catalyst for intermolecular hydroamination of alkynes [2868].

### 2,4-Dinitrophenylhydrazone of the methyl ether $C_{19}H_{22}N_4O_5$ mol. wt. 386.41

m.p. 148° [967].

**Benzyl ether** [92532-18-8] C₁₉H₂₂O₂ mol. wt. 282.38

-Obtained by treatment of 3-(benzyloxy)phenyl-1-hexanol with pyridinium chlorochromate in methylene chloride at r.t. for 1.5 h (98 %) [2199]. -Also refer to: [583, 2197].

oil [2199].

BIOLOGICAL ACTIVITY: Antiallergic and inflammation inhibitor [2197, 2199].

2,4-Dinitrophenylhydrazone	$C_{18}H_{20}N_4O_5$	mol. wt. 372.38
m.p. 196° [2586].		
4-Nitrophenylhydrazone	$C_{18}H_{21}N_3O_3$	mol. wt. 327.38

m.p. 158° [967].

OH

## 1-(4-Hydroxyphenyl)-1-hexanone

Syntheses

-Phenol was energetically mixed with finely divided aluminium chloride (1 mol) and heated to  $100^{\circ}$  (1 mol of hydrochloric acid is evolved); 1 mol of caproyl chloride was then added and heated to  $125-130^{\circ}$  for 1 h (34 %) [2700].

-Also obtained by reaction of caproyl chloride with phenol in the presence of aluminium chloride,

*in methylene chloride for 1 h at  $0^\circ$ , then at r.t. overnight (74 %) [114] or for 14 h at r.t. (45 %) [1910];

*in nitrobenzene first at  $5-10^{\circ}$ , then at r.t. for some hours (81 %) [2970].

-Also obtained by reaction of caproic acid with phenol,

*in the presence of boron trifluoride for 3 h at 65–70° (72.9 %) [1938] or for 2 h at 70° (66 %) [1685];

*in the presence of polyphosphoric acid for 20 min on a boiling water bath (41 %) [2240] or for 10 min at 100° (40 %) [2238];

*in the presence of fused zinc chloride for few min (8 %) [726].

-Also obtained by Fries rearrangement of phenyl caproate,

*in the presence of polyphosphoric acid for 10 min at 100° (36 %) [2238];

*in the presence of aluminium chloride at 140° for 45 min (30 %) [932], in tetrachloroethane [3169] or in nitrobenzene [1116], (43 %) [2947];

*in the presence of aluminium chloride in refluxing carbon disulfide for 5 h. Then, the mixture was kept at  $80-90^{\circ}$  for 2 h and at  $140-150^{\circ}$  for 1 h after solvent elimination [1222].

-Also obtained [2478] by the method [2074].

-Also obtained by anaerobic co-metabolic oxidation of 4-hexylphenol by *Thauera* sp. strain R5 [2861].

-Also obtained by reaction of caproic acid with phenol in the presence of activated acid clay catalyst at  $190^{\circ}$  for 2 h [3277].

-Also refer to: [113, 414, 1415, 1536 (96 %), 2690, 2692, 2772, 2937, 2977, 3454].

b.p._{0.01} 160° [2970], b.p.₁ 167° [932, 1536], b.p.₁ 174–176° [2947], b.p._{2.9} 198° [1938], b.p.₁₀ 207–208° [2700], b.p.₁₅ 211–212° [1685], b.p.₁₅ 220–230° [2478]; white solid [1910]; m.p. 64.2–64.6° [1910], 64–65° [3277], 63–64° [726], 62° [2240], 61° [1536, 1685, 1938, 2700, 3169], 60° [932] (Sadtler standard N° 65673K), 59–60° [2478]; ¹H NMR [114, 1910] (Sadtler standard N° 38624M), ¹³C NMR [114, 1910], IR [1910] (Sadtler standard N° 65673K), UV [1995], MS [1910, 2861]; TLC [1910].

BIOLOGICAL ACTIVITY: Inhibition of  $17-\beta$  hydroxysteroid dehydrogenase 3 [1910]; Non-steroidal inhibitors of  $17-\beta$ -HSD in treatment of hormone-dependent cancers [1909]; Estrogenic activity [2692].

2,4-Dinitrop	henylhydrazor	ne (	$C_{18}H_{20}N_4O_5$	mol. wt. 372.38
m.p. 187°	[3169], 184° [9	932].		
isoNicotinyl	hydrazone	[101720-12-1	] $C_{18}H_{21}N_3O_2$	mol. wt. 311.38
m.p. 205°	[520, 521].			
USE: Chemo	otherapy of lepro	osy [520, 521]		
Semicarbaz	one	C ₁₃ H ₁₉	N ₃ O ₂	mol. wt. 249.31
m.p. 149°	[2240].			
Benzoate		C ₁₉ H ₂₀ O ₃	3	mol. wt. 296.37
m.p. 105.	5° [2700].			
Caproate		C ₁₈ H ₂₆ O	3	mol. wt. 290.40
•	reaction of cap t at 190° for 2 h		phenol in the presence	e of activated acid
m.p. 67–6	68° [3277].			
Sulfate	Refer to: [114]	] (73 %);	¹ H NMR [114],	¹³ C NMR [114].

## Methyl ether [6397-82-6] C₁₃H₁₈O₂ mol. wt. 206.28

-Obtained by Friedel-Crafts acylation of anisole with caproic anhydride,

- *in the presence of large molecular sizes on mesoporous silica catalyst at 453K for 4 h (73 %) [1466], [1480];
- *in the presence of microcrystalline beta zeolite-II for 6 h at  $130^{\circ}$  under argon (75 %) [1594];
- *in the presence of lithium perchlorate at  $60^{\circ}$  for 1.5 h (92 %) [267];
- *in the presence of SbCl₅-LiClO₄ mixture in refluxing methylene chloride for 30 min (92 %) [2176];
- *in the presence of  $(C_6H_5CN)_2PtCl_2$  (2.5 ml%) and AgSbF₆ (5 mol%) (77 %) [1046].
- -Also obtained by direct acylation of 4-bromoanisole with caproaldehyde by palladium catalysis (90 %) [2668].
- -Preparation: potassium carbonate and  $PdCl_2$  were added to a mixture of SDS and water and heated to  $60^{\circ}$  with stirring. Then 4-methoxyphenylboronic acid and caproic anhydride (or caproyl chloride) were added to the solution and the mixture held at  $60^{\circ}$  for 6 h (23–30 %) [3346].
- -Also obtained from hexanoic anhydride and (4-methoxyphenyl)boronic acid using tris(p-methoxyphenyl)phosphane as the ligand (91 %) [1139].
- -Also obtained by reaction of (4-methoxyphenyl)boronic acid with hexanoic anhydride in the presence of a Pd, Ni, Pd, or Cu catalyst [1137].
- -Also obtained by reaction of hexanoic acid with anisole,
- *over three large pore zeolites-beta (BEA), faujasite (FAU) and mordenite (MOR) [3246];
- *in the presence of montmorillonite-enwrapped titanium as a solid acid catalyst [937];
- *in the presence of  $Cs_{2.5}H_{0.5}PW_{12}O_{40}$  at 110° for 5 h (63 %) [1636];
- *in the presence of PPA at 90–100° for 1.5 h [2942] or for 1.5 h at 80° (80 %) [869]; *over HZSM-5 catalyst for 48 h at 423° K (20 %) [3265].
- -Obtained by reaction of dimethyl sulfate with 4-hexanoylphenol in the presence of aqueous sodium hydroxide for 4 h at reflux [2478].
- -Also obtained by direct  $\beta$ -alkylation of secondary alcohols with primary alcohols catalyzed by a CpIr complex (69 %) [1009, 1060].
- -Also obtained treatment of 1-(4-methoxyphenyl)-1-hexyne,
- *with p-toluenesulfonic acid (PTSA) in ethanol at 78° for 60 h (81 %) [2337];
- *with 2-aminophenol in the presence of palladium nitrate in dioxane at  $120^{\circ}$  for 3 h (71 %) [2867].
- -Also obtained by reaction of hexanoyl chloride with anisole in the presence of graphite in refluxing 1,2-dichloroethane for 8 h (66 %) [1723].
- -Also obtained by reaction of 4-methoxybenzyl alcohol with 1-pentene in the presence of RhCl₃.H₂O and 2-amino-4-methylpyridine at 130° for 12 h (77 %) [1556].
- -Also obtained by reaction of 1-pentene with 4-methoxybenzaldehyde in toluene at 150° for 24 h under a mixture of chlorotris(triphenylphosphine)rhodium (5 mol%) and 20 mol% of 2-amino-3-picoline (66 %) [1559].

-Also obtained by Friedel-Crafts acylation reaction of caproic anhydride with anisole, using the combined catalyst system of  $TiCl(OTf)_3$  and TfOH (98 %) [1483].

- -Also obtained by Friedel-Crafts acylation of anisole in the presence of a Lewis acid catalyst while using carboxylic acid or trisubstituted-silyl carboxylate as acylating agent and carrying out the reaction in the presence of p-trifluoromethylbenzoic acid anhydride and, if necessary, a silver salt [2997].
- -Also obtained by zeolite-catalyzed Friedel-Crafts reaction of anisole with caproic acid [3266].

-Also obtained by action of hexanoyl chloride with anisole,

*in the presence of ferric chloride at reflux for 5 h (33 %) [3435];

- *in the presence of aluminium chloride at reflux for 5 h (65 %) [3435].
- -Also refer to: [698, 842, 895 (49.3 %), 913, 1114, 1138, 1200, 1202, 1415, 1425, 1765, 1829, 1840, 1871, 1980, 2016, 2173 (67 %), 2180, 2240, 2406 (82 %), 2485, 2849, 3227 (quantitatif), 3262, 3421].

b.p._{1.6} 72–74° [1265], b.p._{0.25} 104° [3227], b.p._{0.4} 113–114° [2478],

b.p. $_5$  140–142° [3435], b.p. $_5$  141–142° [3479], b.p. $_{0.5}$  145° [869], b.p. $_8$  156° [2735],

b.p.₁₀ 165° [2240], b.p.₁₄ 172–174° [3178], b.p.₁₅ 183° [2478]; white solid [3346];

- m.p. 60° [1485], 41° [2735, 2901, 3479], 39–40° [1186, 1870], 38–39° [895], 38° [2240], 37–38° [869], 36–37° [2016], 34–35° [1978], 34° [2478];
- ¹H NMR [698, 1060, 1186, 1485, 1840, 1870, 1876, 1980, 2668, 2867, 2868, 3227, 3346], ¹³C NMR [698, 1060, 1249, 1485, 1801, 1840, 1876, 1978, 1980, 2668, 2999],

IR [698, 929, 1249, 1485, 1840, 1978, 1980, 2668, 2867, 2868, 2999],

UV [869, 1978], MS [698, 1186, 1485, 1870, 1978, 2668, 3346].

USE: Aquapalladium complex catalyst for intermolecular hydroamination of alkynes [2868].

## 2,4-Dinitrophenylhydrazone of the methyl ether

[102161-19-3]	$C_{19}H_{22}N_4O_5$	mol. wt. 386.41

m.p. 146–147° [869], 145–146° [2016], 141° [869, 3479].

## Phenylhydrazone of the methyl ether C₁₉H₂₄N₂O mol. wt. 296.41

m.p. 28° [2901].

Semicarbazone of the methyl ether [101777-85-9] C₁₄H₂₁N₃O₂ mol. wt. 263.34

m.p. 143° [895], 142.5° [3178], 128–129° [869].

Ethyl ether [35031-74-4] C₁₄H₂₀O₂ mol. wt. 220.31

-Obtained by reaction of hexanoyl chloride with phenetole, *in the presence of ferric chloride at reflux for 5 h (28 %) [3435]; *in the presence of aluminium chloride at reflux for 5 h (61 %) [3435]. -Also refer to: [1829].

oil [3435]; b.p.₂ 140–142° [3435], b.p.₇ 179–182° [1235]; m.p. 45° [1235]; IR [3435]; GLC [3435].

Phenyl ether[695196-65-7] $C_{18}H_{20}O_2$ mol. wt. 268.36-Obtained by treatment of diphenyl oxide with caproyl chloride under Friedel-<br/>Crafts conditions [516].

b.p.₁₅ 236–238° [516]; m.p. 49° [244], 39° [516].

**3-Chloropropyl ether**  $C_{15}H_{21}ClO_2$  mol. wt. 268.78

-Obtained by reaction of 1-bromo-3-chloropropane with 4-hydroxycaprophenone in the presence of potassium carbonate in refluxing 2-butanone (95 %) [969].

**4-Hexanoylphenyl ether** [791137-06-9] C₂₄H₃₀O₃ mol. wt. 366.49

-Obtained by reaction of hexanoyl chloride with diphenyl ether in the presence of aluminium chloride in refluxing carbon disulfide for 30 min (71 %) [463].

m.p. 104–105.5° [463]. ¹H NMR [460], ¹³C NMR [460], MS [460].

#### **Dioxime of the hexanoylphenyl ether** $C_{24}H_{32}N_2O_3$ mol. wt. 396.53

-Preparation: A suspension of the diketone, 100 % excess of hydroxylamine hydrochloride, and powdered anhydrous sodium carbonate in propanol [463].

m.p. 104–105.5° [721], 103–104.5° [463], 87–88° [463]. 87–88° and 103–104.5° (two forms) [463].

## **Dihydrazone of the hexanoylphenyl ether** C₂₄H₃₄N₄O mol. wt. 394.56

m.p. 103–104.5° [721].

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

[862666-34-0]  $C_{12}H_{16}O_3$  mol. wt. 208.26 OH Synthesis OU CO(CH₂)₄CH₃ -Obtained by deprotection of its dimethyl ether with boron tribromide in methylene chloride at 0°. Then, the mixture was stirred overnight at r.t. (87 %) [82].

brown solid [82]; m.p. 52° [82]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82].

USE: Reactant for preparation of lithium gallium/titanium ketone complexes [82].

**Dimethyl ether** [1854-73-5] C₁₄H₂₀O₃ mol. wt. 236.31

-Obtained by treatment of 1-(2,3-dimethoxyphenyl)-1-hexanol in acetone with potassium dichromate in dilute sulfuric acid (Jones reagent) at r.t. for 3 h (31 %) [82].

-Also obtained by treatment of 2,3-dimethoxyphenylpentyl-carbinol with potassium dichromate in dilute sulfuric acid at 30° (57 %) [3148].

colourless oil [82]; b.p.₂₀ 178–180° [3148]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82].

## 2,4-Dinitrophenylhydrazone of the dimethyl ether

[1854-72-4]

m.p. 125° [3148].

## 1-(2,4-Dihydroxyphenyl)-1-hexanone

[3144-54-5]	$C_{12}H_{16}O_3$			mol	. wt. 2	08.26
OH CO(CH ₂ ) ₄ CH ₃	Syntheses -Preparation by	reaction	of	caproic	acid	with

HO

-Preparation by reaction of caproic acid with resorcinol, *in the presence of boron trifluoride at 70° for 2 h

(90 %) [2312] or for 2–3 h between 65 and 85° [503];

*in the presence of zinc chloride (68–78 %) [2501];

*in the presence of polyphosphoric acid at 90° for 30 min [2907].

-Also obtained by reaction of capronitrile with resorcinol (Hoesch reaction) (27 %) [1371].

-Also obtained by reaction of ethyl caproate with resorcinol in the presence of zinc chloride, the mixture was heated for 1 h at its boiling point (60 %) [3160].

-Also obtained by reaction of hexanoyl chloride with resorcinol at  $85-90^{\circ}$  for 20-30 min (87.5 %) [731]. The same result was obtained in the presence of zinc chloride.

 $C_{20}H_{24}N_4O_6$ 

mol. wt. 416.43

-Also obtained by reaction of hexanoyl chloride with resorcinol in the presence of aluminium chloride in 1,2-dichloroethane for 5 h at 65° [284].

-Identification into catalytic hydrothermal treatment of wood biomass at 280° for 15 min in the presence of alkaline solution (NaOH, Na₂CO₃, KOH, K₂CO₃) [1601]. -Also obtained by reduction of 2,4-dihydroxysorbophenone [2907].

-Also refer to: [126, 129, 229 (93 %), 893, 1079, 1239, 1562, 1598, 1599, 1657, 1673, 1798, 1892, 2114, 2498, 2637, 2640, 2692, 2769, 2787, 2841, 2842, 2910, 3010, 3051, 3077, 3099, 3168, 3208, 3449].

white crystals [3160]; colourless needles [229]; b.p._{0.3} 144–146° [229], b.p.₃ 170–175° [1562], b.p.₃ 184–185° [1448], b.p.₆₋₇ 196–197° [2841, 2842, 3099], b.p.₉ 205–208° [1448], b.p.₁₀ 200–210° [2907], b.p.₁₅ 210–220° [731], b.p.₁₄ 217–218° [3160], b.p. 343–345° (d) [3160]; m.p. 58° [503], 56–57° [1562, 3160], 56° [229, 2312], 55–56° [1448], 55° [731], 54.5–56° [893, 1673, 2842]; ¹H NMR [1239], ¹³C NMR [1239]; GC-MS [1601].

BIOLOGICAL ACTIVITY: Nematocide: 4-Hexanoylresorcinol at 100 ppm controlled *Pratylenchus penetrans* [1798]; Responses of *Phytophthora sojae* zoospores to isoflavones and analogs [3161]; Inhibition of polyphenol oxidase (**PPO**) [2030]; Binary classification models for endocrine disrupter effects mediated through the estrogen receptor [2640]; Effect of  $K_2CO_3$  concn. and biomass/water ratio on products distribution in hydrothermal upgrading of biomass [1597]; Effect of RbOH and CsOH on product distribution in catalytic hydrothermal treatment of pine wood biomass [1600]; Antiseptic and germicidal product [2734]; Differentiation-inducing factor 1 (DIF-1) as protease-inhibitory and antiviral compound [129]; Reproductive effects (germination) [126, 238]; Picornavirus activity inhibitor [126]; Estrogenic [2769]; Antiviral [126]; Antifungal [2114, 2769]; Fungicides for hydocarbon lubricants and hydraulic fluids [2498] and for lubricants for optical instruments [2637]; Cytotoxicity [126].

USE: In detn. of uranium by spectrophotometry [2891]; Wine preservation by, [3168].

**2,4-Dinitrophenylhydrazone** [109252-18-8] C₁₈H₂₀N₄O₆ mol. wt. 388.38

-Refer to: [2907].

m.p. 232-235° [2907].

## Diacetate

#### $C_{16}H_{20}O_5$

mol. wt. 292.33

-Obtained by boiling the 2,4-dihydroxycaprophenone with sodium acetate and acetic anhydride for several hours (poor yield) [3160].

thick yellow oil [3160]; b.p.₁₃ 229–232° [3160].

#### **Dimethyl ether** [312488-55-4] C₁₄H₂₀O₃ mol. wt. 236.31

-Preparation by reaction of n-caproyl chloride with 1,3-dimethoxybenzene,

*in the presence of aluminium chloride in methylene chloride at  $0-5^{\circ}$  for 2.5 h (90 %) [2188];

*in the presence of HY-zeolite. Before reaction, the zeolites were activated at  $500^{\circ}$  for 5 h in nitrogen flow [352].

-Also obtained (by-product) by reaction of dimethyl sulfate with 2,4-dihydroxycaprophenone in the presence of 20 % sodium hydroxide solution [3160].

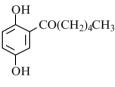
white crystals [3160]; m.p. 36° [2188], 35–36° [3160]; ¹H NMR [2188], IR [2188], MS [2188].

## 1-(2,5-Dihydroxyphenyl)-1-hexanone

[4693-18-9]

 $C_{12}H_{16}O_3$ 

mol. wt. 208.26



Syntheses

 $CO(CH_2)_4CH_3$  -Preparation by reaction of caproic acid with hydroquinone [2102] in the presence of boron trifluoride at 125° for 2 h in a sealed tube (94 %) [2312] or heated for 2 h at 125° [2063].

-Also obtained by reaction of caproyl chloride with hydroquinone in the presence of aluminium chloride in nitrobenzene heated on a water bath for 3 h [1442].

-Also obtained by Fries rearrangement of hydroquinone dicaproate with aluminium chloride for 5 h at 150–160° [1442].

-Also obtained by treatment of its dimethyl ether with hydrobromic acid in refluxing acetic acid for 6 h [1442].

-Also refer to: [1481, 2063, 2102, 3168, 3204].

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m.p. 85–86° [2312], 82–83° [2102], 82° [3204], 81–83° [2063], 81–82° [1442].
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USE: Wine preservation by, [3168].

**Dimethyl ether** [430425-41-5] C₁₄H₂₀O₃ mol. wt. 236.31

-Obtained by reaction of caproyl chloride with hydroquinone dimethyl ether,

*in the presence of aluminium chloride in nitrobenzene, first at r.t. overnight, then the mixture heated on a water bath for 3 h [1442];

*in the presence of HY-zeolite. Before reaction, the zeolites were activated at 500° for 5 h in nitrogen flow [352].

-Also refer to: [1919].

 $b.p._{0.5} \ 132-134^\circ \ [1919], \ b.p._{15} \ 175-180^\circ \ [1442]; \quad m.p. \ 15^\circ \ [1919].$ 

#### 1-(2,6-Dihydroxyphenyl)-1-hexanone

(2-Caproylresorcinol)

OH  $CO(CH_2)_4CH_3$  Syntheses Obtained by Fries rearrangement of 1,3-dihydroxyphenylcaproate (b.p.₁₁ 166–175°) with aluminium chloride innitrobenzene at 60° for 4 h (40 %) [1877].

-Also refer to: [2629, 2672].

m.p. 74° [2672], 142–143° [1877]. **N.B.**: One of the reported melting point is obviously wrong.

Dimethyl ether	[5673-08-5]	$C_{14}H_{20}O_3$	mol. wt. 236.31
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-Obtained by reaction of 2,6-dimethoxybenzoyl chloride with dipentylcadmium (51 %) [2629].

-Also refer to: [2672].

oil [2629]; b.p.₂ 142° [2672], b.p.₁₄ 182–184° [2629]; ¹H NMR [2629], IR [2629].

#### 1-(3,4-Dihydroxyphenyl)-1-hexanone

[4009-78-3]  $C_{12}H_{16}O_3$  mol. wt. 208.26 OH Syntheses -Obtained by reaction of caproic acid with pyrocatechol, *in the presence of zinc chloride at reflux [726]; *in the presence of boron trifluoride for 2.5 h at 150° (55 %) [2312].

-Also obtained by Fries rearrangement of pyrocatechol dicaproate with aluminium chloride in the presence of pyrocatechol at  $135-140^{\circ}$  for 4.5 h (72 %) [2075] or for 5 h (30 %) [283].

-Also obtained by treatment of guaiacol caproate in carbon disulfide at  $90^{\circ}$  for 50 min, then at 135–140° for 2 h after solvent elimination (30–47 %) [2075].

-Also obtained by reaction of guaiacol caproate with aluminium chloride in nitrobenzene [726].

-Also refer to: [1770, 2075, 2508].

b.p.₄ 212–220° [2075], b.p.₁₅ 240–250° [726]; m.p. 94° [2312], 93.8° [2075], 93–94° [726, 2955], 91–93° [283]; ¹H NMR [283], UV [985]; paper chromatography [2508].

BIOLOGICAL ACTIVITY: Protective agent against the lethal effects of X-rays [1809].

#### **Dimethyl ether** [52375-87-8] C₁₄H₂₀O₃ mol. wt. 236.31

-Obtained by Friedel-Crafts acylation reaction of caproic anhydride with veratrole,

*in the presence of a catalyst generated from gallium trichloride and a silver salt (AgClO₄ or AgSbF₆) (high yields) [2175];

*using the combined catalyst system of TiCl(OTf)₃ and TfOH (94 %) [1483];

- *in the presence of SbCl₅-LiClO₄ mixture in refluxing methylene chloride for 30 min (87 %) [2176].
- -Also obtained by reaction of caproyl chloride with veratrole,
- *in the presence of zinc chloride in refluxing carbon disulfide for 4 h (49 %) [1565];
- *in the presence of HY-zeolite. Before reaction, the zeolites were activated at 500° for 5 h in nitrogen flow [352].
- -Also obtained by Friedel-Crafts acylation of veratrole in the presence of a Lewis acid catalyst while using carboxylic acid or trisubstituted-silyl carboxylate as acylating agent and carrying out the reaction in the presence of p-trifluoromethylbenzoic acid anhydride and, if necessary, a silver salt [2997].

-New inhibitor of the CoQ-dependent redox reactions in mitochondria and chromatophores [1729].

-Also refer to: [1112, 1202, 1249, 2172, 2174, 2999, 3056, 3364].

viscous liquid [1565]; b.p.₁ 150° [1565], b.p.₁₇ 176–178° [3056]; ¹H NMR [1249, 2999], ¹³C NMR [1249, 2999], IR [1249, 2999, 3364].

colourless needles [1565]; m.p. 178° [1565].

## 1-(3,5-Dihydroxyphenyl)-1-hexanone

[105401-56-7]	$C_{12}H_{16}O_3$		mol. wt. 208.26
HO CO(CH ₂ ) ₄ CH ₃	Synthesis -Obtained by treatm sodium hydroxide a		
m.p. 106° [1406].			
2,4-Dinitrophenylhydrazor	ne [109248-78-4]	$C_{18}H_{20}N_4O_6$	mol. wt. 388.38

m.p. 222° (d) [1406].

Diacetate	[101430-19-7]	$C_{16}H_{20}O_5$	mol. wt. 292.33

-Preparation by reaction of dipentylcadmium with 3,5-diacetoxybenzoyl chloride in refluxing benzene for 1 h (77 %) [1406].

b.p._{0.8} 189–192° [1406].

#### 2,4-Dinitrophenylhydrazone of the diacetate

[102810-66-2]	$C_{22}H_{24}N_4C$	<b>)</b> ₈	mol. wt. 472.45
m.p. 152° [1406].			
Dimethyl ether	[41497-32-9]	$C_{14}H_{20}O_{3}$	mol. wt. 236.31

-Preparation by reaction of pentylmagnesium bromide with 3,5-dimethoxybenzamide in refluxing ethyl ether for 5 h (88 %) [2990].

-Also obtained by reaction of pentylmagnesium bromide with 3,5-dimethoxybenzonitrile in refluxing THF for 3 h, followed by heating with 6 N HCl at reflux (65 %) [2209].

-Also refer to: [238, 543, 2899].

white solid [2209]; b.p.₇ 175–176° [2990]; m.p. 53° [2990], 49–51° [543]; ¹H NMR [543, 2209], ¹³C NMR [2209], IR [2209], UV [543], MS [2209].

C12H16O4

## 1-(2,3,4-Trihydroxyphenyl)-1-hexanone

(4-Hexanoylpyrogallol)

[43043-26-1]

Syntheses

mol. wt. 224.26

HO HO HO

-Obtained by reaction of caproic acid with pyrogallol in the presence of zinc chloride (Nencki reaction), *at 150° for 45 min (41 %) [1643]; *at 140–145° for 4 h (70 %) [506];

*at 135–140° for 2 h (50 %) [1283]; *at 130–140° for 1.5 h (32 %) [1260]. -Also refer to: [1643, 3168].

white needles [1260]; m.p. 86.5–87° [1260], 86–87° [506], 84° [1643], 72–74° [1283].

BIOLOGICAL ACTIVITY: Protection against whole-body irradiation [1810].

USE: Wine preservation by, [3168].

#### 1-(2,4,5-Trihydroxyphenyl)-1-hexanone

 $[105476-10-6] C_{12}H_{16}O_4$ mol. wt. 224.26 OH CO(CH₂)₄CH₃ -Obtained by reaction of caproyl chloride with 1,2,4-trihydroxybenzene in the presence of aluminium chloride in nitrobenzene [292]. -Also refer to: [291, 771, 1708].

m.p. 113-114° [291], 108-111° [292].

USE: Antioxidant [1708]; Antioxidant for fats, oils and paraffin waxes [292]; Toxicity [1708].

-Obtained by reaction of hexanoyl chloride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride (60–74 %) [771].

-Also obtained by reaction of hexanoic anhydride with 1,2,4-trimethoxybenzene in the presence of aluminium chloride in methylene chloride at  $60^{\circ}$  for 1 h (65 %) [772].

-Also obtained by reaction of caproic acid with 1,2,4-trimethoxybenzene in the presence of polyphosphoric acid for 7 h at  $45-50^{\circ}$  [2695].

white powder [772]; m.p. 82–84° [772, 2695]; ¹H NMR [772, 2695], ¹³C NMR [772], IR [772, 2695], MS [772, 2695].

### 1-(2,4,6-Trihydroxyphenyl)-1-hexanone

(Phlorocaprophenone) (THPH)

OH

HO

[5665-89-4]	$C_{12}H_{16}O_4$		mol. wt. 224.26
OH CO(CH ₂ ) ₄ CH ₃	Syntheses -Obtained by reaction phloroglucinol,	of caproyl	chloride with

*in the presence of aluminium chloride;

*in nitrobenzene (60–70 %) [421], (63 %) [2646], (51 %) [2618];

*in nitrobenzene and carbon disulfide mixture (61 %) [2113], (51 %) [2620];

*in methylene chloride at r.t. [1129];

*in the presence of boron trifluoride etherate, first at  $0^{\circ}$ , then at r.t. for 48 h under nitrogen [2786].

-Also obtained by reaction of hexanoic acid with phloroglucinol in the presence of boron trifluoride etherate [3019].

-Also obtained by reaction of caproyl nitrile with phloroglucinol (Hoesch reaction) [1376, 1699].

-Also obtained by acid hydrolysis of its 2,4-di-β-D-glucopyranoside [1089].

-Also obtained by adding phloroglucinol to a solution of phosphorous oxychloride plus aluminium chloride and stirred under nitrogen. Hexanoic acid was added and the reaction stirred under nitrogen at  $0^{\circ}$  for 8 h, then at  $6^{\circ}$  for 40 h (40–54 %) [3201].

-Also obtained by treatment of its trimethyl ether with boron tribromide in chloroform first at  $-18^{\circ}$ , then at reflux for 3 h [2764].

-Also refer to: [74, 129, 153, 180, 1026, 1038, 1065, 1439, 1776, 1942, 2012, 2771, 3168, 3202].

Isolation from natural sources

-Of *Populus tritis* bud exudate [955].

-In bud exudate of *Populus koreana*, *Populus maximowiczii* and *Populus suaveolens* (Salicaceae) [1164].

yellow amorphous solid [1129]; pale beige needles [2764]; m.p. 120–121° [421, 1376, 1439], 120° [2113, 2646], 118° [1699, 2618, 2620], 108–110° [2764]; ¹H NMR [421, 2764, 3019], ¹³C NMR [3019], IR [421, 2764, 3019], UV [2764, 3019], MS [421, 1129]; GC-MS [1164].

USE: Assembly and photodimerization of bis(pyridyl)ethylene with phloroglucinols [1038]; Wine preservation by, [3168].

BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773, 1776]; Antagonist both thromboxane  $A_2$  and Leukotriene  $D_4$  [3019]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129]; Antifungal [2113]; Differentiation-inducing factor 1 (DIF-1) as protease-inhibitory and antiviral compound [129]; Effect on gibberellin-inducible  $\alpha$ -amylase synthesis in barley aleurone cells [153].

#### Hydrate

 $C_{12}H_{16}O_4, H_2O$ 

mol. wt. 242.28

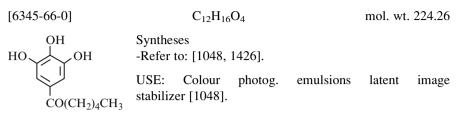
m.p. 95–96° [1376], 95° [1699].

Trimethyl ether	[86360-63-6]	$C_{15}H_{22}O_4$	mol. wt. 266.34

-Refer to: [1065, 2764].

pale yellow oil [2764]; ¹H NMR [2764], IR [2764], UV [2764].

#### 1-(3,4,5-Trihydroxyphenyl)-1-hexanone



**Trimethyl ether** [170489-32-4] C₁₅H₂₂O₄ mol. wt. 266.34

-Refer to: [1425, 1426].

¹H NMR [1426], MS [1426].

### 1-(2,3,4,6-Tetrahydroxyphenyl)-1-hexanone

	$C_{12}H_{16}O_5$	mol. wt. 240.26
HO CO(CH ₂ ) ₄ CH ₃	Synthesis -Refer to: [1353].	
ностран	Tetramethyl ether $C_{16}H_{24}O_5$	mol. wt. 296.36

-Refer to: [1353].

#### 1-(4-Hydroxyphenyl)-4-methyl-1,3,5-hexanetrione

	$C_{13}H_{14}O_4$	mol. wt. 234.25
OH	Synthesis -Refer to: [1168]. <b>Methyl ether</b> [92120-37-1]	
COCH ₂ CO-CH-COCH ₃	$C_{14}H_{16}O_4$	mol. wt. 248.28

-Preparation: Trityl-lithium in THF was injected into 3-methylpentane-2,4-dione under nitrogen at  $-15^{\circ}$ . When the red colour persisted methyl p-methoxybenzoate in THF was injected. The solution was stirred at  $-15^{\circ}$  and trityl-lithium solution added to just maintain the red colour [1168].

yellow crystals [1168]; m.p. 56–57° [1168]; ¹H NMR [1168], IR [1168], UV [1168], MS [1168].

#### 1-(2-Hydroxyphenyl)-2-methylene-1-hexanone

[935277-51-3]	$C_{13}H_{16}O_2$	mol. wt. 204.27
OH CO-C-(CH ₂ ) ₃ CH ₃ II CH ₂	Synthesis -Obtained by stirring a solution of 1-hexyne, RhCl(PPh ₃ ) ₃ , acetonitrit tate in methylene chloride at r.t. argon atmosphere (45 %) [1434].	le and sodium ace-

 $C_{14}H_{18}O_2$ 

¹H NMR [1434], IR [1434].

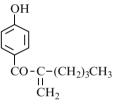
Methyl ether

mol. wt. 218.30

¹H NMR [2881], ¹³C NMR [2881], IR [2881], MS [2881].

## 1-(4-Hydroxyphenyl)-2-methylene-1-hexanone

 $C_{13}H_{16}O_2$ mol. wt. 204.27



[80856-36-6]

	Synthesis
	-Refer to: [1560].
	Methyl ether [473835-69-7]
	$C_{14}H_{18}O_2$ mol. wt. 218.30
3	-Obtained by efficient and selective hydroacylation of
	1-hexyne with p-methoxybenzaldehyde by a chelation-
	assisted catalytic system (76 %) [1560].

### 1-(2-Hydroxyphenyl)-4-methyl-1,3-hexanedione

C₁₃H₁₆O₃ mol. wt. 220.27

Synthesis COCH₂CO-CH-C₂H₅ CH₃
-Obtained by reaction of 2-hydroxyacetophenone with methyl 2-methylbutyrate in the presence of sodium hydride (61.8 %) [2807].

b.p._{0.7} 114–120° [2807]; ¹H NMR [2807], IR [2807], UV [2807].

#### 1-(4-Hydroxyphenyl)-5-methyl-1-hexanone

[161582-00-9]	$C_{13}H_{18}O_2$	mol. wt. 206.28
OH	Syntheses -Refer to: [38, 39]. <b>Methyl ether</b> [92300-78-2]	
CO(CH ₂ ) ₃ CH(CH ₃ ) ₂	$C_{14}H_{20}O_2$	mol. wt. 220.31

-Preparation by reaction of 5-methylhexanoyl chloride with anisole in the presence of aluminium chloride in carbon disulfide under cooling for 70 min, then at r.t. (91 %) [2237].

-Also refer to: [2236].

b.p.₁ 131–133° [2236, 2237].

Semicarbazone of the methyl ether [93762-01-7] C₁₅H₂₃N₃O₂ mol. wt. 277.37

m.p. 124–125° [2237].

### 1-(2,4-Dihydroxyphenyl)-2-methyl-1-hexanone

[406174-64-9]	$C_{13}H_{18}O_3$	mol. wt. 222.28
HO HCO-CH-C	Syntheses -Obtained by reaction of 2- resorcinol in the presence 150° (52 %) [2829]. -Also obtained by enz deacetylation of its 2,4-dia	of zinc chloride for 2 h at zymatic enantioselective
oil [2829]; ¹ H NMR [2829], ¹³ MS [2829]; TLC	C NMR [2829], IR [2829], UV [2 [2829].	829],
Diagotatag	СИО	mal wet 206.26

#### Diacetates

 $C_{17}H_{22}O_5$ 

mol. wt. 306.36

-Refer to: [2829].

*racemic [406174-68-3].

-Obtained by reaction of acetic anhydride with 1-(2,4-dihydroxyphenyl)-2-methyl-1-hexanone in the presence of catalytic amount of N,N-dimethylaminopyridine at 22–25° (90 %) [2829].

oil [2829]; ¹H NMR [2829], ¹³C NMR [2829], IR [2829], UV [2829], MS [2829]; TLC [2829]. *levogyre (-) [406174-80-9].

-Obtained by chemical acetylation of monoacetate [2829].

 $(\alpha)_{\rm D}^{25} = -41.5^{\circ}$  (chloroform) [2829].

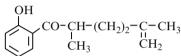
*dextrogyre (+) [406174-76-3].

-Obtained by treatment of racemic ketone with PPL pre-incubated in THF at 40-42° for 10 h in the presence of butanol (60 %) [2829].

 $(\alpha)_{\rm D}^{25} = +37.9^{\circ}$  (chloroform) [2829].

## 1-(2-Hydroxyphenyl)-2-methyl-5-methylene-1-hexanone

$$C_{14}H_{18}O_2$$
 mol. wt. 218.30



Synthesis  $\begin{array}{c} \text{Synthesis} \\ \text{CO-CH-}(\text{CH}_2)_2 - \text{C-CH}_3 \\ \text{CH}_3 \\ \text{CH}_2 \\$ 1,5-hexadiene (6 equiv.) in the presence of RhCl(PPh₃)₃ (0.2 equiv.) in methylene chloride for 24 h at r.t. (95 %) [1435].

¹H NMR [1435].

#### 1-(3-Hydroxyphenyl)-2,2-dimethyl-1-hexanone

[104325-46-4]	$C_{14}H_{20}O_2$		mol. wt. 220.31
OH CH3 CO-C-C4H9 CH3	Synthesis -Refer to: [2197]. <b>Phenoxymethyl ether</b> $C_{21}H_{26}O_3$ -Refer to: [729, 2197].	[104341-05-1]	mol. wt. 326.44

BIOLOGICAL ACTIVITY: Antiallergic and inflammation inhibitor [2197].

Benzyl ether	[103119-42-2]	$C_{21}H_{26}O_2$	mol. wt. 310.44

-Refer to: [583, 729, 2197].

BIOLOGICAL ACTIVITY: Lipoxygenase inhibition by, [583].

#### 2-Ethyl-1-(4-hydroxyphenyl)-1-hexanone

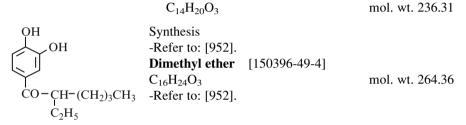
1-(4-hydroxyphenyl)-2-ethylpentyl ketone

$$C_{14}H_{20}O_2$$
 mol. wt. 220.31

Synthesis

-Obtained by Fries rearrangement of phenyl 2-ethylcaproate with aluminium chloride in nitrobenzene for 48 h at  $40^{\circ}$  under an argon atmosphere [1116].

#### 1-(3,4-Dihydroxyphenyl)-2-ethyl-1-hexanone



USE: Starting materials in synthesis of calixbisethylhexyl benzocrown [952].

#### 1-(2,4-Dihydroxyphenyl)-2-ethyl-1-hexanone

b.p.₈ 186° [1806, 1807].

BIOLOGICAL ACTIVITY: Antifungal activity against *Dreschlera oryzae*, *Macrophomina phaseolina*, *Fusarium solani* and *Pythium aphanidermatum* [2702].

**Diacetate** [251463-55-5]  $C_{18}H_{24}O_5$  mol. wt. 320.39

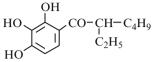
-Obtained by reaction of acetic anhydride with the title ketone in the presence of pyridine (>80 %) [2517].

viscous oil [2517]; ¹H NMR [2517], ¹³C NMR [2517], IR [2517], UV [2517], MS [2517].

 $\bigcup_{\substack{CO-CH-C_4H_9\\C_2H_5}}^{OH}$ 

#### 1-(2,3,4-Trihydroxyphenyl)-2-ethyl-1-hexanone

$$C_{14}H_{20}O_4$$
 mol. wt. 252.31



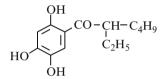
Synthesis  $\begin{array}{ccc} \text{CO-CH-C}_4\text{H}_9 \\ \text{C}_2\text{H}_5 \end{array} \begin{array}{c} \text{-Obtained by reaction of 2-ethylcaproic acid with} \\ \text{pyrogallol in the presence of strongly acidic ion} \\ \text{exchanger Amberlyst-15 at } 120^\circ \text{ for } 24 \text{ h} \end{array}$ (56 %) [231].

¹H NMR [231]. ¹³C NMR [231].

#### 1-(2,4,5-Trihydroxyphenyl)-2-ethyl-1-hexanone

[100864-42-4]

$$C_{14}H_{20}O_4$$
 mol. wt. 252.31



Synthesis -Obtained by reaction of 2-ethylcaproyl chloride with 1,2,4-trihydroxybenzene in the presence of aluminium chloride in nitrobenzene [292] at r.t. for several hrs, and heated 0.5 h at  $65^{\circ}$  [290].

m.p. 94–97° [290, 292].

USE: Antioxidant in fats and oils [290]; Antioxidant for fats, oils and paraffin waxes [292].

#### 1-(2-Hvdroxyphenyl)-3,5,5-trimethyl-1-hexanone

-Refer to: [110, 417].

¹H NMR [628]; TLC [628].

Oxime	[50652-75-0]	$C_{15}H_{23}NO_2$	mol. wt. 249.35

USE: In extn. of copper and nickel from sulfate solns. [110].

## 1.2 Substituted Hydroxyketones

## 1-(3-Bromo-5-fluoro-2-hydroxyphenyl)-1-hexanone

**2,4-Dinitrophenylhydrazone** [1841-68-5]  $C_{18}H_{18}BrFN_4O_5$  mol. wt. 469.27

## m.p. 147 $^{\circ}$ [1550].

## 1-(3-Bromo-5-fluoro-4-hydroxyphenyl)-1-hexanone

[329-44-2] C₁₂H₁₄BrFO₂ mol. wt. 289.14 OH Synthesis

$$F \xrightarrow{OH} Br$$

$$CO(CH_2)_4CH_3$$

Synthesis -Obtained by reaction of bromine with 3-fluoro-4-hydroxyhexanophenone in acetic acid [516]. m.p. 95° [516].

## 1-(3,5-Dibromo-4-hydroxyphenyl)-1-hexanone

[20683-49-2]	$C_{12}H_{14}Br_2O_2$	mol. wt. 350.05
Br Br CO(CH ₂ ) ₄ CH ₃	Syntheses -Obtained by reaction of bromine caprophenone in aqueous acetic acid [5 -Also refer to: [433]. m.p. 68° [516].	

## 1-(3,5-Dibromo-2,4-dihydroxyphenyl)-1-hexanone

[238074-76-5]	$C_{12}H_{14}Br_2O_3$	mol. wt. 366.05
Br HO Br	Syntheses -Obtained by reaction of bron caprophenone in acetic acid for a short time (33 %) [316 -Also refer to: [998].	first at r.t., then at $40-50^{\circ}$

pale yellow needles [3160]; m.p. 102–103° [3160].

#### 1-(3-Chloro-5-fluoro-2-hydroxyphenyl)-1-hexanone

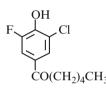
[1644-57-1]	$C_{12}H_{14}CIFO_2$	mol. wt. 244.69
$\begin{array}{c} OH\\CI\\F\\F\end{array} CO(CH_2)_4CH_3\\F\end{array}$	Synthesis -Obtained by Fries rearrangement phenyl caproate with aluminium for 3 h (62 %) [1550]. b.p. ₄ 130° [1550].	

**2,4-Dinitrophenylhydrazone** [1957-52-4] C₁₈H₁₈ClFN₄O₅ mol. wt. 424.82

m.p. 150° [1550].

#### 1-(3-Chloro-5-fluoro-4-hydroxyphenyl)-1-hexanone

C₁₂H₁₄ClFO₂ mol. wt. 244.69



Synthesis Cl -Refer to: [1404]. Methyl ether [371757-70-9]  $C_{13}H_{16}CIFO_2$ mol. wt. 258.72 -Obtained by reaction of caproyl chloride with 2-chloro-6-fluoroanisole in the presence of aluminium chloride in methylene chloride [1404].

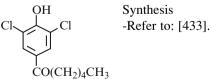
-Also refer to: [2407].

## 1-(3,5-Dichloro-2-hydroxyphenyl)-1-hexanone

[3226-18-4]  $C_{12}H_{14}Cl_2O_2$ mol. wt. 261.15 OH  $CO(CH_2)_4CH_3$  Syntheses -Obtained by Fries rearrangement of 2,4-dichlorophenyl caproate with aluminium chloride [3170]. -Also refer to: [433] m.p. 59.5° [3170]; UV [3170].

## 1-(3,5-Dichloro-4-hydroxyphenyl)-1-hexanone

[131427-28-6] 
$$C_{12}H_{14}Cl_2O_2$$
 mol. wt. 261.15  
OH Synthesis



621

mol. wt. 293.15

#### 1-(4,6-Dichloro-2-hydroxyphenyl)-1-hexanone

#### 1-(3,5-Dichloro-2,6-dihydroxyphenyl)-1-hexanone

 $\begin{array}{c} C_{12}H_{14}Cl_{2}O_{3} & \mbox{mol. wt. } 277.15 \\ Synthesis \\ CO(CH_{2})_{4}CH_{3} & -Refer to: [1261]. \\ b.p. \ 180-185^{\circ} \ [1261]; \ m.p. \ 119-120^{\circ} \ [1261]. \end{array}$ 

# **1-(3,5-Dichloro-2,4,6-trihydroxyphenyl)-1-hexanone** (*TH-DIF-1*)

[118222-71-2]

HC

CO(CH₂)₄CH₃ -Obtained by reaction of sulfuryl chloride (2 equiv.) With 2,4,6-trihydroxycaprophenone in a chloroform/ ethanol mixture at r.t. [1129]. -Also refer to: [129, 1772, 2012].

yellow amorphous solid [1129]; MS [1129].

BIOLOGICAL ACTIVITY: Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129]; Differentiation-inducing factor 1 (DIF-1) as protease-inhibitory and antiviral compound [129].

C12H14Cl2O4

**Trimethyl ether** [861889-69-2]  $C_{15}H_{20}Cl_2O_4$  mol. wt. 335.23 (*TM-DIF-1*)

-Obtained by reaction of methyl p-toluenesulfonate with the titled ketone in the presence of potassium carbonate in acetone at r.t. for 16 h (95 %) [1129]. -Also refer to: [1772].

colourless oil [1129]; ¹H NMR [1129], ¹³C NMR [1129], MS [1129].

BIOLOGICAL ACTIVITY: Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

## 1-(3-Fluoro-4-hydroxy-5-iodophenyl)-1-hexanone

[350-16-3]	$C_{12}H_{14}FIO_2$	mol. wt. 336.14
$I$ $F$ $CO(CH_2)_4CH_3$	Synthesis -Obtained by reaction of iodine caprophenone in ethanol in the pro- oxide [519]. lustrous colourless leaflets [519]; m.p. 102° [519].	

#### 1-(4-Hydroxy-3,5-diiodophenyl)-1-hexanone

 $C_{12}H_{14}I_2O_2$  mol. wt. 444.05



Synthesis -Obtained by reaction of iodine with 4-hydroxycaprophenone in ethanol in the presence of yellow mercuric oxide [516].

 $CO(CH_2)_4CH_3$  colourless prisms [516]; m.p. 69° [516].

#### 1-(5-Bromo-2-hydroxyphenyl)-1-hexanone

[103797-90-6] C₁₂H₁₅BrO₂ mol. wt. 271.15 OH Syntheses

CO(CH₂)₄CH₃ -Preparation by Fries rearrangement of 4-bromophenyl caproate in the presence of aluminium chloride [2797], (69 %) [1640],

*in tetrachloroethane at 120° for 30 min (86 %) [2026]; *without solvent for 30 min at 150–160° [1701].

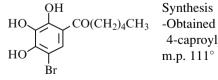
plates [2026]; b.p.₁ 145–150° [2026], b.p._{0.5} 150–155° [1640]; m.p. 60.5° [1701], 58–59° [2026]; IR [1640].

#### **2,4-Dinitrophenylhydrazone** [101728-14-7] C₁₈H₁₉BrN₄O₅ mol. wt. 451.28

m.p. 206° [2026].

#### 1-(5-Bromo-2,3,4-trihydroxyphenyl)-1-hexanone

C₁₂H₁₅BrO₄ mol. wt. 303.15



Synthesis -Obtained by reaction of bromine with 4-caproylpyrogallol in acetic acid [506]. m.p. 111° [506].

#### 1-(3-Chloro-2-hydroxyphenyl)-1-hexanone

b.p.₁ 135–137° [3170]; m.p. 78–80° [3013], 56–57° [1673]. **N.B.**: One of the reported melting point is obviously wrong. UV [3170]; GC [3012, 3013]; polarity of [3013].

**Oxime** [101002-14-6] C₁₂H₁₆ClNO₂ mol. wt. 241.72 -Refer to: [2742, 3077].

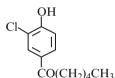
BIOLOGICAL ACTIVITY: As lipoxygenase inhibitor [3077].

## 1-(3-Chloro-4-hydroxyphenyl)-1-hexanone

[3226-35-5]

$$C_{12}H_{15}ClO_2$$

mol. wt. 226.70



Syntheses -Obtained by Fries rearrangement of 2-chlorophenyl caproate with aluminium chloride [3170]. -Also refer to: [381].

m.p. 79–81° [381], 77° [3170]; UV [3170].

## 1-(4-Chloro-2-hydroxyphenyl)-1-hexanone

[50444-92-3]	C ₁₂ H ₁₅ ClO ₂	mol. wt. 226.70
CO(CH ₂ ) ₄ CH ₃	Syntheses -Preparation by Fries rearrangement caproate with aluminium chloride, *without solvent at 130° for 2 h (74 *in nitrobenzene at 25° for 6 h (92 9	%) [2802];

-Also refer to: [2428–2431].

b.p.₃ 150° [2802].

USE: Reaction of, with thionyl chloride in the presence of aluminium chloride, [2430].

**Phenylhydrazone** [77132-64-0]  $C_{18}H_{21}CIN_2O$  mol. wt. 316.83

m.p. 81–82° [2429].

USE: Cyclization of, indole deriv. from, [2429].

2,4-Dinitrophenylhydrazone	$C_{18}H_{19}CIN_4O_5$	mol. wt. 406.80
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m.p. 207° [2802].

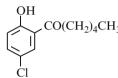
#### Methyl ether C13H17ClO2 mol. wt. 240.73

-Obtained by methylation of the above ketone in the usual way (88 %) [2802].

b.p.33 135° [2802].

#### 1-(5-Chloro-2-hydroxyphenyl)-1-hexanone

C12H15ClO2 [3226-16-2] mol. wt. 226.70



Syntheses CO(CH₂)₄CH₃ -Obtained by Fries rearrangement of 4-chlorophenyl caproate with aluminium chloride [3170], (55.3 %) [1640]. -Also obtained by oxidation of 4-chloro-2-hexylphenol in the presence of selenium oxide in aqueous dioxane [3235].

-Also obtained by reaction of hexanoyl chloride with 4-chlorophenol in the presence of aluminium chloride (50 %) [2680]. -Also refer to: [1702, 1798, 2134].

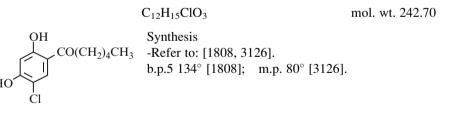
b.p.₁ 129–132° [2680], b.p.₀₇ 135–138° [1640]; m.p. 57–58° [2680], 57° [3170], 56.5° [1702], 56° [3235]; IR [1640], UV [3170].

BIOLOGICAL ACTIVITY: Nematocide [1798].

Oxime	[401935-06-6]	$C_{12}H_{16}CINO_2$	mol. wt. 241.72
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-Topical preparations comprising at least one aryloxime and bisabolol [483].

## 1-(5-Chloro-2,4-dihydroxyphenyl)-1-hexanone

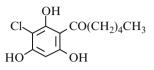


#### 1-(3-Chloro-2,4,6-trihydroxyphenyl)-1-hexanone (TH-DIF-3), (Cl-THPH)

[200878-66-6]

C12H15ClO4

mol. wt. 258.70



Syntheses CO(CH₂)₄CH₃ -Obtained by reaction of sulfuryl chloride (1 equiv.) with 2,4,6-trihydroxycaprophenone in a chloroform/ ethanol mixture at r.t. [1129]. -Also refer to: [126, 129, 1772, 2260].

yellow amorphous solid [1129];

¹H NMR [126], UV [2260], MS [1129, 2260].

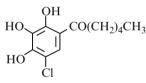
BIOLOGICAL ACTIVITY: Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129]; Differentiation-inducing factor 1 (DIF-1) as protease-inhibitory and antiviral compound [129]; Antiviral [126].

Trimethyl ether [916895-75-5]  $C_{15}H_{21}ClO_4$ mol. wt. 300.78

-Refer to: [1772].

#### 1-(5-chloro-2,3,4-trihydroxyphenyl)-1-hexanone





Synthesis

-Obtained by reaction of chlorine with 4-caproylpyrogallol in acetic acid [506]. m.p. 95–96° [506].

## 1-(3-Fluoro-4-hydroxyphenyl)-1-hexanone

[695196-63-5]

C12H15FO2 mol. wt. 210.25



Synthesis -Obtained by refluxing 3-fluoro-4-methoxyhexanophenone with pyridinium chloride for 15 min [516]. b.p.₁₅ 200° [516]; m.p. 64° [516].

**Methyl ether** [371757-62-9] C₁₃H₁₇FO₂ mol. wt. 224.27

-Obtained by Friedel-Crafts acylation of 2-fluoroanisole with caproyl chloride in the presence of aluminium chloride in carbon disulfide (75–85 %) [516] or in methylene chloride (92 %) [1404].

-Also refer to: [2407].

m.p. 62° [516], 61–62° [1404]; ¹H NMR [1404], ¹³C NMR [1404].

#### 1-(5-Fluoro-2-hydroxyphenyl)-1-hexanone

 $[319-31-3] C_{12}H_{15}FO_2 mol. wt. 210.25$ 

$$CO(CH_2)_4$$

Syntheses

Preparation by Fries rearrangement of 4-fluorophenyl caproate with aluminium chloride [1641] at 130° for 2 h (87%) or at 150° (47%) [2991].
Also refer to: [1549].

b.p.₁₂ 146–147° [2991]; m.p. 49–50° [2991], 31° [1549].

**2,4-Dinitrophenylhydrazone** [860-34-4] C₁₈H₁₉FN₄O₅ mol. wt. 390.37

m.p. 176° [1549].

#### 1-(4-Hydroxy-3-nitrophenyl)-1-hexanone

[70079-25-3]

 $C_{12}H_{15}NO_4$ 



Syntheses

-Obtained by reaction of caproyl chloride with 2-nitrophenol in the presence of aluminium chloride in nitrobenzene for 2.5 h at  $55-60^{\circ}$  (26 %) [465].

 $^{\circ}CO(CH_2)_4CH_3$  -Also obtained by treatment of 4-caproylphenol with concentrated nitric acid in concentrated sulfuric acid at 0° for 1 h 15 min [1222].

m.p. 37.2-37.6° [465], 37-38° [1222].

#### 2,4-Dinitrophenylhydrazone

 $C_{18}H_{19}N_5O_7$ 

mol. wt. 417.38

mol. wt. 237.26

m.p. 162.6–163.2° [465].

#### 1-(2,4,6-Trihydroxy-3-nitrophenyl)-1-hexanone

0° (70–80 %) [3414].

1-hexanone in concentrated sulfuric acid below

-Also refer to: [153].

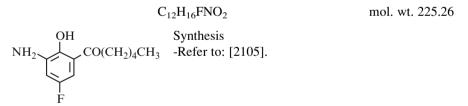
bright yellow needles [3414]; m.p. 50–53° [3414]; ¹H NMR [3414], IR [3414], MS [3414].

BIOLOGICAL ACTIVITY: Germination inhibitory activity [3414]; PET inhibitory activity [3414]. Effect on gibberellin-inducible  $\alpha$ -amylase synthesis in barley aleurone cells [153].

# 1-(3-Amino-5-chloro-2-hydroxyphenyl)-1-hexanone

 $C_{12}H_{16}CINO_{2}$ mol. wt. 241.72 OH Synthesis NH₂  $CO(CH_{2})_{4}CH_{3}$  -Refer to: [2105].

#### 1-(3-Amino-5-fluoro-2-hydroxyphenyl)-1-hexanone

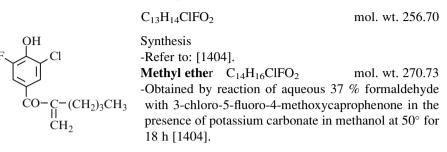


#### 1-(2-Amino-5-hydroxyphenyl)-1-hexanone

[404919-00-2]	$C_{12}H_{17}NO_2$	mol. wt. 207.27
OH CO(CH ₂ ) ₄ CH ₃	Syntheses -Obtained by hydrogenation of 2-nitrophenyl)-2-hexyn-1-one (91 %) [12 -Also refer to: [3331, 3332]. ¹ H NMR [3332].	

USE: For preparation of indoles and indazoles as thyromimetics [1232, 3331].

#### 1-(3-Chloro-5-fluoro-4-hydroxyphenyl)-2-methylene-1-hexanone



#### 1-(2,4-Dihydroxy-6-methylphenyl)-1,3,5-hexanetrione

$C_{13}H_{14}$	$O_5$	mol. wt. 250.25
ŎН	Synthesis	
COCH ₂ COCH ₂ COCH ₃	-Refer to: [1259].	
	Dimethyl ether	[62643-36-1]
HO CH ₃	$C_{15}H_{18}O_5$	mol. wt. 278.30

-Obtained by adding methyl O,O'-dimethylorsellinate (m.p.  $36-38^{\circ}$ ) to the dilithium salt of dilithioacetylacetone in THF under nitrogen at  $0^{\circ}$  and the mixture was stirred at 25° for 16 h (88 %) [1259].

m.p. 63–65° [1259]; ¹H NMR [1259], IR [1259], UV [1259].

11 0

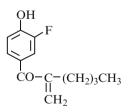
Dibenzyl ether	[38071-42-0]	$C_{27}H_{26}O_5$	mol. wt. 430.50
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-Obtained by treatment of dilithioacetylacetone with methyl O,O'-dibenzylorsellinate in THF for 36 h at 25° (71 %) [1259].

m.p. 55.5–57° [1259]; ¹H NMR [1259], IR [1259], UV [1259].

Hemihydrate of the dibenzyl ether C₂₇H₂₆O₅, 0.5 H₂O mol. wt. 439.51

#### 1-(3-Fluoro-4-hydroxyphenyl)-2-methylene-1-hexanone



Synthesis -Refer to: [1404]. Methyl ether [942037-66-3] C14H17FO2 mol. wt. 236.29  $(CH_2)_3CH_3~$  -Obtained by reaction of aqueous 37 % formaldehyde with 3-fluoro-4-methoxycaprophenone in the presence of potassium carbonate in methanol at 50° for 18 h [1404].

¹H NMR [1404].

. .....

#### 1-(3,5-Dibromo-2,4-dihydroxy-6-methoxyphenyl)-1-hexanone

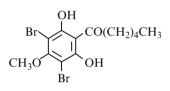
 $[118191-29-0] C_{13}H_{16}Br_2O_4 mtext{mol. wt. 396.08} \\ OH Synthesis \\ -Preparation by halogenation of 1-(2,4-dihydroxy-6-methoxyphenyl)-1-hexanone with bromine in \\ -Preparation by halogenation of 1-(2,4-dihydroxy-6-methoxyphenyl)-1-hexanone with bromine in \\ -Preparation by halogenation of 1-(2,4-dihydroxy-6-methoxyphenyl)-1-hexanone with bromine in \\ -Preparation by halogenation of 1-(2,4-dihydroxy-6-methoxyphenyl)-1-hexanone with bromine in \\ -Preparation by halogenation of 1-(2,4-dihydroxy-6-methoxyphenyl)-1-hexanone with bromine in \\ -Preparation by halogenation of 1-(2,4-dihydroxy-6-methoxyphenyl)-1-hexanone with bromine in \\ -Preparation by halogenation of 1-(2,4-dihydroxy-6-methoxyphenyl)-1-hexanone with bromine in \\ -Preparation by halogenation by halogena$ 

BIOLOGICAL ACTIVITY: Differentiation-inducing factor from the slime mould *Dictyostelium discoideum* [2012].

# **1-(3,5-Dibromo-2,6-dihydroxy-4-methoxyphenyl)-1-hexanone** (*Br-DIF-1*)

[118191-30-3]

 $C_{13}H_{16}Br_2O_4$  mol. wt. 396.08



Syntheses

water [2012].

CO(CH₂)₄CH₃ -Obtained by reaction of pyridinium tribromide with 2,6-dihydroxy-4-methoxyhexanophenone in pyridine and the mixture was stirred for 1 h (62 %) [1129].

> -Preparation by halogenation of 1-(2,6-dihydroxy-4-methoxyphenyl)-1-hexanone with bromine in water [2012].

-Also refer to: [1772, 1773, 2341].

yellow amorphous solid [1129]; ¹H NMR [1129], ¹³C NMR [1129], MS [1129].

BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773]. Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129]; Differentiation-inducing factor from the slime mould *Dictyostelium discoideum* [2012].

#### 1-(3,5-Dichloro-2,4-dihydroxy-6-methylphenyl)-1-hexanone

[118191-31-4]	$C_{13}H_{16}Cl_2O_3$	mol. wt. 291.17
Cl CO(CH ₂ ) ₄ CH ₃	Synthesis -Refer to: [2012].	
HO CI CH ₃	BIOLOGICAL ACTIVITY: inducing factor [2012].	As differentiation

HO

#### 1-(3,5-Dichloro-2,4-dihydroxy-6-methoxyphenyl)-1-hexanone (DIF-1), (2-MIDIF-1)

[118191-28-9]	$C_{13}H_{16}Cl_2O_4$	mol. wt. 307.17
$\begin{array}{c} OH\\ Cl \\ HO \\ Cl \\ Cl \\ \end{array} \begin{array}{c} OH\\ CO(CH_2)_4CH_3 \\ OCH_3 \\ \end{array}$	Syntheses -Obtained by reaction of chlorine 6-methoxyhexanophenone in wat -Also refer to: [74, 128, 129, 1773	er [2012].

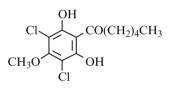
¹H NMR [2012]. MS [2012].

BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773, 1776, 2341]: Differentiation-inducing factor from the slime mould *Dictyostelium* discoideum [2012]; Differentiation-inducing factor 1 (DIF-1) as protease-inhibitory and antiviral compound [129]; As cysteine protease activity modulator [128].

#### 1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-1-hexanone (DIF-1)

[111050-72-7]

C13H16Cl2O4 mol. wt. 307.17



OH Cl  $CO(CH_2)_4CH_3$   $H_3O$  OH Syntheses -Preparation by adding a solution of sulfuryl chlo-ride (2.16 equiv.) in ethanol to a solution of 2,6-dihydroxy-4-methoxyhexanophenone in chloroform at r.t.; then, the solution was stirred for 1 h at r.t. (93 %) [1129].

-Also obtained by reaction of chlorine with 2,6-dihydroxy-4-methoxyhexanophenone in water [2012].

-Also refer to: [74, 126–129, 207, 935, 1135, 1586, 1593, 1775, 1860, 2021, 2248, 2341, 2682, 2729, 2737, 3368, 3395, 3402].

Isolation from natural sources

-Found in Dictyostelium discoideum [318, 1654, 1776]. -From Dictyostecoideum [2869].

yellow amorphous solid [126, 127, 1129]; m.p. 101° [126, 127]; ¹H NMR [1129, 2012], ¹³C NMR [1129], UV [2012], MS [1129, 2012], X-ray data [2012]; GC [2154].

BIOLOGICAL ACTIVITY: Structure and function of polyketide synthase in cellular slime mold for differentiation-inducing factor-1 formation [2682]; β-amyloid level redn. by treatment with differentiation-inducing factor [2207]; Antitumor activities of derivs. of DIF-1 [1677, 1775]; Dictyostelium differentiation-inducing factor-1 derivs. promotion of glucose consumption in mammalian cells [1777]; Cell type specificity

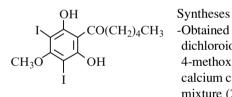
of a diffusible inducer is detd, by a GATA family transcription factor in Dictyostelium [1659]; DIF-1: Stalk-cell differentiation; Anticancer drug [1129]; DIF-1 derivs. for treating diabetes and obesity [1773]; A UDP-glucose deriv, in required for vacuolar autophagic cell death [3136]; Antileukemic activities of Dictyostelium secondary metabolites [1678]; Antitumor substance [1776]; Inhibits progesterone-induced oocyte maturation in *Xenopus laevis* [1776]; Differentiationinducing factor from the slime mould *Dictyostelium discoideum* [2012]; Cell type detn. in *Dictyostelium discoideum* by differentiation-inducing factor DIF-1 [2005]; Novel development rescuing factors secreted by Dictyostelium cells that are involved in restoration of mutant lacking MAP kinase ERK2 [3153]; Potent antiproliferative [2869]; Novel development rescuing factors secreted by Dictyostelium cells that are involved in restoration of mutant lacking MAP kinase ERK2 [3153]; Effects of differentiation-inducing factor-1 (DIF-1) of Dictyostelium discoideum on bacteria, fungi, and an influenza virus [1789]: Differentiation-inducing factor 1 (DIF-1) as protease-inhibitory and antiviral compound [129]; As cysteine protease activity modulator [128]; Differentiation-inducing factor D. discoideum raises intracellular calcium concentration and suppresses cell growth in rat pancreatic AR42J cells [1778]; Prespore-specific gene transcriptional repression by, in *Dictyostelium* [935]. -Also refer to: [1263, 1675, 1774, 1804, 1926, 2309, 2341, 3029].

#### 1-(2,6-Dihydroxy-3,5-diiodo-4-methoxyphenyl)-1-hexanone (I-DIF-1)

[861889-71-6]

C13H16I2O4

mol. wt. 490.08



CO(CH₂)₄CH₃ -Obtained by reaction of benzyltrimethylammonium dichloroiodate (2.09 equiv.) with 2,6-dihydroxy-4-methoxyhexanophenone in the presence of calcium carbonate in a methylene chloride/methanol mixture (2:1) at r.t. for 2 h (57 %) [1129].

-Also refer to: [1772, 1773, 2341].

yellow amorphous solid [1129]; ¹H NMR [1129], ¹³C NMR [1129], MS [1129].

BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

#### 1-(2,4-Dihydroxy-6-methylphenyl)-1,3-hexanedione

$$\begin{array}{c} C_{13}H_{16}O_4 \\ OH \\ HO \\ CH_3 \\ CH_3 \\ CI_5H_{20}O_4 \end{array} \qquad \begin{array}{c} \text{mol. wt. 236.27} \\ \text{mol. wt. 236.27} \\ \text{mol. wt. 236.27} \\ \text{mol. wt. 236.27} \\ \text{mol. wt. 264.32} \\ \text{mol. wt. 264.32} \end{array}$$

-Obtained by treatment of a mixture of orcacetophenone dimethyl ether and ethyl butyrate with pulverised sodium at  $115-120^{\circ}$  for 1.5 h (48 %) [2814].

-Also obtained by treatment of a mixture of orcpropiophenone, butyric anhydride and sodium butyrate at 180–190° for 8 h [3142].

viscous brown oil [3142]; pale yellow oil [2814]; b.p.₂₀₋₂₅ 220–225° [2814].

#### **Copper salt of the dimethyl ether** $(C_{15}H_{19}O_4)_2Cu$ mol. wt. 590.17

-Preparation from the above dimethyl ether and copper acetate [2814].

m.p. 175–177° [2814].

#### 3-Hexanoyl-4-hydroxybenzoic acid

[136039-85-5]	$C_{13}H_{16}$	$O_4$	mol. wt. 236.27
OH CO(CH ₂ ) ₄ CH ₃	Synthesis -Obtained by hydro m.p. 188° [967]. Acetate	blysis of its ethyl ester $C_{15}H_{18}O_5$	[967]. mol. wt. 278.30

-Obtained by acetylation of 3-hexanoyl-4-hydroxybenzoic acid [967].

m.p. 100° [967].

#### Methyl ether

 $C_{14}H_{18}O_4$ 

mol. wt. 250.29

-Obtained by methylation of 4-hydroxy-3-valeroylbenzoic acid [967].

m.p. 160° [967].

#### Ethyl ester

C₁₅H₂₀O₄ mol. wt. 264.32

-Obtained by reaction of hexanoyl chloride with ethyl 4-hydroxybenzoate in the presence of aluminium chloride in tetrachloroethane, then the reaction mixture was heated at  $120^{\circ}$  for 3–4 h [967].

 $b.p._{20} \ 200^\circ \ [967].$ 

#### 5-Hexanoyl-2-hydroxybenzoic acid

[78418-00-5]	$C_{13}H_{16}O_4$	mol. wt. 236.27
OH COOH CO(CH ₂ ) ₄ CH ₃	Syntheses -Obtained by hydrolysis of methyl 5-caproyl ate with boiling 20 % solution of potassium -Also refer to: [689] (82 %). m.p. 117° [730], 116–117° [689].	• •

Methyl ester	[78417-95-5]	$C_{14}H_{18}O_4$	mol. wt. 250.29
meenyr coter		01411804	mon. wt. 250.27

-Obtained by Fries rearrangement of methyl 2-(caproyloxy)benzoate with aluminium chloride in boiling carbon disulfide for 2 h, then the reaction mixture heated at 90–110° for a few min after solvent elimination (82 %) [1259]. -Also refer to: [689] (77 %).

b.p.₂₀ 202–205° [730]; m.p. 58–60° [689], 50–51° [730]; ¹H NMR [689].

#### 2,4,6-Trihydroxy-3-(1-oxohexyl)benzaldehyde

[96573-32-9]	$C_{13}H_{16}O_5$	mol. wt. 252.27
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	OH		
СНО	人	CO(C	CH ₂ ) ₄ CH ₃
			20.0
HO	$\sim$	OH	

Syntheses -Obtained by reaction of ethyl orthoformate with 2,4,6-trihydroxycaprophenone in the presence of aluminium chloride in methylene chloride with cooling in an ice bath for 30 min (70 %) [421].

-Also refer to: [3033].

m.p. 134–136° [421]; ¹H NMR [421], IR [421], MS [421].

BIOLOGICAL ACTIVITY: Inhibitory effects of treatment with this acylphenol on TPA Induced EBV-EA Activation [3033]; As photosynthetic electron transport (PET) [3405].

#### 1-(3-Bromo-2,6-dihydroxy-4-methoxyphenyl)-1-hexanone (Br-DIF-3)

[861889-70-5] C13H17BrO4 mol. wt. 317.18 **Syntheses** OH CO(CH₂)₄CH₃ -Obtained by reaction of pyridinium tribromide Br with 2,6-dihydroxy-4-methoxyhexanophenone in pyridine and the mixture was stirred for 1 h CH₃O (28 %) [1129]. -Also refer to: [1773, 2341].

colourless amorphous solid [1129]; ¹H NMR [1129], ¹³C NMR [1129], MS [1129].

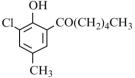
BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

#### 1-(2-Chloro-6-hydroxy-4-methylphenyl)-1-hexanone

[24490-27-5]	$C_{13}H_{17}ClO_2$	mol. wt. 240.73
CH ₃ CH ₃ CH ₃ CH ₃ CO(CH ₂ ) ₄ CH ₃	Synthesis -Refer to: [3138]. Fluorescence spectra [3138].	

#### 1-(3-Chloro-2-hydroxy-5-methylphenyl)-1-hexanone

	$C_{13}H_{17}ClO_2$	mol. wt. 240.73
	Synthesis	
Ъ.	-Refer to [2679]	



CO(CH₂)₄CH₃ -Refer to: [2679]. b.p.₁ 150–152° [2679]; m.p. 25–27° [2679];  $n_D^{30} = 1.5396$  [2679].

#### 1-(5-Chloro-2-hydroxy-3-methylphenyl)-1-hexanone

$C_{13}H_{17}ClO_2$		mol. wt. 240.73
CH ₃ Cl	Synthesis -Refer to: [2679]. b.p. ₁ 149–151° [2679];	$n_D^{25} = 1.5428$ [2679].

#### 1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-hexanone

[196813-77-1]	$C_{13}H_{17}ClO_2$	mol. wt. 240.73
CH ₃ CO(CH ₂ ) ₄ CH ₃	Syntheses -Obtained by reaction of cap 3-methylphenol in the prese *for 1 h at 100° in a sealed t *for 2 h at 70–80° (31 %) or (70 %) [1684].	ence of boron trifluoride, tube (85 %) [1684];

-Also refer to: [1686, 2679, 3138].

b.p.₁ 152–154° [2679]; m.p. 56–57° [1684], 42–44° [2679]; IR (Sadtler standard N° 8988) [1684]; fluorescence spectral data [3138].

#### 1-(3-Chloro-2,4-dihydroxy-6-methoxyphenyl)-1-hexanone

[227946-80-7]	$C_{13}H_{17}ClO_4$	mol. wt. 272.73
CI CO(CH ₂ ) ₄ CH ₃ HO OCH ₃	Syntheses -Refer to: [74, 127, 128, 1771, 2869 ¹ H NMR [127], MS [127].	].

BIOLOGICAL ACTIVITY: In the treatment of cancer (PDE1 inhibitor) [1771]; As cysteine protease activity modulator [128]; Inhibition of enzyme human leukemia K562 cells [74]; Inhibition growth human leukemia K562 cells [74]; Antiviral (HRV-14 and HRV-16) [127]; Cytotoxicity [127].

# **1-(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)-1-hexanone** (*DIF-3*)

[113411-17-9]	$C_{13}H_{17}ClO_4$	mol. wt. 272.73
Cl CO(CH ₂ ) ₄ CH ₃ CH ₃ O OH	ride (1.1 equiv.) in et 2,6-dihydroxy-4-methox	solution of sulfuryl chlo- thanol to a solution of yhexanophenone in chlo- olution was stirred for 1 h
11 6 . 574 010 1650	1677 1772 1772 1777	0011 0060 0001 0150

-Also refer to: [74, 318, 1653, 1677, 1772, 1773, 1777, 2011, 2869, 3031, 3153, 3256].

colourless amorphous solid [1129]; ¹H NMR [1129], ¹³C NMR [1129], MS [1129]; GC [2154]. BIOLOGICAL ACTIVITY: Antitumor activities of derivs. of DIF-1 [1677]; DIF-3 (3 M); Dictyostelium differentiation-inducing factor-1 derivs. promotion of glucose consumption in mammalian cells [1777]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129]; DIF-1 derivs. for treating diabetes and obesity [1773]; Involvement of GSK-3β and DYRK1B in differentiation-inducing factor-3-induced phosphorylation of cyclin D1 in HeLa cells [3030]; Novel development rescuing factors secreted by Dictyostelium cells that are involved in restoration of mutant lacking MAP kinase ERK2 [3153]; Cell differentiation regulation by, in Dictyostelium discoideum [1653]; Differentiation-inducing factor, from Dictyostelium discoideum, characterization of, [2011].

#### 1-(5-Chloro-4,6-dihydroxy-2-methoxyphenyl)-1-hexanone

[227946-81-8]	$C_{13}H_{17}ClO_4$	mol. wt. 272.73
HO $CI$ $CO(CH_2)_4CH_3$ $CO(CH_2)_4CH_3$ $CO(CH_2)_4CH_3$	Synthesis -Refer to: [128]. ¹ H NMR [127]; MS [127]. BIOLOGICAL ACTIVITY: As activity modulator [128].	s cysteine protease

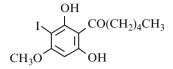
### 1-(3-Fluoro-4-hydroxyphenyl)-5-methyl-1-hexanone

	$C_{13}H_{17}FO_2$	mol. wt. 224.27
ОН	Synthesis	
, ↓ , F	-Refer to: [38].	
	Methyl ether [161581-	.92-6]
$\mathbf{i}$	$C_{14}H_{19}FO_2$	mol. wt. 238.30
$CO(CH_2)_3CH(CH_3)_2$	-Refer to: [38].	

# 1-(2,6-Dihydroxy-3-iodo-4-methoxyphenyl)-1-hexanone

(I-DIF-3)

[861889-72-7]



Syntheses

CO(CH₂)₄CH₃ -Obtained by reaction of benzyltrimethylammonium dichloroiodate (1.1 equiv.) with 2,6-dihydroxy-4-methoxyhexanophenone in the presence of calcium carbonate in a methylene chloride/methanol mixture (2:1) at r.t. for 2 h [1129].

-Also refer to: [1773, 2341].

yellow amorphous solid [1129]; ¹H NMR [1129], ¹³C NMR [1129], MS [1129].

BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

# 1-(2-Hydroxy-3-methylphenyl)-1-hexanone

[132858-60-7]  $C_{13}H_{18}O_2$  mol. wt. 206.28 OH Syntheses -Obtained by Fries rearrangement of o-cresyl caproate with aluminium chloride for 30 min at 160–180° (60 %) [726] or (37 %) [1644]. -Also refer to: [2270, 2679]. b.p. t 131–132° [2679]. b.p. ts 16 150–154° [2270]. b.p. ts 152–154° [726, 1644]:

b.p.₁ 131–132° [2679], b.p._{15–16} 150–154° [2270], b.p.₁₅ 152–154° [726, 1644]; m.p. 23° [726];  $n_D^{20} = 1.5338$  [2679].

2,4-Dinitrophenylhydrazone	$C_{19}H_{22}N_4O_5$	mol. wt. 386.41
m.p. 178–179° [2270].		

#### Phenylhydrazone

 $C_{19}H_{24}N_2O$ 

mol. wt. 296.41

m.p. 93–94° [726].

#### 1-(2-Hydroxy-4-methylphenyl)-1-hexanone

 $[52122-70-0] C_{13}H_{18}O_2$ mol. wt. 206.28 OH CO(CH₂)₄CH₃ Syntheses -Preparation by Fries rearrangement of 3-methylphenyl caproate with aluminium chloride, *without solvent for 2 h at 160° (93 %) [726], at 140– 150° [906], 10–20 min at 120–140° (91 %) [243] or 2 h at 120–148° (73 %) [1644];

*in nitrobenzene for 24 h at 25° (62 %) [243] or at 25–30° for 48 h (64 %) [244]; *in the presence of a mixture of graphite and methanesulfonic acid at 160° for 30 min (80 %) [2833].

-Also obtained by reaction of caproic acid with m-cresol,

*in the presence of boron trifluoride for 6 h at 80° (88 %) [1685];

*in the presence of polyphosphoric acid for 1 h at 80° (22 %) [2916];

*in the presence of a mixture of graphite and methanesulfonic acid at  $120^{\circ}$  for 2 h (88 %) [2834] or at 140° for 5 min (90 %) [2833].

-Also refer to: [108–110, 1452, 2833, 3077, 3270].

b.p. $_{0.1}$  90–115° [109], b.p. $_{0.4}$  101° [2916], b.p. $_1$  115–117° [906], b.p. $_3$  134–135° [243], b.p. $_2$  135–137° [2679], b.p. $_{15}$  162–164° [726, 1644], b.p. $_{18}$  171–173° [1685];

m.p. 23-24° [726, 1644], 20-21° [1685];

¹H NMR [2834], ¹³C NMR [2834], IR [2834];  $n_D^{25} = 1.5339$  [2679].

Oxime	[50652-74-9]	$C_{13}H_{19}NO_2$	mol. wt. 221.30

-Refer to: [108, 483, 2742, 3077].

USE: Topical preparations comprising at least one aryloxime and bisabolol [483]; In copper extraction from aqueous solutions [108]; In extn. of copper and nickel from sulfate solns. [110]; Chelation with, of metals, [109].

BIOLOGICAL ACTIVITY: As lipoxygenase inhibitor [3077].

#### Phenylhydrazone

#### $C_{19}H_{24}N_2O$

mol. wt. 296.41

m.p. 97–97.5° [243], 94° [2916], 92–93° [726].

#### 1-(2-Hydroxy-5-methylphenyl)-1-hexanone

1-(2-Hydroxy-5-n	netnyipnenyi)-1-nexano	ne		
[101002-28-2]	$C_{13}H_{18}$	O ₂	mol. wt. 206.28	
OH CO(CH ₂ ) ₄ C	caproate with alun [726], 10 min at 1 [1644] or 72 h at 2	•		
	[2679], b.p. ₁₅ 150–152° $n_D^{25} = 1.5315$ [2679].	[726, 1644], b.p. ₁₅ 16	53° [2647];	
Oxime	[99283-86-0]	$C_{13}H_{19}NO_2$	mol. wt. 221.30	
-Refer to: [2742, 3	3013, 3077].			
GC [3013]; p	olarity of [3013].			
BIOLOGICAL A	CTIVITY: As lipoxygena	use inhibitor [3077].		
<b>Oxime</b> ( <i>E</i> )	$C_{13}H_{19}NC$	$\mathbf{D}_2$	mol. wt. 221.30	
MS [1921].				
Phenylhydrazone	c C ₁₉ H ₂	₄ N ₂ O	mol. wt. 296.41	
m.p. 110–112°	[726].			
Methyl ether	$C_{14}H_{20}C_{14}$	$D_2$	mol. wt. 220.31	
-Preparation by reaction of caproyl chloride with 4-methylanisole in the presence of aluminium chloride in carbon disulfide (72 %) [515].				
pale yellow oil	[515]; b.p. ₁₂ 176–179°	[515].		
1-(4-Hydroxy-2-methylphenyl)-1-hexanone				
[132858-61-8]	$C_{13}H_{18}$	O ₂	mol. wt. 206.28	
OH CH ₃ CO(CH ₂ ) ₄ CH ₃	Syntheses -Obtained by reaction presence of polyphosph			

-Also obtained by Fries rearrangement of 3-methylphenyl caproate with aluminium chloride in nitrobenzene at  $25-30^{\circ}$  for 48 h (4 %) [244]. -Also refer to: [1685].

m.p. 77–77.5° [2916], 76–76.5° [244], 72° [1685].

**Phenyl ether** [791615-78-6] C₁₉H₂₂O₂ mol. wt. 282.38

-Obtained by adding a mixture of m-phenoxytoluene and hexanoyl chloride to a suspension of aluminium chloride in methylene chloride at  $0^{\circ}$ , then the mixture stirred for 1.5–2 h at 3–5° (33 %) [2503].

b.p.₃ 220–222° [2503]; ¹H NMR [2503], IR [2503], MS [2503].

#### 1-(4-Hydroxy-3-methylphenyl)-1-hexanone

 $\label{eq:c132858-62-9} [132858-62-9] \qquad \qquad \text{mol. wt. } 206.28$ 

Syntheses

-Obtained by Fries rearrangement of 2-methylphenyl caproate with aluminium chloride at 160° (25 %) [726] or 160–180° (20 %) [1644].

CO(CH₂)₄CH₃ b.p.₂ 180–182° [2679], b.p.₁₅ 200–205° [726, 1916];

m.p. 79-80° [726, 2679].

OH

CH₃

Methyl ether	[141036-68-2]	$C_{14}H_{20}O_2$	mol. wt. 220.31
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-Obtained by Friedel-Crafts acylation reaction of caproic anhydride with 2-methoxytoluene,

*using the combined catalyst system of TiCl(OTf)₃ and TfOH (97 %) [1483];

*in the presence of SbCl₅-LiClO₄ mixture in refluxing methylene chloride for 30 min (88 %) [2176].

-Also obtained by Friedel-Crafts acylation of 2-methylanisole in the presence of a Lewis acid catalyst while using carboxylic acid or trisubstituted-silyl carboxylate as acylating agent and carrying out the reaction in the presence of p-trifluoromethylbenzoic anhydride and, if necessary, a silver salt [2997]. -Also refer to: [2172, 2999].

m.p. 34° [2999]; ¹H NMR [2999], ¹³C NMR [2999], IR [2999].

Benzoate	$C_{20}H_{22}O_3$	mol. wt. 310.39	m.p. 59–60° [726].
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#### 1-(2,5-Dihydroxy-4-methylphenyl)-1-hexanone

$C_{13}H_{18}O_{3}$		mol. wt. 222.28
CH ₃ CO(CH ₂ ) ₄ CH ₃	Synthesis -Refer to: [1755]. <b>Dimethyl ether</b> [83893-24-7] C ₁₅ H ₂₂ O ₃	mol. wt. 250.34

-Prepared by Friedel-Crafts acylation (85 %) [1755].

b.p.₃ 183° [1755]; ¹H NMR [1755], IR [1755].

# 1-(4,5-Dihydroxy-2-methylphenyl)-1-hexanone

	$C_{13}H_1$	₈ O ₃				mol. wt. 2	22.28
HO H	Synthesis -Refer to: [ Dimethyl e	-		-81			
CH ₃	$C_{15}H_{22}O_3$		-	-		mol. wt. 2	50.34
CO(CH ₂ ) ₄ CH ₃	-Obtained	by	reaction	of	caproyl	chloride	with
	homoverat	role	in the pres	ence	of alumi	nium chlori	ide in
	carbon dis	ulfide	(73 %) [3	05].			

-Also obtained by methylation of 1-(4,5-dihydroxy-2-methylphenyl)-1-hexanone [305].

b.p.₈ 190° [305]; m.p. 48° [305].

Semicarbazone of the dimethyl ether	[3787-68-6]	$C_{16}H_{25}N_3O_3$	mol. wt. 307.39
m.p. 128° [305].			
Phenylhydrazone of the dimethyl ether	[3307-23-1]	$C_{21}H_{28}N_2O_2$	mol. wt. 340.47

m.p. 132° [305].

# 1-(2-Hydroxy-4-methoxyphenyl)-1-hexanone

[372486-19-6]	$C_{13}H_{18}O_3$	mol. wt. 222.28
OH	Syntheses	f. dim sheet
CO(CH ₂ ) ₄ CH ₃	-Obtained by reaction 2,4-dihydroxycaproph	of dimethyl sulfate with enone,
CH ₃ O	1	% sodium hydroxide solution
	(72 %) [3160];	

*in the presence of potassium carbonate in refluxing acetone for 4–6 h (85-90 %) [2501].

colourless oil [3160]; b.p._{12–13} 189–192° [3160].

Oxime [111625-07-1] C₁₃H₁₉NO₃ mol. wt. 237.30 GC [3012].

#### 1-(2-Hydroxy-5-methoxyphenyl)-1-hexanone

$$\begin{array}{c} C_{13}H_{18}O_3 & \text{mol. wt. } 222.28\\ OH & Synthesis\\ \hline CO(CH_2)_4CH_3 & -Refer to: [285].\\ \hline Oxime & [140943-13-1]\\ C_{13}H_{19}NO_3 & \text{mol. wt. } 237.30\\ OCH_2 \end{array}$$

-Refer to: [285].

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

#### 1-(4-Hydroxy-3-methoxyphenyl)-1-hexanone

[114541-98-9]

$$C_{13}H_{18}O_3$$

mol. wt. 222.28



Syntheses

-Obtained by refluxing a mixture (about 165°) of caproic acid, guaiacol and fused zinc chloride (Nencki reaction) [726]. -Also obtained by Fries rearrangement of 2-methoxyphenyl caproate with aluminium chloride [1990] in nitrobenzene (50 %) [726].

-Also obtained by adding DDQ to 1-(4-hydroxy-3-methoxyphenyl)-1-hexanol in benzene and the reaction mixture stirred for 4 h at r.t. [2989]. -Also refer to: [1427, 1429, 2075].

b.p.₁₅ 212–215° [726]; m.p. 60–62° [726], 53° [2989]; ¹H NMR [2989].

BIOLOGICAL ACTIVITY: Chloleretic [2989].

#### Benzoate

C₂₀H₂₂O₄

mol. wt. 326.39

m.p. 54–55° [726].

#### 1-(2,4-Dihydroxy-6-methoxyphenyl)-1-hexanone

[200878-64-4]  $C_{13}H_{18}O_4$ 

mol. wt. 238.28

Syntheses

HO CO(CH₂)₄CH₃

CO(CH₂)₄CH₃ -Obtained by reaction of hexanonitrile with 3,5-dihydroxy-anisole in the presence of zinc chlo-OCH₃ ride and hydrochloric acid (Hoesch reaction) [2012].

-Also refer to: [126-129].

m.p. 109° [126, 127]; ¹H NMR [126, 127], MS [126, 127]. BIOLOGICAL ACTIVITY: Differentiation-inducing factor 1 (DIF-1) as protease-inhibitory and antiviral compound [129]; As cysteine protease activity modulator [128]; Picornavirus activity inhibitor [126]; 3C protease activity inhibitor [126]; Antiviral [126, 127]; Cytotoxicity [126].

Syntheses

#### 1-(2,6-Dihydroxy-4-methoxyphenyl)-1-hexanone (DMPH)

[142234-79-5]

CH₂O

C13H18O4 mol. wt. 238.28

OH

OH

.CO(CH₂)₄CH₃ -Obtained by reaction of hexanoyl chloride with 5-methoxyresorcinol in the presence of aluminium chloride in methylene chloride at r.t. for 3 h (58 %) [1129].

> -Also obtained by reaction of hexanonitrile with 3,5-dihydroxyanisole in the presence of zinc chloride and hydrochloric acid (Hoesch reaction) [2012].

-Also refer to: [126–129, 1773, 2341, 3475].

m.p. 121° [126, 127]; ¹H NMR [126, 127, 3475], ¹³C NMR [3475], IR [3475], UV [3475], MS [126, 127, 3475]; GC-MS [1164].

Isolation from natural sources

-From Syzygium levinei (Myrtaceae) [3475].

-From clove Syzygium aromaticum (Myrtaceae) [2108].

-Of Populus tritis bud exudate [955].

-In bud exudate of Populus koreana, Populus maximowiczii and Populus suaveolens (Salicaceae) [1164].

BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129]; Differentiation-inducing factor 1 (DIF-1) as protease-inhibitory and antiviral compound [129]; As cysteine protease activity modulator [128]; Caspase-3, inhibition of [127]; Picornavirus activity inhibitor [126]; 3C protease activity inhibitor [126]; Antiviral [126, 127]; Cytotoxicity [126].

# 1-(2,4,6-Trihydroxy-3-methylphenyl)-1-hexanone

[101268-53-5]	$C_{13}H_{18}O_4$	mol. wt. 238.28
OH CH ₃ HO OH OH	Synthesis -Obtained by reaction of phloroglucinol (Hoesch re -Also refer to: [1440]. m.p. 138–139° [1440, 144	eaction) [1441].

#### 1-(2,3,4-Trihydroxy-6-methoxyphenyl)-1-hexanone

[227946-82-9]	$C_{13}H_{18}O_5$	mol. wt. 254.28
HO HO CO(CH ₂ ) ₄ CH ₃		autoina motosca
HO OCH3	BIOLOGICAL ACTIVITY: As activity modulator [128].	cysteine protease

# 1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1,3,5-hexanetrione

01411605	moi. wt. 204.20
CH ₃ O ^{OH} CH ₃ O ^{CH} CH ₃ COCH ₂ COCH ₂ COCH ₃ COCH ₃ COCH ₂ COCH ₃ COCH	Synthesis -Obtained by hydrogenolysis of 1-(2-benzyloxy-4-methoxy-6-methyl- phenyl)-1,3,5-hexanetrione using a Pd-charcoal catalyst proceeded smoothly (93 %) [1259].

**Benzyl ether** [62643-39-4] C₂₁H₂₂O₅ mol. wt. 354.40

-Obtained by acylation of dilithioacetylacetone with methyl 2-O-benzyl-4-O-methylorsellinate for 15 h at  $25^{\circ}$  (74 %) [1259].

yellow crystals [1259]; m.p. 58.5–60.5° [1259]; ¹H NMR [1259], IR [1259], UV [1259].

#### 1-(2-Hydroxy-4,5-dimethoxyphenyl)-1,3,5-hexanetrione

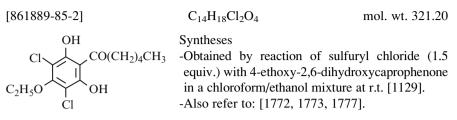
[856348-12-4]	C ₁₄	$H_{16}O_6$	mol. wt. 280.28
CH ₃ O OH OCH ₃ O	COCH ₂ COCH ₂ COCH ₃	Syntheses -Refer to: [573, 834]. m.p. 100–102° [573].	

#### 1-(2,4-Dimethoxy-6-hydroxyphenyl)-1,3,5-hexanetrione

[76631-00-0]	C ₁₄	$H_{16}O_6$		mo	1. wt	. 280	.28
CH ₃ O	COCH ₂ COCH ₂ COCH ₃	Synthesis -Preparation of 2,4-penta 4-dimethoxy- [2699].	anedione	with	me	thyl	2,

apricot orange crystals [2699]; m.p. 95–98° [2699]; ¹H NMR [2699], IR [2699], MS [2699]. mol wt 264.28

# **1-(3,5-Dichloro-4-ethoxy-2,6-dihydroxyphenyl)-1-hexanone** (*Et-DIF-1*)



Yellow amorphous solid [1129]; MS [1129].

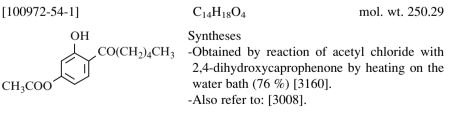
BIOLOGICAL ACTIVITY: Et-DIF-1; Dictyostelium differentiation-inducing factor-1 derivs. promotion of glucose consumption in mammalian cells [1777]; DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

### 1-(5-Acetyl-2-hydroxyphenyl)-1-hexanone

[92757-67-0]	$C_{14}H_{18}O_3$	mol. wt. 234.30
OH CO(CH ₂ ) ₄ CH ₃	Syntheses -Obtained by Fries rearrangement a acetophenone with aluminium chlorid solvent at 150° for 3 h (37 %) [1305].	
COCH ₃	-Also obtained by Friedel-Craft p-hydroxyacetophenone with caproyl sence of aluminium chloride in tetrach for 4 h (39 %) [1305].	chloride in the pre-

m.p. 52° [1305]; ¹H NMR [1305], IR [1305].

#### 1-[4-(Acetyloxy)-2-hydroxyphenyl]-1-hexanone



colourless oil [3160]; b.p.₁₄ 213–215° [3160]; m.p. 32.5–33.5° [3008].

#### 1-(2-Hydroxy-4,6-dimethoxyphenyl)-1,3-hexanedione

$$[60658-72-2] C_{14}H_{18}O_5 mol. wt. 266.29$$

$$OH Synthesis$$

$$OH COCH_2COC_3H_7 OCH_3 OC$$

colourless needles [2602]; m.p. 104–105° [2602]; ¹H NMR [2602], IR [2602], UV [2602].

#### 1-(3-Chloro-6-hydroxy-2,4-dimethylphenyl)-1-hexanone

[100792-79-8]	$C_{14}H_{19}ClO_2$	mol. wt. 254.76
$CH_{3} \xrightarrow{CH_{3}} CO(CH_{2})_{4}CH_{3}$	Synthesis -Preparation by Fries rea 3,5-dimethylphenyl caprox ride in carbon disulfide at for 2 h after solvent elimin	ate with aluminium chlo- 80° for 2 h, then at 110°

m.p. 80° [3114].

m.p. 152° [3114].

Allyl ether	[101598-23-6]	$C_{17}H_{23}ClO_2$	mol. wt. 294.82
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-Obtained by reaction of allyl bromide with the title ketone in the presence of potassium carbonate in refluxing acetone for 6 h (69 %) [3114].

b.p.₂ 190° [3114].

USE: Insecticide [3114].

#### 1-(5-Chloro-3-ethyl-2-hydroxyphenyl)-1-hexanone

[53347-28-7]	$C_{14}H_{19}ClO_2$	mol. wt. 254.76
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Synthesis

OH Synthesis  $C_2H_5$   $CO(CH_2)_4CH_3$  -Obtained by Fries rearrangement of 4-chloro-2-ethylphenyl caproate in the presence of aluminium chloride at 120° for 1.5 h (83 %) [2763].

b.p. $_{0.2}$  120–123.5° [2763];  $n_D^{20} = 1.5363$  [2763].

BIOLOGICAL ACTIVITY: Bacteriostatic [2763].

# 1-(3-Chloro-4-ethoxy-2, 6-dihydroxyphenyl)-1-hexanone

(*Et-DIF-3*)

[861889-93-2]	$C_{14}H_{19}ClO_4$	mol. wt. 286.76
$Cl \downarrow CO(CH_2)_4CH_3$ $C_2H_5O OH$	equiv.) with	n of sulfuryl chloride (1.5 4-ethoxy-2,6-dihydroxyca- loroform/ethanol mixture at

-Also refer to: [1772, 1773, 1777].

Colourless amorphous solid [1129]; MS [1129].

BIOLOGICAL ACTIVITY: Et-DIF-3; Dictyostelium differentiation-inducing factor-1 derivs. promotion of glucose consumption in mammalian cells [1777]; DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

### 1-(4-Ethyl-2-hydroxyphenyl)-1-hexanone

(	$C_{14}H_{20}O_2$	mol. wt. 220.31
OH CO(CH ₂ ) ₄ CH ₃	Syntheses -Obtained by Fries rearrangemenn-caproate (1 equiv.), *in the presence of aluminium of in nitrobenzene at 25° for 6 h (1) *in the presence of aluminium of in refluxing carbon disulfide for at 130° after solvent elimination	hloride (1.3 equiv.) (88 %) [2801]; nloride (2.8 M), first or 2 h, then for 2 h

b.p.₁₈ 170° [2801].

#### 2,4-Dinitrophenylhydrazone

m.p. 146° [2801].

#### Methyl ether

 $C_{15}H_{22}O_2$ 

 $C_{20}H_{24}N_4O_5$ 

mol. wt. 234.34

mol. wt. 400.43

-Obtained by reaction of dimethyl sulfate with 1-(4-ethyl-2-hydroxyphenyl)-1-hexanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating on the water bath for 5 h (90 %) [2801].

b.p.36 165° [2801].

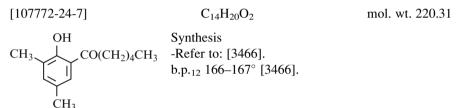
#### 1-(5-Ethyl-2-hydroxyphenyl)-1-hexanone

$$\begin{array}{ccc} C_{14}H_{20}O_2 & \mbox{mol. wt. } 220.31 \\ \hline OH & Synthesis \\ -Obtained by Fries rearrangement of 4-ethylphenyl caproate with aluminium chloride at 100° for 2 h \\ (82 \%) [2800]. \\ b.p._{10} 162° [2800]. \end{array}$$

**2,4-Dinitrophenylhydrazone**  $C_{20}H_{24}N_4O_5$  mol. wt. 400.43

m.p. 178° [2800].

#### 1-(2-Hydroxy-3,5-dimethylphenyl)-1-hexanone



#### 1-(2-Hydroxy-3,6-dimethylphenyl)-1-hexanone

#### 1-(2-Hydroxy-4,5-dimethylphenyl)-1-hexanone

[856349-95-6]	$C_{14}H_{20}O_{2}$	2		mol. wt. 22	20.31
CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₂ CO(CH ₂ ) ₄ CH ₃	Synthesis -Obtained 1 3,4-dimethylpl ride at 110° w b.p. ₄ 180° [311	henyl ithout	caproate	rearrangement with aluminium (84 %) [3117].	of chlo-

# 2,4-Dinitrophenylhydrazone C₂₀H₂₄N₄O₅

m.p. 197° [3117].

mol. wt. 400.43

mol. wt. 400.43

#### 1-(2-Hydroxy-4,6-dimethylphenyl)-1-hexanone

$C_{14}H_{20}O_2$			mol. wt. 22	0.31	
CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃	*in the pres in nitroben *in the prese in refluxing	ylpheny ence of zene at ence of g carboi	aluminiu 25° for 6 aluminiu n disulfide	rearrangement ate (1 equiv.), m chloride (1.3 equ h (83 %) [2801]; n chloride (2.8 M), e for 2 h, then for 2 on (75 %) [2801].	first

b.p.20 180° [2801].

Methyl ether	$C_{15}H_{22}O_2$	mol. wt. 234.34
--------------	-------------------	-----------------

-Obtained by reaction of dimethyl sulfate with 1-(2-hydroxy-4,6-dimethylphenyl)-1-hexanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating on the water bath for 5 h (76 %) [2801].

b.p.56 180° [2801].

2,4-Dinitrophenylhydrazone	$C_{20}H_{24}N_4O_5$	mol. wt. 400.43
----------------------------	----------------------	-----------------

m.p. 190° [2801].

#### 1-(2-Hydroxy-4-methylphenyl)-5-methyl-1-hexanone

[88555-60-6]	$C_{14}H_{20}O_2$	mol. wt. 220.31
CH ₃ CO(CH ₂ ) ₃ CH(CH ₃ ) ₂	Synthesis -Obtained by reaction of isohe with m-cresol in the presen- chloride in nitrobenzene at 4	ce of aluminium
¹ H NMR [98], IR [98].		

[90], IK [90]

#### $C_{20}H_{24}N_4O_5$ 2,4-Dinitrophenylhydrazone

-Refer to: [98].

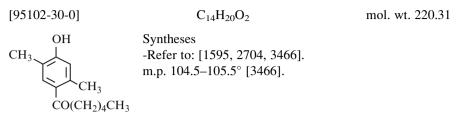
m.p. 208-209° [98].

Methyl ether	[88555-61-7]	$C_{15}H_{22}O_2$	mol. wt. 234.34
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-Obtained by treatment of the title ketone with dimethyl sulfate in the presence of sodium hydroxide in refluxing ethanol for 3 h [98].

¹H NMR [98], IR [98].

#### 1-(4-Hydroxy-2,5-dimethylphenyl)-1-hexanone



USE: Colour developer for thermal recording materials [1595].

#### 1-(4-Hydroxy-3,5-dimethylphenyl)-1-hexanone

[148516-07-8]	$C_{14}H_{20}O_2$		mol. wt. 220.31
CH ₃ CH ₃ CO(CH ₂ ) ₄ CH ₃	Syntheses -Obtained by reac 2,6-dimethylphenol previously [2871], ( -Also refer to: [244,	according to the 50 %) [119].	
m.p. 108–109° [346	5], 97.5–99° [119], 97–9	99° [244]; ¹ H NM	IR [119], IR [119].
Methyl ether	[104008-49-3]	$C_{15}H_{22}O_2$	mol. wt. 234.34

-Refer to: [3004].

#### 1-(2,4-Dihydroxy-3,5-dimethylphenyl)-1-hexanone

(Tetrahydrosorbicillin)

	OH
CH ₃	CO(CH ₂ ) ₄ CH ₃
HO'	Ý
	CH ₃

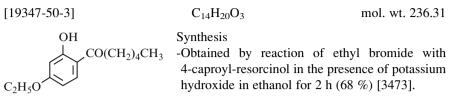
Syntheses -Obtained by reaction of caproic acid with 3,5-dimethyl-resorcinol in the presence of boron trifluoride etherate at 120° for 1 h [2269].

-Also obtained by hydrogenation of sorbicillin (21 %) [3137].

-Also refer to: [743].

pale yellow crystals [2269]; m.p. 69–70° [743], 66–69° [3137], 66–68° [2269]; ¹H NMR [2269, 3137], ¹³C NMR [2269], IR [2269, 3137], UV [3137], MS [2269, 3137].

#### 1-(4-Ethoxy-2-hydroxyphenyl)-1-hexanone



b.p._{0.5} 142–144° [3473].

#### 1-(5-Ethoxy-2-hydroxyphenyl)-1-hexanone

[140943-32-4]	$C_{14}H_{20}O_3$	mol. wt. 236.31
OH CO(CH ₂ ) ₄ CH ₃	Synthesis -Refer to: [285]. <b>Oxime</b> [140943-19-7]	
OC ₂ H ₅	C ₁₄ H ₂₁ NO ₃ -Refer to: [285].	mol. wt. 251.33

#### 1-(5-Ethyl-2,4-dihydroxyphenyl)-1-hexanone

[95102-17-3] 
$$C_{14}H_{20}O_3$$
 mol. wt. 236.31  
OH Syntheses  
-Refer to: [2651, 2704].  
b.p.₁₀ 220–225° [2651]; m.p. 93–94° [2651].

USE: Colour developer, for thermal recording materials [2704].

#### 1-(2-Hydroxy-4-methoxy-3-methylphenyl)-1-hexanone

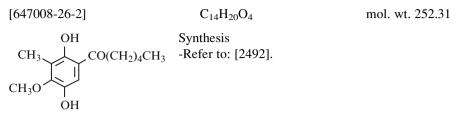
[647008-30-8]  $C_{14}H_{20}O_3$  mol. wt. 236.31 OH Synthesis CH₃ CO(CH₂)₄CH₃ -Refer to: [2492]. CH₃O

USE: Preparation of trichodimerol and related vertinoid polyketides [2492].

Acetate[647008-31-9] $C_{16}H_{22}O_4$ mol. wt. 278.35

USE: Preparation of trichodimerol and related vertinoid polyketides [2492].

#### 1-(2,5-Dihydroxy-4-methoxy-3-methylphenyl)-1-hexanone



#### 1-(4-Ethoxy-2,6-dihydroxyphenyl)-1-hexanone

BIOLOGICAL ACTIVITY: Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

#### 1-(2-Hydroxy-4,6-dimethoxyphenyl)-1-hexanone

[1142936-17-1]	$C_{14}H_{20}O_4$	mol. wt. 252.31
CH ₃ O CH ₃ O OCH ₃ O OCH ₃ O	1-(2,4,6-trihydroxyphen	of dimethyl sulfate with yl)-1-hexanone in the carbonate in acetone at hitrogen (32 %) [2786].

m.p. 74–75° [2786]; ¹H NMR [2786], ¹³C NMR [2786], MS [2786].

#### 1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-hexanone

[134081-94-0]	$C_{14}H_{20}O_5$	mol. wt. 268.31
CH ₃ O OH CH ₃ O OH OCH ₃ O	3,4-dimethoxy-2-(4-m phenyl]-1-hexanone wi	tment of 1-[6-hydroxy- nethylphenylsulfonyloxy) ith potassium carbonate in 1-3 h (87 %) [1353].

m.p. 96.5–97° [1353]; ¹H NMR [1353].

Dibenzyl ether	$C_{28}H_{32}O_5$	mol. wt. 448.56

-Refer to: [1353].

# 1-(8-Hydroxy-5-quinolinyl)-1-hexanone

[246531-45-3]	$C_{15}H_{17}NO_2$	mol. wt. 243.31
OH V CO(CH ₂ ) ₄ CH ₃	Synthesis -Refer to: [1722]. <b>Hydrochloride</b> C ₁₅ H ₁₇ NO ₂ , HCl	mol. wt. 279.77
m.p. 172–174	° [1725]; ¹ H NMR [1725], IR [1725].	

USE: Ion-flotation collector [1725].

C ₁₅ H ₁₆ LiNO ₂	mol. wt. 249.24
	C ₁₅ H ₁₆ LiNO ₂

-Refer to: [1722].

# 1-(8-Hydroxy-7-quinolinyl)-1-hexanone

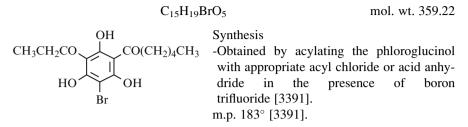
[246531-46-4]	$C_{15}H_{17}NO_2$	mol. wt. 243.31
CH ₃ (CH ₂ ) ₄ CO	Synthesis -Refer to: [1722].	

#### 1-(2,4-Dihydroxy-3-quinolinyl)-1-hexanone

[54289-79-1]	$C_{15}H_{17}NO_3$	mol. wt. 259.30
OH OH	2,4-dihydroxyquinoline in	of caproyl chloride with n the presence of aluminium on the steam bath for 3 h
m.p. 183–184° [3123];	UV [3123].	

BIOLOGICAL ACTIVITY: Antibacterial properties (Staphylococcus aureus and Escherichia coli) [3123].

#### 1-[3-Bromo-5-(1-oxopropyl)-2,4,6-trihydroxyphenyl]-1-hexanone



### 1-[2-(Acetyloxy)-4-hydroxyphenyl]-2-methyl-1-hexanone (-)

[406174-72-9]	$C_{15}H_{20}O_4$	mol. wt. 264.32
HO	Synthesis -Obtained by enzymatic deacetylation of diester in PPL (54 %) [2829].	enantioselective the presence of

oil [2829];

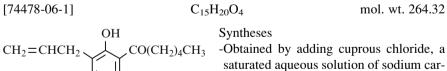
¹H NMR [2829], ¹³C NMR [2829], IR [2829], UV [2829], MS [2829]; TLC [2829];  $(\alpha)_D^{25} = -41.6^{\circ}$  (chloroform) [2829].

#### 1-[2,5-Dihydroxy-3,4,6-trimethylphenyl]-1,5-hexanedione

 $C_{15}H_{20}O_{4}$ mol. wt. 264.32 OH CH₃ CH₃ CH₃ OH CH₃ CO(CH₂)₃COCH₃ -Refer to: [2642]. ¹H NMR [2642], ¹³C NMR [2642].

#### 1-[2,4,6-Trihydroxy-3-(2-propenyl)phenyl]-1-hexanone

2-Caproyl-4-(propen-2-yl)phloroglucinol (20) [1026].



-Obtained by adding cuprous chloride, a saturated aqueous solution of sodium carbonate and allyl chloride to a solution of phlorocaprophenone in ethyl ether at r.t., then to keep the mixturefor 12 h at this temperature [1026].

-Also obtained by reaction of allyl chloride with phlorovalerophenone in an alkaline two-phase (aqueous-ether) system and catalyzed by CuCl [838, 3193]. -Also refer to: [2111].

m.p. 118–120° [3193]; ¹³C NMR [1026, 3193], IR [1026].

BIOLOGICAL ACTIVITY: Bactericidal and fungicidal [1026, 3193]; Fungicide [2111].

#### 1-(5-Chloro-2-hydroxy-4-methylphenyl)-2-ethyl-1-hexanone

$$[57080-96-3] C_{15}H_{21}ClO_2 mol. wt. 268.78$$
  
OH Synthesis  
-Refer to: [1903].  
CH₃ Cl  $L_{2}H_5$  b.p.₁₋₂ 150–160° [1903].

**Oxime**[57080-95-2] $C_{15}H_{22}CINO_2$ mol. wt. 283.80

-Refer to: [1903].

#### 1-(2-Hydroxy-3-nitrophenyl)-3,5,5-trimethyl-1-hexanone

[176043-79-1]	$C_{15}H_{21}NC$	<b>D</b> ₄	mol. wt. 279.33
NO ₂ COCH ₂ -	$CH_3$ - $C - CH_3$ $CH_3$	Synthesis -Refer to: [628].	

#### 1-(2-Hydroxy-5-nitrophenyl)-3,5,5-trimethyl-1-hexanone

 $[154737-34-5] C_{15}H_{21}NO_4$ mol. wt. 279.33 OH  $CH_3$   $CH_3$   $CH_3$  Syntheses -Obtained by treatment of 1-(2-hydroxyphenyl)-3,5,5-trimethyl-1-hexanone with sodium nitrite in trifluoroacetic acid for 2 h at r.t. (45 %) [628]. -Refer to: [417].

m.p. 94–96° [628]; ¹H NMR [628].

#### 1-(2,4,6-Trihydroxy-3-nitro-5-propylphenyl)-1-hexanone

[119692-02-3]	$C_{15}H_{21}NO_6$	mol. wt. 311.33
$\begin{array}{c} OH\\ NO_2 \\ HO \\ C_3H_7 \end{array} CO(CH_2)_4CH_3 \\ OH \\ C_3H_7 \end{array}$	Syntheses -Obtained by adding a mix and acetic acid to the hydroxy-3-propyl-phenyl) acid at 60° for 30 min (30	solution of 1-(2,4,6-tri- -1-hexanone in acetic

-Also refer to: [153].

m.p. 52–53° [3414]; ¹H NMR [3414], IR [3414], MS [3414].

BIOLOGICAL ACTIVITY: Germination inhibitory activity [3414]; PET inhibitory activity [3414]; Effect on gibberellin-inducible  $\alpha$ -amylase synthesis in barley aleurone cells [153].

#### 2-Ethyl-1-(2-hydroxy-4-methylphenyl)-1-hexanone

[57080-99-6]	$C_{15}H_{22}O_2$	mol. wt. 234.34
ОН СН3 СН3	Synthesis -Refer to: [1903]. <b>Oxime</b> [57080-97-4] C ₁₅ H ₂₃ NO ₂	mol. wt. 249.35

-Refer to: [1903].

#### 2-Ethyl-1-(2-hydroxy-5-methylphenyl)-1-hexanone

[74604-17-4]	$C_{15}H_{22}O_2$	mol. wt. 234.34
$\bigcup_{\substack{I \\ CH_3}}^{OH} CO - CH - C_4H_9$	Synthesis -Obtained by Fries rearrangement of 2-ethylhexanoate (b.p. _{0.02} 90°) [2520]. b.p. _{0.02} 90° [2520]; IR [2520], UV [2520].	4-methylphenyl

**Oxime** [51528-15-5] C₁₅H₂₃NO₂ mol. wt. 249.35

-Refer to: [2520, 3445].

crystals [2520]; m.p. 64–66° [2520]; IR [2520], UV [2520].

USE: Solvent extraction of copper (II) [2520].

# 1-(2-Ethyl-6-hydroxy-4-methylphenyl)-1-hexanone

	$C_{15}H_{22}O_2$	mol. wt. 234.34
CH ₃ CH ₃ OH CO(CH ₂ ) ₄ CH ₃ CO(CH ₂ ) ₄ CH ₃	5-methylphenyl capro *without solvent at 13	s rearrangement of 3-ethyl- bate with aluminium chloride, 0° for 2 h (82 %) [2802]; ° for 6 h (83 %) [2802].

b.p.9 178° [2802].

#### Methyl ether

**er**  $C_{16}H_{24}O_2$  mol. wt. 248.37

-Obtained by methylation of the above ketone in the usual way (75 %) [2802].

b.p.48 215° [2802].

# 1-[4-Hydroxy-3-(1-methylethyl)phenyl]-1-hexanone

[95102-36-6]	$C_{15}H_{22}O_2$	mol. wt. 234.34
OH CH(CH ₃ ) ₂	Syntheses -Refer to: [1595, 2704]. USE: Colour developer for thermal recording	materials [1595].
$CO(CH_2)_4CH_3$		

# 1-(4-Hydroxyphenyl)-3,5,5-trimethyl-1-hexanone

$C_{15}H_{22}$	₂ O ₂	mol. wt. 234.34
$\bigcup_{CO-CH_2}^{CH_3} \bigcup_{CH_3}^{CH_3} \bigcup_{CH_2-CH_2-CH_2-CH_3}^{CH_3} \bigcup_{CH_3}^{CH_3}$	Synthesis -Refer to: [698]. <b>Methyl ether</b> $C_{16}H_{24}O_2$ -Obtained by reaction of with 3,5,5-trimethylhexanal caproaldehyde) in the prese (2 mol%), Pd phosphine (6 and pyrrolidine in DMA a (73 %) [698].	(3,5,5-trimethyl- ence of Pd(dba) ₂ mol%), 4 Å MS

¹H NMR [698], ¹³C NMR [698], IR [698], MS [698].

#### 1-(2,4-Dihydroxy-5-propylphenyl)-1-hexanone

$$\begin{array}{c} C_{15}H_{22}O_{3} \\ OH \\ + CO(CH_{2})_{4}CH_{3} \\ + OH \\ CH_{2}CH_{2}CH_{2}CH_{3} \end{array} \qquad \begin{array}{c} \text{mol. wt. 250.34} \\ \text{Synthesis} \\ - \text{Refer to: [2651].} \\ \text{b.p.}_{9} \ 225 - 230^{\circ} \ [2651]; \\ \text{m.p. 53^{\circ} [2651].} \end{array}$$

#### 1-(2-Hydroxy-4-propoxyphenyl)-1-hexanone

 $\begin{array}{cccc} [92730-24-0] & C_{15}H_{22}O_3 & \mbox{mol. wt. } 250.34 \\ OH & Synthesis \\ -Obtained by reaction of propyl bromide with \\ 4-caproyl-resorcinol in the presence of potassium \\ Hydroxide in ethanol for 2 h (60 \%) [3473]. \end{array}$ 

b.p.₂ 169–171° [3473]; m.p. 28° [3473].

#### 1-(3-Ethyl-2,4-dihydroxyphenyl)-2-methyl-1-hexanone

 $[820215-94-9] C_{15}H_{22}O_3 mtext{mol. wt. } 250.34$ 

 $\begin{array}{ccc} OH & CH_3 & Isolation from natural sources \\ C_2H_5 & CO-CH-C_4H_9 & -From unidentified fungal strain \\ HO & CRM-51006 [2317]. \\ -Novel strain MT90049 (KCTC 18043p), novel compound produced therefrom and use thereof [65]. \end{array}$ 

¹H NMR [2317], ¹³C NMR [2317].

BIOLOGICAL ACTIVITY: CRM-51006 as new phospholipase C inhibitor [65, 2317].

#### 1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-hexanone

[134081-63-3]	$C_{15}H_{22}O_5$	mol. wt. 282.34
CH ₃ O CH ₃ O CH ₃ O OCH ₃	crude 2,3,4,6-tetrame	selective demethylation of thoxyhexanophenone with in acetonitrile at $50^{\circ}$ for

m.p. 62.5–64° [1353]; ¹H NMR [1353].

mol. wt. 372.46

**p-Toluenesulfonate** [134081-78-0] C₂₂H₂₈O₇S mol. wt. 436.53

-Obtained by reaction of p-toluenesulfonyl chloride with 2-hydroxy-3,4,6-trimethoxy-hexanophenone in the presence of potassium carbonate in refluxing acetone for 6-14 h (96 %) [1353].

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m.p. 91.5–92.5° [1353]; <sup>1</sup>H NMR [1353].
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Methyl ether	$C_{16}H_{24}O_5$	mol. wt. 296.36
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-Obtained by reaction of caproyl chloride with 1,2,3,5-tetramethoxybenzene in the presence of aluminium chloride in ethyl ether at  $0^{\circ}$  for 30 min [1353].

#### 1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-hexanone

[134081-70-2]	$C_{15}H_{22}O_5$	mol. wt. 282.34
CH ₃ O CH ₃ O OCH ₃ OCH ₃	Syntheses -Obtained by hydrogenatio 2,3,4-trimethoxycaprophe dium on charcoal in ethy until the uptake of hydroge -Also refer to: [1351].	none over 10 % palla- l acetate/methanol (1:1)

C22H28O5

m.p. 46.5–48° [1353]; ¹H NMR [1353].

# **Benzyl ether**

-Refer to: [1353].

#### 1-(3-Amino-2-hydroxyphenyl)-3,5,5-trimethyl-1-hexanone

[176043-97-3]	$C_{15}H_{23}NC$	$D_2$	mol. wt. 249.35
OH NH ₂ COCH ₂ - CH - CH CH ₃	$\begin{array}{c} CH_3 \\ H_2 - \overset{1}{C} - CH_3 \\ H_3 \end{array}$	Synthesis -Refer to: [628].	

#### 1-(5-Amino-2-hydroxyphenyl)-3,5,5-trimethyl-1-hexanone

$$[154737-35-6] C_{15}H_{23}NO_2 mol. wt. 249.35$$
  
OH  
COCH₂ - CH - CH₂ -  $\stackrel{I}{C}$  - CH₃  
CH₃ - Refer to: [417, 628].  
NH₂

#### 1-(2-Acetyl-7-hydroxy-4-benzofuranyl)-1-hexanone

[59445-81-7]	$C_{16}H_{12}$	$_{3}O_{4}$	mol. wt. 274.32
OH O CO(CH ₂ ) ₄ CH ₃	•	nent of its methyl ethe in methylene chloride	
Methyl ether	[59445-72-6]	$C_{17}H_{20}O_4$	mol. wt. 288.34

-Obtained by reaction of caproyl chloride with 2-acetyl-7-methoxybenzofuran with aluminium chloride (2.4 mol) in methylene chloride at r.t. for 24 h (90 %) [682].

m.p. 101° [682].

CH₃

#### 1-(8-Hydroxy-2-methyl-5-quinolinyl)-1-hexanone

$$[217815-21-9] C_{16}H_{19}NO_2 mtext{mol. wt. } 257.33$$

Synthesis

Synthesis

reaction -Obtained by of hexanovl chloride with 8-hydroxyquinaldine in the presence of aluminium chloride in nitrobenzene or 1,2-dichloroethane first at 10-30°, then at 70-80° for 20 h [2261].

m.p. 60–62° [2261]; MS [2261].

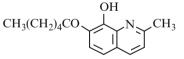
#### 1-(8-Hydroxy-2-methyl-7-quinolinyl)-1-hexanone

 $CO(CH_2)_4CH_3$ 

OH

C16H10NO2

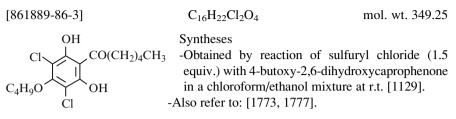
mol. wt. 257.33



-Obtained by reaction of hexanoyl chloride with 8-hydroxyquinaldine in the presence of aluminium chloride in nitrobenzene or 1,2-dichloroethane first at  $10-30^{\circ}$ , then at 70-80° for 20 h [2261].

m.p. 56° [2261]; MS [2261].

# **1-(4-Butoxy-3,5-dichloro-2,6-dihydroxyphenyl)-1-hexanone** (*Bu-DIF-1*)



Yellow amorphous solid [1129]; MS [1129].

BIOLOGICAL ACTIVITY: DIF-1(3 M); Dictyostelium differentiation-inducing factor-1 derivs. promotion of glucose consumption in mammalian cells [1777]; DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

# 1-[2-(Acetyloxy)-4-hydroxyphenyl]-2-ethyl-1-hexanone

$[251463-60-2]  C_{16}H_{22}O_4  mtext{mol. wt. } 278.$
---------------------------------------------------------

OH  $OCOCH_3$   $CO - CH - C_4H_9$   $C_2H_5$ 

Synthesis -Obtained by selective deacetylation of 2,4-diacetoxy-phenyl-1-ethylpentyl ketone mediated by porcine pancreatic lipase (PPL) in THF at 42–45° for 48 h in the presence of n-butanol (45 %) [2517].

viscous oil [2517]; ( $\alpha$ )_D²³ =  $-6.6^{\circ}$  (chloroform) [2517]; ¹H NMR [2517], ¹³C NMR [2517], IR [2517], UV [2517], MS [2517].

# 1-[3-(2-Butenyl)-2,4,6-trihydroxyphenyl]-1-hexanone

[74478-07-2]	$C_{16}H_{2}$	₂ O ₄	mol. wt. 278.35
CH ₃ CH=CHCH ₂	OH CO(CH ₂ ) ₄ CH ₃	Synthesis -Refer to: [2111].	
но		BIOLOGICAL ACTIV [2111].	VITY: Fungicide

#### 1-[2,4,6-Trihydroxy-3-(2-methyl-2-propenyl)phenyl]-1-hexanone

[74478-08-3] 
$$C_{16}H_{22}O_4$$
 mol. wt. 278.35  
 $CH_2 = C - CH_2$  OH  $CO(CH_2)_4CH_3$  -Refer to: [2111].  
HO OH USE: Fungicide [2111].

# 1-(6,7-Dihydroxy-5-propyl-1,3-benzodioxol-4-yl)-1-hexanone

$$\begin{array}{ccc} C_{16}H_{22}O_5 & \text{mol. wt. } 294.35 \\ OH & Synthesis \\ OH & -Refer to: [2179]. \\ \hline Dimethyl ether & [82652-26-4] \\ CO(CH_2)_4CH_3 & C_{18}H_{26}O_5 & \text{mol. wt. } 322.40 \end{array}$$

-Obtained by reaction of caproyl chloride with dihydrodillapiole in the presence of fused zinc chloride in methylene chloride for 3-4 h in an ice bath (70 %) [2179].

colourless solid [2179]; ¹H NMR [2179], IR [2179].

USE: Synergistic insecticidal activity of, with pyrethrum [2179].

#### 1-[3-Bromo-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-hexanone

[101254-34-6]	$C_{16}H_{23}BrO_2$	mol. wt. 327.26
$\begin{array}{c} OH\\ Br \\ \downarrow \\ C(CH_3)_3 \end{array} CH_2)_4CH_3$	Synthesis -Preparation by Fries rearrang butylphenyl caproate in the chloride at 110° for 2 h (68 b.p. ₉ 210° [3113].	e presence of aluminium
2,4-Dinitrophenylhydraz	one $C_{22}H_{27}BrN_4O_5$	mol. wt. 507.38

$2,4$ -Diniti opnenymyurazone $C_{22}n_{27}Dn_{4}O_5$ mol. wt. $507$ .	2,4-Dinitrophenylhydrazone	$C_{22}H_{27}BrN_4O_5$	mol. wt. 507.3
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m.p. 136° [3113].

Allyl ether	[108719-92-2]	$C_{19}H_{27}BrO_2$	mol. wt. 367.33
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-Obtained by reaction of allyl bromide with the title ketone in the presence of potassium carbonate in refluxing acetone for 8 h (76 %) [3113].

b.p.₁ 165° [3113].

USE: Insecticide [3113].

1 4 20 4 25

mol. wt. 322.40

# 2,4-Dinitrophenylhydrazone of the allyl ether $C_{25}H_{31}BrN_4O_5$ mol. wt. 547.75

m.p. 129° [3113].

# 1-[3-Chloro-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-hexanone

$$\begin{array}{cccc} [101254-66-4] & C_{16}H_{23}ClO_2 & \text{mol. wt. } 282.81 \\ \\ OH & Synthesis \\ Obtained by Fries rearrangement of 2-chloro-4-tert-butylphenyl caproate with aluminium chloride at 110° (71 %) [3119]. \\ \\ C(CH_3)_3 & b.p._{13} 163° [3119]. \end{array}$$

**2,4-Dinitrophenylhydrazone** [102475-97-8]  $C_{22}H_{27}CIN_4O_5$  mol. wt. 462.93

m.p. 163° [3119].

# 1-(4-Butoxy-3-chloro-2,6-dihydroxyphenyl)-1-hexanone

(*Bu-DIF-3*)

[861889-94-3]

C₁₆H₂₃ClO₄ mol. wt. 314.81

Cl  $CO(CH_2)_4CH$   $CO(CH_2)_4CH$   $C_4H_9O$  OH

Syntheses

CO(CH₂)₄CH₃ -Obtained by reaction of sulfuryl chloride (1.5 equiv.) with 4-butoxy-2,6-dihydroxycaprophenone OH in a chloroform/ethanol mixture at r.t. [1129]. -Also refer to: [1773, 1777].

Yellow amorphous solid [1129]; MS [1129].

BIOLOGICAL ACTIVITY: Bu-DIF-3; Dictyostelium differentiation-inducing factor-1 derivs. promotion of glucose consumption in mammalian cells [1777]; DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

# 1-[4-Hydroxy-3-(1,1-dimethylethyl)phenyl]-1-hexanone

$$C_{16}H_{24}O_2$$
 mol. wt. 248.37

OH C(CH₃)₃ CO(CH₂)₄CH₃

Synthesis -Refer to: [2704] (Japanese patent). m.p. 114° [2704]. USE: As colour developer [2704].

#### 1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-hexanone

[856349-97-8]	$C_{16}H_{24}O_2$	mol. wt. 248.37
(CH ₃ ) ₂ CH CO(CH ₂ ) ₄ CH ₃	Synthesis -Obtained by Fries rearrang caproate with aluminium (71 %) [2803].	
b.p. ₃₈ 176° [2803].		
<b>2,4-Dinitrophenylhydrazone</b> m.p. 188° [2803].	$C_{22}H_{28}N_4O_5$	mol. wt. 428.49

#### 1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-hexanone

b.p.₁₃ 220° [2660]; m.p. 103° [2660], 101° [2704].

USE: As colour developer [2704]; In preparation of thermographic recording material [1595].

# Methyl ether (VII) $C_{17}H_{26}O_2$ mol. wt. 262.39

-Obtained by reaction of caproyl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (70 %) [2660].

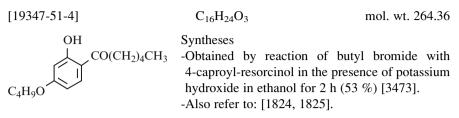
b.p.₁₆ 196–198° [2660];  $n_D^{22.5} = 1.5205$  [2660].

#### 1-(2-Hydroxy-4-methylphenyl)-3,5,5-trimethyl-1-hexanone

[52122-73-3]	$C_{16}H_{24}O_2$		mol. wt. 248.37
OH CH ₃ COCH ₂ -CH-CH ₂ CH ₃ COCH ₂ -CH-CH ₂	$- \overset{CH_3}{-} \overset{-}{C} - CH_3$ $\overset{-}{C} H_3$	Syntheses -Refer to: [108, 109 <b>Oxime</b> [52122-64 C ₁₆ H ₂₅ NO ₂	]. -2] mol. wt. 263.38

USE: In copper extraction from aqueous solutions [108]; Chelation with, of metals, [109].

# 1-(4-Butoxy-2-hydroxyphenyl)-1-hexanone



b.p.2 176-180° [3473].

# 1-(5-Butoxy-2-hydroxyphenyl)-1-hexanone

[140943-37-9]	$C_{16}H_{24}O_3$	mol. wt. 264.36
OH CO(CH ₂ ) ₄ CH ₃	Synthesis -Refer to: [285]. <b>Oxime</b> [140943-23-3]	
OC ₄ H ₉	C ₁₆ H ₂₅ NO ₃	mol. wt. 279.38

-Refer to: [285].

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

# 1-(4-Butoxy-2,6-dihydroxyphenyl)-1-hexanone

[861889-81-8]	$C_{16}H_{24}O_4$	mol. wt. 280.36
OH	Synthesis	
CO(CH ₂ ) ₄ CH	-Preparation by reaction	of caproyl chloride with
[ζ Ι]	1 0	yl ether in the presence of
C ₄ H ₉ O OH	aluminium chloride in	methylene chloride at
	r.t. [1129].	

BIOLOGICAL ACTIVITY: Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

# 1-[2,4,6-Trihydroxy-3-(2-methylpropyl)phenyl]-1-hexanone

 $[66711-56-6] C_{16}H_{24}O_4 mol. wt. 280.36$   $(CH_3)_2CHCH_2 + CO(CH_2)_4CH_3 -Refer to: [2719].$  HO OH BIOLOGICAL ACTIVITY: Fungicide [2719].

#### 1-[3-Amino-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-hexanone

[85052-50-2]	$C_{16}H_{25}NO_2$	mol. wt. 263.38
OH NH ₂ CO(CH ₂ ) ₄ CH ₃	Synthesis -Refer to: [2105].	
C(CH ₃ ) ₃	Hydrochloride $[85052-49-9]$ $C_{16}H_{25}NO_2$ , HCl-Refer to: [2105].	mol. wt. 299.84

# 1-(2',3'-Difluoro-4'-hydroxy[1,1'-biphenyl]-4-yl)-1-hexanone

$C_{17}H_{18}F_2$	$O_2$	mol. wt. 204.34
HO-CO(CH ₂ ) ₄ CH ₃	Synthesis -Refer to: [25 <b>Octyl ether</b> $C_{26}H_{34}F_2O_2$	578]. [126163-53-9] mol. wt. 416.55

USE: Preparation of, for liquid-crystal media [2578].

# **1-[3,5-Dichloro-4-(cyclopentyloxy)-2,6-dihydroxyphenyl]-1-hexanone** (*CP-DIF-1*)

[861889-87-4]

 $C_{17}H_{22}Cl_2O_4$  mol. wt. 361.26

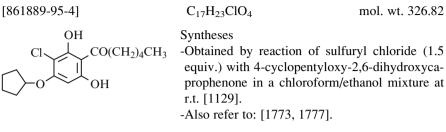
OH
Cl CO(CH ₂ ) ₄ CH ₃
С ОН
Cl

Syntheses -Obtained by reaction of sulfuryl chloride (1.5 equiv.) with 4-cyclopentyloxy-2,6-dihydroxycaprophenone in a chloroform/ethanol mixture at r.t. [1129]. -Also refer to: [1773, 1777].

Yellow amorphous solid [1129]; MS [1129].

BIOLOGICAL ACTIVITY: CP-DIF-1; Dictyostelium differentiation-inducing factor-1 derivs. promotion of glucose consumption in mammalian cells [1777]; DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

# **1-[3-Chloro-4-(cyclopentyloxy)-2,6-dihydroxyphenyl]-1-hexanone** (*CP-DIF-3*)



Yellow amorphous solid [1129]; MS [1129].

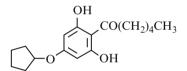
BIOLOGICAL ACTIVITY: CP-DIF-3; Dictyostelium differentiation-inducing factor-1 derivs. promotion of glucose consumption in mammalian cells [1777]; DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

# 1-[4-(Cyclopentyloxy)-2,6-dihydroxyphenyl]-1-hexanone

[861889-82-9]

 $C_{17}H_{24}O_4$ 

mol. wt. 292.37



Synthesis -Preparation by reaction of caproyl chloride with phloroglucinol monocyclopentyl ether in the presence of aluminium chloride in methylene chloride at r.t. [1129].

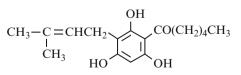
BIOLOGICAL ACTIVITY: Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

# 1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-hexanone

2-Caproyl-4-(3-methylbuten-2-yl)phloroglucinol (5)

 $C_{17}H_{24}O_4$ 

mol. wt. 292.37



# Syntheses

-Obtained by adding cuprous chloride, a saturated aqueous solution of sodium carbonate and prenyl bromide to a solution of phlorocaprophenone in ethyl ether at r.t., then to keep the mixture for 12 h at this temperature [1026]. -Also prepared by adding prenyl bromide to a suspension of the sodium salt of phlorocaprophenone in benzene, then the mixture obtained was refluxed for 3 h [1026].

-Also obtained by reaction of prenyl chloride with phlorovalerophenone in an alkaline two-phase (aqueous-ether) system and catalyzed by CuCl [838, 3193].

-Also prepared by adding at 5° a solution of sodium methoxide in methanol, followed by isopentenyl bromide in ethyl ether to a solution of phlorocaprophenone in benzene and ethyl ether, then the mixture was stirred at r.t. for 6 h (15 %) [2113].

m.p. 135° [2113], 122–123° [3193].

**N.B.**: One of the reported melting point is obviously wrong. ¹³C NMR [1026, 3193], IR [1026].

BIOLOGICAL ACTIVITY: Antifungal [2113]; Bactericidal and fungicidal [1026, 3193].

# 1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methylphenyl]-1-hexanone

C ₁₇	H ₂₆ O ₂ m	nol. wt. 262.39
(CH ₃ ) ₃ C CO(CH ₂ ) ₄ CH ₃ CH ₃	Syntheses -Obtained by Fries rearrangement of 5-methylphenyl caproate, *in the presence of aluminium equiv.) in nitrobenzene at 25° for [3118];	chloride (1.5

*in the presence of aluminium chloride (3 equiv.) at 110° for 2 h (78 %) [3118].

b.p.8 146° [3118].

**2,4-Dinitrophenylhydrazone**  $C_{23}H_{30}N_4O_5$  mol. wt. 442.52

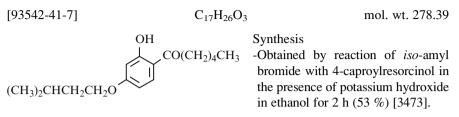
m.p. 197° [3118].

### 2-Ethyl-1-[2-hydroxy-4-(1-methylethyl)phenyl]-1-hexanone

[57080-92-9]	$C_{17}H_{26}O_2$	mol. wt. 262.39
6/ Y	$\begin{array}{c} \text{Synthesis} \\ \text{CH}-\text{C}_{4}\text{H}_{9} \\ \text{C}_{2}\text{H}_{5} \\ \end{array} \begin{array}{c} \text{-Refer to: [1903].} \\ \textbf{Oxime} \\ \text{[57080-91-8]} \\ \text{C}_{17}\text{H}_{27}\text{NO}_{2} \end{array}$	mol. wt. 277.41

-Refer to: [1903].

# 1-[2-Hydroxy-4-(isopentyloxy)phenyl]-1-hexanone



b.p._{0.5} 161–162° [3473]; m.p. 36–37° [3473]; IR [3473].

### 1-[2-Hydroxy-4-(pentyloxy)phenyl]-1-hexanone

 $[101002-32-8] C_{17}H_{26}O_3 mol. wt. 278.39$ OH CO(CH₂)₄CH₃ Synthesis -Obtained by reaction of pentyl bromide with 2,4-dihydroxycaprophenone in the presence of potassium carbonate in refluxing acetone for 20 h [284].

m.p. 41-42° [284].

Oxime	[101002-19-1]	$C_{17}H_{27}NO_3$	mol. wt. 293.41

-Obtained by reaction of hydroxylamine hydrochloride with 1-[2-hydroxy-4-(pentyloxy)phenyl]-1-hexanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

-Also refer to: [2742].

m.p. 80° [284, 3077]; ¹H NMR [284].

BIOLOGICAL ACTIVITY: Topical preparations comprising at least one aryloxime and bisabolol [483]; As lipoxygenase inhibitor [3077].

### 1-[2,4,6-Trihydroxy-3-(3-methylbutyl)phenyl]-1-hexanone

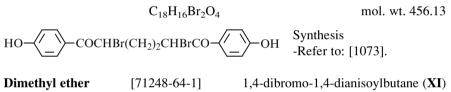
[74478-05-0]	$C_{17}H_{26}C_{17}$	$\mathbf{D}_4$	mol. wt. 294.39
(CH ₃ ) ₂ CHCH ₂ CH ₂ HC	OH CO(CH ₂ ) ₄ CH ₃ OH	Syntheses -Preparation by of 2,4,6-trihydro 2-butenyl)-isovaler presence of $PtO_2$ ir an hydrogen atmos 1 h (82 %) [2113].	rophenone in the methanol under

-Also refer to: [2111].

m.p. 159° [2113].

BIOLOGICAL ACTIVITY: Antifungal [2111, 2113].

### 1-(2,4,6-Trihydroxy-3-pentylphenyl)-1-hexanone



 $C_{20}H_{20}Br_2O_4$ 

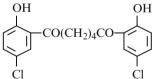
mol. wt. 484.18

-Obtained by reaction of bromine with 1,6-bis(4-methoxyphenyl)-1,6-hexanedione in hot carbon tetrachloride (67–90 %) [1073].

m.p. 158–169° (d) [1073].

# 1,6-Bis-(5-chloro-2-hydroxyphenyl)-1,6-hexanedione

 $[101735-99-3] C_{18}H_{16}Cl_2O_4 mtext{mol. wt. 367.23}$ 



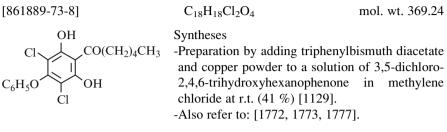
OH Synthesis -Obtained by Fries rearrangement of bis-4-chlorophenyl adipate with aluminium chloride at 150° for 1 h (36 %) [3235]. Cl m.p. 196.5° [3235].

# 1-(4'-Bromo-2'-fluoro[1,1'-biphenyl]-4-yl)-2-hydroxy-1-hexanone

[792709-04-7]  $C_{18}H_{18}BrFO_2$  mol. wt. 365.24 Br - -  $CO(CH_2)_4CH_3$  - Refer to: [2553].

mol. wt. 282.33

# **1-(3,5-Dichloro-2,6-dihydroxy-4-phenoxyphenyl)-1-hexanone** (*Ph-DIF-1*)



Yellow amorphous solid [1129]; MS [1129].

BIOLOGICAL ACTIVITY: Ph-DIF-1; Dictyostelium differentiation-inducing factor-1 derivs. promotion of glucose consumption in mammalian cells [1777]; DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

# 6-(4-Hydroxyphenyl)-1-phenyl-1,6-hexanedione

$$C_{18}H_{18}O_3$$
HO-CO(CH₂)₄CO-Synthesis  
-Refer to: [3247].

Methyl ether [51067-61-9] C₁₉H₂₀O₃ mol. wt. 296.36

-Refer to: [3247 (30 %), 3249].

off-white solid [3247]; m.p. 111–112.5° [3247]; ¹H NMR [3247], ¹³C NMR [3247], IR [3247], MS [3247]; phosphorescence spectroscopy [3249].

# 1,6-Bis(2-hydroxyphenyl)-1,6-hexanedione

[109471-10-5]

 $C_{18}H_{18}O_4$  mol. wt. 298.34



Syntheses -Obtained by Fries rearrangement of phenyl adipate with aluminium chloride, -in the presence of solvents:

*in the tetrachloroethane/nitrobenzene mixture (500 ml/125 ml) for 4 h at 50–60° [902];

*in nitrobenzene for 4 h at  $50^{\circ}$ , then 30 min at  $60^{\circ}$  [902];

*in tetrachloroethane at  $80^{\circ}$  [902].

-without solvent at 120° [902].

m.p. 160-161° [902].

#### 1,6-Bis(4-hydroxyphenyl)-1,6-hexanedione

 $\begin{bmatrix} 20837-37-0 \end{bmatrix} \qquad C_{18}H_{18}O_4 \qquad \text{mol. wt. } 298.34$  $HO \longrightarrow CO(CH_2)_4CO \longrightarrow OH \qquad Syntheses$ 

-Obtained by Fries rearrangement of phenyl adipate with aluminium chloride,

-in the presence of solvents:

*in the tetrachloroethane/nitrobenzene mixture (500 ml/125 ml) for 4 h at 50–60 $^{\circ}$  [902];

*in nitrobenzene for 4 h at  $50^{\circ}$ , then 30 min at  $60^{\circ}$  [902];

*in tetrachloroethane at  $80^{\circ}$  [902].

-without solvent at  $120^{\circ}$  [902].

-Also refer to: [470, 471, 1148, 1176, 2530].

m.p. 240–242° [902], 237–240° [1148].

**Diacetate** [114399-84-7] C₂₂H₂₂O₆ mol. wt. 382.41

-Obtained by reaction of acetic anhydride with 1,6-bis(4-hydroxyphenyl)-1,6-hexanedione [902].

m.p. 142° [902].

**Dimethyl ether** [4280-49-3] 
$$C_{20}H_{22}O_4$$
 mol. wt. 326.39

-Obtained by reaction of dimethyl sulfate with 1,6-bis(4-hydroxyphenyl)-1,6-hexanedione in the presence of 2 N sodium hydroxide [902].

-Also obtained by reaction of adipic acid dichloride with anisole in the presence of aluminium chloride, (60 %) [210],

*in carbon disulfide at a temperature  $< 60^{\circ}$  [1073];

*in nitrobenzene/tetrachloroethane mixture (1:2) at  $0^{\circ}$  (55 %) [593];

*in dichloromethane at  $0^{\circ}$  for 4 h (93 %) [1724];

*without solvent at  $< 40^{\circ}$  (75 %) [905].

-Also obtained by reaction of adipic anhydride with anisole in the presence of aluminium chloride in refluxing carbon disulfide for 3 h (14 %) [2494].

-Also obtained by adding dropwise adiponitrile over 30 min to an ether solution of the Grignard reagent prepared from p-bromoanisole and magnesium. Then, the solution was refluxed for 4 h (74 %) [3247].

-Also refer to: [41, 348, 681 (35 %), 843, 1014 (55 %), 1148, 1688, 1833, 2168, 2519, 2577, 2596 (85 %), 3197].

colourless prisms [2494]; m.p. 147–149° [2577], 147° [902], 146–147° [1148], 145–146° [2596], 145° [593], 144.5–145.5° [210], 144–146° [1014], 144–145° [843, 905], 144° [2494], 142° [681], 141.5–142.5° [1073, 3247], 140–142° [2519], 140° [3197], 137–138° [1724], 136° [1688]; ¹H NMR [348, 681, 1724, 3247], ¹³C NMR [681, 3247], IR [681, 843, 1724, 3247], UV [3247], MS [681, 3247].

Dihydrazone of the dimethyl ether	$C_{20}H_{26}N_4O_2$	mol. wt. 354.45

m.p. 109–111° [905].

**Diethyl ether** C22H26O4 mol. wt. 354.45 [88167-05-9]

-Refer to: [41, 2467, 2494, 3197].

colourless plates [2494]; m.p. 131–132° [3197], 127° [2494].

# 1-(2-Hydroxyphenyl)-6-(4-hydroxyphenyl)-1,6-hexanedione

[101790-34-5]

 $C_{18}H_{18}O_4$ 

HO CO(CH₂)₄CC

-Obtained by Fries rearrangement of phenyl adipate with aluminium chloride, -in the presence of solvents:

*in the tetrachloroethane/nitrobenzene mixture (500 ml/125 ml) for 4 h at 50-60° [902]:

Syntheses

*in nitrobenzene for 4 h at 50°, then 30 min at  $60^{\circ}$  [902];

*in tetrachloroethane at 80° [902].

-without solvent at 120° [902].

m.p. 190-192° [902].

# 1,6-Bis(2,4-dihydroxyphenyl)-1,6-hexanedione

[19343-47-6]

 $C_{18}H_{18}O_{6}$ 

Syntheses

mol. wt. 330.34



-Obtained by adding resorcinol into adiponitrile and hydrogen chloride in ethyl ether in the presence of zinc chloride.

Then, the diketimine dichlorhydrate obtained was hydrolyzed by boiling water (40 %) [2674].

-Also refer to: [1735, 2504, 3374].

m.p. 285–286° [3374], 285° (d) [2504, 2674]; UV [2504].

mol. wt. 298.34

# **Di-2,4-Dinitrophenylhydrazone** [115963-79-6] $C_{30}H_{26}N_8O_{12}$ mol. wt. 690.58

C₁₈H₁₈O₆

m.p. 360° [2674].

[91453-26-8]

# 1,6-Bis(2,5-dihydroxyphenyl)-1,6-hexanedione

 $\begin{array}{c} OH & HO \\ \hline \\ -CO(CH_2)_4CO & \hline \\ HO & OH \end{array}$ 

Syntheses -Obtained by treatment of 1,6-bis(2-hydroxy-5-methoxyphenyl)-1,6-hexanedione with boron tribromide in methylene chloride (85 %) [2103].

-Also refer to: [2331].

yellow solid [411]; m.p. 236–238° [2103].

#### **Tetramethyl ether** [10365-23-8] C₂₂H₂₆O₆ mol. wt. 386.45

-Obtained by reaction of adipic acid with hydroquinone dimethyl ether in the presence of thionyl chloride (32 %) [2331].

-Also obtained by reaction of adipic acid dichloride (1 mol) with hydroquinone dimethyl ether (2.5 mol) in the presence of aluminium chloride (1 mol) (65 %) [1575]. -Also obtained by reaction of adipic acid dichloride with hydroquinone dimethyl ether in the presence of aluminium chloride in carbon disulfide or methylene chloride (53–56 %).

-Also refer to: [1575, 3175] (5 %).

needles [2331]; white needles [3175]; m.p. 112–113° [3175], 110° [1575], 105–106° [2331].

# 1,6-Bis(3,4-dihydroxyphenyl)-1,6-hexanedione

 $\begin{array}{ccc} C_{18}H_{18}O_6 & \text{mol. wt. 330.34} \\ HO & OH & \text{Synthesis} \\ HO & OH & -CO(CH_2)_4CO & OH & -CO(CH_2)_4CO & -CO(CH$ 

-Obtained by hydrogenating 1-veratroyl-4-(3,4-dimethoxy-6-bromophenyl)butane with Raney Nickel and KOH in ethanol at 12 atm. and  $63^{\circ}$  (90 %) [3061].

-Also obtained by hydrogenating its oxime in acetic acid, in the presence of 10 % Pd/C in a Parr apparatus at r.t. and 45–50 psig for 12–36 h (68 %) [1014].

-Obtained by reaction of adipyl chloride with veratrole in the presence of aluminium chloride in carbon disulfide first at  $0^{\circ}$ , then at reflux for 3 h [1124]. -Also refer to: [1013, 2342, 3364 (70 %)].

m.p. 149–150° [1014, 1124]; ¹H NMR [2342], ¹³C NMR [2342], IR [3364]. mol. wt. 330.34

Dioxime of the tetramethyl ether [50766-28-4]  $C_{22}H_{28}N_2O_6$  mol. wt. 416.47

-Refer to: [1012, 1014].

m.p. 154° [1012], 151–153° [1014].

**Tetraethyl ether** [50766-18-2] C₂₆H₃₄O₆ mol. wt. 442.55

-Obtained by hydrogenating its oxime in acetic acid, in the presence of 10 % Pd/C in a Parr apparatus at r.t. and 45–50 psig for 12–36 h (39 %) [1014]. -Also refer to: [1012].

m.p. 135–138° [1012], 132–134° [1014].

Dioxime of the tetraethyl ether [50766-34-2] C₂₆H₃₆N₂O₆ mol. wt. 472.58

m.p. 135-139° [1014].

**Tetrapropyl ether** [50766-19-3] C₃₀H₄₂O₆ mol. wt. 498.66

-Obtained by hydrogenating its oxime in acetic acid, in the presence of 10 % Pd/C in a Parr apparatus at r.t. and 45–50 psig for 12–36 h (48 %) [1014]. -Also refer to: [1012].

m.p. 147–149° [1012], 144–146° [1014].

Dioxime of the tetrapropyl ether [50766-35-3] C₃₀H₄₄N₂O₆ mol. wt. 528.69

m.p. 142–143° [1014].

**Tetrabutyl ether** [50766-20-6] C₃₄H₅₀O₆ mol. wt. 554.77

-Obtained by hydrogenating its oxime in acetic acid, in the presence of 10 % Pd/C in a Parr apparatus at r.t. and 45–50 psig for 12–36 h (61 %) [1014]. -Also refer to: [1012].

m.p. 126–128° [1012], 123–125° [1014].

Dioxime of the tetrabutyl ether [50766-36-4]  $C_{34}H_{52}N_2O_6$  mol. wt. 584.80

m.p. 123–124° [1014].

### **Dimethylenedioxy** [6268-56-0] C₂₀H₁₈O₆ mol. wt. 354.36

1,6-Bis(3,4-methylenedioxyphenyl)-1,6-hexanedione

-Obtained by adding 1,6-bis(3,4-methylenedioxyphenyl)-1,6-hexanediol to a suspension of chromium (VI) oxide in pyridine and Celite at r.t. under an argon atmosphere. After the mixture was stirred for 8 h at r.t. (77 %) [1132]. -Also refer to: [1014, 2693 (38 %)].

m.p. 179.4–180.4° [2693], 179–180° [1014]; ¹H NMR [1132], IR [1132].

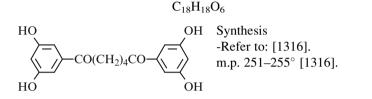
#### 

m.p. 316-317° (d) [2693].

#### 

m.p. 117–121° [1014].

# 1,6-Bis(3,5-dihydroxyphenyl)-1,6-hexanedione



# 1,6-Bis(2,3,4-trihydroxyphenyl)-1,6-hexanedione

 $\begin{array}{cccc} C_{18}H_{18}O_8 & \text{mol. wt. 362.33} \\ HO & OH & HO & OH \\ HO & \leftarrow & CO(CH_2)_4CO & \leftarrow & OH \\ HO & HO & HO & CO(CH_2)_4CO & \leftarrow & CO(CH_2)_4CO \\ Hexamethyl ether & CO(CH_2)_4CO & CO(CH_2)_4CO & CO(CH_2)_4CO \\ Hexamethyl ether & CO(CH_2)_4CO & CO(CH_2)_4CO \\ HO & HO & CO(CH_2)_4CO & CO(CH_2)_4CO \\ HO & HO & CO(CH_2)_4CO & CO(CH_2)_4CO \\ HO & HO & CO(CH_2)_4CO \\ HO$ 

-Obtained by reaction of dimethyl sulfate with 1,6-bis(2-hydroxy-3,4-dimethoxyphenyl)-1,6-hexanedione in the presence of 30 % sodium hydroxide (65–90 %) [1574]. -Also refer to: [1575].

m.p. 116° [1574, 1575].

### 1,6-Bis(2,4,6-trihydroxyphenyl)-1,6-hexanedione



mol. wt. 330.34

#### 1-(4'-Bromo[1,1'-biphenyl]-4-yl)-2-hydroxy-1-hexanone

[792708-89-5] 
$$C_{18}H_{19}BrO_2$$
 mol. wt. 347.25  
Br  $CO(CH_2)_4CH_3$  Synthesis  
HO

# 1-(3'-Bromo-4'-hydroxy[1,1'-biphenyl]-4-yl)-1-hexanone

$$\begin{array}{ccc} C_{18}H_{19}BrO_2 & \text{mol. wt. } 347.25 \\ Br & & Synthesis \\ HO & & CO(CH_2)_4CH_3 & \text{Refer to: } [2993]. \\ \textbf{Methyl ether} & [83258-17-7] \\ C_{19}H_{21}BrO_2 & \text{mol. wt. } 361.27 \end{array}$$

-Refer to: [2993].

# **1-(3-Chloro-2,6-dihydroxy-4-phenoxyphenyl)-1-hexanone** (*Ph-DIF-3*)

Syntheses

[861889-74-9]

C₁₈H₁₉ClO₄ mol. wt. 334.80

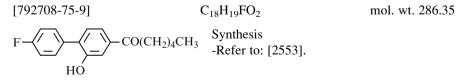
Cl  $CO(CH_2)_4CH$   $CO(CH_2)_4CH$   $Cc_6H_5O$  OH

CO(CH₂)₄CH₃
 Preparation by adding triphenylbismuth diacetate and copper powder to a solution of 3-chloro-2,4,6-trihydroxy-hexanophenone in methylene chloride at r.t. [1129].
 Also refer to: [1772, 1773, 1777].

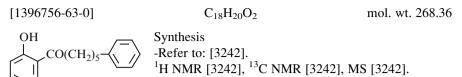
colourless amorphous solid [1129]; MS [1129].

BIOLOGICAL ACTIVITY: Ph-DIF-3; Dictyostelium differentiation-inducing factor-1 derivs. promotion of glucose consumption in mammalian cells [1777]; DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

#### 1-(4'-Fluoro[1,1'-biphenyl]-4-yl)-2-hydroxy-1-hexanone

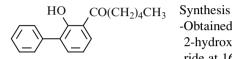


### 1-(2-Hydroxyphenyl)-5-phenyl-1-hexanone



# 1-[1,1'-Biphenyl]-3-yl-2-hydroxy-1-hexanone

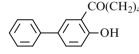
 $C_{18}H_{20}O_2$ mol. wt. 268.36



-Obtained (by-product) by Fries rearrangement of 2-hydroxydiphenyl caproate with aluminium chloride at 160° for 30–45 min (7 %) [1256].

# 1-[1,1'-Biphenyl]-3-yl-4-hydroxy-1-hexanone

C₁₈H₂₀O₂ mol. wt. 268.36



O(CH₂)₄CH₃ Synthesis

-Obtained bv Fries rearrangement of 4-hydroxydiphenyl caproate with aluminium chloride at 160° for 30-45 min (47 %) [1256]. m.p. 86–88° [1256].

mol. wt. 297.40

# 1-[1,1'-Biphenyl]-4-yl-2-hydroxy-1-hexanone

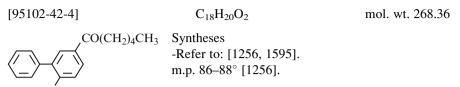
[792705-86-3]	$C_{18}H_{20}O_2$	mol. wt. 268.36
HO CO(CH ₂ ) ₄ CH ₃	Synthesis -Refer to: [2553]. Acetate [792708-61-3] C ₂₀ H ₂₂ O ₃	mol. wt. 310.39

-Refer to: [2553].

#### **O-Methyloxime**

[792708-48-6]  $C_{19}H_{23}NO_2$ -Refer to: [2553].

#### 1-(6-Hydroxy[1,1'-biphenyl]-3-yl)-1-hexanone



USE: Colour developer for thermal recording materials [1595].

### 1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-1-hexanone

 $C_{18}H_{20}O_{2} \qquad \qquad \text{mol. wt. 268.36} \\ HO \longrightarrow CO(CH_{2})_{4}CH_{3} \qquad \qquad Synthesis \\ -Obtained by Fries rearrangement of$ 4-hexanoyl-oxybiphenyl with aluminiumchloride in nitrobenzene, first at 20° for $12 h, then at 60° for 1 h [522]. \\ \end{array}$ 

**Methyl ether** [56116-78-0] 
$$C_{19}H_{22}O_2$$
 mol. wt. 282.38

-Obtained from hexanoyl chloride and 4-methoxybiphenyl [522]. -Also refer to: [514, 847, 2994].

smooth nacreous [522]; b.p.₁₅ 258–262° [514, 522]; m.p. 123° [514, 522].

#### Various ethers (10)

-Preparations and liquid crystalline properties of, [847].

Ethyl ether	[56116-87-1]	$C_{20}H_{24}O_2$	mol. wt. 296.41	Refer to: [847].
Propyl ether	[56116-95-1]	$C_{21}H_{26}O_2$	mol. wt. 310.44	Refer to: [847].
Butyl ether	[56117-03-4]	$C_{22}H_{28}O_2$	mol. wt. 324.46	Refer to: [847].
Pentyl ether	[56117-12-5]	$C_{23}H_{30}O_2$	mol. wt. 338.49	Refer to: [847].
Hexyl ether	[56117-21-6]	$C_{24}H_{32}O_2$	mol. wt. 352.52	

-Obtained by reaction of hexanoyl chloride with 4-hexyloxybiphenyl in the presence of aluminium chloride in nitrobenzene (23 %) [1799].

-Also refer to: [847].

HO

Heptyl ether	[56117-30-7]	$C_{25}H_{34}O_2$	mol. wt. 366.54	Refer to: [847].
Octyl ether	[56117-38-5]	$C_{26}H_{36}O_2$	mol. wt. 380.57	Refer to: [847].
Nonyl ether	[56117-47-6]	$C_{27}H_{38}O_2$	mol. wt. 394.60	Refer to: [847].
Decyl ether	[56117-56-7]	$C_{28}H_{40}O_2$	mol. wt. 408.62	Refer to: [847].
Dodecyl ether	[56117-65-8]	$C_{30}H_{44}O_2$	mol. wt. 436.68	Refer to: [847].
Hexanoate	[72057-94-4]	$C_{24}H_{30}O_3$	mol. wt. 366.49	Refer to: [847].

-Obtained by Friedel-Crafts reaction of 4-hydroxybiphenyl with caproyl chloride in the presence of aluminium chloride (72 %) [287].

# 1-(2-Hydroxy-4-phenoxyphenyl)-1-hexanone

[307000-38-0]	$C_{18}H_{20}O_3$	mol. wt. 284.35	
OH CO(CH ₂ ) ₄ CH ₃	Syntheses -Refer to: [250, 1345].		
C ₆ H ₅ O	USE: For composition of antiperspirant [250].	deodorant and	
BIOLOGICAL ACTIVITY A	ntimicrobial [1345]		

BIOLOGICAL ACTIVITY: Antimicrobial [1345].

# 1-(2-Butyl-6-hydroxy-5-benzofuranyl)-1-hexanone

[1002158-21-5]	$C_{18}H_{24}O_3$		r	nol. wt	. 288.39
HO CH ₃ (CH ₂ ) ₄ CO	-Obtained (butylethyn	yl)-1,5 quiv.)	treatment -diacetoxybe in THF/MeC	nzene	with

¹H NMR [1875], ¹³C NMR [1875], IR [1875], MS [1875].

# 1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-hexanone

[3118-34-1]	$C_{18}H_{26}O_5$		mol. wt. 322.40
CH ₃ (CH ₂ ) ₄ CO	OH CO(CH ₂ ) ₄ CH ₃	Syntheses -Obtained by reaction of with phloroglucinol in boron trifluoride etherate	the presence of
		-Also obtained by react chloride with phloroglu sence of aluminium c	icinol in the pre-

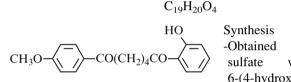
benzene for 3 days at r.t. (5–10 %) [421].

-Also obtained by reaction of caproic anhydride with phloroglucinol in the presence of boron trifluoride etherate on heating for 4 h [457]. -Also refer to: [457, 600, 644, 962, 2911, 3018, 3033].

m.p. 105° [421], 97–98° [457, 2911]; ¹H NMR [421, 3019], ¹³C NMR [3019], IR [421, 3019], UV [3019], MS [421].

BIOLOGICAL ACTIVITY: Antagonist both thromboxane  $A_2$  and Leukotriene  $D_4$  [3019]; Inhibitory effects of treatment with this acylphenol on TPA Induced EBV-EA Activation [3033]; Antiviral activity towards the replication of vesicular stomatitis virus in HEL cell cultures [644]; Germination inhibition in cress (*Lepidium sativum*) seeds [421]; Industrial bactericidal and fungicidal agent, algicide and biofouling inhibitor [962]; For treatment of immune dysfunction associated with human immunodeficiency virus infection [600]; Anthelmintic [457].

### 6-(2-Hydroxyphenyl)-1-(4-methoxyphenyl)-1,6-hexanedione

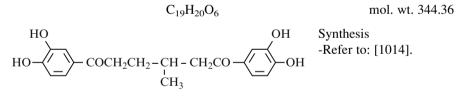


mol. wt. 312.36

Obtained by reaction of dimethyl sulfate with 1-(2-hydroxyphenyl)-6-(4-hydroxyphenyl)-1,6-hexanedione in the presence of 2 N NaOH [902].

m.p. 118° [902].

### 1,6-Bis(3,4-dihydroxyphenyl)-3-methyl-1,6-hexanedione



**Tetramethyl ether** [50766-25-1]  $C_{23}H_{28}O_6$  mol. wt. 400.47

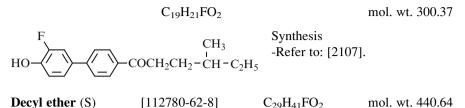
-Obtained by hydrogenating its oxime in acetic acid, in the presence of 10 % Pd/C in a Parr apparatus at r.t. and 45–50 psig for 12–36 h (38 %) [1014]. -Also refer to: [1012].

m.p. 91–94° [1014], 83–86° [1012].

Dioxime of the tetramethyl ether [50766-42-2]  $C_{23}H_{30}N_2O_6$  mol. wt. 430.50

m.p. 127–130° [1014].

#### 1-(3'-Fluoro-4'-hydroxy[1,1'-biphenyl]-4-yl)-4-methyl-1-hexanone



USE: Liq.-crystal compnds. contg., for display devices [2107].

# 1-[2-Hydroxy-4-[(3-nitrophenyl)methoxy]phenyl]-1-hexanone

C₁₉H₂₁NO₅ [103981-28-8] mol. wt. 343.38 **Synthesis** OH CO(CH₂)₄CH₃ -Obtained by reaction of 3-nitrobenzyl NO₂ bromide 2,4-dihydroxycaprowith CH₂-C phenone in the presence of potassium cesium carbonate and carbonate (cat.) [1657]. Methyl ether [103981-30-2] C₂₀H₂₃NO₅ mol. wt. 357.41

-Refer to: [1657].

### 1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1-hexanone

[143287-04-1]	$C_{19}H_{22}O_3$	mol. wt. 298.38
Co(CH ₂ ) ₄ CH ₃ C ₆ H ₅ CH ₂ O	Synthesis -Obtained by reaction of ben 2,4-dihydroxycaprophenone of potassium carbonate in 1 for 20 h [284].	in the presence
-Also refer to: [3410].		

m.p. 62–67° [284].

Oxime [143286-81-1]

 $C_{19}H_{23}NO_3$ 

mol. wt. 313.40

-Obtained by reaction of hydroxylamine hydrochloride with 1-[2-hydroxy-4-(phenylmethoxy)phenyl]-1-hexanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 88–94° [284]; ¹H NMR [284].

-Refer to: [3410].

#### 1-[2-Hydroxy-5-(phenylmethoxy)phenyl]-1-hexanone

	$C_{19}H_{22}O_3$	mol. wt. 298.38
ОН	Synthesis	
CO(CH ₂ ) ₄ CH ₃	-Refer to: [3409].	
	Methyl ether [186246-05-9]	
$\mathbf{i}$	$C_{20}H_{24}O_3$	mol. wt. 312.40
OCH ₂ C ₆ H ₅	-Refer to: [3409, 3411].	

# 1-[3-Hydroxy-2-(phenylmethoxy)phenyl]-1-hexanone

	$C_{19}H_{22}O_3$		mol. wt. 298.38
OH OCH ₂ C ₆ H ₅	Synthesis -Refer to: [341	1]. [195158-14-6]	
CO(CH ₂ ) ₄ CH ₃	•	[195156-14-0]	mol. wt. 312.40

-Refer to: [3411].

# 1,1'-(2,4,6-Trihydroxy-5-methyl-1,3-phenylene)bis-1-hexanone

[3118-38-5]	C ₁₉ H	H ₂₈ O ₅	mol. wt. 336.43
CH ₃ (CH ₂ ) ₄ CO HO	OH CO(CH ₂ ) ₄ CH ₃ OH CH ₃	with 2-methylphlor sence of boron tri heating for 4 h [457	n of caproic anhydride oglucinol in the pre- ifluoride etherate on ]. 00, 2730, 2731, 2911].

m.p. 108–110° [457, 2911].

BIOLOGICAL ACTIVITY: For treatment of immune dysfunction associated with human immunodeficiency virus infection [600]; Mitochondria of ascarid in response to [2730]; Anthelmintic [457]; Phosphorylation response to, in round-worm mitochondria, [2731].

# 1-(2,5-Dihydroxy-3,4,6-trimethylphenyl)-5-methyl-2-(1-methylethyl)-1-hexanone

$$[357172-20-4] C_{19}H_{30}O_3 mol. wt. 306.45$$
  
OH Synthesis  
CH₃ CO-CH-(CH₂)₂CH(CH₃)₂ -Refer to: [2352].  
CH₃ CH₃ CH(CH₃)₂

# 1,6-Bis(2-hydroxy-4-methylphenyl)-1,6-hexanedione

[13320-65-5]  $C_{20}H_{22}O_4$ HO OH CO(CH₂)₄CO CH₃ -CH₃

Syntheses -Obtained by Fries rearrangement of m-cresyl adipate with aluminium chloride, first at 100°, then at 165° for 50 min [2774].

mol. wt. 326.39

-Also refer to: [618].

m.p. 122–123° [2774]; ¹H NMR [618].

### 1,6-Bis(2-hydroxy-5-methylphenyl)-1,6-hexanedione

[13282-24-1] C20H22O4 mol. wt. 326.39 Syntheses HO OH -Obtained by Fries rearrangement of di O(CH₂)₄CO (4-methyl-phenyl) adipate with aluminium chloride. CH₃ CH-

*without solvent at  $130^{\circ}$  for 4 h (28 %) [3107];

*in refluxing chlorobenzene for 6 h (72 %) [3107].

m.p. 163–164° [3107]; IR [3107].

# 1,6-Bis(4-hydroxy-2-methylphenyl)-1,6-hexanedione

[5550-54-9]

Synthesis

-OH

-Obtained by reaction of adipyl chloride with m-cresol in the presence of aluminium chloride in nitrobenzene at 0° [593].

mol. wt. 326.39

m.p. 114° [593].

$$\begin{array}{cccc} [5550-54-9] & C_{20}H_{22}O_4 \\ & & CH_3 & CH_3 & S_2 \\ & & & & & & & \\ HO & & & & & & & \\ HO & & & & & & & \\ HO & & & & & & & \\ HO & & & & & & & \\ HO & & & & & & & \\ HO & & & & & & & \\ HO & & & & & & & \\ HO & & & & & & & \\ HO & & \\ HO & & \\ HO & & & \\$$

mol. wt. 356.42

**Dioxime** [5538-13-6]

m.p. 131° [593].

Dimethyl ether	[5550-56-1]	$C_{22}H_{26}O_4$	mol. wt. 354.45
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 $C_{20}H_{24}N_2O_4$ 

-Obtained by reaction of adipyl chloride with m-cresol methyl ether in the presence of aluminium chloride in a nitrobenzene/tetrachloroethane mixture (1:1) at  $0^{\circ}$  [593].

m.p. 130° [593].

**Oxime of the dimethyl ether** [5538-16-9] C₂₂H₂₈N₂O₄ mol. wt. 384.48

m.p. 145° [593].

## 1,6-Bis(4-hydroxy-3-methylphenyl)-1,6-hexanedione

 $[6016-44-0] C_{20}H_{22}O_4$ mol. wt. 326.39  $CH_3 \qquad CH_3 \qquad CH_3 \qquad Obtained by reaction of adipyl chloride with o-cresol in the presence of aluminium chloride in nitrobenzene at 0° (10 %) [593].$ 

**Dioxime** [5538-08-9] C₂₀H₂₄N₂O₄ mol. wt. 356.42

m.p. 175° [593].

<b>Differing ether</b> $[5536-10-5]$ $C_{22}11_{26}O_4$ fillor, wt. 5	methyl ether	mol. wt. 354.45	$C_{22}H_{26}O_4$	[5538-10-3]
-----------------------------------------------------------------------	--------------	-----------------	-------------------	-------------

-Obtained by reaction of adipyl chloride with o-cresol methyl ether in the presence of aluminium chloride in a nitrobenzene/tetrachloroethane mixture (1:1) at  $0^{\circ}$  [593].

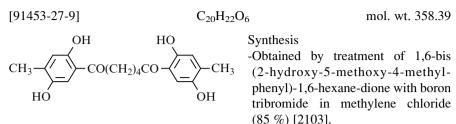
-Also refer to: [1014 Method D (81 %), 2246, 3378].

m.p. 164–165° [3378], 163° [2246], 160–161° [1014], 160° [593]; ¹H NMR [3378], IR [3378], UV [2246].

# Dioxime of the dimethyl ether [5550-53-8] $C_{22}H_{28}N_2O_4$ mol. wt. 384.48

m.p. 185° [593], 178–179° [1014].

#### 1,6-Bis(2,5-dihydroxy-4-methylphenyl)-1,6-hexanedione



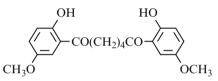
yellow solid [2103]; m.p. 137–139° [2103]; ¹H NMR [2103], IR [2103].

#### 1,6-Bis(2-hydroxy-5-methoxyphenyl)-1,6-hexanedione

[10365-28-3]

 $C_{20}H_{22}O_{6}$ 

mol. wt. 358.39



Syntheses -Obtained by reaction of adipoyl dichloride with p-dimethoxybenzene in the presence of aluminium chloride [1575] in refluxing 1,2-dichloroethane

for 8 h [2103].

-Also refer to: [1575, 3175].

yellow crystals [3175]; m.p. 157–158° [3175], 157° [1575].

Diacetate [10365-33-0] C₂₄H₂₆O₈ mol. wt. 442.47

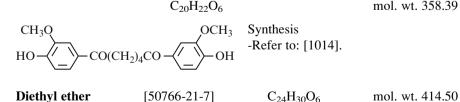
-Obtained by reaction of acetic anhydride (4 vol) with the title ketone in the presence of 1-2 drops concentrated sulfuric acid, the mixture warmed 5 min [1575].

m.p. 152° [1575].

-Refer to Method D: [1014] (85 %).

m.p. 130–131° [1014].

### 1,6-Bis(4-hydroxy-3-methoxyphenyl)-1,6-hexanedione



**Dioxime of the diethyl ether** [50766-37-5] C₂₄H₃₂N₂O₆ mol. wt. 444.53 m.p. 187–189° [1014].

 Dibenzyl ether
 [50766-22-8]
  $C_{34}H_{34}O_6$  mol. wt. 538.64

-Refer to Method D: [1014] (70 %).

m.p. 163–164° [1014].

Dioxime of the dibenzyl ether [50766-38-6]  $C_{36}H_{40}N_2O_6$  mol. wt. 596.72 m.p. 125–126° [1014].

# 3-[4-(2-Chloroethyl)phenyl]-1-(2-hydroxyphenyl)-1-hexanone

[70206-42-7]	$C_{20}H_{23}ClO_2$	mol. wt. 330.85
OH CO-CH ₂ -CH-C ₃ H ₇	Synthesis -Obtained from degradation ¹ H NMR [3205], IR [3205]	

### 3-[4-(2-Chloroethyl)phenyl]-2-hydroxy-1-(2-hydroxyphenyl)-1-hexanone

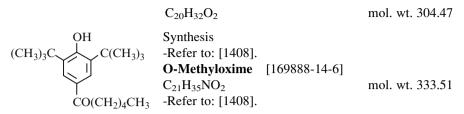
### 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1,2-hexanedione

. CH₂CH₂Cl

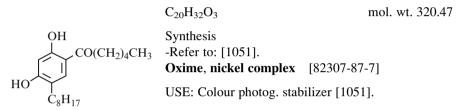
[96251-00-2]	$C_{20}H_{30}O_3$	mol. wt. 318.46	
$(CH_3)_3C$ $(CH_3)_3C$ $(CH_3)_3$ $(CCH_3)_3$ $(CCCCC_4H_9)$	Syntheses -Obtained by oxygenation of 4-hydroxyphenyl]-1-hexyne lene chloride at 0° (70 %) [ -Also refer to: [2007, 2289].	e with Co(Salpr) in methy- 2290].	
1 1			

colourless prisms [2290]; m.p. 187–188° [2289, 2290]; ¹H NMR [2007, 2289, 2290], ¹³C NMR [2007, 2289, 2290], IR [2289, 2290], UV [2290].

# 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-hexanone



# 1-(2,4-Dihydroxy-5-octylphenyl)-1-hexanone



# 1-[4-[(2-Ethylhexyl)oxy]-2-hydroxyphenyl]-1-hexanone

[80851-64-5]	$C_{20}H_{3}$	₂ O ₃	mol. wt. 320.47
C ₂ H ₅ C ₄ H ₉ -CH-CH ₂ O		Synthesis -Refer to: [1050]. <b>Oxime, nickel complex</b>	[80848-70-0]

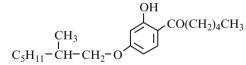
USE: Photog. stabilizer compns. contg. phenol derivs. and, for magenta images [1050].

# 1-[2-Hydroxy-4-(sec-octyloxy)phenyl]-1-hexanone

[127313-65-9]

 $C_{20}H_{32}O_3$ 

mol. wt. 320.47



H Synthesis  $CO(CH_2)_4CH_3$  -Refer to: [3427]. Oxime [127313-53-5]  $C_{20}H_{33}NO_3$  mol. wt. 335.49

USE: Copper extn. reactivity of [3427].

mol. wt. 372.46

#### 1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-1-hexanone

[134082-02-3]	$C_{21}H_{26}O_5$	mol. wt. 358.43
CH ₃ O CH ₃ O CH ₃ O OCH ₂ C ₆ H ₅	Synthesis -Obtained by elimination of one ph in 2-position by treatment of 1- 3,4-dimethoxyphenyl)-1-hexanone trated hydrochloric acid and ace 2–3 h (81 %) [1353].	(2,6-dibenzyloxy- e with concen-
m.p. 104.5–105.5° [1353];	¹ H NMR [1353].	

C22H28O5

Methyl ether

-Refer to: [1353].

# 1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]-1-hexanone

[134081-86-0]	$C_{21}H_{26}O_7S$	mol. wt. 422.50
$CH_{3O} \xrightarrow{OH} CO(CH_{2})_{4}CH_{3}$ $CH_{3O} \xrightarrow{OCH_{3}} OSO_{2}C_{6}H_{4}CH_{3}(p)$	Synthesis -Obtained by treatment 3,4,6-trimethoxyphenyl)-1-h aluminium bromide in acc 2–3 h (80 %) [1353].	exanone with 25 %

m.p. 53–54° [1353]; ¹H NMR [1353].

# 1,6-Bis(2-hydroxy-5-methoxy-4-methylphenyl)-1,6-hexanedione

[344574-57-8] C22H26O6 mol. wt. 386.45 OH Synthesis HO -Obtained by reaction of adipoyl CO(CH₂)₄CO CH₃ CH₃ dichloride with 2,5-dimethoxytoluene in the presence of aluminium CH₂C OCH₂ chloride in refluxing 1,2-dichloro-

**Dimethyl ether** 

[180578-80-7]

 $C_{24}H_{30}O_6$  mol. wt. 414.50

ethane for 8 h [2103].

-Obtained by reaction of adipic acid dichloride with 2,5-dimethoxytoluene in the presence of aluminium chloride in dichloromethane first at  $0^{\circ}$  under an argon atmosphere, then at r.t. for 16 h (61 %) [2570], (56 %) [1402].

colourless needles [1402]; m.p. 167° [1402], 164–165° [2570]; ¹H NMR [2570], ¹³C NMR [2570], IR [2570].

#### 1,6-Bis(2-hydroxy-3,4-dimethoxyphenyl)-1,6-hexanedione

[10351-90-3] mol. wt. 418.44 C22H26O8



Syntheses -Obtained by reaction of adipic acid dichloride with pyrogallol trimethyl ether in the presence of aluminium

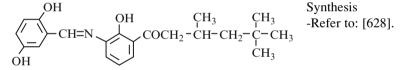
chloride [3058] in tetrachloroethane [1574]. -Also refer to: [1575, 3058].

m.p. 175° [1574, 1575], 174–175° [3058].

# 1-[3-[[(2,5-Dihydroxyphenyl)methylene]amino]-2-hydroxyphenyl]-3,5,5-trimethyl-1-hexanone

- 1-[3-(2.5-Dihydroxybenzylideneamino)-2-hydroxyphenyl]-3,5,5-trimethyl-1-hexanone
- 1-[3-[(2,5-Dihydroxybenzylidene)amino]-2-hydroxyphenyl]-3,5,5-trimethyl-1-hexanone

mol. wt. 369.46 [176044-11-4] C22H27NO4



BIOLOGICAL ACTIVITY: As inhibitor of EGF receptor-assocd. tyrosine kinase [628].

# 1-[5-[[(2,5-Dihydroxyphenyl)methylene]amino]-2-hydroxyphenyl]-3,5,5-trimethyl-1-hexanone

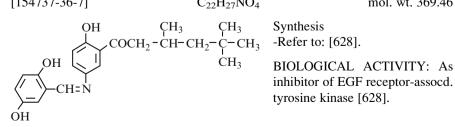
1-[5-(2,5-Dihydroxybenzylideneamino)-2-hydroxyphenyl]-3,5,5-trimethyl-1-hexanone

1-[5-[(2,5-Dihydroxybenzylidene)amino]-2-hydroxyphenyl]-3,5,5-trimethyl-1-hexanone

[154737-36-7]

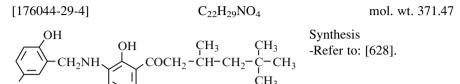
#### $C_{22}H_{27}NO_4$

mol. wt. 369.46



# 1-[3-[[(2,5-Dihydroxyphenyl)methyl]amino]-2-hydroxyphenyl]-3,5,5-trimethyl-1-hexanone

1-[3-(2,5-Dihydroxybenzylamino)-2-hydroxyphenyl]-3,5,5-trimethyl-1-hexanone



BIOLOGICAL ACTIVITY: As inhibitor of EGF receptor-assocd. tyrosine kinase [628].

# 1-[5-[[(2,5-Dihydroxyphenyl)methyl]amino]-2-hydroxyphenyl]-3,5,5-trimethyl-1-hexanone

1-[5-(2,5-Dihydroxybenzylamino)-2-hydroxyphenyl]-3,5,5-trimethyl-1-hexanone

 $[154736-91-1] C_{22}H_{29}NO_4 mol. wt. 371.47$   $OH CH_2 CH_3 CH_3 CH_3 Syntheses -Refer to: [417, 628].$   $HO CH_3 BIOLOGICAL ACTIVITY: As inhibitor of EGF receptor-assocd. tyrosine kinase [628].$ 

### 1-[4-(Decyloxy)-2-hydroxyphenyl]-1-hexanone

[101002-33-9]  $C_{22}H_{36}O_3$  mol. wt. 348.53 OH  $C_{10}H_{21}O$  OH  $C_{10}H_{21}O$  CO(CH₂)₄CH₃ Obtained by reaction of decyl bromide with 2,4-dihydroxycaprophenone in the presence of potassium carbonate in refluxing acetone for 20 h [284].

-Also refer to: [3077].

m.p. 45-47° [284].

**Oxime** [101002-20-4] C₂₂H₃₇NO₃ mol. wt. 363.54

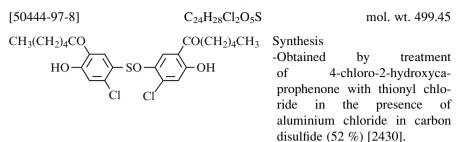
-Obtained by reaction of hydroxylamine hydrochloride with 1-[4-(decyloxy)-2-hydroxyphenyl]-1-hexanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

-Also refer to: [3077].

m.p. 58–60° [284, 3077]; ¹H NMR [284].

USE: Topical preparations comprising at least one aryloxime and bisabolol [483]; As lipoxygenase inhibitor [3077].

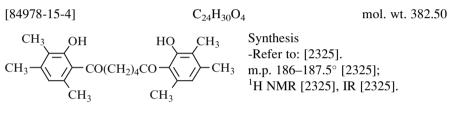
#### 1,1'-[Sulfonylbis(6-chloro-4-hydroxy-3,1-phenylene)]bis-1-hexanone



m.p. 123° [2430]; IR [2430].

USE: Antifungal [2430].

# 1,6-Bis(2-hydroxy-3,4,6-trimethylphenyl)-1,6-hexanedione



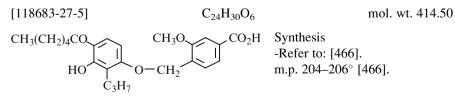
# 1,1'-(4,4'-Dihydroxy[1,1'-biphenyl]-3,3'-diyl)-1-hexanone

aluminium chloride.

*in the presence of sodium chloride at 140° (99 %) [2091]; *in refluxing chlorobenzene for 24 h (53 %) [2377].

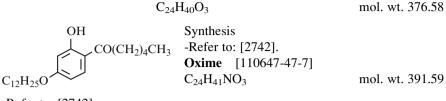
m.p. 92.5–93.5° [2377]; IR [2377].

# 4-[3-Hydroxy-4-(1-hexanoyl)-2-propylphenoxymethyl]-3-methoxybenzoic acid



BIOLOGICAL ACTIVITY: Antagonist of the peptidoleukotrienes [466].

#### 1-[4-(Dodecyloxy)-2-hydroxyphenyl]-1-hexanone



-Refer to: [2742].

# 1-[5-(Dodecyloxy)-2-hydroxyphenyl]-1-hexanone

 $\begin{array}{cccc} [140466-94-0] & C_{24}H_{40}O_3 & \text{mol. wt. 376.58} \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\$ 

### 1-(3,5-Dihexyl)-2,4,6-(trihydroxyphenyl)-1-hexanone

# 1,1'-[Methylenebis(2,3,4-trihydroxy-5,1-phenylene)]bis-1-hexanone

C25H32O8

 $HO \qquad OH \qquad HO \qquad OH$  $HO \qquad CH_2 \qquad OH$  $CH_3(CH_2)_4CO \qquad CO(CH_2)_4CH_3$  mol. wt. 460.52

Synthesis

-Obtained by treatment of 4-caproyl-pyrogallol with 35 % aqueous solution of formaldehyde in refluxing ethanol for 10 min [506].

m.p. 171° [506].

# 1-(2-Hydroxy-4-tetradecylphenyl)-1-hexanone

[118469-92-4]

 $C_{26}H_{44}O_2$ 

mol. wt. 388.63

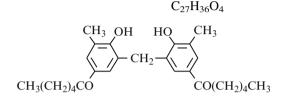
OH  

$$CO(CH_2)_4CH_3$$
 -Refer to: [3323].  
 $m.p. 44-45^{\circ}$  [3323].

#### 1-(2-Hydroxy-5-tetradecylphenyl)-1-hexanone

 $\begin{array}{cccc} [118469-84-4] & C_{26}H_{44}O_2 & \text{mol. wt. } 388.63 \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\$ 

# 1,1'-[Methylenebis(2-hydroxy-3-methyl-5,1-phenylene)]bis-1-hexanone

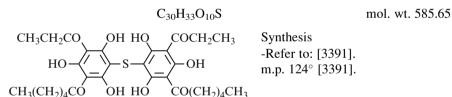


mol. wt. 424.58

Syntheses -Obtained by reaction of hexanoyl chloride with bis(2-hydroxy-3-methyl-phenyl) methane according to the method described previously [2871], (55 %) [119].

m.p. 141–142° [119]; ¹H NMR [119], IR [119].

### 1,1'-Thiobis[2,4,6-trihydroxy-3-(1-oxopropyl)-5,1-phenylene]bis-1-hexanone



### 1,1'-Methylenebis(2,4,6-trihydroxy-3,5,1-phenylene)bis-1-hexanone

 $\begin{bmatrix} 68223-34-7 \end{bmatrix} \qquad \begin{array}{c} C_{37}H_{52}O_{10} \\ CH_3(CH_2)_4CO & OH & HO & CO(CH_2)_4CH_3 \\ HO & -CH_2 & -OH \\ CH_3(CH_2)_4CO & OH & HO & CO(CH_2)_4CH_3 \\ \end{bmatrix}$ 

Syntheses

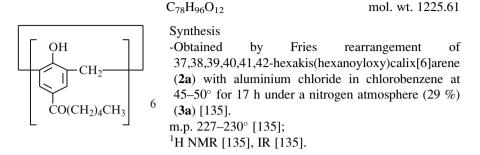
-Preparation by condensation of two molecules of acylphloroglucinol with formaldehyde or methoxymethyl acetate [3391].

mol. wt. 656.81

-Also refer to: [1571].

m.p. 116–118° [3391], 95–97° [1571].

#### 5,11,17,23,29,35-Hexahexanoyl-37,38,39,40,41,42-hexahydroxycalix [6]arene



# 2 Aromatic Hydroxyketones Derived from Various Halogenohexanoic Acids

# 2.1 Unsubstituted Hydroxyketones

# 2,2,3,3,4,4,5,5,6,6,6-Undecafluoro-1-(2-hydroxyphenyl)-1-hexanone

[173979-31-2]	$C_{12}H_5F_{11}O_2$	mol. wt. 390.15
OH COCF ₂ CF ₂ CF ₂ CF ₂ CF ₃	anisole with 48 % h acetic acid at 110° fo	nt of 4-(perfluoro-n-hexyl) ydrobromic acid in glacial r 22 days (30 %) [626].

¹H NMR [626], ¹⁹F NMR [626], IR [626], MS [626].

# 2,2,3,3,4,4,5,5,6,6,6-Undecafluoro-1-(4-hydroxyphenyl)-1-hexanone

[173979-30-1]	$C_{12}H_5F_{11}O_2$	mol. wt. 390.15
ОН	Synthesis	
$\checkmark$	-Obtained by treatment of 4-(	perfluoro-n-hexyl)anisole

with 48 % hydrobromic acid in glacial acetic acid at 110° for 6 days (79 %) [626].

COCF₂CF₂CF₂CF₂CF₃ b.p.₇₀ 185° [626]; m.p. 67° [626];

¹H NMR [626], ¹⁹F NMR [626], IR [626], MS [626].

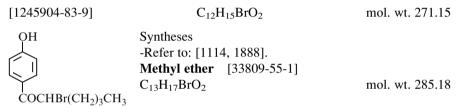
### 2-Chloro-1-(2-hydroxyphenyl)-1,3-hexanedione

$$\begin{array}{cccc} C_{12}H_{13}ClO_3 & \text{mol. wt. } 240.69 \\ \hline OH & Synthesis \\ \hline COCHClCOC_3H_7 & -Refer to: [3400]. \\ \hline Methyl ether & [1001024-96-9] \\ \hline C_{13}H_{15}ClO_3 & \text{mol. wt. } 254.71 \end{array}$$

-Obtained by reaction of N-chlorosuccinimide with 1-(2-hydroxyphenyl)-1,3-hexanedione in refluxing carbon tetrachloride for 4 h at 75–80° (81 %) [3400].

yellowish oil [3400]; ¹H NMR [3400], ¹³C NMR [3400], IR [3400], MS [3400]; GC-MS [3400].

# 2-Bromo-1-(4-hydroxyphenyl)-1-hexanone



-Obtained by treatment of 1-(4-methoxyphenyl)-1-hexanone with bromine,

*in ethyl ether and glacial acetic acid at r.t. (89 %) [1114];

*in a mixture of ethyl ether/dioxane at r.t. (46 %) [3232].

-Also obtained by treatment of 1-(4-methoxyphenyl)-1-hexanone with bromine in the presence of a catalytic amount of aluminium chloride (nearly quantitative yield) [2157].

-Also refer to: [1186, 1870, 2157].

b.p.₁₄ 182–184° [3232]; m.p. 54–55° [1186, 1870], 51–52° [1114]; ¹H NMR [1114, 1186, 1870, 2157, 3232], ¹³C NMR [2157], IR [3232], MS [1114, 1186, 1870].

# 6-Bromo-1-(2-hydroxyphenyl)-1-hexanone

[51821-14-8]	$C_{12}H_{15}BrO_2$	mol. wt. 271.15
OH CO(CH ₂ ) ₄ CH ₂ Br	Synthesis -Obtained by demethylation of boron tribromide in methylene (73 %) [1430]. m.p. 44.5–45° [1430]; ¹ H NMR [1	chloride at $-20^{\circ}$

-Obtained by interaction of 6-bromohexanenitrile with the Grignard reagent derived from o-bromoanisole, followed by acid hydrolysis (52 %) [1430].

IR [1430].

# 6-Bromo-1-(3-hydroxyphenyl)-1-hexanone

C	$_2H_{15}BrO_2$	mol. wt. 271.15
HO CO(CH ₂ ) ₄ CH ₂ Br	Synthesis -Refer to: [154]. ¹ H NMR [154].	

# 6-Bromo-1-(4-hydroxyphenyl)-1-hexanone

 $[188973-67-3] C_{12}H_{15}BrO_2 mol. wt. 271.15$ 

OH CO(CH₂)₄CH₂Br Syntheses -Obtained by Friedel-Crafts acylation of phenol with 6-bromohexanoyl chloride in the presence of aluminium chloride [2459] in methylene chloride at 25° [2919].

 $CO(CH_2)_4CH_2Br$  -Also refer to: [154, 217, 1314, 1473, 2459, 3419].

¹H NMR [154, 3419].

USE: Polymeric prodrugs having temporary linkages to amino groups [1314].

**Methyl ether** [57840-61-6] C₁₃H₁₇BrO₂ mol. wt. 285.18

-Obtained by Friedel-Crafts acylation of anisole,

*with 6-bromohexanoic acid using alkylhalosilanes and indium halides or triflates [217];

*with 6-bromohexanoyl chloride in the presence of aluminium chloride (85 %) [1509], in 1,2-dichloroethane at  $-10^{\circ}$  for 1 h (87 %) [2943], at 25° [2919] or in dichloromethane at  $-10^{\circ}$  for 1–2 h under nitrogen (73 %) [572].

-From 6-bromo-1-fluoro-1-(p-methoxyphenyl)-1-hexene in  $CDCl_3$  or neat  $(H_2O)$  [90].

-Also refer to: [572, 620, 1123, 1473, 1509, 1630, 2298, 2950].

m.p. 50–52° [2943], 43–44° [572], 41–43° [620]; ¹H NMR [572, 1509, 2943], ¹³C NMR [2943], MS [1509, 2943]; GC-MS [1509].

Phenyl ether	[164396-78-5]	$C_{18}H_{19}BrO_2$	mol. wt. 347.25
D 6 (717)			

-Refer to: [717].

#### 6-Bromo-1-(2,4-dihydroxyphenyl)-1-hexanone

$$\begin{array}{c} C_{12}H_{15}BrO_{3} \\ OH \\ CO(CH_{2})_{4}CH_{2}Br \\ HO \end{array} \begin{array}{c} \text{Syntheses} \\ -\text{Refer to: [154, 308].} \\ ^{1}\text{H NMR [154].} \end{array}$$

**Dimethyl ether** [118018-78-4]  $C_{14}H_{19}BrO_3$  mol. wt. 315.20

-Refer to: [308, 1123].

#### 6-Bromo-1-(2,6-dihydroxyphenyl)-1-hexanone

 $[1111652-08-4] C_{12}H_{15}BrO_3 mol. wt. 287.15$  OH Synthesis  $CO(CH_2)_4CH_2Br -Refer to: [1219] (Chinese patent).$  OH

USE: Preparation of low swelling sulfonated polyimide proton exchange membrane for fuel cell [1219].

#### 6-Bromo-1-(3,4-dihydroxyphenyl)-1-hexanone

$$C_{12}H_{15}BrO_3$$
 mol. wt. 287.15

 $\begin{array}{ccc} OH & Synthesis \\ OH & -Refer to: [2919]. \\ \hline \\ Dimethyl ether & [123014-46-0] \\ C_{14}H_{19}BrO_3 & mol. wt. 315.20 \end{array}$ 

-Obtained by Friedel-Crafts acylation of veratrole with 6-bromohexanoyl chloride in the presence of aluminium chloride in methylene chloride at 25° [2919]. -Also refer to: [154, 560, 561, 1473, 1542, 1543, 3419].

m.p. 44–45° [1543], 38–40° [154, 3419]; ¹H NMR [1543], ¹³C NMR [1543].

#### Methylenedioxy

 $C_{13}H_{15}BrO_3$ 

mol. wt. 299.16

¹H NMR [3419].

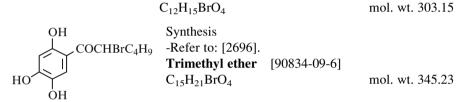
#### 2-Bromo-1-(3,4-dihydroxyphenyl)-1-hexanone

$$\begin{array}{ccc} C_{12}H_{15}BrO_{3} & \mbox{mol. wt. } 287.15 \\ OH & Synthesis \\ \hline OH & -Refer to: [2657]. \\ \hline Dibenzyl ether & C_{26}H_{27}BrO_{3} & \mbox{mol. wt. } 467.40 \\ -Obtained by reaction of N-bromosuccinimide with \end{array}$$

3,4-(dibenzyloxy)caprophenone in carbon tetrachloride in the presence of benzoyl peroxide at  $50^{\circ}$  (85–90 %) [2657].

m.p. 98° [2657].

#### 2-Bromo-1-(2,4,5-trihydroxyphenyl)-1-hexanone



-Obtained by reaction of bromine with 2,4,5-trimethoxycaprophenone in acetic acid at  $35-40^{\circ}$ , then at  $25^{\circ}$  for 40 min [2695]. -Also refer to: [2696].

m.p. 55–58° [2696], 55–56° [2695]; ¹H NMR [2695, 2696], IR [2695, 2696], MS [2695, 2696].

#### 6-Chloro-1-(2-hydroxyphenyl)-1-hexanone

[501083-62-1]	$C_{12}H_{15}Cle$	$O_2$	mol. wt. 226.70
OH CO(CH ₂ ) ₄ CH ₂	Synthesis Cl -Refer to: [2459]. ¹ H NMR [2459], N	MS [2459].	
Methyl ether	[501083-60-9]	C ₁₃ H ₁₇ ClO ₂	mol. wt. 240.73
-Refer to: [2459].			

¹H NMR [2459], MS [2459].

#### 6-Chloro-1-(3-hydroxyphenyl)-1-hexanone

[501083-64-3]	$C_{12}H_{15}ClO_2$	mol. wt. 226.70
OH CO(CH ₂ ) ₄ CH ₂ Cl	Synthesis -Refer to: [2459]. ¹ H NMR [2459], MS [2459].	

Methyl ether[258882-50-7] $C_{13}H_{17}CIO_2$ mol. wt. 240.73

-Preparation by treatment of 6-chloro-1-(3-methoxyphenyl)-1-hexanol with chromium trioxide in dilute sulfuric acid (Jones' reagent) in acetone first at  $0^{\circ}$ , then at r.t. for 6 h [2460].

-Also refer to: [2459].

¹H NMR [2460], MS [2460].

#### 6-Chloro-1-(4-hydroxyphenyl)-1-hexanone

	$C_{12}H_{15}C_{12}$	C10 ₂	mol. wt. 226.70
OH	Synthesis -Refer to: [228	71.	
$\square$		[278619-91-3]	mol. wt. 240.73
CO(CH ₂ ) ₄ CH ₂ Cl			

-Obtained by Friedel-Crafts acylation of anisole,

*with tert-butyl 6-chlorohexanoate in the presence of indium tribromide using dimethylchlorosilane (1 t) (64 %) [2287];

*with 6-chlorohexanoic acid using alkylhalosilanes, and indium halides or triflates [217].

b.p._{0.1} 130° [2287]; ¹H NMR [2287], ¹³C NMR [2287], IR [2287], MS [2287].

#### 6-Chloro-1-(3,4-dihydroxyphenyl)-1-hexanone

	$C_{12}H_{15}ClO_3$	mol. wt. 242.70
	Synthesis	
OH	-Refer to: [2560].	
	Dimethyl ether [19347-74-1]	

	The second se		
	Dimethyl ether	[19347-74-1]	
$\mathbf{i}$	$C_{14}H_{19}ClO_3$		mol. wt. 270.76
CO(CH ₂ ) ₄ CH ₂ Cl	-Refer to: [2560].		

# 2.2 Substituted Hydroxyketones

#### 6-Bromo-1-(2,3-dichloro-4-hydroxyphenyl)-1-hexanone

$$C_{12}H_{13}BrCl_2O_2$$
 mol. wt. 340.04

OH Synthesis Cl -Refer to: [3333]. Methyl ether [53107-64-5] Cl  $C_{13}H_{15}BrCl_2O_2$  mol. wt. 354.07 CO(CH₂)₄CH₂Br

-Obtained by reaction of 6-bromohexanoyl chloride with 2,3-dichloroanisole in the presence of aluminium chloride (21 %) [3333]. -Also refer to: [732, 736–738, 740, 2052, 2053].

m.p. 52–53° [2051, 2052], 50° [3333].

USE: Prepn. and cyclization of, [736, 737, 2053]; Prepn. and reaction of, with formaldehyde [738, 2052].

#### 2-Bromo-1-(3,5-dibromo-2,4-dihydroxyphenyl)-1-hexanone

 $[238074-78-7] C_{12}H_{13}Br_{3}O_{3} mol. wt. 444.95$ OH Synthesis Br CO-CH-(CH₂)₃CH₃ -Refer to: [998]. HO Br

#### 6,6,6-Trifluoro-1-(3-hydroxyphenyl)-1-hexanone

[104325-65-7]	$C_{12}H_{13}F_{3}O_{2}$	mol. wt. 246.23
OH CO(CH ₂ ) ₄ CF ₃	Syntheses -Refer to: [2197, 2198]. USE: In preparation of antiinflammatory agents [2198].	and antiallergic

#### 6-Bromo-1-(2-chloro-4-hydroxyphenyl)-1-hexanone

	$C_{12}H_{14}BrClO_2$	mol. wt. 305.60
он	Synthesis	
$\checkmark$	-Refer to: [1123].	
	Methyl ether [118108-79-5]	
C1	$C_{13}H_{16}BrClO_2$	mol. wt. 319.63
CO(CH ₂ ) ₄ CH ₂ Br	-Refer to: [1123].	

#### 6-Bromo-1-(3-chloro-4-hydroxyphenyl)-1-hexanone

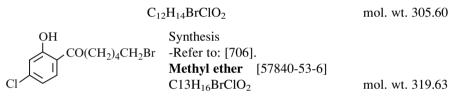
$$\begin{array}{ccc} C_{12}H_{14}BrClO_2 & mol. wt. 305.60 \\ OH & Synthesis \\ \hline Cl & -Refer to: [2919]. \\ \hline Methyl ether & [188973-65-1] \\ C_{13}H_{16}BrClO_2 & mol. wt. 319.63 \\ \hline CO(CH_2)_4CH_2Br \end{array}$$

-Obtained by Friedel-Crafts acylation of 2-chloroanisole with 6-bromohexanoyl chloride in the presence of aluminium chloride in methylene chloride at  $25^{\circ}$  [2919].

-Also refer to: [154, 3419].

m.p. 104-106° [154, 3419].

#### 6-Bromo-1-(4-chloro-2-hydroxyphenyl)-1-hexanone



-Refer to: [706].

### 6-Bromo-1-(5-chloro-2-hydroxyphenyl)-1-hexanone

[173055-27-1]  $C_{12}H_{14}BrClO_2$  mol. wt. 305.60 OH Synthesis CO(CH₂)₄CH₂Br -Refer to: [2623].

#### 6-Bromo-1-(3-fluoro-4-hydroxyphenyl)-1-hexanone

	$C_{12}H_{14}BrFO_2$	mol. wt. 289.14
OH ↓ F	Synthesis -Refer to: [2919].	
	<b>Methyl ether</b> [188973-66-2] C ₁₃ H ₁₆ BrFO ₂	mol. wt. 303.17
CO(CH ₂ ) ₄ CH ₂ Br		

-Obtained by Friedel-Crafts acylation of 2-fluoroanisole with 6-bromohexanoyl chloride in the presence of aluminium chloride in methylene chloride at 25° [2919]. -Also refer to: [154, 1473, 3419].

m.p. 47–48° [154, 3419].

#### 6-Bromo-1-(5-fluoro-2-hydroxyphenyl)-1-hexanone

[173055-25-9]  $C_{12}H_{14}BrFO_2$  mol. wt. 289.14 OH Synthesis CO(CH₂)₄CH₂Br -Refer to: [2623].

6-Bromo-1-(2-fluoro-4,5-dihydroxyphenyl)-1-hexanone

	$C_{12}H_{14}BrFO_3$		mol. wt. 305.14
OH HO	Synthesis -Refer to: [560].	[102015 04 0]	
F CO(CH ₂ ) ₄ CH ₂ Br	<b>Dimethyl ether</b> $C_{14}H_{18}BrFO_3$ -Refer to: [560].	[123015-34-9]	mol. wt. 333.20

m.p. 81-83° [558, 559]; MS [558, 559].

#### 2-Bromo-1-(3-bromo-4-hydroxyphenyl)-1-hexanone

	$\mathrm{C}_{12}\mathrm{H}_{14}\mathrm{Br}_{2}\mathrm{O}_{2}$		mol. wt. 350.05
OH ↓ ,Br	Synthesis -Refer to: [441].		
DI DI	Methyl ether (2S)	[306972-96-3]	
COCHBr(CH ₂ ) ₃ CH ₃	$\mathrm{C}_{13}\mathrm{H}_{16}\mathrm{Br}_{2}\mathrm{O}_{2}$		mol. wt. 364.08

-Preparation of nonracemic  $\alpha$ -bromoketones by stereoselective bromination of aryl alkyl tartrate acetals followed by hydrolysis (67 %, 66 % ee) [441].

m.p. 58–60° [441]; ¹H NMR [441], IR [441], MS [441].

#### 6-Chloro-1-(3-fluoro-4-hydroxyphenyl)-1-hexanone

	$C_{12}H_{14}ClFO_2$	mol. wt. 244.69
OH ↓ ,F	Synthesis -Refer to: [217].	
	<b>Methyl ether</b> [927911-86-2] C ₁₃ H ₁₆ ClFO ₂	mol. wt. 258.72
CO(CH ₂ ) ₄ CH ₂ Cl		

-Obtained by Friedel-Crafts acylation of 2-fluoroanisole with 6-chlorohexanoic acid using alkylhalosilanes, and indium halides or triflates [217].

-Also obtained by reaction of 6-chlorohexanoic acid with 2-fluoroanisole in the presence of  $HSiMe_2Cl$  and  $InCl_3$  in 1,2-dichloroethane, first 1 h at r.t., then heated at 80° for 4 h (72 %) [218].

m.p. 35–37° [218]; ¹H NMR [218], ¹³C NMR [218], IR [218], MS [218].

#### 6-Chloro-1-(4-fluoro-2-hydroxyphenyl)-1-hexanone

[263545-26-2]	$C_{12}H_{14}ClFO_2$	mol. wt. 244.69
OH CO(CH ₂ ) ₄ CH ₂ Cl	Syntheses -Refer to: [1662, 1663]. <b>Oxime</b> [263545-27-3]	
F	Oxime [263545-27-3] $C_{12}H_{15}CIFNO_2$	mol. wt. 259.71

-Refer to: [1662, 1663].

#### 6-Chloro-1-(2,3-dichloro-4-hydroxyphenyl)-2-methylene-1-hexanone

	$C_{13}H_{13}Cl_3O_2$	mol. wt. 307.60
$\bigcup_{\substack{I \\ CO - C - (CH_2)_3 CH_2 CI \\ CH_2}}^{OH}$	Syntheses -Refer to: [732, 738, 2052]. <b>Methyl ether</b> [54343-87-2] C ₁₄ H ₁₅ Cl ₃ O ₂ -Refer to: [732, 738, 2052].	mol. wt. 321.63

-Prepn. and cyclization of, [738, 2052].

### 2-Bromo-1-(2-hydroxy-5-methylphenyl)-1-hexanone

С	$_{13}H_{17}BrO_2$	mol. wt. 2
$\bigcup_{\substack{I \\ CH_3}}^{OH} CO-CH-(CH_2)_3CH_3$	Synthesis -Refer to: [181]. m.p. 30.5–31.5° [181].	

### 6-Bromo-1-(2-hydroxy-5-methylphenyl)-1-hexanone

[173055-29-3]  $C_{13}H_{17}BrO_2$  mol. wt. 285.18 OH Synthesis CO(CH₂)₄CH₂Br -Refer to: [2623].

285.18

#### 6-Bromo-1-(4-hydroxy-3-methylphenyl)-1-hexanone

 $\begin{array}{cccc} [868521-08-8] & C_{13}H_{17}BrO_2 & \mbox{mol. wt. } 285.18 \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\$ 

#### 6-Bromo-1-(2-hydroxy-4-methoxyphenyl)-1-hexanone

 $[173055-23-7] C_{13}H_{17}BrO_3 mol. wt. 301.18$ OH Syntheses CO(CH₂)₄CH₂Br -Refer to: [308, 2623, 3284]. CH₃O

#### 6-Bromo-1-(2-hydroxy-5-methoxyphenyl)-1-hexanone

[173055-21-5]	$C_{13}H_{17}BrO_3$	mol. wt. 301.18
$\bigcup_{OCH_3}^{OH} CO(CH_2)_4 CH_2 Br$	Syntheses -Refer to: [2623, 2918].	

## 6-Chloro-1-(2-hydroxy-5-methylphenyl)-1-hexanone

[51317-86-3]	$C_{13}H_{17}ClO_2$	mol. wt. 240.73
OH CO(CH ₂ ) ₄ CH ₂ Cl CH ₃	•	of 6-chlorocaproic acid with boron trifluoride (86 %) [2311]. .64 [2311].

#### 6-Chloro-1-(4-hydroxy-3-methylphenyl)-1-hexanone

	$C_{13}H_{17}ClO_2$	mol. wt. 240.73
OH CH ₃	Synthesis -Refer to: [218].	
CO(CH ₂ ) ₄ CH ₂ Cl	Methyl ether [927911-85-1] $C_{14}H_{19}ClO_2$	mol. wt. 254.76

-Obtained by reaction of 6-chlorohexanoic acid with 2-methylanisole in the presence of  $HSiMe_2Cl$  and  $InCl_3$  (or  $InBr_3$ ) in 1,2-dichloroethane, first 1 h at r.t., then heated at 80° for 4 h (78 %) (or 83 %) [218].

m.p. 44–46° [218]; ¹H NMR [218], ¹³C NMR [218], IR [218], MS [218].

#### 6-Bromo-1-(3,4-dihydroxy-2,5-dimethylphenyl)-1-hexanone

	$C_{14}H_{19}BrO_3$		mol. wt. 315.20
CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CO(CH ₂ ) ₄ CH ₂ Br	Synthesis -Refer to: [560]. <b>Dimethyl ether</b> $C_{16}H_{23}BrO_3$ -Refer to: [560].	[123015-22-5]	mol. wt. 343.26

MS [558, 559].

#### 6-Bromo-1-(2-hydroxy-4,5-dimethoxyphenyl)-1-hexanone

[173055-31-7]	$C_{14}H_{19}BrO_4$	mol. wt. 331.20
CH ₃ O OCH ₃ O OCH ₃ O	Syntheses -Refer to: [2623, 2918]. ¹ H NMR [2918].	

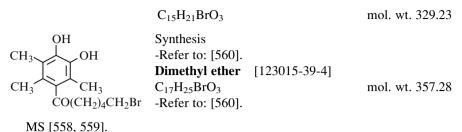
#### 6-Bromo-1-(2-hydroxy-4,6-dimethoxyphenyl)-1-hexanone

[173055-33-9]	$C_{14}H_{19}BrO_4$	mol. wt. 331.20
OH CO(CH ₂ ) ₄ CH ₂ Br CH ₃ O OCH ₃	Synthesis -Refer to: [2623].	

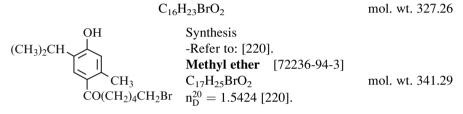
#### 2-Bromo-3,5,5-trimethyl-1-(4-hydroxyphenyl)-1-hexanone

C ₁₅	$H_{21}BrO_2$	mol. wt. 313.23
$\bigcup_{\substack{I \\ CO-CH-CH-CH_2-C-CH_3 \\ CH_3 \\ CH_3}}^{OH} H_1^{CH_3} H_2^{CH_3} H_$	Synthesis -Refer to: [1140]. <b>Pentyl ether</b> [97744-24-6] $C_{20}H_{31}BrO_2$ -Refer to: [1140].	mol. wt. 383.37

#### 6-Bromo-1-(3,4-dihydroxy-2,5,6-trimethylphenyl)-1-hexanone



#### 6-Bromo-1-[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-hexanone



-Obtained by reaction of 6-bromohexanoyl chloride with thymol methyl ether in the presence of aluminium chloride in methylene chloride at r.t. (90 %) [220].

BIOLOGICAL ACTIVITY: Amebicidal and bactericidal and molluscicidal [220].

#### 6-Bromo-1-[3,4-dihydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-hexanone

C ₁₆	H ₂₃ BrO ₃		mol. wt. 343.26
OH (CH ₃ ) ₂ CH OH	Synthesis -Refer to: [560].		
	Dimethyl ether	[123015-21-4]	
$CO(CH_2)_4CH_2Br$	$C_{18}H_{27}BrO_3$ -Refer to: [560].		mol. wt. 371.31

#### 5-Chloro-1-(2,6-dihydroxy-3,4-dimethoxyphenyl)-2,4,5-trimethyl-1-hexanone

[1049661-48-4]	C ₁₇ H ₂₅ ClO ₅	mol. wt. 344.83
CH ₃ O CH ₃ O CH ₃ O OH	$\begin{array}{ccc} CH_3 & CH_3 & CH_3 & Synthesis\\ CH-CH_2-CH-C-CH_3 & -Refer to: [802].\\ Cl\end{array}$	

#### 6-Bromo-1-(4-hydroxy[1,1'-biphenyl]-3-yl)-1-hexanone

$$\begin{array}{ccc} C_{18}H_{19}BrO_2 & \text{mol. wt. } 347.25 \\ \hline \\ CO(CH_2)_4CH_2Br & Synthesis \\ -Refer to: [308]. \\ \hline \\ Methyl \ ether & [187396-83-4] \\ C_{19}H_{21}BrO_2 & \text{mol. wt. } 361.27 \end{array}$$

-Refer to: [308].

# 3 Aromatic Hydroxyketones Derived from 6-Oxohexanoic Acid

## 3.1 Unsubstituted Hydroxyketones

#### 6-(2-Hydroxyphenyl)-6-oxo-1-hexanoic acid

[100118-19-2]	$C_{12}H_{14}O_4$	mol. wt. 222.24
---------------	-------------------	-----------------

-Obtained by Fries rearrangement of phenyl adipate with aluminium chloride,

-in the presence of solvents:

*in the tetrachloroethane/nitrobenzene mixture (500 ml/125 ml) for 4 h at 50–60° [902];

*in nitrobenzene for 4 h at  $50^{\circ}$ , then 30 min at  $60^{\circ}$  [902];

Syntheses

*in tetrachloroethane at 80° [902].

-without solvent at  $120^{\circ}$  [902].

-Also obtained by treatment of hexahydrodiphenyl oxide with chromium trioxide in acetic acid on a water bath (30 %) [444].

-Also obtained by diazotization of  $\delta$ -o-aminobenzoylvaleric acid [2419].

-Also obtained by degradation of 2,3-dihydropentachromone (m.p.  $120-121^{\circ}$ ) with KOH in dilute ethanol (69 %) [1211].

-Also refer to: [936 (80 %), 3370].

b.p.₁₂ 240–242° [444]; m.p. 94° [444, 902, 2419], 93–94° [1211], 90° [936].

Oxime	[100391-90-0]	$C_{12}H_{15}NO_4$	mol. wt. 237.26
m.p. 128°	[444].		
Semicarbazo	ne [100706-52-3]	$C_{13}H_{17}N_3O_4$	mol. wt. 279.30
m.p. 186°	[444].		

<b>Phenylhydrazone</b> m.p. 173° [444, 24	C ₁₈ H ₂₀ 419].	N ₂ O ₃	mol. wt. 312.37
<b>Benzoate</b> m.p. 82° [444].	$C_{19}H_{18}O_5$		mol. wt. 326.35
Methyl ether -Obtained by treatme -Also refer to: [2495 m.p. 82° [444, 249		$C_{13}H_{16}O_4$ methyl iodide [444].	mol. wt. 236.27
Semicarbazone of th m.p. 175–176° [44	·	$C_{14}H_{19}N_3O_4$	mol. wt. 293.32
Methyl ester of the m.p. 28° [444].	methyl ether	$C_{14}H_{18}O_4$	mol. wt. 250.29

#### 6-(4-Hydroxyphenyl)-6-oxo-1-hexanoic acid

[5537-75-7]	$C_{12}H_{14}O_4$	mol. wt. 222.24
HO — CO(CH ₂ ) ₄ CO ₂ H	Syntheses -Obtained by Fries adipate with alumini	rearrangement of phenyl um chloride,

-in the presence of solvents:

*in the tetrachloroethane/nitrobenzene mixture (500 ml/125 ml) for 4 h at 50–60° [902];

*in nitrobenzene for 4 h at  $50^{\circ}$ , then 30 min at  $60^{\circ}$  [902];

*in tetrachloroethane at  $80^{\circ}$  [902].

-without solvent at  $120^{\circ}$  [902].

-Also obtained by reaction of adipyl chloride with phenol in the presence of aluminium chloride in nitrobenzene, first at  $0-5^\circ$ , then at r.t. for 12 h (36 %) [593]. -Also obtained by reaction of  $\delta$ -carbomethoxyvaleroyl chloride with phenol in the presence of aluminium chloride in chlorobenzene, first at  $10-15^\circ$ , then at  $60^\circ$  for 6 h (45 %) [2519].

-Also refer to: [445, 1073, 3135].

m.p. 150° [593], 149–150° [2519], 148° [902, 3135], 147° [445].

#### **2,4-Dinitrophenylhydrazone** [5485-73-4] C₁₈H₁₈N₄O₇ mol. wt. 402.36

m.p. 166° [593].

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Methyl ether [5537-76-8] C<sub>13</sub>H<sub>16</sub>O<sub>4</sub> mol. wt. 236.27
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-Obtained by reaction of adipyl chloride with anisole in the presence of aluminium chloride in nitrobenzene/tetrachloroethane mixture (1:2) at  $0^{\circ}$  (45 %) [593].

-Also obtained (by-product) in the preparation of 1,6-bis(4-methoxyphenyl)-1,6-hexanedione (12–16 %) [1073].

-Also obtained by reaction of  $\delta$ -carbomethoxyvaleroyl chloride with anisole in the presence of aluminium chloride,

*in a mixture of tetrachloroethane and chlorobenzene at 0–5° for 3 h and left overnight (79 %) [2519];

*in a mixture of tetrachloroethane and nitrobenzene (79 %) [2519];

*in tetrachloroethane at  $0^{\circ}$  for 3–4 h (95 %) [2392].

-Also obtained by reaction of adipic anhydride with anisole in the presence of aluminium chloride,

*in refluxing carbon disulfide for 3 h (23 %) [2494];

*in a mixture of nitrobenzene and tetrachloroethane for two days at  $5^{\circ}$  (33 %) [2519].

-Also obtained by treatment of its methyl ester in THF with 2 N sodium hydroxide at r.t. overnight (87 %) [1724].

-Also refer to: [178, 242, 701, 988, 1131, 2168, 3195].

colourless plates [2494];

m.p. 129° [593], 128–129° [2494], 128° [2392], 127–129° [242], 127–127.5° [2168, 3195], 127° [988, 2494], 126° [1073], 123–125° [178], 122–124° [2519];

¹H NMR [701, 1724], IR [242, 701, 1724].

BIOLOGICAL ACTIVITY: Delivery of human zinc insulin [1131].

Methyl ester of the methyl ether [29389-23-9]  $C_{14}H_{18}O_4$  mol. wt. 250.29

-Obtained by reaction of adipic acid monochloride monomethyl ester with anisole in the presence of aluminium chloride in methylene chloride at  $0^{\circ}$  for 30 min [1724].

-Refer to: [1274, 1512].

m.p. 65° [1274], 64–65° [1512]; ¹H NMR [1512, 1724], ¹³C NMR [1512], IR [1512, 1724], MS [1512].

Ethyl ester of the methyl ether [42916-80-3]  $C_{15}H_{20}O_4$  mol. wt. 264.32

-Obtained by reaction of ethyl 5-(chloroformyl)pentanoate with anisole in the presence of aluminium chloride in tetrachloroethane (50 %) [2689]. -Also refer to: [878 (49 %), 1865, 2689, 3254].

b.p._{0.05} 142–155° [878]; m.p. 54° [3254], 52–54° [2689], 44–45° [878]; UV [3254], ESR spectroscopy [3254]; phosphorescence spectroscopy [3254]. **Ethyl ether** [854659-09-9]  $C_{14}H_{18}O_4$  mol. wt. 250.29

-Obtained by reaction of adipic acid dichloride with phenetole in the presence of aluminium chloride in carbon disulfide first at  $-10^{\circ}$ , then at r.t. for 5 h (16 %) [3196].

-Also refer to: [2494].

colourless plates [2494]; m.p. 111–112° [3196], 110° [2494].

Ethyl ester [119348-65-1]  $C_{14}H_{18}O_4$  mol. wt. 250.29

-Obtained by treatment of 6-(4-hydroxyphenyl)-6-oxo-1-hexanoic acid in ethanol with methanesulfonic acid at  $25^{\circ}$  for 25 h (74 %) [878].

m.p. 66–67° [878].

#### 6-(2,4-Dihydroxyphenyl)-6-oxo-1-hexanoic acid

[133535-20-3]	$C_{12}H_{14}O_5$	mol. wt. 238.24
HO HO HO	Syntheses -Obtained by reaction of adip in the presence of zinc chl (20 %) [445]. -Also refer to: [2606, 3370].	

C13H16O5

 $b.p._{0.5} \ 250^{\circ} \ [445]; \quad m.p. \ 171^{\circ} \ [445], \ 169^{\circ} \ [2606].$ 

#### Methyl ester

b.p._{0.4} 230° [445]; m.p. 113° [445].

#### 6-(2,5-Dihydroxyphenyl)-6-oxo-1-hexanoic acid

pale yellow leaflets [469]; m.p. 130–131.5° [3175], 130° [469].

#### 2,4-Dinitrophenylhydrazone C₁₈H

 $C_{18}H_{18}N_4O_8$ 

mol. wt. 418.36

orange plates [469]; m.p. 194° [469].

[445] 160° [**2**606]

mol. wt. 252.27

#### **Dimethyl ether** [79381-16-1] C₁₄H₁₈O₅ mol. wt. 266.29

-Obtained by reaction of polymeric adipic anhydride with hydroquinone dimethyl ether in the presence of aluminium chloride (53 %) [2331].

-Also obtained by reaction of adipoyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride,

*in carbon disulfide at  $< 10^{\circ}$  (48 %) [3175];

*in nitrobenzene at  $0^{\circ}$  for 5 h (60 %) [3128].

-Also refer to: [3056].

needles [2331]; white prisms [3175]; colourless solid [3128]; m.p. 80.5–82° [3175], 78–80° [2331, 3056], 75–77° [3128]; ¹H NMR [3128], ¹³C NMR [3128].

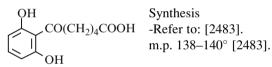
#### Methyl ester of the dimethyl ether [374808-50-1] C₁₅H₂₀O₅ mol. wt. 280.32

-Obtained by treatment of dimethyl ether with methanol in the presence of concentrated sulfuric acid (2 drops) at reflux for 18 h (94 %) [3128].

pale yellow oil [3128]; ¹H NMR [3128], ¹³C NMR [3128].

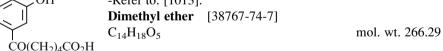
#### 6-(2,6-Dihydroxyphenyl)-6-oxo-1-hexanoic acid

 $C_{12}H_{14}O_5$  mol. wt. 238.24



#### 6-(3,4-Dihydroxyphenyl)-6-oxo-1-hexanoic acid

 $C_{12}H_{14}O_5 \qquad \text{mol. wt. } 238.24$ Synthesis OH -Refer to: [1013].



-Obtained by heating of its methyl ester below with 10 % aqueous NaOH on the steam bath for 1.5 h (90 %) [1013].

-Also obtained (by-product) by reaction of adipyl chloride with veratrole in the presence of aluminium chloride in carbon disulfide first at  $0^{\circ}$ , then at reflux for 3 h [1124].

-Also obtained by treatment of ethyl 6-(3,4-dimethoxyphenyl)-6-oxo-1-hexanoate with 10 % ethanolic NaOH at r.t. for 3 h (97 %) [306]. -Also refer to: [1080, 2982, 3057, 3363, 3364].

m.p. 123–125° [306], 123° [2982, 3057], 122–123.5° [3363], 122–123° [1013, 1124], 111–115° [1080]; ¹H NMR [306], IR [1013, 3364]. Methyl ester of the dimethyl ether [57641-18-6] C₁₅H₂₀O₅ mol. wt. 280.32

-Obtained by reaction 6-chloro-6-oxohexanoic acid methyl ester with 1,2-dimethoxybenzene in the presence of aluminium chloride in 1,1,2,2-tetrachloroethane first at  $0-3^{\circ}$  for 2.5 h, then overnight to  $2^{\circ}$  (82 %) [1013].

b.p._{0.1} 175–176° [1013]; m.p. 65–66° [1013]; IR [1013].

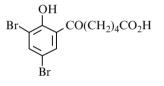
**Ethyl ester of the dimethyl ether** [167159-65-1] C₁₆H₂₂O₅ mol. wt. 294.35 -Refer to: [306].

m.p. 50–52° [306]; ¹H NMR [306].

#### 3.2 Substituted Hydroxyketones

6-(3,5-Dibromo-2-hydroxyphenyl)-6-oxo-1-hexanoic acid

$$C_{12}H_{12}Br_2O_4$$
 mol. wt. 380.03



Synthesis CO(CH₂)₄CO₂H -Obtained by treatment of 1-(2-hydroxyphenyl)-6-oxo-1-hexanoic acid with a potassium bromate/ potassium bromide mixture in dilute hydrochloric acid [936]. m.p. 128° [936].

#### 6-(3,5-Dibromo-4-hydroxyphenyl)-6-oxo-1-hexanoic acid

[100121-89-9]	$\mathrm{C}_{12}\mathrm{H}_{12}\mathrm{Br}_{2}\mathrm{O}_{4}$	mol. wt. 380.03
Br HO Br	Synthesis -Refer to: [2471]. m.p. 158° [2471].	

#### 6-(5-Chloro-2-hydroxyphenyl)-6-oxo-1-hexanoic acid

C12H13ClO4 [857480-74-1] mol. wt. 256.69 Syntheses  $CO(CH_2)_4CO_2H$  -Obtained by Fries rearrangement of bis(4-chlorophenyl) adipate (m.p. 110.5°) with aluminium chloride (1 mol) at 150° for 1 h (31 %) [3235]. -Also obtained by hydrolysis of 6-chloro-2,3-"dihydropentachromone" [2495].

m.p. 136° [2495, 3235].

#### 6-(4-Hydroxy-3-nitrophenyl)-6-oxo-1-hexanoic acid

$$\begin{array}{c} C_{12}H_{13}NO_6 & \text{mol. wt. } 267.24 \\ \hline NO_2 & \text{Synthesis} \\ HO \longrightarrow CO(CH_2)_4CO_2H & \text{Refer to: } [2494]. \\ \hline \textbf{Methyl ether} \\ C_{13}H_{15}NO_6 & \text{mol. wt. } 281.26 \end{array}$$

-Obtained by treatment of  $\delta$ -anisoylvaleric acid in concentrated sulfuric acid with potassium nitrate between -5 and  $0^{\circ}$  (70 %) [2494].

pale yellow plates; m.p. 107-109° [2494].

**Ethyl ether** [867134-00-7] C₁₄H₁₇NO₆ mol. wt. 295.29

-Refer to: [2494].

colourless prisms [2494]; m.p. 110° [2494].

#### 6-(3-Amino-4-hydroxyphenyl)-6-oxo-1-hexanoic acid

C ₁₂	$H_{15}NO_4$	mol. wt. 237.26
$HO \longrightarrow CO(CH_2)_4CO_2H$	Synthesis -Refer to: [2494]. <b>Methyl ether</b> $C_{13}H_{17}NO_4$	mol. wt. 251.28

-Obtained by adding a hot solution of 4-methoxy-3-nitrobenzoylvaleric acid in dilute ammonia to a boiling solution of ferrous sulfate to which an excess ammonia been added. The whole was boiled 10 min (41 %) [1531].

brown plates; m.p. 116° [2494].

Ethyl ether

#### $C_{14}H_{19}NO_4$

mol. wt. 265.31

-Refer to: [2494].

pink plates; m.p. 102° [2494].

#### 6-(2-Hydroxy-5-methylphenyl)-6-oxo-1-hexanoic acid

	$C_{13}H_{16}O_4$	mol. wt. 236.27
OH CO(CH ₂ ) ₄ COOH CH ₃	Synthesis -Refer to: [1827]. UV [1827].	

#### 6-(3-Hydroxy-4-methylphenyl)-6-oxo-1-hexanoic acid

[124016-88-2]  $C_{13}H_{16}O_4$  mol. wt. 236.27 HO CO(CH₂)₄COOH Synthesis -Refer to: [2972].

#### 6-(4-Hydroxy-2-methylphenyl)-6-oxo-1-hexanoic acid

 $C_{13}H_{16}O_4$  mol. wt. 236.27

HO 
$$\leftarrow$$
 CO(CH₂)₄CO₂H

Synthesis -Obtained by reaction of adipyl chloride with m-cresol in the presence of aluminium chloride in nitrobenzene at  $0^{\circ}$  [593].

m.p. 100° [593].

[5538-11-4]

**2,4-Dinitrophenylhydrazone** [5538-12-5] C₁₉H₂₀N₄O₇ mol. wt. 416.39 m.p. 185° [593].

Methyl ether	[5538-14-7]	$C_{14}H_{18}O_4$	mol. wt. 250.29
Methyl ether	[3336-14-7]	$C_{14}\Pi_{18}O_4$	11101. wt. 2.

-Obtained by reaction of adipyl chloride with m-cresol methyl ether in the presence of aluminium chloride in a nitrobenzene/tetrachloroethane mixture (1:1) at  $0^{\circ}$  [593].

b.p._{0.2} 160° [593].

#### 2,4-Dinitrophenylhydrazone of the methyl ether

[5550-55-0]	$C_{20}H_{22}N_4O_7$	mol. wt. 430.42

m.p. 175° [593].

m.p. 165° [593].

#### 6-(4-Hydroxy-3-methylphenyl)-6-oxo-1-hexanoic acid

[5538-07-8]	$C_{13}H_{16}O_4$		mol. wt. 236.27
HO-CO(CH ₂ ) ₄ CO ₂ H	Synthesis -Obtained by reaction of adipyl chloride with o-cresol in the presence of aluminium chloride in nitrobenzene at 0° (90 %) [593].		
m.p. 125° [593].			
2,4-Dinitrophenylhydrazone	[5550-52-7]	$C_{19}H_{20}N_4O_7$	mol. wt. 416.39

716

**Methyl ether** [5485-77-8] C₁₄H₁₈O₄ mol. wt. 250.29

-Obtained by reaction of adipyl chloride with o-cresol methyl ether in the presence of aluminium chloride in a nitrobenzene/tetrachloroethane mixture (1:1) at  $0^{\circ}$  [593].

m.p. 95° [593].

#### 2,4-Dinitrophenylhydrazone of the methyl ether

 $\begin{array}{ll} \mbox{[5538-09-0]} & C_{20}H_{22}N_4O_7 & \mbox{mol. wt. 430.42} \\ \mbox{m.p. } 162^\circ \mbox{[593]}. \end{array}$ 

#### 6-(2,4-Dihydroxy-5-methylphenyl)-6-oxo-1-hexanoic acid

[1344146-37-7]	$C_{13}H_{16}O_5$	mol. wt. 252.27
HO CH ₃ CO(CH ₂ ) ₄ COOH	Synthesis -Refer to: [1940]. m.p. 210–212° [1940]; ¹³ C NMR [1940], IR [19	¹ H NMR [1940], 940], MS [1940].

BIOLOGICAL ACTIVITY: Cytotoxic [1940].

#### 6-(2-Hydroxy-3,4,6-trimethylphenyl)-6-oxo-1-hexanoic acid

[58185-73-2]	$C_{15}H_{20}O_4$	mol. wt. 264.32
CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CO(CH ₂ ) ₄ COOH CH ₃	Synthesis -Refer to: [2149, 2325]. m.p. 146–148° [2149, 2325], 2325], IR [2149, 2325].	¹ H NMR [2149,

#### 6-(3-Cyclohexyl-4-hydroxyphenyl)-6-oxo-1-hexanoic acid

$C_{12}$	${}_{8}\mathrm{H}_{24}\mathrm{O}_{4}$		mol. wt.	304.39
HO CO(CH ₂ ) ₄ CO ₂ H	Synthesis -Preparation by ether [496]. m.p. 137° [496].	-	of its	methyl

#### Methyl ether

C₁₉H₂₆O₄ mol. wt. 318.41

-Obtained by reaction of  $\delta$ -carbomethoxyvaleryl chloride with o-cyclohexylanisole in the presence of aluminium chloride in benzene, first for 4 h between 3 and 8°, then at r.t. for 18 h. After hydrolysis, the residual crude methyl ester, after solvent elimination, was saponified with sodium hydroxide in refluxing methanol (50 %) [496].

colourless crystals [496]; m.p. 96° [496].

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# Chapter 5 Heptanones

# 1 Aromatic Hydroxyketones Derived from Heptanoic Acids

# 1.1 Unsubstituted Hydroxyketones

#### 1-(2-Hydroxyphenyl)-1-heptanone

[22362-59-0]

 $C_{13}H_{18}O_2$ 

mol. wt. 206.28

OH CO(CH₂)₅CH₃

-Obtained by Fries reaction of phenyl enanthate with aluminium chloride, *(58 %) [726];

*in nitrobenzene at 37.5° for 44.5 h (19 %) [244];

*without solvent at 140° for 45 min (51 %) [932].

**Svntheses** 

-Also obtained by reaction of enanthoyl chloride with phenol in the presence of aluminium chloride,

*without solvent, first at 100°, then at 125–130° for 1 h (48 %) [2700];

*in nitrobenzene, first at 0°, then at r.t. overnight (33 %) [2700].

- -Also obtained by stirring a solution of salicylaldehyde, 1-hexene,  $RhCl(PPh_3)_3$  [1435], acetonitrile and sodium acetate in methylene chloride at r.t. for 8 h under an argon atmosphere (86 %) [1434].
- -Also obtained by treatment of 4-chromanone with n-butyllithium in hexane/THF first at  $0^{\circ}$  for 4 h, then at r.t. overnight (16.3 %) [248].

-Also obtained by zeolite-catalyzed Friedel-Crafts reaction of anisole with heptanoic acid [3266].

-Also refer to: [1447, 2259, 2478, 2584, 3066].

R. Martin, J.-P. Buisson, Aromatic Hydroxyketones: Preparation & Physical Properties, DOI 10.1007/978-3-319-14185-5_5

mol. wt. 296.41

yellow semi-solid mass [248]; b.p._{0.1} 94° [932], b.p._{0.4} 104–106° [2478], b.p.₁₀ 155–156° [2700], b.p.₂₀ 172–174° [726]; m.p. 24° [726], 9.8° [2700]; ¹H NMR [248, 1435, 1447, 3066], IR [1447, 3066], UV [1996], MS [248, 1447]; TLC [248].  $n_{D}^{25} = 1.5205$  [2478],  $n_{D}^{25} = 1.5211$  [932].

USE: Cyclization of, with chloroacetamide or chloroacetonitrile [2584].

## **Oxime** $C_{13}H_{19}NO_2$ mol. wt. 221.30

-Obtained by treatment of 4-chromanone oxime with n-butyllithium in hexane/THF first at  $0^{\circ}$  for 4 h, then at r.t. overnight (54.8 %) [248].

white crystals [248]; m.p. 88–90° [248]; ¹H NMR [248], ¹³C NMR [248], IR [248], MS [248].

2,4-Dinitrophenylhydrazone	$C_{19}H_{22}N_4O_5$	mol. wt. 386.41
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m.p. 153° [932].

#### Phenylhydrazone

m.p. 91–92° [726].

Methyl ether	[118476-18-9]	$C_{14}H_{20}O_2$	mol. wt. 220.31

 $C_{19}H_{24}N_2O$ 

-Obtained by hydration of 1-(2-methoxyphenyl)-1-heptyne on treatment with sodium sulfide followed by aqueous HCl in methanol at  $65^{\circ}$  for 60 h (95 %) [611]. -Also refer to: [1648, 1649 (3 %)].

Ethyl ether	[52922-74-4]	$C_{15}H_{22}O_2$	mol. wt. 234.34

-Obtained (by-product) by adding levulinic acid to the mixture of o-ethoxybenzoyl chloride and triethylamine at  $0-5^{\circ}$ . After, the reaction with hexylmagnesium bromide was carried at  $-20^{\circ}$  (11 %) [130].

#### 1-(3-Hydroxyphenyl)-1-heptanone

[132858-49-2]	$C_{13}H_{18}O_2$	mol. wt. 206.28
OH CO(CH ₂ ) ₅ CH ₃	Syntheses -Synthesis of 3-hydroxyheptanophe organocadmium derivatives (60 %) [ -Also obtained by treatment of its aluminium chloride in benzene by bath for 2 h (80 %) [966].	2586]. methyl ether with

-Also obtained by diazotization of 3-aminoheptanophenone (42 %) [551].

-Also obtained by treatment of its methyl ether with aluminium bromide in boiling benzene for 4–5 h [551].

-Also obtained by heating an emulsion of N,N-diethyl-2-heptanoyl-6-methoxybenzamide in 48 % hydrobromic acid and acetic acid under reflux for 48 h (20 %) [2321].

-Also refer to: [967].

orange oil [2321]; b.p.₁ 165–167° [2586], b.p._{0.6–0.7} 170–177° [551]; m.p. 66° [551], 62° [966, 967, 2586]; ¹H NMR [2321], ¹³C NMR [2321], IR [2321].

**4-Nitrophenylhydrazone**  $C_{19}H_{23}N_3O_3$  mol. wt. 341.41

m.p. 143° [967].

**2,4-Dinitrophenylhydrazone** [110051-30-4] C₁₉H₂₂N₄O₅ mol. wt. 386.41

m.p. 193° [551], 191° [2586].

Acetate	$C_{15}H_{20}O_3$	mol. wt. 248.32	b.p. ₁ 144–146° [2586].
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Methyl ether	[100863-37-4]	$C_{14}H_{20}O_2$	mol. wt. 220.31
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- -Obtained by treatment of 3-hydroxyenantophenone with dimethyl sulfate in 2 N sodium hydroxide on a water bath for 90 min (35 %) [551].
- -Also obtained by adding a solution of hexylmagnesium bromide in ethyl ether to a solution of N,3-dimethoxy-N-methylbenzamide in THF and the reaction solution refluxing for 6 h (61 %) [436].
- -Also obtained by condensation of hexylmagnesium bromide with m-methoxybenzamide in ethyl ether. The reaction was carried out at  $60^{\circ}$  for 48 h under hydrogen atmosphere (60-70 %) [966].
- -Also obtained by hydration of 1-(3-methoxyphenyl)-1-heptyne on treatment with sodium sulfide followed by aqueous HCl in methanol at  $65^{\circ}$  for 60 h (<25 %) [611].
- -Also obtained by treatment of 1-(3-methoxyphenyl)-1-heptanol with  $CrO_3$  in aqueous sulfuric acid in acetone at 5°, then at r.t. for 30 h (98 %) [635].
- -Also refer to: [2789].

colourless oil [436]; pale yellow oil [551]; b.p._{0.6} 139–144° [551], b.p.₂₀ 235° [966, 967]; ¹H NMR [436, 635, 2789], ¹³C NMR [635, 2789], MS [635, 2789];  $n_{D}^{20} = 1.5153$  [551],  $n_{D}^{35} = 1.5171$  [967].

#### 2,4-Dinitrophenylhydrazone of the methyl ether

 $[102458-44-6] C_{20}H_{24}N_4O_5 mol. wt. 400.43$ 

m.p. 110.5–111° [551], 107° [967].

#### 1-(4-Hydroxyphenyl)-1-heptanone

[14392-72-4]

OH

 $C_{13}H_{18}O_2$ 

mol. wt. 206.28

Syntheses

-Phenol was energetically mixed with finely divided aluminium chloride (1 mol) and heated to  $100^{\circ}$  (1 mol of hydrochloric acid is evolved); 1 mol of n-enantoyl chloride was then added and heated to  $125-130^{\circ}$  for 1 h (41 %) [2700].

-Also obtained by reaction of heptanoyl chloride with phenol in the presence of aluminium chloride in methylene chloride for 14 h at r.t. (45 %) [1910].

-Also obtained by reaction of enanthic acid with phenol in the presence of zinc chloride (9 %) [726].

-Also obtained by Fries reaction of phenyl enanthate with aluminium chloride,

*in nitrobenzene (60 %) [379] at 37.5° for 44.5 h (72 %) [244] at 38° for 2 days (45 %) [414] or at 40° for 48 h under an argon atmosphere (73.4 %) [1116];

*without solvent at  $140^{\circ}$  for 45 min (34 %) [932].

-Also obtained [2478] by the method [2074].

-Also refer to: [62, 499, 513, 914, 1057, 1536 (92 %), 1872].

b.p._{0.4} 166° [2478], b.p.₁ 171° [932, 1536], b.p.₉ 214° [2700], b.p.₁₅ 220° [726]; white solid [1910];

m.p. 95.2–95.7° [1910], 93–94° [726], 92–94° [414], 92° (Sadtler standard N° 65674K),

91–92° [379], 91–91.5° [2700], 90° [932, 1116], 88° [2478];

¹H NMR [1910] (Sadtler standard N° 38625M), ¹³C NMR [1910],

IR [1910] (Sadtler standard N° 65674K), UV [1995], MS [1910]; TLC [1910].

BIOLOGICAL ACTIVITY: Inhibition of  $17-\beta$  hydroxysteroid dehydrogenase 3 [1910]; Fungicides, for coatings, for kitchens and ship bulls [499].

2,4-Dinitrophenylhydrazone	$C_{19}H_{22}N_4O_5$	mol. wt. 386.41
m.p. 199–200° [379], 174° [932].		

Nicotinylhydrazone [102011-49-4] C₁₉H₂₃N₃O₂ mol. wt. 325.41

m.p. 155° [520, 521].

USE: In chemotherapy of leprosy [520, 521].

#### isoNicotinylhydrazone [108984-69-6] $C_{19}H_{23}N_3O_2$ mol. wt. 325.41

m.p. 203° [520, 521].

USE: In chemotherapy of leprosy [520, 521].

# Acetate $C_{15}H_{20}O_3$ mol. wt. 248.32 b.p.9 194–195° [2700]; m.p. 46.5° [2700]. Benzoate $C_{20}H_{22}O_3$ mol. wt. 310.39 m.p. 96.5–97° [2700], 92–93° [726]. 100

Methyl ether	[69287-13-4]	$C_{14}H_{20}O_2$	mol. wt. 220.31
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-Obtained by adding a solution of hexylmagnesium bromide in ethyl ether to a solution of N,4-dimethoxy-N-methylbenzamide in THF and the reaction solution refluxing for 6 h (89 %) [436].

- -Other preparation: n-hexyl triphenylphosphonium bromide and lithium iodide were suspended in THF. The reaction flask was wrapped with aluminium foil. At ice-water temperature, butyllithium in hexane was added, producing the red solution of the phosphorane. After 20 min, THF containing 4-methoxybenzonitrile was added. The reaction mixture was then maintained at 50° for 8 h (95 %) [3014]. -Also obtained by Friedel-Crafts acylation of anisole with n-heptanoic acid in the presence of  $[Eu(NTf_2)_3]$  at 250° for 6 h (87 %) [2709], (84 %) [1649].
- -Also obtained by direct acylation of 4-bromoanisole with enanthic aldehyde by palladium catalysis (88 %) [2668].
- -Also obtained by reaction of heptanoyl chloride with 4-methoxyphenylmagnesium chloride in the presence of Fe(acac)₃ as catalyst in THF at  $-78^{\circ}$  (99 %) [2739].
- -Also obtained by Friedel-Crafts acylation of anisole using metal triflates in ionic liquid [2654].
- -Obtained by reaction of dimethyl sulfate with 4-heptanoylphenol in the presence of aqueous sodium hydroxide for 4 h at reflux [2478].
- -Also obtained by reaction of 4-methoxybenzyl alcohol with 1-hexene in the presence of RhCl₃, x H₂O, polystyrene-based diphenylphosphine and PPh₃ with co-catalyst 2-amino-4-picoline in toluene at 130° for 72 h (58–68 %) [1555].

-Also obtained by reaction of heptanoic acid with anisole,

- *in the presence of  $HNTf_2$  in refluxing toluene for 36 h, using a Dean-Stark apparatus (89 %) [1648];
- *in the presence of  $Eu(NTf_2)_3$  at 250° for 6 h (84 %) [1648];
- *over HZSM-5 catalyst for 48 h at 423° K (7 %) [3265];
- *in the presence of an ultrastable Y zeolite (USY) catalyst [1941];
- *in the presence of HY zeolite as catalyst [3264].
- -Also obtained by hydration of 1-(4-methoxyphenyl)-1-heptyne on treatment with sodium sulfide followed by aqueous HCl in methanol at  $65^{\circ}$  for 60 h (92 %) [611].
- -Also obtained by zeolite-catalyzed Friedel-Crafts reaction of anisole with heptanoic acid [3266].

-Also refer to: [605, 698 (74 %), 1009, 1200, 1202, 1558 (79 %), 1679, 1749 (81 %), 1750, 2080, 2399, 2478, 2866, 2901, 3214, 3262].
b.p._{0.4} 120–121° [2478], b.p.₁₇ 192° [2901], b.p.₂₅ 203° [251], b.p.₅₀ 240° [2399]; white solid [1648];
m.p. 44–45° [2739], 43° [251], 41–43° [3214], 40° [2901], 39–40° [2478], 38.6–39.8° [1750], 38–39° [1648];
¹H NMR [436, 698, 1649, 1749, 1750, 2668, 2709, 2739, 3014], ¹³C NMR [698, 2668, 2739, 3014], IR [698, 1749, 1750, 2668, 2709, 2739], MS [698, 2668, 2739]; n_D²⁸ = 1.5114 [2399].

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2-Bromoethyl ether	[60985-68-4]	$C_{15}H_{21}BrO_2$	mol. wt. 313.23
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USE: Preparation and condensation with (methylphenethyl)amine [2484].

Propyl ether	$C_{16}H_{24}O_2$	mol. wt. 248.37
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-Obtained by reaction of heptanoyl chloride with phenyl propyl ether in the presence of zinc chloride at reflux for 30 min (30.6 %) [1237].

b.p.₁₅ 217–221° [1237]; m.p. 31–32° [1237].

**Oxime of the propyl ether**  $C_{16}H_{25}NO_2$  mol. wt. 263.38

m.p. 53.5° [1237].

1,1,2,2-Tetrafluoroethyl ether	[56426-10-9]	$C_{15}H_{18}F_4O_2$	mol. wt. 306.44
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USE: Reaction of, with halogenated heterocycles [297].

**4-Heptanoylphenyl ether** [149454-86-4] C₂₆H₃₄O₃ mol. wt. 394.55

-Obtained by reaction of heptanoyl chloride with diphenyl ether in the presence of aluminium chloride in refluxing carbon disulfide for 30 min (85 %) [463].

m.p. 102–103° [463].

#### Dioxime of the 4-heptanoylphenyl ether $C_{26}H_{36}N_2O_3$ mol. wt. 424.58

-Preparation: A suspension of the diketone, 100 % excess of hydroxylamine hydrochloride, and powdered anhydrous sodium carbonate in butanol [463].

m.p. 102–103° [721], 95–96° [463]. m.p. 95–96° [463].

#### Dihydrazone of the 4-heptanoylphenyl ether C₂₆H₃₈N₄O mol. wt. 422.61

b.p._{0.02} 212–218° [721]; m.p. 95–96° [721].

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

$$[862666-35-1] C_{13}H_{18}O_3 mtext{mol. wt. } 222.28$$

Synthesis

OH HO

 $CO(CH_2)_5CH_3$  -Obtained by deprotection of its dimethyl ether with boron tribromide in methylene chloride at  $0^{\circ}$ . Then, the mixture was stirred overnight at r.t. (70 %) [82].

brown solid [82]; m.p. 46° [82]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82],

USE: Reactant for preparation of lithium gallium/titanium ketone complexes [82].

-Obtained by treatment of 1-(2,3-dimethoxyphenyl)-1-heptanol in acetone with potassium dichromate in dilute sulfuric acid (Jones reagent) at r.t. for 3 h (28 %) [82].

-Also obtained by treatment of 2,3-dimethoxyphenylhexylcarbinol with potassium dichromate in dilute sulfuric acid at 30° (73.5 %) [3148].

colourless oil [82]; b.p.₁₀ 179–181° [3148]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82].

#### 2,4-Dinitrophenylhydrazone of the dimethyl ether

[1854-68-8]	$C_{21}H_{26}N_4O_6$	mol. wt. 430.46
m.p. 127° [3148].		

C13H18O3

#### 1-(2,4-Dihydroxyphenyl)-1-heptanone

(*Res-oenanthophenone*)

[27883-47-2]

Syntheses

-Obtained by reaction of enanthic nitrile with resorcinol (Hoesch reaction) [1608].

mol. wt. 222.28

-Also obtained by reaction of heptanoic acid with resorcinol in the presence of zinc chloride (68-78 %) [2501].

-Also obtained by reaction of heptanoyl chloride with resorcinol in the presence of aluminium chloride in 1,2-ethylene chloride for 5 h at 65° [284].

-Also refer to: [893, 1500, 1655, 1673, 1798, 2112, 2114, 2790, 2842, 3168].

b.p.₆₋₇ 204–206° [893, 2842]; m.p. 48–50° [893, 1673, 2842], 48–49° [1608].

USE: Polyamide fibers modified with, transparency of, [2790]; Wine preservation by, [3168].

BIOLOGICAL ACTIVITY: Antiseptic and germicidal product [2734]; Antifungal [2112, 2114]; Nematocide [1798].

Hemihydrate 
$$C_{13}H_{18}O_3, 0.5 H_2O$$
 mol. wt. 231.29

m.p. 41-43° [1608].

Oxime	[165740-65-8]	$C_{13}H_{19}NO_3$	mol. wt. 237.30
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USE: Silver halide photog. material [1472].

#### 1-(2,5-Dihydroxyphenyl)-1-heptanone

-Preparation by reaction of heptanoic acid with hydroquinone in the presence of boron trifluoride for 2 h at 125° [2063]. -Also refer to: [3168, 3460, 3462].

m.p. 118° [1442], 50° [2063].

N.B.: One of the reported melting point is obviously wrong.

USE: Electroluminescent devices employing complex fluorene-containing compounds [3461]; Wine preservation by, [3168].

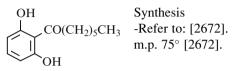
Oxime	[165740-62-5]	$C_{13}H_{19}NO_3$	mol. wt. 237.30
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USE: Silver halide photog. material [1472].

-Preparation by reaction of heptanoyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride in nitrobenzene at r.t. overnight [1442]. -Also refer to: [550 (by-product), 3460, 3462]. b.p.₅ 175–180° [1442]; m.p. 34–35° [1442].

USE: Electroluminescent devices employing complex fluorene-containing compounds [3461].

#### 1-(2,6-Dihydroxyphenyl)-1-heptanone



#### 1-(3,4-Dihydroxyphenyl)-1-heptanone

(4-Heptanoylcatechol)

 $\label{eq:c13} [2525-08-8] \qquad \qquad \text{C}_{13}\text{H}_{18}\text{O}_{3} \qquad \qquad \text{mol. wt. } 222.28$ 

Syntheses



-Obtained by adding heptanoyl chloride (1 mol) to a solution of pyrocatechol (1 mol) and aluminium chloride (1.5 mol) in carbon disulfide. After heating at  $40-50^{\circ}$  the solvent was removed and the residue heated at 150° for 4 h (50 %) [1283].

-Also obtained by treatment of guaiacol heptanoate with aluminium chloride in carbon disulfide at  $90^{\circ}$  for 50 min, then at  $135-140^{\circ}$  for 2 h after solvent elimination [2075].

-Also refer to: [2508].

m.p. 93–94° [1283], 78–79° [2075]; paper chromatography [2508].

<b>O-Methyloxime</b>	[474668-95-6]	$C_{14}H_{21}NO_3$	mol. wt. 251.33
Refer to: [3177]			

-Refer to: [3177].

Dimethyl ether	[101100-86-1]	$C_{15}H_{22}O_3$	mol. wt. 250.34
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-Obtained by reaction of heptoyl chloride with veratrole in the presence of zinc chloride in refluxing carbon disulfide for 4 h and then left overnight (60 %) [2836], (59 %) [1486].

-Also refer to: [2505].

b.p.₄ 178–180° [2836], b.p.₁ 180° [1486].

#### 

white needles [2836]; m.p. 141° [1486], 140–141° [2836].

#### 1-(3,5-Dihydroxyphenyl)-1-heptanone

 $\begin{array}{cccc} [39192-54-6] & C_{13}H_{18}O_3 & \mbox{mol. wt. } 222.28 \\ OH & Syntheses \\ -Obtained by treatment of its diacetate with 5 % sodium hydroxide (quantitatif yield) [1880], at reflux for 4.5 h (62 %) [1406]. \end{array}$ 

Isolation from natural sources

-From the roots and stems of *Ardisia virens* Kurz (Myrsinaceae) [607]. -From the roots of *Ardisia cornudentata* Mez. [604].

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m.p. 94° [1406], 88–89° [1880].
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#### **2,4-Dinitrophenylhydrazone** [102161-23-9] C₁₉H₂₂N₄O₆ mol. wt. 402.41

m.p. 230° [1406].

**Diacetate** [39192-52-4] C₁₇H₂₂O₅ mol. wt. 306.36

-Preparation by reaction of dihexylcadmium with 3,5-diacetoxybenzoyl chloride (quantitatif yield) [1880], in refluxing benzene for 1 h (82 %) [1406].

b.p._{0.8} 205° [1406].

#### 2,4-Dinitrophenylhydrazone of the diacetate

 $[102655-30-1] C_{23}H_{26}N_4O_8 mol. wt. 486.48$ 

m.p. 132° [1406].

Dimethyl ether	[39192-51-3]	$C_{15}H_{22}O_3$	mol. wt. 250.34
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-Preparation by reaction of hexylmagnesium bromide with 3,5-dimethoxybenzamide in refluxing ethyl ether for 5 h (85 %) [2990].

-Preparation by reaction of hexyl bromide with 3,5-dimethoxybenzonitrile [2017]. -Preparation by reaction of hexylmagnesium bromide with (3,5-dimethoxyphenyl)-

*N*-methoxy-*N*-methylcarboxamide (90 %) [1252].

-Also obtained by adding a solution of chromic acid in dilute sulfuric acid (Jone's reagent) to a cold solution (0 °C) of 1-(3,5-dimethoxyphenyl)-1-heptanol in acetone. Then, the reaction mixture was stirred at r.t. for 30 min (79.6 %) [2394]. -Also refer to: [1880 (quantitatif yield), 2144, 2395, 2396, 2575, 2899].

white solid [1252]; b.p.₃ 161–161.5° [2990]; m.p. 30.5–31° [2990], 27–28° [1252]; ¹H NMR [1252, 2394], ¹³C NMR [1252], IR [1252], MS [1252];  $n_D^{25} = 1.5175$  [2990].

#### 1-[2,3,4-Trihydroxyphenyl]-1-heptanone

mol. wt. 238.28  $C_{13}H_{18}O_{4}$ [43043-27-2] OH **Synthesis** CO(CH₂)₅CH₃ -Refer to: [1260, 1500, 3168]. HO. m.p. 78–78.5° [1260]. HO

USE: Wine preservation by, [3168].

#### 1-[2,4,6-Trihydroxyphenyl]-1-heptanone

[43043-31-8]  $C_{13}H_{18}O_4$ mol. wt. 238.28 OH Syntheses CO(CH₂)₅CH₃ -Obtained by reaction of enanthic nitrile with phloroglucinol (Hoesch reaction) [1608]. -Also obtained by reaction of heptanoyl chloride with OН HO phloroglucinol in the presence of aluminium

*in nitrobenzene for 3 days at r.t. (60-70 %) [421]; *in nitrobenzene and carbon disulfide mixture (58 %) [2113]. -Also refer to: [1026, 1439, 2111, 2719, 3168].

chloride,

m.p. 108° [2113], 107–108° [1608], 107° [421, 1439]; ¹H NMR [421], IR [421], MS [421].

USE: Assembly and bis(pyridyl)ethylene with phloroglucinols [1038]; Win

BIOLOGICAL ACTIVITY: Antifungal [2111, 2113, 2719]; Anthelmintic and bactericide [1439].

13111804, 1120 1101. wt. 230.30	Monohydrate	$C_{13}H_{18}O_4, H_2O$	mol. wt. 256.30
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C13H18O4

m.p. 98–100° [1608].

#### Polymer with formaldehyde

-Refer to: [1366].

#### 1-(3,4,5-Trihydroxyphenyl)-1-heptanone

[100079-25-2]
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Syntheses -Refer to: [151, 2315].

BIOLOGICAL ACTIVITY: As platelet aggregation inhibitor and antiallergic agent [2315].

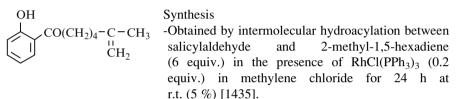
mol. wt. 238.28

Trimethyl ether	$C_{16}H_{24}O_4$	mol. wt. 280.36
-Obtained by treatment of KOH at 40° [151].	ethyl 3,4,5-trimethoxybenzoyl	amylacetate with 5 % alc.
b.p.4 175–176° [151];	m.p. 29° [151].	

Tribenzyl ether	[100079-28-5]	$C_{34}H_{36}O_4$	mol. wt. 444.66
-Refer to: [2315].			

### 1-(2-Hydroxyphenyl)-6-methylene-1-heptanone

C14H18O2 mol. wt. 218.30



¹H NMR [1435].

#### 1-(4-Hydroxyphenyl)-2-methyl-1-heptanone (+)

[120837-02-7] (+)

C14H20O2

mol. wt. 220.31

### 1-(3,5-Dihydroxyphenyl)-2-methyl-1-heptanone

C₁₄H₂₀O₃ mol. wt. 236.31 Synthesis OH  $\begin{array}{c} \text{-Refer to: [25].} \\ \text{CH}_{3} \\ \text{CO-CH-C}_{5}\text{H}_{11} \end{array} \begin{array}{c} \text{-Refer to: [25].} \\ \text{Dimethyl ether} \\ \text{C}_{16}\text{H}_{24}\text{O}_{3} \end{array}$ mol. wt. 264.36

-Preparation from 3,5-dimethoxybenzamide and BrMgCH(CH₃)C₅H₁₁ in ethyl ether (93 %) [479], (82 %) [25, 31]. -Also refer to: [870, 2348].

 $\begin{array}{l} b.p_{\cdot 0.2} \ 130^{\circ} \ [479], \ b.p_{\cdot 0.2} \ 133 - 138^{\circ} \ [870], \ b.p_{\cdot 1} \ 147^{\circ} \ [25, \ 31]; \\ {}^{1} H \ NMR \ [479]; \quad n_{D}^{20} = 1.5136 \ [25]. \end{array}$ 

USE: Preparation of soft cannabinoid analogues as potential antiglaucoma agents [479]; Preparation and Grignard reaction with methyl iodide [2348].

#### 1-(4-Hydroxyphenyl)-2,6-dimethyl-1-heptanone

#### 3-Ethyl-1-(3,4,5-trihydroxyphenyl)-1-heptanone-

[353499-11-3]	$C_{15}H_{22}O_4$	mol. wt. 266.34
HO HO COCH ₂ -CH-(CH ₂ ) ₃ CH ₃ $\stackrel{l}{CH_2CH_3}$	Syntheses -Refer to: [1969, 2688]. USE: Developing agent for graphic plates [2688].	r making litho-
$CI_2CI_3$		

#### 1-(4-Hydroxyphenyl)-5-phenyl-1-heptanone

 $\begin{array}{cccc} [14392-75-7] & C_{19}H_{22}O_2 & \mbox{mol. wt. } 282.38 \\ & & & \\ & & \\ & & \\ HO & & \\ & & \\ HO & & \\ & & \\ HO & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & &$ 

# 1.2 Substituted Hydroxyketones

#### 1-(3-Bromo-5-fluoro-2-hydroxyphenyl)-1-heptanone

$$[2342-47-4] C_{13}H_{16}BrFO_2 mol. wt. 303.17$$

$$OH Synthesis -Obtained by Fries rearrangement of 2-bromo-
4-fluorophenyl enanthate with aluminium chloride at 130–140° for 3 h (90 %) [1550].$$

$$b.p._{0,2-0,5} 148^{\circ} [1550].$$

**2,4-Dinitrophenylhydrazone** [2317-60-4] C₁₉H₂₀BrFN₄O₅ mol. wt. 483.29 m.p. 140–141° [1550].

#### 1-(3,5-Dibromo-4-hydroxyphenyl)-1-heptanone

[20683-50-5]	$C_{13}H_{16}Br_2O_2$	mol. wt. 364.08
$Br$ $H$ $Br$ $CO(CH_2)_5CH_3$	Synthesis -Obtained by reaction of bromine enanthophenone in aqueous acetic acid m.p. 71° [516].	• •

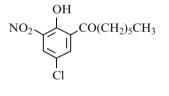
#### 1-(3-Chloro-5-fluoro-2-hydroxyphenyl)-1-heptanone

[2342-49-6]	$C_{13}H_{16}ClFO_2$	mol. wt. 258.72
$\begin{array}{c} OH\\Cl \leftarrow \\F\\ \end{array} CO(CH_2)_5CH_3\\ \\F\\ \end{array}$	Synthesis -Obtained by Fries rearrang rophenyl enanthate with 130–140° for 3 h (90 %) [1 b.p. _{0.5} 140° [1550].	aluminium chloride at

**2,4-Dinitrophenylhydrazone** [1814-29-5]  $C_{19}H_{20}ClFN_4O_5$  mol. wt. 438.84

m.p. 132° [1550].

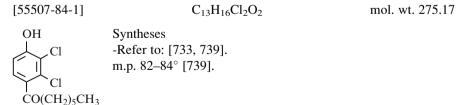
#### 1-(5-Chloro-2-hydroxy-3-nitrophenyl)-1-heptanone



Synthesis CO(CH₂)₅CH₃ -Preparation by treatment of 5-chloro-2-hydroxyheptanophenone with concentrated nitric acid (60 %) in acetic acid in the presence of one drop of concentrated sulfuric acid for 25 min at r.t. (92 %) [2105].

¹H NMR [2105], MS [2105].

#### 1-(2,3-Dichloro-4-hydroxyphenyl)-1-heptanone



USE: Preparation and etherification of, with bromoacetate [733].

#### 1-(4-Hydroxy-3,5-diiodophenyl)-1-heptanone

$$\begin{array}{ccc} C_{13}H_{16}I_2O_2 & \text{mol. wt. } 458.08 \\ \hline OH & \text{Synthesis} \\ I & -\text{Obtained by reaction of iodine with } 4-\text{hydroxy-} \\ & \text{enanthophenone in ethanol in the presence of yellow mercuric oxide [516].} \\ CO(CH_2)_5CH_3 & \text{colourless needles [516]; m.p. } 50^\circ \text{ [516].} \end{array}$$

#### 1-(2-Hydroxyphenyl)-1,6-heptanedione

 $\begin{array}{cccc} [1237740-92-9] & C_{13}H_{16}O_3 & \text{mol. wt. } 220.27 \\ OH & Synthesis \\ -Refer to: [498]. \\ & ^1H \ NMR \ [498], \ ^{13}C \ NMR \ [498], \ IR \ [498], \ MS \ [498]. \end{array}$ 

#### 1-(3-Bromo-2-hydroxyphenyl)-1-heptanone

	$C_{13}H_{17}BrO_2$	mol. wt. 285.18
ОН	Synthesis	
Br CO(CH ₂ ) ₅ CH ₃	-Refer to: [3036].	
	Methyl ether [952103-47-8]	
$\bigtriangledown$	$C_{14}H_{19}BrO_2$	mol. wt. 299.21

-Refer to: [2214, 3036].

USE: Preparation of optically active-4-phenylthiazole derivs. [3036]; Preparation of thrombopoietin receptor agonist by yeast [2214].

#### 1-(5-Bromo-2-hydroxyphenyl)-1-heptanone

	$C_{13}H_{17}BrO_2$	mol. wt. 285.18
OH CO(CH ₂ ) ₅ CH ₃	heptanoate with aluminium chloride [2	
Br	<b>2,4-Dinitrophenylhydrazone</b> C ₁₉ H ₂₁ BrN ₄ O ₅	mol. wt. 465.30

m.p. 189° [2798].

#### 1-(3-Chloro-4-hydroxyphenyl)-1-heptanone

$$\begin{array}{cccc} & & C_{13}H_{17}ClO_2 & & mol. \ wt. \ 240.73 \\ & \\ OH & Synthesis \\ & \\ \hline Cl & -Refer \ to: \ [789]. \\ & \\ & \\ Methyl \ ether & \ [250686-92-1] \\ & \\ Cl_{14}H_{19}ClO_2 & \\ & \\ Cl(CH_2)_5CH_3 \end{array}$$

m.p. 24° [1160].

USE: News nematics with negative dielectric anisotropy [789].

#### 1-(4-Chloro-2-hydroxyphenyl)-1-heptanone

	$C_{13}H_{17}ClO_2$	mol. wt. 240.73
OH CO(CH ₂ ) ₅ CH ₃	Syntheses -Preparation by Fries rearrangement of heptoate with aluminium chloride, *without solvent at 130° for 2 h (83 % *in nitrobenzene at 25° for 6 h (83 %	%) [2802];

b.p.₃₀ 200° [2802].

**2,4-Dinitrophenylhydrazone** C₁₉H₂₁ClN₄O₅ mol. wt. 420.85

m.p. 189° [2797].

#### $C_{14}H_{19}ClO_2$ Methyl ether mol. wt. 254.76

-Obtained by methylation of the above ketone in the usual way (90 %) [2802].

b.p.₃₆ 180° [2802].

#### 1-(5-Chloro-2-hydroxyphenyl)-1-heptanone

[85052-18-2]  $C_{13}H_{17}ClO_2$ mol. wt. 240.73

OH

Syntheses CO(CH₂)₅CH₃ -Obtained by Fries rearrangement of 4-chlorophenyl enanthate with titanium tetrachloride at  $100^{\circ}$  for 18 h (72 %) [2105]. -Also refer to: [1702].

b.p.₅ 140–144° [1702]; m.p. 43.5° [1702]; ¹H NMR [2105]; MS [2105].

#### 1-(2,4,6-Trihydroxy-3-nitrophenyl)-1-heptanone

[119691-97-3]	C ₁₃ H ₁₇ NO ₆	mol. wt. 283.28
OH NO ₂ HO OH OH	Syntheses -Obtained by adding hexane, concentrated sulfuric acid and 0° to a solution of 1-(2,4 1-heptanone in concentrated 0° (70–80 %) [3414].	fuming nitric acid at ,6-trihydroxyphenyl)-

-Also refer to: [3406].

Amorphous [3414]; ¹H NMR [3414], IR [3414], MS [3414].

BIOLOGICAL ACTIVITY: Germination inhibitory activity [3414]; PET inhibitory activity [3414].

#### 1-(3-Amino-5-chloro-2-hydroxyphenyl)-1-heptanone

[85052-73-9]	$C_{13}H_{18}CINO_2$	mol. wt. 255.74
OH NH ₂ Cl CO(CH ₂ ) ₅ CH ₃	Synthesis -Obtained by reduction of 1-(5- 3-nitro-phenyl)-1-heptanone trichloride [2105]. <b>Hydrochloride</b> [85052-45-5]	• •
	$C_{13}H_{18}CINO_2$ , HCl	mol. wt. 292.20

#### 1-(3-Amino-5-fluoro-2-hydroxyphenyl)-1-heptanone

 $\begin{array}{c} C_{13}H_{18}FNO_2 \\ \text{one of } MH_2 & \text{one of } MH_2 \\ & \text{one of } H_2 \\ & \text{one$ 

#### 1-(3,5-Dichloro-2,4-dihydroxy-6-methoxyphenyl)-1-heptanone

[118191-32-5]	$C_{14}H_{18}Cl_2O_4$	mol. wt. 321.20
$\begin{array}{c} OH \\ Cl \\ HO \\ Cl \\ Cl \\ OCH_3 \\ \end{array}$	Synthesis -Obtained by reaction of chlorine y 6-methoxyheptanophenone in wa ¹ H NMR [2012], MS [2012].	with 2,4-dihydroxy- ter [2012].

BIOLOGICAL ACTIVITY: Differentiation-inducing factor from the slime mould *Dictyostelium discoideum* [2012].

# **1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-1-heptanone** (*DIF-1*) (+1)

[118191-33-6]

Cl

CH₃O

OH

Syntheses

CO(CH₂)₅CH₃ -Preparation by adding a solution of sulfuryl chloride (1.5 equiv.) in ethanol to a solution of 2,6-dihydroxy-4-methoxyheptanophenone in chloroform at r.t.; then, the solution was stirred at r.t. [1129].

-Also obtained by reaction of chlorine with 2,4-dihydroxy-6-methoxyheptanophenone in water [2012].

-Also refer to: [1772, 1773, 2341].

yellow amorphous solid [1129]; ¹H NMR [2012], MS [1129, 2012].

BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129]; Differentiation-inducing factor from the slime mould *Dictyostelium discoideum* [2012].

#### 3-Heptanoyl-4-hydroxybenzoic acid

 $\begin{array}{ccc} C_{14}H_{18}O_4 & \text{mol. wt. 250.29} \\ OH & Synthesis \\ -Obtained by hydrolysis of its ethyl ester [967]. \\ m.p. 183^{\circ} [967]. \\ Acetate & C_{16}H_{20}O_5 & \text{mol. wt. 292.33} \\ COOH \end{array}$ 

-Obtained by acetylation of 3-heptanoyl-4-hydroxybenzoic acid [967].

m.p. 101° [967].

#### Methyl ether

-Obtained by methylation of 3-heptanoyl-4-hydroxybenzoic acid [967].

m.p. 163° [967].

#### Ethyl ester

C₁₆H₂₂O₄ mol. wt. 278.35

-Obtained by reaction of heptanoyl chloride with ethyl 4-hydroxybenzoate in the presence of aluminium chloride in tetrachloroethane, then the reaction mixture was heated at  $120^{\circ}$  for 3–4 h [967].

b.p.₂₂ 210° [967]; 
$$n_D^{35.5} = 1.5115$$
 [967].

#### 2,4,6-Trihydroxy-3-(1-oxoheptyl)benzaldehyde

[96573-33-0]	$C_{14}H_{18}O_5$	mol. wt. 266.29
OH CHO HO OH OH	Syntheses -Obtained by reaction of eth 2,4,6-trihydroxyenantopheno aluminium chloride in met cooling in an ice bath for 30	ne in the presence of hylene chloride with

-Also refer to: [3406].

m.p. 103–105° [421]; ¹H NMR [421], IR [421], MS [421].

BIOLOGICAL ACTIVITY: As photosynthetic electron transport (PET) [3405].

#### 1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-heptanone

(	$C_{14}H_{19}ClO_2$		
CH ₃ CO(CH ₂ ) ₅ CH ₃	Synthesis -Refer to: [3138]. Fluorescence [3138].		

**1-(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)-1-heptanone** (*DIF-3*) (+1)

[861889-89-6]	$C_{14}H_{19}ClO_4$	mol. wt. 286.76
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Syntheses

CO(CH₂)₅CH₃ -Obtained by reaction of sulfuryl chloride (1.5 equiv.) with 2,6-dihydroxy-4-methoxyhepta-OH nophenone in a chloroform/ethanol mixture at r.t. [1129].

-Also refer to: [1772].

CH₃O

colourless amorphous solid [1129]; MS [1129].

BIOLOGICAL ACTIVITY: Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

mol. wt. 254.76

mol. wt. 310.44

#### 1-(2-Hydroxy-3-methylphenyl)-1-heptanone

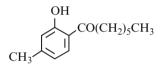
[1000598-85-5]	$C_{14}H_{20}O_2$	mol. wt. 220.31
CH ₃ CO(CH ₂ ) ₅ CH ₃	Syntheses -Obtained by acylation (53 %) [1627]. -Also refer to: [2270].	of organometallic reagents
colourless oil [1627]; b.j ¹ H NMR [1627], ¹³ C NM	p. _{14–15} 161–162° [2270]; R [1627], MS [1627].	

 $\label{eq:constraint} \textbf{2,4-Dinitrophenylhydrazone} \qquad C_{20}H_{24}N_4O_5 \qquad \qquad \text{mol. wt. 400.43}$ 

m.p. 151-152° [2270].

#### 1-(2-Hydroxy-4-methylphenyl)-1-heptanone

C₁₄H₂₀O₂ mol. wt. 220.31



CO(CH₂)₅CH₃ -Preparation by Fries reaction of m-tolyl enanthate with aluminium chloride,

*without solvent at 160° for 2 h (84 %) [726], at 140–150° [906] or at 120–140° for 10–20 min (67 %) [243];

*in nitrobenzene at 25° for 24 h (73 %) [243] or at 25–30° for 66 h (82 %) [244].

C20H26N2O

b.p.₁ 123–124° [906], b.p.₄ 148° [243], b.p.₁₅ 172–174° [726]; m.p. 18° [726].

Syntheses

Phenylhydrazone

-Refer to: [243].

m.p. 82-83° [243].

#### 1-(2-Hydroxy-5-methylphenyl)-1-heptanone

 $[74604-13-0] C_{14}H_{20}O_2 mtext{mol. wt. } 220.31$ 

OH	
$\checkmark$	.CO(CH ₂ ) ₅ CH ₃
Ý	
CH ₃	

Syntheses

-Preparation by Fries rearrangement of p-cresyl enanthate with aluminium chloride,
*for 10 min at 120° (86 %) [2647];
*in tetrachloroethane at 120° [2520];
*without solvent at 130–150° [2520].

-Also refer to: [244, 1437, 1438, 2647].

b.p._{0.1} 113° [2520], b.p.₈ 158° [244], b.p.₁₀ 168° [2647]; ¹H NMR [1437], IR [1437, 2520], UV [2520], MS [1437].

Oxime	[74604-06-1]	$C_{14}H_{21}NO_2$	mol. wt. 235.33
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m.p. 88° [2520]; IR [2520], UV [2520].

USE: Solvent extraction of copper (II) [2520].

Phenylhydrazone	$C_{20}H_{26}N_2O$		mol. wt. 310.44
m.p. 107–108° [244	m.p. 107–108° [244].		
Methyl ether	[194359-57-4]	$C_{15}H_{22}O_2$	mol. wt. 234.34
-Refer to: [1437, 1438	·].		

¹H NMR [1437].

#### 1-(4-Hydroxy-2-methylphenyl)-1-heptanone

$C_{14}H_{20}O_2$	mol. wt. 220.31
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**Synthesis** OH -Obtained by Fries reaction of m-tolyl enanthate with aluminium chloride in nitrobenzene at  $25-30^{\circ}$  for 66 h (10 %) [244]. CH₂ m.p. 67-67.5° [244]. CO(CH₂)₅CH₃

#### 1-(4-Hydroxy-3-methylphenyl)-1-heptanone

[95102-27-5] C14H20O2 mol. wt. 220.31 OH Syntheses -Obtained by Friedel-Crafts acylation of o-cresol with CH₃ n-enantoyl chloride in the presence of aluminium chloride in nitrobenzene at 30° for 24 h (92 %) [2704].

 $CO(CH_2)_5CH_3$  -Also refer to: [1595].

USE: Colour developer, for thermal recording materials [1595].

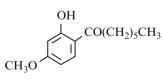
#### 1-(2-Hydroxy-4-methoxyphenyl)-1-heptanone

[143286-89-9] C14H20O3 mol. wt. 236.31 Syntheses OH CO(CH₂)₅CH₃ -Obtained by reaction of methyl bromide with

2,4-dihydroxyenanthophenone in the presence of potassium carbonate in refluxing acetone for 20 h [284].

-Also obtained by reaction of dimethyl sulfate with 2,4-dihydroxyenanthophenone in the presence of potassium carbonate in refluxing acetone for 4-6 h (85-90 %) [2501].

m.p. 24° [284].



-Obtained by reaction of hydroxylamine hydrochloride with 1-(2-hydroxy-4-methoxyphenyl)-1-heptanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 76° [284]; ¹H NMR [284].

#### 1-(2-Hydroxy-5-methoxyphenyl)-1-heptanone

 $C_{14}H_{20}O_3$ 

mol. wt. 236.31

Syntheses

-Obtained by reaction of enanthyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride in refluxing carbon disulfide for 8 h [550]. -Also refer to: [161, 1515].

yellow crystals [550]; m.p. 43–44° [1515], 34° [550], 33–33.5° [161]; ¹H NMR [1515].

**2,4-Dinitrophenylhydrazone**  $C_{20}H_{24}N_4O_6$  mol. wt. 416.43

m.p. 145° [550].

#### 1-(4-Hydroxy-3-methoxyphenyl)-1-heptanone

 $[114515-52-5] C_{14}H_{20}O_3 mol. wt. 236.31$  OH Synthesis -Obtained by adding DDQ to 1-(4-hydroxy-3-methoxy-phenyl)-1-heptanol in benzene and the reaction mixture stirred for 4 h at r.t. [2989].  $CO(CH_2)_5CH_3 m.p. 49^{\circ} [2989]; ^1H NMR [2989].$ 

BIOLOGICAL ACTIVITY: Chloleretic [2989].

#### 1-(2,6-Dihydroxy-4-methoxyphenyl)-1-heptanone

BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

#### 1-(2,4,6-Trihydroxy-3-methylphenyl)-1-heptanone

	$C_{14}H_{20}O_4$	mol. wt. 252.31
CH ₃ HO OH CO(CH ₂ ) ₅ CH ₃	Synthesis -Obtained by react 3-methyl-phloroglue	tion of enanthic nitrile with cinol (Hoesch reaction) [1608].

m.p. 143–144° [1608].

#### 3-Heptanoyl-2-hydroxy-5-methylbenzoic acid

 $C_{15}H_{20}O_{4}$ mol. wt. 264.32 OH Synthesis  $\begin{array}{c|c} CH_3(CH_2)_5CO & \begin{array}{c} CO_2H \\ \hline \\ CH_3 \end{array} \begin{array}{c} CO_2H \\ CH_3 \end{array} \begin{array}{c} CH_3 \end{array} \begin{array}{c} CO_2H \\ CH_3 \end{array} \begin{array}{c} CH_3 \end{array} \begin{array}{c}$ 

#### C16H22O4 mol. wt. 278.35 Methyl ester

-Preparation by Fries rearrangement of methyl 2-(heptanoyloxy)-5-methylbenzoate with aluminium chloride in carbon disulfide for 2 h at  $60^{\circ}$  (90 %) [3235].

b.p._{0.2} 204–210° [3235]; m.p. 51–52° [3235].

#### 1-(3-Chloro-6-hydroxy-2,4-dimethylphenyl)-1-heptanone

[102003-68-9]	$C_{15}H_{21}ClO_2$	mol. wt. 268.78
CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃	Synthesis -Preparation by Fries ream 3,5-dimethylphenyl heptar chloride in carbon disulfide 110° for 2 h after solvent eli	hoate with aluminium e at $80^{\circ}$ for 2 h, then at

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m.p. 80° [3114].
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Semicarbazone	[107151-50-8]	$C_{16}H_{24}ClN_3O_2$	mol. wt. 325.84
m.p. 154° [3114].			

Allyl ether	[101892-45-9]	$C_{18}H_{25}ClO_2$	mol. wt. 308.85
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-Obtained by reaction of allyl bromide with the title ketone in the presence of potassium carbonate in refluxing acetone for 6 h (87 %) [3114].

b.p.₂ 187° [3114].

USE: Insecticide [3114].

mol. wt. 248.37

#### 3-Ethyl-1-(4-hydroxy-3-nitrophenyl)-1-heptanone

[214058-35-2] 
$$C_{15}H_{21}NO_4$$
 mol. wt. 279.33  
OH Synthesis  
-Refer to: [1315].  
COCH₂ - CH - C₄H₉  
 $C_2H_5$ 

#### 1-(3-Ethyl-4-hydroxyphenyl)-1-heptanone

[95185-66-3]  $C_{15}H_{22}O_2$  mol. wt. 234.34 OH Synthesis -Refer to: [2704].

#### 1-(4-Ethyl-2-hydroxyphenyl)-1-heptanone

(	$C_{15}H_{22}O_2$	mol. wt. 234.34
OH C ₂ H ₅ CO(CH ₂ ) ₅ CH ₃	Syntheses -Obtained by Fries rearran enanthate (1 equiv.), *in the presence of alumir in nitrobenzene at 25° fo *in the presence of alumir	nium chloride (1.3 equiv.) r 6 h (91 %) [2801];

first in refluxing carbon disulfide for 2 h, then for 2 h at  $130^{\circ}$  after solvent elimination (88 %) [2801].

b.p.4 176° [2801].

CO(CH₂)₅CH₃

### $\label{eq:constraint} \textbf{2,4-Dinitrophenylhydrazone} \qquad C_{21}H_{26}N_4O_5 \qquad \qquad \text{mol. wt. 414.46}$

m.p. 143° [2801].

#### Methyl ether

-Obtained by reaction of dimethyl sulfate with 1-(4-ethyl-2-hydroxyphenyl)-1-heptanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating on the water bath for 5 h (90 %) [2801].

 $C_{16}H_{24}O_2$ 

b.p.40 195° [2801].

#### 1-(5-Ethyl-2-hydroxyphenyl)-1-heptanone

$$\begin{array}{ccc} C_{15}H_{22}O_2 & \text{mol. wt. 234.34} \\ OH & Synthesis \\ \hline CO(CH_2)_5CH_3 & \text{-Obtained by Fries rearrangement of 4-ethylphenyl} \\ & \text{heptanoate with aluminium chloride at 100° for 2 h} \\ & (80 \%) [2800]. \\ & \text{b.p.9 178° [2800].} \end{array}$$

 $C_{21}H_{26}N_4O_5$ 

2,4-Dinitrophenylhydrazone

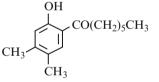
m.p. 129° [2800].

#### 1-(2-hydroxy-3,5-dimethylphenyl)-1-heptanone

	$C_{15}H_{22}O_2$			mol.	wt. 234	1.34
ОН	Synthesis					
$CH_3$ $\downarrow$ $CO(CH_2)_5CH_3$	-Obtained	by	Fries	rearranger	nent	of
Ϋ́, Ι	2,4-dimethyl	phenyl	heptanoa	ate (b.p. ₁₆	180-18	32°)
$\mathbf{i}$	with aluminium chloride (74 %) [184].					
ĊH ₃	pale yellow;	b.p. ₁₆	186-190	° [184].		

#### 1-(2-hydroxy-4,5-dimethylphenyl)-1-heptanone

 $C_{15}H_{22}O_2$ [50342-14-8] mol. wt. 234.34



Syntheses -Obtained by Fries rearrangement of 3,4-dimethylphenyl enanthate with aluminium chloride at 110° without solvent (83 %) [3117]. -Refer to: [2665].

b.p.₉ 220° [3117].

USE: Cyclization of, with chloroacetal [2665].

**2,4-Dinitrophenylhydrazone** 
$$C_{21}H_{26}N_4O_5$$
 mol. wt. 414.46

m.p. 211° [3117].

mol. wt. 414.46

#### 1-(2-Hydroxy-4,6-dimethylphenyl)-1-heptanone

[855899-90-0]	$C_{15}H_{22}O_2$	mol. wt. 234.34
CH ₃ CH ₃ OH CO(CH ₂ ) ₅ CH ₃ CH ₃	3,5-dimethylpheny *in the presence of in nitrobenzene at *in the presence of in refluxing carbor	Fries rearrangement of el enanthate (1 equiv.), aluminium chloride (1.3 equiv.) 25° for 6 h (73 %) [2801]; aluminium chloride (2.8 M), first a disulfide for 2 h, then for 2 h at elimination (63 %) [2801].
b.p. ₅ 160° [2801].		( <i>iii</i> ) [2001].

**2,4-Dinitrophenylhydrazone**  $C_{21}H_{26}N_4O_5$  mol. wt. 414.46

m.p. 165° [2801].

#### Methyl ether $C_{16}H_{24}O_2$ mol. wt. 248.37

-Obtained by reaction of dimethyl sulfate with 1-(2-hydroxy-4,6-dimethylphenyl)-1-heptanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating on the water bath for 5 h (61 %) [2801].

b.p.36 205° [2801].

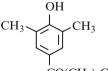
#### 1-(4-Hydroxy-2,5-dimethylphenyl)-1-heptanone

[95102-39-9]	$C_{15}H_{22}O_2$	mol. wt. 234.34
CH ₃ CO(CH ₂ ) ₅ CH ₃	Syntheses -Refer to: [1595, 2704]. USE: Colour developer, for materials [2704].	thermal recording

#### 1-(4-hydroxy-3,5-dimethylphenyl)-1-heptanone

Synthesis

C₁₅H₂₂O₂ mol. wt. 234.34

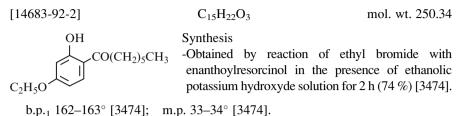


-Obtained by Fries rearrangement of 2,6-dimethylphenyl heptanoate (*vic-m-Xylenyl heptoate*) (b.p.₁₂ 162–164°) in the presence of aluminium chloride (65 %) [184].

CO(CH₂)₅CH₃

pale yellow [184]; b.p.₁₂ 220–230° [184]; m.p. 92–93° [184].

#### 1-(4-Ethoxy-2-hydroxyphenyl)-1-heptanone

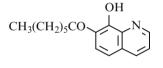


1-(8-Hydroxy-5-quinolinyl)-1-heptanone

# $\begin{array}{cccc} [62189-88-2] & C_{16}H_{19}NO_2 & \text{mol. wt. } 257.33 \\ OH & Synthesis \\ & & -\text{Refer to: } [2360]. \\ & & & \text{m.p. } 69-70^\circ \ [2360]; \ \ ^1\text{H NMR } [2360], \text{ IR } [2360]. \\ \end{array}$

#### 1-(8-Hydroxy-7-quinolinyl)-1-heptanone

[60697-65-6] C₁₆H₁₉NO₂ mol. wt. 257.33



Syntheses -Obtained by acylation of 8-quinolinol with n-heptanoyl chloride in the presence of aluminium chloride (20 %) [2361]. -Refer to: [2360, 2362].

m.p. 75–76° [2362]; ¹H NMR [2362], IR [2362].

#### **Copper complex**

[62153-16-6]

-Refer to: [2360].

**Ethyl ether** [62189-86-0]  $C_{18}H_{23}NO_2$  mol. wt. 285.38 -Obtained by ethylation of 7-heptanoyl-8-quinolinol with ethyl sulfide (71 %) [2360].

b.p.₂ 188–194° [2360];  $^{1}\text{H}$  NMR [2360], IR [2360];  $n_{D}^{20}=1.5614$  [2360].

#### 1-[2,4,6-Trihydroxy-3-(1-oxopropyl)phenyl]-1-heptanone

[129227-94-7] C16H22O5 mol. wt. 294.35 **Synthesis** OH .CO(CH₂)₅CH₃ -Obtained from 2,4,6-trihydroxy-3-(1-oxoheptyl)benzaldehyde [3406]. HO OH COCH₂CH₃

#### 1-(2-Ethyl-6-hydroxy-4-methylphenyl)-1-heptanone

	$C_{16}H_{24}O_2$	mol. wt. 248.37
CH ₃ CH ₃ CH ₃ CH ₃ CO(CH ₂ ) ₅ CH ₃ CO(CH ₂ ) ₅ CH ₃	5-methylphenyl enant *without solvent at 13	s rearrangement of 3-ethyl- hate with aluminium chloride, 0° for 2 h (80 %) [2802]; ° for 6 h (84 %) [2802].

b.p.2 200° [2802].

#### Methyl ether

C17H26O2 -Obtained by methylation of the above ketone in the usual way (76 %) [2802].

b.p.35 220° [2802].

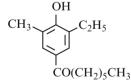
2,4-Dinitrophenylhydrazone C₂₂H₂₈N₄O₅ mol. wt. 428.49

m.p. 184° [2802].

#### 1-(3-Ethyl-4-hydroxy-5-methylphenyl)-1-heptanone

Synthesis

$$C_{16}H_{24}O_2$$
 mol. wt. 248.37



-Obtained by Fries rearrangement 2-ethylof 6-methylphenyl n-heptanoate (b.p.₁₂ 172–174°) in the presence of aluminium chloride (70 %) [184].

b.p.₁₂ 230–240° [184]; m.p. 56–57.5° [184].

mol. wt. 262.39

#### 1-(4-Hydroxy-3-(1-methylethyl)phenyl)-1-heptanone

USE: As colour developer [2704]; In preparation of thermographic recording material [1595].

#### 1-(2-Hydroxy-4-propoxyphenyl)-1-heptanone

[14683-93-3]  $C_{16}H_{24}O_3$  mol. wt. 264.37 OH Synthesis -Obtained by reaction of n-propyl bromide with enanthoylresorcinol in the presence of ethanolic potassium hydroxyde solution for 2 h (72 %) [3474].

 $b.p._1 \ 164-165^\circ \ [3474]; \quad m.p. \ 44^\circ \ [3474].$ 

#### 1-(6,7-Dihydro-4-hydroxy-6-methylfuro[2,3-f]-1,3-benzodioxol-8-yl)-1-heptanone

	$C_{17}H_{22}O_5$	mol. wt. 306.36
ОН	Synthesis	
$\downarrow$ ,0, ,CH ₃	-Refer to: [2179].	
	Methyl ether [82652-35-5]	
	(Heptanoyl furapiole)	
CO(CH ₂ ) ₅ CH ₃	$C_{18}H_{24}O_5$	mol. wt. 320.39

-Obtained by reaction of heptanoyl chloride with furapiole in the presence of fused zinc chloride in methylene chloride for 3–4 h in an ice bath (70 %) [2179].

colourless plates; m.p. 58° [2179]; ¹H NMR [2179], IR [2179].

USE: Synergistic insecticidal activity of, with pyrethrum [2179].

#### 1-(6,7-Dihydroxy-5-propyl-1,3-benzodioxol-4-yl)-1-heptanone

	$C_{17}H_{24}O_5$	mol. wt. 308.37
OH	Synthesis	
OF CH	-Refer to: [2179].	
< T I	<b>Dimethyl ether</b> [82652-27-5]	
$O \sim C_{3H_7}$	(Heptanoyl dihydrodillapiole)	
CO(CH ₂ ) ₅ CH ₃	$C_{19}H_{28}O_5$	mol. wt. 336.43

-Obtained by reaction of heptanoyl chloride with dihydrodillapiole in the presence of fused zinc chloride in methylene chloride for 3–4 h in an ice bath (70 %) [2179].

colourless solid [2179]; ¹H NMR [2179], IR [2179].

USE: Synergistic insecticidal activity of, with pyrethrum [2179].

#### 1-[3-Bromo-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-heptanone

[101577-83-7]	$C_{17}H_{25}BrO_2$	mol. wt. 341.29
$\begin{array}{c} OH\\ Br \\ \downarrow \\ \downarrow \\ C(CH_3)_3 \end{array} CO(CH_2)_5CH_3$	Synthesis -Preparation by Fries rearrangement butylphenyl heptanoate in the pre- chloride at 110° for 2 h (65 %) [2 b.p. ₂ 200° [3113].	esence of aluminium

2,4-Dinitrophenylhydrazone	$C_{23}H_{29}BrN_4O_5$	mol. wt. 521.41

m.p. 140-141° [3113].

Allyl ether [102465-47-4] C₂₀H₂₉BrO₂ mol. wt. 381.35

-Obtained by reaction of allyl bromide with the title ketone in the presence of potassium carbonate in refluxing acetone for 8 h (68 %) [3113].

b.p.₁ 164° [3113].

USE: Insecticide [3113].

#### 1-[5-(1,1-Dimethylethyl)-2-hydroxy-3-nitrophenyl]-1-heptanone

[85052-35-3]	$C_{17}H_{25}NO_4$	mol. wt. 307.39
$\begin{array}{c} OH\\ NO_2 \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\$	Synthesis -Obtained by nitration of heptanophenone (65 %) [2 process [1475]. ¹ H NMR [2105].	

#### 1-[3-(1,1-Dimethylethyl)-4-hydroxyphenyl]-1-heptanone

[95185-65-2] 
$$C_{17}H_{26}O_2$$
 mol. wt. 262.39  
OH Synthesis  
-Refer to: [2704].  
 $CO(CH_2)_5CH_3$ 

#### 1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-heptanone

[85052-34-2] 
$$C_{17}H_{26}O_2$$
 mol. wt. 262.39  
OH Synthesis  
-Refer to: [2105].  
C(CH₃)₃  
1.12 Hydravy 3 methyl 6 (1 methylethyl)phenyll 1 heptenone

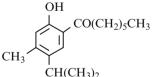
#### 1-[2-Hydroxy-3-methyl-6-(1-methylethyl)phenyl]-1-heptanone C.-H. O.

$C_{17}H_{26}O_2$	mol. wt. 262.39
Synthesis -Obtained by Frie heptanoate with (73 %) [2798]. b.p. ₃ 176° [2798].	es rearrangement of carvacryl aluminium chloride at 120°

m.p. 122° [2798].

#### 1-[2-Hydroxy-4-methyl-5-(1-methylethyl)phenyl]-1-heptanone

**2,4-Dinitrophenylhydrazone** C₂₃H₃₀N₄O₅



Synthesis CO(CH₂)₅CH₃ -Preparation by Fries rearrangement of 3-methyl-4-(1-methylethyl)phenyl heptanoate (20) with titanium tetrachloride in nitromethane at  $20^{\circ}$  for 170 h (95 %) (26) [1997].

mol. wt. 442.52

pale yellow crystals [1997]; m.p. 38° (Sadtler standard N° 79765K) [1997]; ¹H NMR (Sadtler standard N° 52708M) [1997],

IR (Sadtler standard N° 79765K) [1997], UV [1997], MS [1997].

#### 1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-heptanone

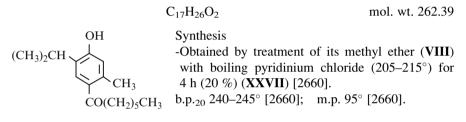
[855956-24-0]	$C_{17}H_{26}O_2$	mol. wt. 262.39
(CH ₃ ) ₂ CH CO(CH ₂ ) ₅ CH ₃ CH ₃	Synthesis -Obtained by Fries rearra heptanoate with aluminiu (84 %) [2803]. b.p. ₂ 173° [2803].	ngement of thymyl Im chloride at 120°

 $C_{23}H_{30}N_4O_5$ 

2,4-Dinitrophenylhydrazone

m.p. 174° [2803].

#### 1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-heptanone



Methyl ether (VIII)

 $C_{18}H_{28}O_2$ 

mol. wt. 276.42

mol. wt. 442.52

-Obtained by reaction of enanthyl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (52 %) [2660].

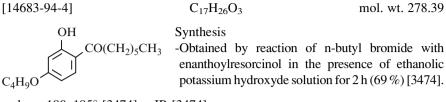
b.p.₁₈ 209–211° [2660];  $n_D^{23} = 1.5185$  [2660].

#### 1-[6-Hydroxy-2-methyl-3-(1-methylethyl)phenyl]-1-heptanone

[129375-15-1]	$C_{17}H_{26}O_2$	mol. wt. 262.39
OH CO(CH ₂ ) ₅ CH ₃	Synthesis -Preparation by treatment of	
CH ₃	2-hydroxy-6-methyl-5-(1-methyle (29) with aluminium chloride in 24 h (22 gl) (22) [1007]	
$CH(CH_3)_2$ m p. 65° (Sadtler st	24 h (82 %) ( <b>32</b> ) [1997].	

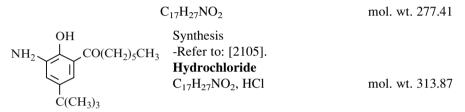
m.p. 65° (Sadtler standard N° 79805K) [1997]; ¹H NMR (Sadtler standard N° 52746M) [1997], IR (Sadtler standard N° 79805K) [1997], UV [1997], MS [1997].

#### 1-(4-Butoxy-2-hydroxyphenyl)-1-heptanone



b.p.₄ 190–195° [3474]; IR [3474].

#### 1-[3-Amino-5-(1,1-methylethyl)-2-hydroxyphenyl]-1-heptanone



-Obtained from the 5-tert-butyl-2-hydroxy-3-nitroheptanophenone (52 %) [2105].

m.p. 125–126° [2105]; ¹H NMR [2105], IR [2105], MS [2105]; TLC [2105].

#### 1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-heptanone

2-Heptanoyl-4-(3-methylbuten-2-yl)phloroglucinol (12) [1026].

[69916-11-6]  $C_{18}H_{26}O_4$  mol. wt. 306.40 OH Syntheses  $CH_3 - C = CHCH_2$   $CO(CH_2)_5CH_3$  -Obtained by adding cuprous chloride, a  $CH_3$   $CH_3$ 

CO(CH₂)₅CH₃ -Obtained by adding cuprous chloride, a saturated aqueous solution of sodium carbonate and prenyl bromide to a solution of phloroheptanophenone in ethyl ether at r.t., then to keep the mixture for 12 h at this temperature [1026].

-Also prepared by adding prenyl bromide to a suspension of the sodium salt of phloroheptanophenone in benzene, then the mixture obtained was kept overnight at  $0^{\circ}$  [2110] or refluxed for 3 h [1026].

-Also obtained by reaction of prenyl chloride with phloroheptanophenone in an alkaline two-phase (aqueous-ether) system and catalyzed by CuCl [838, 3193].

-Also prepared by adding at  $5^{\circ}$  a solution of sodium methoxide in methanol, followed by isopentenyl bromide in ethyl ether to a solution of phloroheptanophenone in benzene and ethyl ether; then the mixture was stirred at r.t. for 6 h (15 %) [2113]. m.p. 157° [2113], 136–138° [3193]. **N.B.**: One of the reported melting point is obviously wrong. ¹³C NMR [1026, 3193], IR [1026].

BIOLOGICAL ACTIVITY: Antifungal against *Trichophyton* species [2113]; Bactericidal and fungicidal [1026, 3193].

#### N,N-Diethyl-2-heptanoyl-6-hydroxybenzamide

	C ₁₈ H ₂₇ NO ₃	mol. wt. 305.40
ОН	Synthesis	
$CON(C_2H_5)_2$	-Refer to: [2321].	
	Methyl ether [334698-83-8]	
CO(CH ₂ ) ₅ CH ₃	C ₁₉ H ₂₉ NO ₃	mol. wt. 319.44

-Obtained by adding a solution of N,N-diethyl-2-(1-hydroxyheptyl)-6-methoxybenzamide in methylene chloride to a suspension of PDC in the same solvent under an argon atmosphere at r.t. and the mixture stirred for 15 h (89 %) [2321].

colourless oil [2321]; ¹H NMR [2321], ¹³C NMR [2321], IR [2321].

#### 1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methylphenyl]-1-heptanone

C ₁₈	H ₂₈ O ₂ mol. wt. 276.42
(CH ₃ ) ₃ C CO(CH ₂ ) ₅ CH ₃ CH ₃	Syntheses -Obtained by Fries rearrangement of 2-tert-butyl- 5-methylphenyl enanthate, *in the presence of aluminium chloride (1.5 equiv.) in nitrobenzene at 25° for 6 h (76 %) [3118];

*in the presence of aluminium chloride (3 equiv.) at 110° for 2 h (72 %) [3118].

b.p.₁₂ 155° [3118].

<b>2,4-Dinitrophenylhydrazone</b> $C_{24}H_{32}N_4O_5$ mol. wt. 456	56.54
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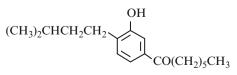
m.p. 187° [3117, 3118].

## 1-[3-Hydroxy-4-(3-methylbutyl)phenyl]-1-heptanone

[101873-72-7]

 $C_{18}H_{28}O_2$ 

mol. wt. 276.42



Synthesis -Obtained by diazotization of 3-amino-4-iso-amylheptanophenone [551].

m.p. 98–98.5° [551].

#### **2,4-Dinitrophenylhydrazone** [113927-77-8] C₂₄H₃₂N₄O₅ mol. wt. 456.54

m.p. 165° [551].

#### 1-[2-Hydroxy-4-(3-methylbutoxy)phenyl]-1-heptanone

[14683-95-5]

C18H28O3

Synthesis

(45 %) [3474].

mol. wt. 292.42

mol. wt. 312.36

OH CO(CH₂)₅CH₃ (CH₃)₂CHCH₂CH₂O

b.p.₂ 180–182° [3474].

#### 1,7-Bis(5-chloro-2,4-dihydroxyphenyl)-1,7-heptanedione

[26086-80-6] C19H18Cl2O6 mol. wt. 413.25 OH HO Syntheses -Obtained by reaction of pimelic acid

dichloride with 4-chlororesorcinol in the presence of aluminium chloride [589, 591].

-Obtained by reaction of isoamyl bromide with enanthoylresorcinol

in the presence of ethanolic potassium hydroxyde solution for 2 h

m.p. 203° [589, 591].

Tetramethyl ether	[26086-83-9]	$C_{23}H_{26}Cl_2O_6$	mol. wt. 469.36
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-Obtained by reaction of pimelic acid dichloride with 4-chlororesorcinol dimethyl ether in the presence of aluminium chloride [589, 591].

 $C_{19}H_{20}O_4$ 

m.p. 163° [589, 591]; IR [589].

#### 1,7-Bis(2-hydroxyphenyl)-1,7-heptanedione

[10401-04-4]



Syntheses -Obtained by Fries rearrangement of phenyl pimelate with aluminium chloride (15-20 %) [1576].

-Also refer to: [1575].

m.p. 90° [1575, 1576].

OH

#### 1,7-Bis(3-hydroxyphenyl)-1,7-heptanedione

-Also refer to: [1575].

m.p. 136° [1575, 1576].

#### 1,7-Bis(4-hydroxyphenyl)-1,7-heptanedione

[20837-38-1]

 $C_{19}H_{20}O_4$ 

mol. wt. 312.36

mol. wt. 344.36

-Refer to: [584, 1148, 1576].

Syntheses

Syntheses

m.p. 187–189° [1148], 55° [584].

N.B.: One of the reported melting point is obviously wrong.

#### **Dimethyl ether** [10365-60-3] C₂₁H₂₄O₄ mol. wt. 340.42

-Obtained by reaction of pimelic acid dichloride with anisole in the presence of aluminium chloride (80–90 %) [1576].

-Also refer to: [584, 1148, 1575].

m.p. 100° [584, 1575, 1576], 95–97° [1148].

#### 1,7-Bis(2,4-dihydroxyphenyl)-1,7-heptanedione

-Obtained by adding resorcinol into the dinitrile of pimelic acid and hydrogen chloride in ethyl ether in the presence of zinc chloride.

Then, the diketimine dihydrochloride obtained was hydrolyzed by boiling water (76 %) [2674].

-Also obtained by reaction of pimelic acid dichloride with resorcinol in the presence of aluminium chloride [591].

-Also refer to: [589, 1735, 2504].

m.p. 166° (d) [2504, 2674], 165° [589, 591]; UV [2504]. **2,4-Dinitrophenylhydrazone** [32354-10-2] C₃₁H₂₈N₈O₁₂ mol. wt. 704.61 m.p. 213° [589, 2674].

**Tetramethyl ether** [32246-62-1] C₂₃H₂₈O₆ mol. wt. 400.47

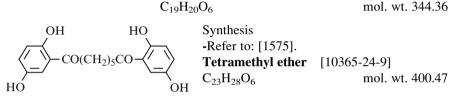
-Refer to: [589].

m.p. 123° [589].

Dioxime of the tetramethyl ether [32246-63-2]  $C_{23}H_{30}N_2O_6$  mol. wt. 430.50

m.p. 164° [589].

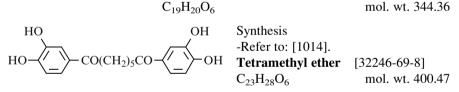
#### 1,7-Bis(2,5-dihydroxyphenyl)-1,7-heptanedione



-Obtained by reaction of pimelic acid dichloride (1 mol) with hydroquinone dimethyl ether (2.5 mol) in the presence of aluminium chloride (1 mol) (58 %) [1575].

m.p. 76° [1575].

#### 1,7-Bis(3,4-dihydroxyphenyl)-1,7-heptanedione



-Obtained by reaction of pimelic acid dichloride with pyrocatechol dimethyl ether in the presence of aluminium chloride [591].

-Also obtained by hydrogenating of its oxime in acetic acid in the presence of 10 % Pd/C in a Parr apparatus at r.t. and 45–50 psig for 12–36 h (68 %) [1014]. -Also refer to: [589, 1012, 2342].

m.p. 95° [589, 591], 94–95° [1012], 91–93° [1014]; ¹H NMR [2342], ¹³C NMR [2342].

#### Dioxime of the tetramethyl ether [50766-29-5] $C_{23}H_{30}N_2O_6$ mol. wt. 430.50

m.p. 115–117° [1014].

#### 1,7-Bis(2,3,4-trihydroxyphenyl)-1,7-heptanedione

$$\begin{array}{cccc} C_{19}H_{20}O_8 & \text{mol. wt. 376.36} \\ HO & OH & HO & OH & Synthesis \\ HO & & & & & & \\ HO & & \\ HO & & & \\ HO & & \\ HO & & & \\ HO & & \\ HO$$

-Obtained by reaction of pimelic acid dichloride with pyrogallol trimethyl ether in the presence of aluminium chloride [591].

-Also obtained by reaction of dimethyl sulfate with 1,7-bis(2-hydroxy-3,4-dimethoxyphenyl)-1,7-heptanedione the presence of 30 % sodium hydroxide (65–90 %) [1574].

-Also refer to: [1575] (Japanese paper).

m.p. 114° [591], m.p. 60° [1574, 1575].

**N.B.**: One the reported melting point is obviously wrong.

#### 1-(3'-Bromo-4'-hydroxy[1,1'-biphenyl]-4-yl)-1-heptanone

-Obtained by acylation of 2-bromo-4-phenylanisole with nonanoyl chloride (64 %) [2992].

#### 1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-1-heptanone

[390358-13-1]  $C_{19}H_{22}O_2$  mol. wt. 282.38 HO  $\leftarrow$   $CO(CH_2)_5CH_3$  Syntheses -Obtained by Fries rearrangement of 4-heptanoyl-oxybiphenyl with aluminium chloride in nitrobenzene, first at 20° for 12 h, then at 60° for 1 h [522].

-Also refer to: [1518].

USE: For preparation of biphenylyl sulfamates as steroid sulfatase inhibitors for estrogen-dependent diseases [1518].

Acetate	[126516-15-2]	$C_{21}H_{24}O_3$	mol. wt. 324.42
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-Refer to: [847, 2080].

#### Various ethers (11)

-Preparations and liquid crystalline properties of, [847].

Methyl ether	[56116-79-1]	$C_{20}H_{24}O_2$	mol. wt. 296.41
-Obtained from hep -Also refer to: [514	otanoyl chloride and 4 , 847].	-methoxybipheny	1 [522].
smooth spangles	[522]; b.p. ₁₂ 250–2	252° [514, 522];	m.p. 118° [514, 522].
<b>Ethyl ether</b> -Refer to: [847].	[56116-88-2]	$C_{21}H_{26}O_2$	mol. wt. 310.44
Propyl ether -Refer to: [847].	[56116-96-2]	$C_{22}H_{28}O_2$	mol. wt. 324.46
Butyl ether -Refer to: [847].	[56117-04-5]	$C_{23}H_{30}O_2$	mol. wt. 338.49
<b>Pentyl ether</b> -Refer to: [847].	[56117-13-6]	$C_{24}H_{32}O_2$	mol. wt. 352.52
Hexyl ether -Refer to: [847].	[56117-22-7]	$C_{25}H_{34}O_2$	mol. wt. 366.54
Heptyl ether -Refer to: [847].	[56117-31-8]	$C_{26}H_{36}O_2$	mol. wt. 380.57
Octyl ether -Refer to: [847].	[56117-39-6]	$C_{27}H_{38}O_2$	mol. wt. 394.60
Nonyl ether -Refer to: [847].	[56117-48-7]	$C_{28}H_{40}O_2$	mol. wt. 408.62
<b>Decyl ether</b> -Refer to: [847].	[56117-57-8]	$C_{29}H_{42}O_2$	mol. wt. 422.65
<b>Dodecyl ether</b> -Refer to: [847].	[56117-66-9]	$C_{31}H_{46}O_2$	mol. wt. 450.71

#### 1-(6-Hydroxy[1,1'-biphenyl]-3-yl)-1-heptanone

[95102-35-5]	$C_{19}H_{22}O_2$	mol. wt. 282.38
CO(CH ₂ ) ₅ CH ₃	Syntheses -Refer to: [1595, 2704]. USE: Colour developer, f materials [2704].	or thermal recording

#### 1-(5-Chloro-3-hexyl-2-hydroxyphenyl)-1-heptanone

[855921-00-5]	$C_{19}H_{29}ClO_2$	mol. wt. 324.89
OH	Synthesis	
$C_6H_{13}$ $CO(CH_2)_5CH_3$	-Obtained by Fries rearran	gement of 4-chloro-
	2-hexyl-phenyl enanthate v	
$\mathbf{i}$	ride at $120^{\circ}$ for 1 h (44 %)	[3235].
C1	oil [3235]; b.p. _{0.2} 198–205	° [3235].

#### 1-[4-(Hexyloxy)-2-hydroxyphenyl]-1-heptanone

CO(CH₂)₅CH₃

 $[143286-90-2] C_{19}H_{30}O_3 mtext{mol. wt. 306.45}$ 

Synthesis

-Obtained by reaction of hexyl bromide with 2,4-dihydroxyheptanophenone in the presence of potassium carbonate in refluxing acetone for 20 h [284].

m.p. 27.5–28.5° [284].

OH

C₆H₁₃O

Oxime [143286-59-3]	$C_{19}H_{31}NO_3$	mol. wt. 321.46
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-Obtained by reaction of hydroxylamine hydrochloride with 1-[4-(hexyloxy)-2-hydroxyphenyl]-1-heptanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 45–48° [284]; ¹H NMR [284].

#### 1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-2-methyl-1-heptanone

[186041-43-0] [186041-40-6] (S)	$C_{20}H_{24}O_2$	mol. wt. 296.41
	CH $-C_5H_{11}$ Synthesis -Refer to: [2278]. CH ₃	

#### 1-[2-Hydroxy-5-(phenylmethoxy)phenyl]-1-heptanone

[719315-64-7]	$C_{20}H_{24}O_3$	mol. wt. 312.40
OH CO(CH ₂ ) ₅ CH ₃ CO(CH ₂ ) ₅ CH ₃ OCH ₂ C ₆ H ₅	Syntheses -Refer to: [3460–3462]. USE: Electroluminescent devices fluorene-containing compounds [346	1,0,0,1

#### 1,1'-(2,4-Dihydroxy-1,3-phenylene)bis-1-heptanone

 $\begin{array}{c} C_{20}H_{30}O_4 & \text{mol. wt. 334.46} \\ OH & Synthesis \\ CH_3(CH_2)_5CO & CO(CH_2)_5CH_3 & -Also obtained by Fries rearrangement of resorcinol dilenanthate with aluminium chloride in nitrobenzene for 8 h at 40–50° (25 %) [379]. \end{array}$ 

m.p. 48-49° [379].

2,4-Dinitrophenylhydrazone

m.p. 196° [379].

#### 1,1'-(4,6-Dihydroxy-1,3-phenylene)bis-1-heptanone

	$C_{20}H_{30}O_4$	mol. wt. 334.46
HO CO(CH ₂ ) ₅ CH ₃	Synthesis -Also obtained by Fries rearranger dilenanthate with aluminium chlori for 8 h at 40–50° (25 %) [379]. m.p. 48–49° [379].	

 $C_{26}H_{34}N_4O_7$ 

 $C_{26}H_{34}N_4O_7$ 

#### 2,4-Dinitrophenylhydrazone

m.p. 196° [379].

#### 1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-heptanone

[3118-36-3]	C ₂₀ I	$H_{30}O_5$	mol. wt. 350.46
CH ₃ (CH ₂ ) ₅ CO	OH CO(CH ₂ ) ₅ CH ₃	Syntheses -Obtained by reaction of ride with phloroglucinol aluminium chloride in 3 days at r.t. (5–10 %) [4	in the presence of nitrobenzene for

mol. wt. 514.57

mol. wt. 514.57

-Also obtained by reaction of heptanoic anhydride with phloroglucinol in the presence of boron trifluoride etherate on heating for 4 h [457]. -Also refer to: [457, 600, 644, 2911, 3033].

m.p. 98–99° [421], 96–98° [457, 2911]: ¹H NMR [421], IR [421], MS [421].

BIOLOGICAL ACTIVITY: Inhibitory effects of treatment with this acylphenol on TPA Induced EBV-EA Activation [3033]; Antiviral activity towards the replication of vesicular stomatitis virus in HEL cell cultures [644]; Germination inhibition in cress (Lepidium sativum) seeds [421]; For treatment of immune dysfunction associated with human immuno deficiency virus infection [600]; Anthelmintic [457].

#### 1,7-Bis(2-hydroxy-3-methylphenyl)-1,7-heptanedione

[10365-66-9]  $C_{21}H_{24}O_4$ mol. wt. 340.42 CH₃ OH Syntheses CH₃ HO -Obtained by Fries rearrangement of o-cresol O(CH₂)₅CC pimelate with aluminium chloride (15-20 %) [1576].

-Also refer to: [1575].

m.p. 76° [1575, 1576].

#### 1,7-Bis(2-hydroxy-4-methylphenyl)-1,7-heptanedione

[13221-24-4] 
$$C_{21}H_{24}O_4$$
 mol. wt. 340.42  
OH HO Syntheses  
-Obtained by Fries rearrangement of  
m-cresol pimelate with aluminium  
chloride (70–80 %) [1576].

-Also refer to: [1575].

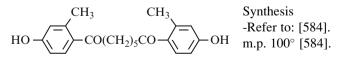
m.p. 127° [1575, 1576].

#### 1,7-Bis(4-hydroxy-2-methylphenyl)-1,7-heptanedione

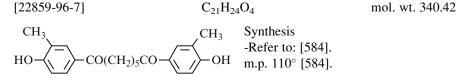
[22860-00-0]



mol. wt. 340.42



#### 1,7-Bis(4-hydroxy-3-methylphenyl)-1,7-heptanedione



.42

of

#### 1,7-Bis(2-hydroxy-5-methylphenyl)-1,7-heptanedione

[111162-35-7]	(	$C_{21}H_{24}O_4$	mol. wt. 340.42
OH CO(CH ₂ ) ₅ C	HO CO-CH ₃	(4-methyl-phenyl) pin	rearrangement of di nelate with aluminium chlorobenzene for 6 h

-Also refer to: [584].

m.p.  $140^{\circ}$  [584],  $100-101^{\circ}$  [3107]. **N.B.**: One of the reported melting points is obviously wrong. IR [3107].

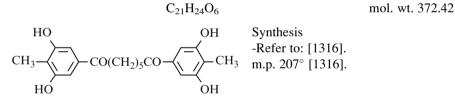
Dimethyl ether	[10400-50-7]	$C_{23}H_{28}O_4$	mol. wt. 368.47
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-Obtained by reaction of pimelic acid dichloride with p-cresol methyl ether in the presence of aluminium chloride (50–70 %) [1576].

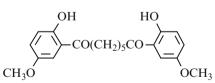
-Also refer to: [1575].

m.p. 82° [1575, 1576].

#### 1,7-Bis(3,5-dihydroxy-4-methylphenyl)-1,7-heptanedione



#### 1,7-Bis(2-hydroxy-5-methoxyphenyl)-1,7-heptanedione



Synthesis

C21H24O6

mol. wt. 372.42

-Obtained by reaction of pimelic acid dichloride with p-dimethoxybenzene in the presence of aluminium chloride [1575].

m.p. 107° [1575].

[10365-29-4]

Diacetate	[10365-34-1]	$C_{25}H_{28}O_8$	mol. wt. 456.49
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-Obtained by reaction of acetic anhydride (4 vol) with the title ketone in the presence of 1–2 drops concentrated sulfuric acid, the mixture warmed 5 min [1575].

m.p. 92° [1575].

#### 1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-2,6-dimethyl-1-heptanone

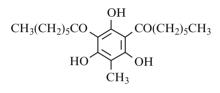
$$[117693-02-4] C_{21}H_{26}O_2 mol. wt. 310.44$$

$$HO \longrightarrow CO - CH - (CH_2)_3 - CH - CH_3 + Refer to: [2095].$$
Benzyl ether [117693-01-3] C_{28}H_{32}O_2 mol. wt. 400.56

-Refer to: [2095].

#### 1,1-(2,4,6-Trihydroxy-5-methyl-1,3-phenylene)bis-1-heptanone

$$[3118-39-6] C_{21}H_{32}O_5 mol. wt. 364.48$$



Syntheses CO(CH₂)₅CH₃ -Obtained by reaction of heptanoic OH anhydride with 2-methylphloroglucinol in the presence of boron trifluoride etherate on heating for 4 h [457]. -Also refer to: [457, 600, 2911].

m.p. 108–110° [457, 2911].

BIOLOGICAL ACTIVITY: For treatment of immune dysfunction associated with human immuno deficiency virus infection [600]; Anthelmintic [457].

#### 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-heptanone

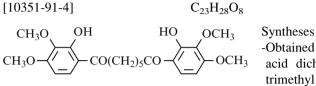
	$C_{21}H_{34}O_2$	mol. wt. 318.50
OH (CH ₃ ) ₃ C $\downarrow$ C(CH ₃ ) ₃	Syntheses -Refer to: [3103, 3104].	
(CH ₃ ) ₃ C C(CH ₃ ) ₃	m.p. 76–78° [3103, 3104]; ¹ H NMR [3103, 3104], ¹³ C NMR	[3104], IR [3103,
CO(CH ₂ ) ₅ CH ₃	3104].	[]; [;

#### 1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methyl-5-(1-methylethyl)phenyl]-1-heptanone

[129375-14-0]	$C_{21}H_{34}O_2$	mol. wt. 318.50
$(CH_3)_3C \underbrace{+}_{CH_3}CO(CH_2)_5CH_3$ $CH(CH_3)_2$	Synthesis -Obtained by Fries rearran methylethyl)-5-methyl-4-( heptanoate (23) with tir in chlorobenzene at 100° [1997].	1-methylethyl)phenyl tanium tetrachloride

pale yellow oil [1997]; b.p._{0.2} 156–157° [1997]; m.p. 29–30° (Sadtler standard N° 79804K) [1997]; ¹H NMR (Sadtler standard N° 52745M) [1997], IR (Sadtler standard N° 79804K) [1997], UV [1997], MS [1997].

#### 1,7-Bis(2-hydroxy-3,4-dimethoxyphenyl)-1,7-heptanedione



mol. wt. 432.47

-Obtained by reaction of pimelic acid dichloride with pyrogallol trimethyl ether in the presence of aluminium chloride in tetrachloroethane [1574].

-Also refer to: [589, 1575].

m.p. 116° [1574, 1575], 114° [589].

#### 1-(4-Butoxy-5-hexyl-2-hydroxyphenyl)-1-heptanone

[143287-07-4]  $C_{23}H_{38}O_3$  mol. wt. 362.55 OH  $C_4H_9O$   $C_{6}H_{13}$   $C_{23}H_{38}O_3$  mol. wt. 362.55 Synthesis -Obtained (12b) by reaction of 1-bromobutane with 1-(2,4-dihydroxy-5-hexylphenyl)-1-heptanone in the presence of potassium carbonate in refluxing acetone for 20 h [284]. m.p. 32–33° [284].

C26H34O4

#### 1,1'-(4,4'-Dihydroxy[1,1'-biphenyl]-3,3'-diyl)-1-heptanone

[104098-36-4]

CH₃(CH₂)₅CO HO – OH

mol. wt. 410.55

*in the presence of sodium chloride at 140° (85 %) [2091]; *in refluxing chlorobenzene for 24 h (52 %) [2377].

m.p. 93–94° [2377]; IR [2377].

## 2 Aromatic Hydroxyketones Derived from Various Halogenoheptanoic Acids

#### 2.1 Unsubstituted Hydroxyketones

#### 2-Bromo-1-(4-hydroxyphenyl)-1-heptanone

	$C_{13}H_{17}BrO_2$		mol. wt. 285.18
$\bigcup_{i=1}^{OH} Br \\ Br \\ CO-CH-C_5H_{11}$		[97744-27-9] -dimethylethoxy)phenyl]-	1-heptanone mol. wt. 341.29
Defer tex [1140]			

-Refer to: [1140].

Methyl ether	[1033774-79-6]	$C_{14}H_{19}BrO_2$	mol. wt. 299.21
-Refer to: [2987].			

#### 2.2 Substituted Hydroxyketones

#### 7-Bromo-1-(2,3-dichloro-4-hydroxyphenyl)-1-heptanone

	$C_{13}H_{15}BrCl_2O_2$	mol. wt. 354.07
ОН	Synthesis	
,CI	-Refer to: [3333].	
	Methyl ether [53107-72-5]	
Cl	$C_{14}H_{17}BrCl_2O_2$	mol. wt. 368.10
CO(CH ₂ ) ₅ CH ₂ Br		

-Obtained by reaction of 7-bromoheptanoyl chloride with 2,3-dichloroanisole in the presence of aluminium chloride (58 %) [3333]. -Also refer to: [740, 2051–2053].

m.p. 57° [2051, 2052, 3333].

USE: Preparation and cyclization of, [2053]; Preparation and reaction of, with formaldehyde [2052].

#### 7-Bromo-1-(5-chloro-2-hydroxyphenyl)-1-heptanone

[206051-19-6]	$C_{13}H_{16}BrClO_2$	mol. wt. 319.63
OH CO(CH ₂ ) ₅ CH ₂ Br	Synthesis -Refer to: [3284].	
CI	Methyl ether $[187396-82-3]$ $C_{14}H_{18}BrClO_2$ -Refer to: $[308, 3284]$ .	mol. wt. 333.65

#### 2-Bromo-1-(2-hydroxy-5-methylphenyl)-1-heptanone

 $\begin{array}{c} C_{14}H_{19}BrO_2 & \text{mol. wt. 299.21} \\ OH & Synthesis \\ \downarrow & CO - CH - (CH_2)_4CH_3 & -Refer to: [181]. \\ & Br & Fluorescence [181]. \\ & CH_3 \end{array}$ 

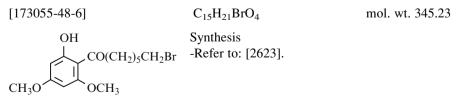
#### 7-Bromo-1-(2-hydroxy-5-methoxyphenyl)-1-heptanone

[173055-36-2]	$C_{14}H_{19}BrO_3$	mol. wt. 315.20
OH	Synthesis	
$\downarrow$ CO(CH ₂ ) ₅ CH ₂ Br	-Refer to: [2623].	
$\mathbf{Y}$		
OCH ₃		

#### 7-Bromo-1-(2-hydroxy-4,5-dimethoxyphenyl)-1-heptanone

 $[173055-42-0] C_{15}H_{21}BrO_4$ mol. wt. 345.23 OH Synthesis CO(CH₂)₅CH₂Br -Refer to: [2623]. CH₃O OCH₃

#### 7-Bromo-1-(2-hydroxy-4,6-dimethoxyphenyl)-1-heptanone



#### 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-7-bromo-1-heptanone

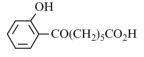
mol. wt. 397.39 [158869-48-8]  $C_{21}H_{33}BrO_2$ **Synthesis** OH -Refer to: [1689].  $C(CH_3)_3$  $(CH_3)_3C$ CO(CH₂)₅CH₂Br

#### 3 Aromatic Hydroxyketones Derived from 7-Oxoheptanoic Acids

#### 3.1 Unsubstituted Hydroxyketones

#### 7-(2-Hydroxyphenyl)-7-oxo-1-heptanoic acid

[57262-58-5] mol. wt. 236.27 C13H16O4



Syntheses -Obtained by treatment of tetrahydroxanthone  $CO(CH_2)_5CO_2H$  (m.p. 104°) with 30 % KOH in refluxing ethanol for 1 h [1211].

-Also refer to: [1196].

colourless plates [1211]; m.p. 97–99° [1211], 94° [1196]; ¹H NMR [1196], IR [1196], UV [1196].

[133535-19-0]  $C_{14}H_{18}O_4$ Methyl ester mol. wt. 250.29 -Refer to: [1211]. colourless plates [1211]; b.p.₃ 182–184° [3370]; m.p. 40–42° [1211], 37.5–38.5° [3370].

#### 7-(4-Hydroxyphenyl)-7-oxo-1-heptanoic acid

$C_{13}H_{16}O_4$		mol. wt. 236.27
HO CO(CH ₂ ) ₅ COOH	Syntheses -Refer to: [445, 1196]. m.p. 140–141° [445];	IR [1196], UV [1196].

#### 7-(2,4-Dihydroxyphenyl)-7-oxo-1-heptanoic acid

4-(6-Carboxyhexanoyl)resorcinol [2184]

[30414-64-3] 
$$C_{13}H_{16}O_5$$
 mol. wt. 252.27  
OH Syntheses  
-Obtained by reaction of pimelic acid with resor-  
cinol in the presence of zinc chloride at 140° for 5 h  
(50 %) [445].

Also obtained by passing rapidly for 3 h hydrogen chloride into a mixture of resorcinol, methyl 6-cyanohexanoate, anhydrous zinc chloride and ether at  $0^{\circ}$ . The mixture was kept at 0° overnight and the resulting red oil was boiled with water for 30 min (20.4 %) [2184].

plates [2184]; m.p. 131–131.5° [2184], 126–127° [2606], 126° [589], 124–126° [445]; IR [2184], UV [2184].

#### Methyl ester

C14H18O5

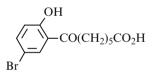
mol. wt. 266.29

b.p.₀₄ 250–255° [445].

#### 3.2 Substituted Hydroxyketones

#### 7-(5-Bromo-2-hydroxyphenyl)-7-oxo-1-heptanoic acid

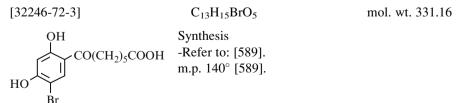
C13H15BrO4 mol. wt. 315.16



Synthesis -Obtained by treatment of 7-bromotetrahydroxanthone  $CO(CH_2)_5CO_2H$  (m.p. 151°) with 30 % KOH in refluxing dilute ethanol for 1 h [1211].

colourless plates [1211]; m.p. 120° [1211].

#### 7-(5-Bromo-2,4-dihydroxyphenyl)-7-oxo-1-heptanoic acid



#### 7-(5-Chloro-2-hydroxyphenyl)-7-oxo-1-heptanoic acid

 $[22994-79-2] C_{13}H_{15}ClO_4 mol. wt. 270.71$  OH Syntheses -Obtained by treatment of 7-chlorotetrahydroxanthone (m.p. 154°) with 30 % KOH in refluxing dilute ethanol for 1 h [1211]. -Also refer to: [584].

colourless plates [1211]; m.p. 122° [1211], 119° [584].

#### 7-(5-Chloro-2,4-dihydroxyphenyl)-7-oxo-1-heptanoic acid

 $[32340-75-3] C_{13}H_{15}ClO_5 mol. wt. 286.71$ OH Synthesis -Refer to: [589]. HO Cl m.p. 147–148° [589].

#### 7-(2,4-Dihydroxy-6-methylphenyl)-3,5,7-trioxo-1-heptanoic acid

 $\begin{array}{c} C_{14}H_{14}O_7 & \text{mol. wt. 294.26} \\ OH & \text{Syntheses} \\ -Obtained from 3,5,7,9,11,13-hexaoxotetradecanoic acid [1253].} \\ HO & CH_3 & -Also refer to: [1259, 1287]. \end{array}$ 

USE: Precursor of 2,2',4,4',6-pentahydroxy-6'-methylbenzophenone in the biosynthesis of griseofulvin [1253].

**Dimethyl ether** [62643-23-6] C₁₆H₁₈O₇ mol. wt. 322.31

-Obtained by adding 1-(2,4-dimethoxy-6-methylphenyl)-1,3,5-hexanetrione to lithium diisopropylamide in THF at 0° under nitrogen. After 10 min,  $CO_2$  was bubbled into the red-brown reaction mixture for 5 min. The solvent was evaporated in vacuo and the residue was acidified with cold, dilute HCl (19 %) [1259].

m.p. 84–85.5° [1259]; ¹H NMR [1259], IR [1259], UV [1259].

Methyl ester of the dimethyl ether [38071-49-7] C₁₇H₂₀O₇ mol. wt. 336.34

-Obtained by treatment of methyl 7-(2-hydroxy-4-methoxy-6-methylphenyl)-3,5,7-trioxoheptanoate with diazomethane in ethyl ether for 30 s (95 %) [1259, 1287].

yellow oil [1259]; ¹H NMR [1259], IR [1259], UV [1259].

Dibenzyl ether[38071-43-1] $C_{28}H_{26}O_7$ mol. wt. 474.51-Refer to: [1259].Methyl ester of the dibenzyl ether[38071-44-2] $C_{29}H_{28}O_7$ mol. wt. 488.54-Refer to: [1259, 1287].IR [1259], UV [1259].Methyl ester[38071-45-3] $C_{15}H_{16}O_7$ mol. wt. 308.29-Refer to: [1259].

-Obtained by hydrogenolysis of methyl ester of dibenzyl ether [1287].

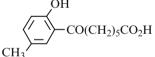
#### 7-(5-Chloro-2-hydroxy-6-methylphenyl)-7-oxo-1-heptanoic acid

	$C_{14}H_{17}ClO_4$	1	mol. wt. 284.74
OH CO(CH ₂ ) ₅ COOH CH ₃	Synthesis -Refer to: [584]. m.p. 148° [584]		

#### 7-(2-hydroxy-5-methylphenyl)-7-oxo-1-heptanoic acid

$$C_{14}H_{18}O_4 \qquad \text{mol. wt. 250.29}$$

$$H \qquad \qquad \text{Synthesis}$$



C

Synthesis				
-Obtained	by	treatment	of	7-methyltetra-
hydroxanthone (m.p. 102–103°) with 30 % KOH				
in refluxing dilute ethanol for 1 h [1211].				

colourless plates [1211]; m.p. 87° [1211].

#### 7-(4-Hydroxy-2-methylphenyl)-7-oxo-1-heptanoic acid

[22811-84-3]	$\mathrm{C}_{14}\mathrm{H}_{18}\mathrm{O}_{4}$	mol. wt. 250.29
HO	Synthesis -Refer to: [584]. m.p. 105° [584].	

#### 7-(2-Hydroxy-4-methoxy-6-methylphenyl)-3,5,7-trioxo-1-heptanoic acid

$$\begin{array}{c} C_{15}H_{16}O_7 & \text{mol. wt. 308.29} \\ OH & Synthesis \\ COCH_2COCH_2COCH_2CO_2H & -Refer to: [1259]. \\ OH & Hethyl ester \\ CH_3O & CH_3 & C_{16}H_{18}O_7 & \text{mol. wt. 322.31} \end{array}$$

-Obtained by hydrogenolysis of its benzyl ether (86 %) [1259].

Benzyl ether of the methyl ester [62643-41-8] C₂₃H₂₄O₇ mol. wt. 412.44

-Obtained by treatment of 1-(2-benzyloxy-4-methoxy-6-methylphenyl)-1,3,5-hexanetrione with lithium diisopropylamide in THF for 15 min at 0° followed by gaseous CO₂ (28 %) [1259].

yellow oil [1259]; ¹H NMR [1259], IR [1259], UV [1259], MS [1259].

#### 7-(4-Hydroxy-2-methoxy-6-methylphenyl)-3,5,7-trioxo-1-heptanoic acid

C ₁₅ H ₁₆ O ₇	mol. wt. 308.29
ОН	Synthesis -Refer to: [1253].
CH ₃ COCH ₃ COCH ₂ CO ₂ H	USE: Precursor of griseophenone C in the biosynthesis of griseofulvin [1253].

#### 7-(2-Hydroxy-3,4,5-trimethylphenyl)-7-oxo-1-heptanoic acid

[84978-19-8]	$C_{16}H_{22}O_4$	mol. wt. 278.35
CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃	Synthesis -Refer to: [2325]. m.p. 105° [2325];	¹ H NMR [2325], IR [2325].

#### 7-(2-Hydroxy-3,4,6-trimethylphenyl)-7-oxo-1-heptanoic acid

[58185-75-4]	$C_{16}H_{22}O_4$	mol. wt. 278.35
OH CH ₃ CO(CH ₂ ) ₅ COOH	Synthesis -Refer to: [2149, 2325].	
CH ₃ CH ₃	m.p. 119–125° [2149, 2325]; 2325], IR [2149, 2325].	¹ H NMR [2149,

## Chapter 6 Octanones

#### 1 Aromatic Hydroxyketones Derived from Octanoic Acids

#### 1.1 Unsubstituted Hydroxyketones

#### 1-(2,4,6-Trihydroxyphenyl)-1,3,5,7-octanetetraone

USE: Precursor of 2,2',4,4',6-pentahydroxy-6'-methylbenzophenone in the biosynthesis of griseofulvin [1253].

**Trimethyl ether** [76631-05-5] C₁₇H₂₀O₇ mol. wt. 336.34

-Preparation by acylation of dianion of 2,4-pentanedione with methyl 2,4,6-trimethoxyphenyl-3-oxopropanoate (42 %) [2699].

-Also obtained by acylation of trianion of 2,4,6-heptanetrione with methyl 2,4,6-trimethoxy-benzoate (65 %) [2699].

yellow crystals [2699]; m.p. 176–179° [2699]; ¹H NMR [2699], IR [2699], MS [2699].

**Tribenzyl ether** [72327-93-6] C₃₅H₃₂O₇ mol. wt. 564.63

-Preparation by acylation of trianion of 2,4,6-heptanetrione with methyl 2,4,6-tribenzoxybenzoate (65 %) [2699].

orange-red oil [2699]; ¹H NMR [2699], IR [2699], MS [2699].

#### 1-(2-Hydroxyphenyl)-1-octanone-2,2-d2

[956239-90-0] C₁₄H₁₈D₂O₂ mol. wt. 222.58 OH **Synthesis** 

 $COCD_2(CH_2)_5CH_3$  -Refer to: [173].

#### 1-(2-Hydroxyphenyl)-1-octanone

[3226-27-5]

OH

 $CO(CH_2)_6CH_3$  -Obtained by Fries rearrangement of phenyl caprylate in the presence of, *aluminium chloride in nitrobenzene at 50° for 18 h (30 %)

[775, 776] or in tetrachloroethane [3169] at 120° [2520] or at 80° for 12–15 h (37.7 %) [1585];

*titanium tetrachloride in tetrachloroethane at 70° for 6 h (22 %) [2552];

*ferric chloride in tetrachloroethane at 70° for 6 h (19 %) [2552];

MS [173].

Syntheses

*without solvent at 130–150° [2520] or at 140° for 45 min (41 %) [932].

-Also obtained by photo-Fries rearrangement of phenyl caprylate in cyclohexane, heptane, benzene or n-amyl alcohol in a sealed tube with an unfiltered Hanovia 450 w medium pressure lamp [2067].

-Also obtained by Friedel-Crafts reaction of caprylic chloride with phenol in the presence of aluminium chloride at 125–130° for 1 h (45 %) [2700],

*in nitrobenzene at 50° for 18 h (25 %) [775];

*in tetrachloroethane at 55° for 4 h (50 %) [2548], at 100° for 6 h (54 %) [2550], at 70° for 3 h (21 %) [2549], at 403–413 K for 6 h (36 %) [3348];

*in carbon disulfide at reflux  $(46^\circ)$  (33%) [2550];

*in skellysolve B at 65° (44 %) [2550].

-Also obtained by zeolite-catalyzed Friedel-Crafts reaction of anisole with octanoic acid [3266].

-Also refer to: [2658].

b.p.₁ 97–99° [2548], b.p._{0.1} 104° [932], b.p._{0.01} 110.5–114° [1585], b.p._{0.02} 112° [2520],

b.p.₁ 115–120° [2549, 2550], b.p._{0.4} 123.5–124.5° [3169], b.p.₁₁ 169–170° [2700]; m.p. 22.3° [2700], 22° [2520];

¹H NMR [2067], IR [2067, 2520], UV [1996, 2067, 2520], MS [2067];

 $n_{D}^{25} = 1.5151 [1585], n_{D}^{25.5} = 1.5169 [2700];$ 

X-ray data [3348]; TLC [1994]; GLC [2067]; GC [2067].

USE: In a process for the preparation of 2-hydroxy-3-(C6-10 n-alkyl) benzaldoximes for use in the solvent extraction and recovery of copper from mineral and other wastes [3162].

## **2,4-Dinitrophenylhydrazone** [17744-54-6] $C_{20}H_{24}N_4O_5$ mol. wt. 400.43

m.p. 145° [3169], 144.5–145° [2549], 140–141° [2548], 140° [932].

**Oxime** [57835-34-4] C₁₄H₂₁NO₂ mol. wt. 235.33

-Refer to: [1142, 1585, 2077, 2520].

m.p. 80° [2520], 76.5–77.5° [1585]; IR [2520], UV [2520].

USE: Blocking agent, binders containing blocked PAPI and phenolic resins for wood composites [2077]; Solvent extraction of copper (II) [2520]; For extn. of metals [1142].

Methyl ether	$C_{15}H_{22}O_2$	mol. wt. 234.34
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-Obtained by oxidation of 1-(2-methoxyphenyl)-1-octanol with CrO₃ (0.05 equiv.) and t-BuOOH (3 equiv.) in methylene chloride at r.t. for 23 h (79 %) [2204].

oil [2204]; ¹H NMR [2204].

#### 1-(3-Hydroxyphenyl)-1-octanone

[778630-63-0]	$C_{14}H_{20}O_2$	mol. wt. 220.31
ОН	Syntheses	 

-Obtained by treatment of its methyl ether with aluminium chloride in benzene by heating on a water bath for 2 h (80 %) [966].

mol. wt. 400.43

 $C_{20}H_{24}N_4O_5$ 

-Also refer to: [967].

m.p. 58° [966, 967].

CO(CH₂)₆CH₃

#### 2,4-Dinitrophenylhydrazone

m.p. 113° [967].

Methyl ether	[136116-43-3]	$C_{15}H_{22}O_2$	mol. wt. 234.34
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-Obtained by condensation of heptylmagnesium bromide with m-methoxybenzamide in ethyl ether. The reaction was carried out at  $60^{\circ}$  for 48 h under hydrogen atmosphere (60-70 %) [966].

-Also obtained by oxidation of 1-(3-methoxyphenyl)-1-octanol with  $CrO_3$  (0.05 equiv.) and t-BuOOH (4 equiv.) in methylene chloride at r.t. for 23 h (95 %) [2204].

-Preparation by silyl chromate-catalyzed oxidation of allyl ether (96 %) [2203]. -Also refer to: [967].

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oil [2204]; b.p.<sub>15</sub> 160° [966, 967]; <sup>1</sup>H NMR [2204]; n_D^{31.5} = 1.4700 [967].
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mol. wt. 414.46

m.p. 90° [967].

#### 1-(4-Hydroxyphenyl)-1-octanone

[2589-73-3]

OH

 $C_{14}H_{20}O_2$ 

mol. wt. 220.31

Syntheses

CO(CH₂)₆CH₃

-Preparation by Fries rearrangement of phenyl caprylate, *with aluminium chloride in nitrobenzene [1585], (60 %) [2947], at 38° for 2 days (59 %) [414] at 50° for 18 h (55 %) [775, 776] or in tetrachloroethane [3169] at 80° for 12–15 h (42–44.6 %) [1585] or without solvent at 140° for 45 min (34 %) [932];

*in the presence of aluminium chloride in refluxing carbon disulfide for 5 h. Then, the mixture was kept at  $80-90^{\circ}$  for 2 h and at  $140-150^{\circ}$  for 1 h after solvent elimination [1222];

*with ferric chloride in tetrachloroethane at  $70^{\circ}$  for 6 h (52 %) [2552];

*with titanium tetrachloride in tetrachloroethane at 70° for 6 h (19 %) [2552];

*with boron trifluoride for 3 h at 75–80° (58.2 %) [1938].

-Also obtained by photo-Fries rearrangement of phenyl caprylate in cyclohexane, heptane, benzene or n-amyl alcohol in a sealed tube with an unfiltered Hanovia 450 w medium pressure lamp [2067].

-Also obtained by reaction of caprylyl chloride with phenol in the presence of aluminium chloride,

*without solvent at 125–130° for 1 h (38 %) [2700];

*in nitrobenzene at 50° for 18 h (67 %) [775] or at r.t. overnight (57 %) [1769];

*in methylene chloride for 1 h at  $0^{\circ}$ , then at r.t. overnight (100 %) [114] or for 14 h at r.t. (35 %) [1910];

*in tetrachloroethane at  $70^{\circ}$  for 3 h (49 %) [2549] at 55° for 4 h (12 %) [2548].

-Also obtained by reaction of caprylic acid with phenol in the presence of activated acid clay catalyst at  $190^{\circ}$  for 2 h [3277].

-Also refer to: [61, 62, 113, 1087, 1116, 1395, 1536, 1892, 1977, 2658, 2692, 2704, 3371].

b.p.₄ 178–181° [1769], b.p.₁ 183° [932, 1536], b.p._{2.1} 210° [1938], b.p.₁₀ 218–221° [2947], b.p.₁₀ 224–225° [2700];

white solid [1910];

m.p. 72.9–73.2° [1910], 64.5° [2704], 63° [775, 776], 62.5–63.5° [2548], 62–63.5° [3277],

62–63° [2549], 62–62.5° [2550], 62° [2700], 61–62° [1769, 3169], 59.5–61° [1585], 59° [932, 1536], [1938] 58–60° [414];

C₂₁H₂₆N₄O₅

¹H NMR [114, 1910, 1977, 2067], ¹³C NMR [114, 1910, 1977], IR [1910, 2067], UV [1995, 2067], MS [1910, 2067];
TLC [1910, 1994]; GLC [2067]; GC [2067]; pKa [2590]; dipole moment [2590].

USE: In a process for the preparation of 2-hydroxy-3-(C6-10 n-alkyl) benzaldoximes for use in the solvent extraction and recovery of copper from mineral and other wastes [3162]; Activator for peroxygen bleach in laundry detergent for mud-soiled clothing [3371]; As colour developer [2704].

BIOLOGICAL ACTIVITY: Inhibition of  $17-\beta$  hydroxysteroid dehydrogenase 3 [1910]; Estrogenic [2692].

Oxime [84498-21-5]  $C_{14}H_{21}NO_2$  mol. wt. 235.33

-Refer to: [1142].

USE: For extn. of metals [1142].

- **2,4-Dinitrophenylhydrazone** [17765-30-9] C₂₀H₂₄N₄O₅ mol. wt. 400.43 m.p. 178° [3169], 176–178° [2548], 171° [932].
- **Nicotinylhydrazone** [102240-72-2]  $C_{20}H_{25}N_3O_2$  mol. wt. 339.44

m.p. 148° [521, 520].

USE: In chemotherapy of leprosy [520, 521].

isoNicotinylhydrazone [102240-70-0]  $C_{20}H_{25}N_3O_2$  mol. wt. 339.44 m.p. 206° [520, 521].

USE: In chemotherapy of leprosy [520, 521].

Phosphate (57 %) [114].

¹H NMR [114], ¹³C NMR [114], ³¹P NMR [114].

Sulfate [114]; ¹H NMR [114].

Methyl ether	[62170-25-6]	$C_{15}H_{22}O_2$	mol. wt. 234.34
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-Surfactant-type iron complex-catalyzed mild oxidation of 4-octylanisole using aqueous TBHP as oxidant in water at  $30^{\circ}$  for 50 h (84 %) [2217].

-Also obtained by direct acylation of 4-bromoanisole with caprylic aldehyde by palladium catalysis (87 %) [2668].

-Also obtained by oxidation of 4-n-octylanisole at  $30^{\circ}$  in the presence of tert-butyl hydroperoxide and Fe₂O(O₃SOC₁₂H₂₅)₄ [1714].

-Nickel-catalyzed cross-coupling of 4-iodo-1-(4-methoxyphenyl)-1-butanone with n-butylmagnesium chloride in DMA at  $-35^{\circ}$  for 30 min (68 %) [3212].

-Also obtained by reaction of octanoic acid with anisole,

- *over three large pore zeolites-beta (BEA) [282], faujasite (FAU) and mordenite (MOR) [3246];
- *in the presence of  $Cs_{2.5}H_{0.5}PW_{12}O_{40}$  at 110° for 5 h (33 %) [1636];
- *on the solid surface of alumina in the presence of trifluoroacetic anhydride for 120 min at r.t. (94 %) [2563];
- *over HZSM-5 catalyst for 48 h at 423° K (4 %) [3265];

*in the presence of an ultrastable Y zeolite (USY) catalyst [1941];

- *in the presence of HY zeolite as catalyst [3264];
- *in the presence of zeolite H-beta [1185].

-Also obtained by acylation of anisole with octanoyl chloride in the presence of strongly acidic mesoporous aluminosilicates prepared from zeolite seeds [2865].

-Also obtained by Friedel-Crafts acylation of anisole in the presence of zeolites [281].

-Also obtained by reaction of octanoic anhydride with anisole in the presence of Yb  $(NTf_2)_3$  in nitromethane for 2 h at 25° (99 %) [2272].

- -Also obtained by oxidation of 1-(4-methoxyphenyl)-1-octanol with  $CrO_3$  (0.05 equiv.) and t-BuOOH (4 equiv.) in methylene chloride at r.t. for 17 h (94 %) [2204].
- -Also obtained by zeolite-catalyzed Friedel-Crafts reaction of anisole with octanoic acid [3266].

-Also refer to: [279, 280, 698 (61 %), 929, 1009, 1120 (72 %), 1897, 2016].

white solid [1120];

m.p. 52.5–53° [2016], 48–49° [2204], 45.3–46.7° [1897], 44° [1120]; ¹H NMR [698, 1120, 1864, 1897, 2204, 2217, 2272, 2563, 2668, 3212]; ¹³C NMR [698, 1120, 1864, 1897, 2204, 2217, 2668, 3212], IR [698, 929, 1864, 2217, 2563, 2668], MS [698, 1864, 1897, 2668].

#### 2,4-Dinitrophenylhydrazone of the methyl ether C21H26N4O5 mol. wt. 414.46

m.p. 140–140.5° [2016].

#### Benzoate

#### $C_{21}H_{24}O_3$

mol. wt. 324.42

m.p. 107-108° [2700].

Caprylate	[102946-00-9]	$C_{22}H_{34}O_{3}$	mol. wt. 346.51
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-Obtained by reaction of caprylic acid with phenol in the presence of activated acid clay catalyst at  $190^{\circ}$  for 2 h [3277].

-Also obtained (by-product) by Fries rearrangement of phenyl octanoate, *with titanium tetrachloride in tetrachloroethane for 6 h at 70° (10 %) [2552]; *with stannic chloride in tetrachloroethane for 7 h at 150° (5 %) [2552].

m.p. 56.5–57.5° [2551], 56–57° [3277].

#### **Phenyl ether** $C_{20}H_{24}O_2$ mol. wt. 296.41

-Obtained by treatment of diphenyl oxide with octanoyl chloride under Friedel-Crafts conditions [516].

m.p. 35° [516].

**4-Octanoylphenyl ether** [38051-39-7] C₂₈H₃₈O₃ mol. wt. 422.61

-Obtained by reaction of octanoyl chloride with diphenyl ether in the presence of aluminium chloride in refluxing carbon disulfide for 30 min (63 %) [463].

m.p. 103° [463]

**Dioxime of the 4-octanoylphenyl ether**  $C_{28}H_{40}N_2O_3$  mol. wt. 452.64

-Preparation: A suspension of the diketone, 100 % excess of hydroxylamine hydrochloride, and powdered anhydrous sodium carbonate in butanol [463].

m.p. 102° [721], 76° [463]

**Dihydrazone of the 4-octanoylphenyl ether** C₂₈H₄₂N₄O mol. wt. 450.67

b.p._{0.02} 220–230° [721]; m.p. 76° [721].

**N-Diethylaminoethyl ether** [14392-78-0] C₂₀H₃₃NO₂ mol. wt. 319.49

-A solution of 4-hydroxyoctanophenone in ethanol was added to a solution of sodium in ethanol; the mixture was refluxed 1 h, cooled, and a solution of  $ClCH_2CH_2N(C_2H_5)_2$  added (80 %) [414, 415].

b.p._{0.005} 156–166° [414, 415];  $n_D^{21} = 1.51$  [414, 415].

#### Hydrochloride of the N-diethylaminoethyl ether

[102462-84-0]	$C_{20}H_{33}NO_2$ , HCl	mol. wt. 355.95
m.p. 95° [2227].		

#### Fumarate of the N-diethylaminoethyl ether

[14392-93-9]  $C_{20}H_{33}NO_2, C_4H_4O_4$  mol. wt. 435.56 m.p. 76–78° [415].

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

C14H20O3 mol. wt. 236.31 [862666-36-2] OH Syntheses  $CO(CH_2)_6CH_3$  -Obtained by deprotection of its dimethyl ether with HO.

boron tribromide in methylene chloride at  $0^{\circ}$ . Then, the mixture was stirred overnight at r.t. (71 %) [82].

-Also obtained (by-product) by Fries rearrangement of pyrocatechol dicaprylate with aluminium chloride in the presence of pyrocatechol for 4.5 h at 135-140° [2075].

brown solid [82]; b.p.₄ 210–220° [2075]; m.p. 87–88° [2075], 47° [82]: ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82],

USE: Reactant for preparation of lithium gallium/titanium ketone complexes [82].

**Dimethyl ether** [1854-64-4] C₁₆H₂₄O₃ mol. wt. 264.36

-Obtained by treatment of 1-(2,3-dimethoxyphenyl)-1-octanol in acetone with potassium dichromate in dilute sulfuric acid (Jones reagent) at r.t. for 3 h (49 %) [82].

-Also obtained by treatment of 2,3-dimethoxyphenylheptylcarbinol with potassium dichromate in dilute sulfuric acid at  $30^{\circ}$  (63 %) [3148].

-Also obtained from 2,3-dimethoxybenzoyl chloride by dialkylcadmium synthesis (41 %) [822].

-Also refer to: [2601].

colourless oil [82]; b.p._{0.1} 114–115° [822], b.p._{0.2} 128–129° [2601], b.p.₁₅ 198–201° [3148]; ¹H NMR [82, 822], ¹³C NMR [82], IR [82, 822], MS [82].

#### 2,4-Dinitrophenylhydrazone of the dimethyl ether

[1854-63-3]	$C_{22}H_{28}N_4O_6$	mol. wt. 444.49
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m.p. 121° [3148].

[37622-68-7]

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#### 1-(2,4-Dihydroxyphenyl)-1-octanone

$C_{14}H_{20}O_3$	mol. wt. 236.31
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OH  $CO(CH_2)_6CH_3$ HO

$$_{14}H_{20}O_3$$
 mol. wt.

Syntheses

-Obtained by refluxing a mixture of caprylic acid, resorcinol and fused zinc chloride [2702] at 125–130° for 2 h (75 %) [3124].

-Also obtained by reaction of caprylic nitrile with resorcinol (Hoesch reaction) [1608].

-Also obtained by reaction of octanoyl chloride with resorcinol in the presence of aluminium chloride in 1,2-dichloroethane for 5 h at 65° [284]. -Also refer to: [651, 893, 1515, 2112, 2114, 2673, 2704, 2842, 3168, 3345].

b.p.₄ 191–201° [3124], b.p._{6–7} 214–216° [893, 2842]; m.p. 66° [2704], 62.5–64° [893, 2842], 62° [2673], 60–61° [3124], 58–59° [1608], 55–56° [1515]; ¹H NMR [1515], ¹³C NMR [1515], MS [1515].

USE: As colour developer [2704]; Wine preservation by, [3168].

BIOLOGICAL ACTIVITY: Antiseptic and germicidal product [2734]; Antifungal [651, 2112, 2114].

Hemihydrate	C ₁₄ H ₂₀ O ₃ , 0.5 H ₂ O	mol. wt. 245.32
menningurace	$c_{14112003}, c_{151120}$	mon. wt. 215.52

m.p. 56–58° [1608].

**2,4-Dinitrophenylhydrazone** [94759-02-1] C₂₀H₂₄N₄O₆ mol. wt. 416.43

m.p. 202° [2673].

Dimethyl ether	[6565-75-9]	$C_{16}H_{24}O_{3}$	mol. wt. 264.36
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-Obtained by reaction of octanoic acid with resorcinol dimethyl ether in the presence of polyphosphoric acid and heated on a water bath for 30 min (72 %) [2424].

-Also obtained by reaction of octanoyl chloride with resorcinol dimethyl ether in the presence of HY-zeolite. Before reaction, the zeolites were activated at 500° for 5 h in nitrogen flow [352].

-Also obtained by reaction of 2-chloro-1-(trimethylsilyl)-1-octanone with resorcinol dimethyl ether in the presence of titanium tetrachloride (84 %) [1354].

b.p._{0.5} 140° [1354], b.p.₂ 148–150° [2424]; ¹H NMR [1354], ¹³C NMR [1354], IR [1354].

#### Azine

 $C_{28}H_{40}N_2O_4$ 

mol. wt. 468.64

-Refer to: [1793].

USE: Polyolefins stabilization [1793].

mol. wt. 416.43

#### 1-(2,5-Dihydroxyphenyl)-1-octanone

 $C_{14}H_{20}O_3$ mol. wt. 236.31 [4693-19-0]

OH

**Syntheses** 

CO(CH₂)₆CH₃ -Obtained by reaction of caprylic acid with hydroquinone in the presence of a catalytic amount of trifluoromethanesulfonic acid [2985].

> -Also obtained by reaction of caprylic acid with hydroquinone in the presence of boron trifluoride in

1,2-dichloroethane at  $50-55^{\circ}$ . The reaction mixture was allowed to stand overnight, then heated for 5–6 h on a steam bath (69 %) [142].

- -Also obtained by treatment of 2-hydroxy-5-methoxyoctanophenone with aluminium bromide in refluxing benzene for 6 h (86 %) [770].
- -Also obtained by treatment of 2-hydroxy-5-pentyloxyoctanophenone with aluminium chloride in carbon disulfide for 3 days at r.t. (77 %) [770].

-Also obtained by hydrolysis of 1-(2-hydroxy-5-octanoyloxyphenyl)-1-octanone with 10 % HCl in ethanol (quantitative yield) [2370].

-Preparation by reaction of octanoic acid with hydroquinone in the presence of boron trifluoride for 2 h at 125° [2063].

-Also refer to: [285], [3168, 3043, 3394] (Japanese patents).

vellow crystals [142]; yellow plates [2370]; m.p. 86–87° [2063, 2370], 86° [770], 84.5–86° [142]; IR [2370].

USE: Wine preservation by, [3168].

<b>Oxime</b> [140943-07-3] C	₁₄ H ₂₁ NO ₃ mol. wt. 251.33
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-Refer to: [285].

2,4-Dinitrophenylhydrazone	$C_{20}H_{24}N_4O_6$
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m.p. 186° [770].

Dimethyl ether	[132859-07-5]	$C_{16}H_{24}O_{3}$	mol. wt. 264.36
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-Obtained by reaction of octanoic acid chloride with hydroquinone dimethyl ether, *in the presence of aluminium chloride [2859] in carbon disulfide first at 0°, kept overnight, heated to the boil [2874];

*in the presence of HY-zeolite. Before reaction, the zeolites were activated at  $500^{\circ}$ for 5 h in nitrogen flow [352].

m.p. 42–45° [2874]; ¹H NMR [2859], MS [2859].

#### 1-(2,6-Dihydroxyphenyl)-1-octanone

 $[13936-91-9] C_{14}H_{20}O_3 mtext{mol. wt. } 236.31$ 

OH CO(CH₂)₆CH₃ OH Syntheses -Preparation by treating resorcinol-4,6-dicarboxylic acid dimethyl ester with caprylyl chloride in the presence of aluminium chloride, followed by hydrolysis and decarboxylation of the compound obtained (30 %) [862].

-Also refer to: [2672].

m.p. 77.5–78° [2672], 58–59° [862]. **N.B.**: One of the reported melting point is obviously wrong.

**Dimethyl ether**  $C_{16}H_{24}O_3$  mol. wt. 264.36

C14H20O3

b.p._{1.5} 163–165° [2672].

#### 1-(3,4-Dihydroxyphenyl)-1-octanone

[37622-78-9]

OH

OH

Syntheses

-Obtained by Fries rearrangement of pyrocatechol dicaprylate with aluminium chloride in the presence of pyrocatechol at  $135-140^{\circ}$  for 4.5 h (50 %) [2075] or for 5 h (20 %) [283].

 $CO(CH_2)_6CH_3$  -Also obtained by reaction of octanoyl chloride with pyrocatechol in the presence of aluminium chloride in nitrobenzene, first at 5–7° for 2 h, then at r.t. for 2–3 days (54 %) [1726].

-Also refer to: [3056, 3110].

b.p.₂ 210–215° [283], b.p.₄ 210–220° [3056], b.p.₅ 225° [2075]; m.p. 95.5–96.5° [283, 2075]; ¹H NMR [283, 1726].

**Diethyl ether** [810661-49-5] C₁₈H₂₈O₃ mol. wt. 292.42

-Preparation by direct acylation of pyrocatechol diethyl ether with octanoic acid, in the presence of 6 mol% of AlPW₁₂O₄₀ (aluminium dodecatungstophosphate) as catalyst in the presence of trifluoroacetic anhydride (TFAA) for 1.5 h (94 %) [1009].

light yellow solid [1009]; m.p. 43° [1009]; ¹H NMR [1009], ¹³C NMR [1009], IR [1009], MS [1009]; TLC [1009]; GC [1009].

mol. wt. 236.31

#### **Dimethyl ether** [93157-10-9] C₁₆H₂₄O₃ mol. wt. 264.36

-Obtained by reaction of octanoyl chloride with veratrole in the presence of zinc chloride in refluxing carbon disulfide,

*for 6 h and then left overnight (49 %) [2420];

*for 4 h and then left overnight (59 %) [2836].

-Also obtained by reaction of octanoyl chloride with veratrole in the presence of HY-zeolite. Before reaction, the zeolites were activated at  $500^{\circ}$  for 5 h in nitrogen flow [352].

-Also refer to: [3364].

silky white flakes [2836]; b.p.₄ 165–168° [2420], b.p.₆ 180–183° [2836]; m.p. 50° [2836]; IR [3364].

#### 2,4-Dinitrophenylhydrazone of the dimethyl ether

$C_{22}H_{28}N_4O_6$		mol. wt. 444.49
–131° [2420].		
[107778-10-9]	$C_{17}H_{27}N_3O_3$	mol. wt. 321.42
m.p. 133° [2836].		
$C_{28}H_{32}O_3$		mol. wt. 416.56
	-131° [2420]. [107778-10-9] m.p. 133° [2836].	$-131^{\circ} [2420].$ $[107778-10-9] \qquad C_{17}H_{27}N_{3}O_{3}$ m.p. 133° [2836]. $C_{28}H_{32}O_{3}$

-Refer to: [2657]; m.p. 53° [2657].

#### 1-(3,5-Dihydroxyphenyl)-1-octanone

[1257228-28-6]	$C_{14}H_{20}O_3$	mol. wt. 236.31
ОН	Syntheses -Refer to: [25, 1893].	
HO CO(CH ₂ ) ₆ CH ₃	BIOLOGICAL ACTIVITY: affinity [1893].	Receptor binding
Dimethyl ether	$C_{16}H_{24}O_3$	mol. wt. 264.36

-Preparation in the usual way: [25 (60 %), 2990]. -Also refer to: [29].

b.p._{0.5} 155° [25], b.p.₂ 180° [29]; m.p. 36–37° [29].

#### 1-(2,3,4-Trihydroxyphenyl)-1-octanone

4-Octanoylpyrogallol

[43043-28-3]  $C_{14}H_{20}O_4$  mol. wt. 252.31 OH Syntheses HO CO(CH₂)₆CH₃ -Obtained by reaction of octanoic acid with pyrogallol in the presence of zinc chloride (Nencki reaction) at 135–140° for 2 h (40 %) [1283].

-Also obtained by reaction of octanoyl chloride with pyrogallol in the presence of aluminium chloride in nitrobenzene, first at  $5-7^{\circ}$  for 2 h, then at r.t. for 2–3 days (57 %) [1726].

-Also refer to: [1660, 2511, 3168].

m.p. 73–74° [1283]; ¹H NMR [1726].

USE: Wine preservation by, [3168].

#### 1-(2,4,5-Trihydroxyphenyl)-1-octanone

[107821-60-3]	$C_{14}H_{20}O_4$	mol. wt. 252.31
HO OH OH OH	Syntheses -Preparation by Friedel-Cra rearrangement of 1,2,4-tra aluminium chloride in nitra -Also obtained by reaction 2,4-trihydroxybenzene in ta chloride in nitrobenzene,	ioctanoyloxybenzene with obenzene [291]. of octanoyl chloride with

*at r.t. several hrs, and heated 0.5 h at 65° [290]; *first at 25°, then at 60° for 45 min [292]. -Also refer to: [1708].

m.p. 113–114° [290–292].

USE: Antioxidant [1708]; Antioxidant for fats and oils [290, 291]; Antioxidant for fats, oils and paraffin waxes [292].

BIOLOGICAL ACTIVITY: Toxicity [1708].

#### 1-(2,4,6-Trihydroxyphenyl)-1-octanone

[43043-32-9]	$C_{14}H_{20}O_4$	mol. wt. 252.31
HO OH CO(CH ₂ ) ₆ CH ₃	phloroglucinol (Hoesch	on of octanoyl chloride with

*of boron trifluoride etherate, first at  $0^{\circ}$ , then at r.t. for 48 h under nitrogen [2786]; *of aluminium chloride in nitrobenzene first between 5 and  $10^{\circ}$ , then at r.t. overnight (40 %) [2276].

-Also obtained by reaction of octanoyl chloride with phloroglucinol in the presence of aluminium chloride in nitrobenzene and carbon disulfide mixture (60 %) [2113], (45 %) [2620].

-Also refer to: [1026, 1065, 1441, 2111, 3168].

m.p. 128–129° [2276], 128° [2620], 125° [1441, 2113], 124° [1608]. IR [2276], MS [2276].

USE: Wine preservation by, [3168].

BIOLOGICAL ACTIVITY: Antifungal [2111, 2113].

Monohydrate	$C_{14}H_{20}O_4$	H ₂ O	mol. wt. 270.39
m.p. 106° [1608], 1	05–106° [1441].		
Trimethyl ether	[441353-19-1]	$C_{17}H_{26}O_4$	mol. wt. 294.39
-Refer to: [1065].			

#### 2-Ethyl-1-(4-hydroxyphenyl)-1-octanone (+)

 $\begin{array}{cccc} [120837\text{-}04\text{-}9] \ (+) & C_{16}H_{24}O_2 & \text{mol. wt. } 248.37 \\ \\ OH & Syntheses \\ & -\text{Refer to: } [1318, 1321]. \end{array}$ 

### 1.2 Substituted Hydroxyketones

 $CO - CH - (CH_2)_5 CH_3$  $L_2H_5$ 

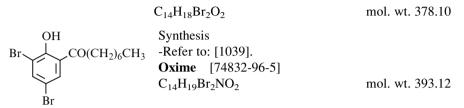
#### 1-(3-Bromo-5-fluoro-2-hydroxyphenyl)-1-octanone

[2262-19-3]	$C_{14}H_{18}BrFO_2$	mol. wt. 317.20
$\begin{array}{c} OH\\ Br & \downarrow \\ & \downarrow \\ & \downarrow \\ & F \end{array} CO(CH_2)_6CH_3 \end{array}$		/

 $\textbf{2,4-Dinitrophenylhydrazone} \hspace{0.2cm} [2414-79-1] \hspace{0.2cm} C_{20}H_{22}BrFN_4O_5 \hspace{0.2cm} mol. \hspace{0.2cm} wt. \hspace{0.2cm} 497.32 \hspace{0.2cm}$ 

m.p. 150° [1550].

#### 1-(3,5-Dibromo-2-hydroxyphenyl)-1-octanone



USE: Extn. properties with respect to Cu and Mo were detd [1039].

#### 1-(3,5-Dibromo-4-hydroxyphenyl)-1-octanone

[20683-51-6]	$C_{14}H_{18}Br_2O_2$	mol. wt. 378.10
Br HO Br	Synthesis -Obtained by bromination in acet BIOLOGICAL ACTIVITY: In activity [2339].	

#### 1-(3,5-Dibromo-2,4-dihydroxyphenyl)-1-octanone

[76092-85-8]	$\mathrm{C}_{14}\mathrm{H}_{18}\mathrm{Br}_{2}\mathrm{O}_{3}$	mol. wt. 394.10
OH Br CO(CH ₂ ) ₆ CH ₃	Synthesis -Refer to: [2112].	
HOBr	BIOLOGICAL ACTIVITY: contg., [2112].	Antifungal compns.

#### 1-(3-Chloro-5-fluoro-2-hydroxyphenyl)-1-octanone

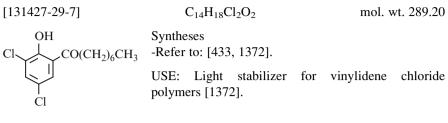
[2262-21-7]	$C_{14}H_{18}ClFO_2$	mol. wt. 272.75
Cl F CO(CH ₂ ) ₆ CH ₃	Synthesis -Obtained by Fries rearrangement of 2 phenyl caprylate with aluminium chl for 3 h (70 %) [1550]. b.p. _{2.5–3} 160° [1550].	

 $\textbf{2,4-Dinitrophenylhydrazone} \hspace{0.1in} [2194-77-6] \hspace{0.1in} C_{20}H_{22}ClFN_4O_5 \hspace{0.1in} \text{mol. wt. } 452.87$ 

m.p. 120° [1550].

785

#### 1-(3,5-Dichloro-2-hydroxyphenyl)-1-octanone



 $\label{eq:oxime} \textbf{Oxime} \qquad [74832-95-4] \qquad C_{14}H_{19}Cl_2NO_2 \qquad \text{mol. wt. } 304.22$ 

USE: Extn. properties with respect to Cu and Mo were detd. [1039].

#### 1-(3,5-Dichloro-4-hydroxyphenyl)-1-octanone

[131427-27-5]  $C_{14}H_{18}Cl_2O_2$  mol. wt. 289.20 OH Synthesis Cl + Cl - Cl - Refer to: [433]. $CO(CH_2)_6CH_3$ 

#### 1-(3,5-Dichloro-2,6-dihydroxyphenyl)-1-octanone

[910457-86-2]	$C_{14}H_{18}Cl_2O_3$	mol. wt. 305.20
Cl Cl Cl CO(CH ₂ ) ₆ CH ₃ OH Cl	Synthesis -Refer to: [2373] (Japanese patent).	

#### 1-(5-Bromo-2-hydroxyphenyl)-1-octanone

[105465-02-9]	$C_{14}H_{19}BrO_2$	mol. wt. 299.21
OH CO(CH ₂ ) ₆ CH ₃ Br	Syntheses -Obtained by Fries rearrangement of caprylate with aluminium chloride (77 -Also refer to: [3404]. b.p. _{0.65} 190–195° [1640]; IR [1640].	

#### 1-(4-Chloro-2-hydroxyphenyl)-1-octanone

	$C_{14}H_{19}ClO_2$	mol. wt. 254.76
OH CO(CH ₂ ) ₆ CH ₃		of 3-chlorophenyl
	caprylate with aluminium chloride, *without solvent at 130° for 2 h (64 G	%) [2802];
	*in nitrobenzene at $25^{\circ}$ for 6 h (75 %	) [2802].

b.p.18 188° [2802].

#### Methyl ether

C₁₅H₂₁ClO₂ mol. wt. 268.78

-Obtained by methylation of the above ketone in the usual way (90 %) [2802].

b.p.19 110° [2802].

#### **2,4-Dinitrophenylhydrazone** $C_{20}H_{23}ClN_4O_5$ mol. wt. 434.88

m.p. 175° [2802].

#### 1-(5-Chloro-2-hydroxyphenyl)-1-octanone

[52196-48-2] C₁₄H₁₉ClO₂ mol. wt. 254.76

-Also refer to: [1702, 1798].

b.p._{0.65} 152–154° [1640], b.p.₂ 145–147° [2680]; m.p. 64–65° [2680], 64° [3170], 63° [1702]; IR [1640], UV [3170].

BIOLOGICAL ACTIVITY: Nematocide [1798].

USE: Ionization of, in methylpyrrolidinone [1763].

#### 1-(2-Fluoro-4-hydroxyphenyl)-1-octanone

[136964-18-6]

 $C_{14}H_{19}FO_2$ 

mol. wt. 238.30

OH Syntheses -Refer to: [2363, 3041]. CO(CH₂)₆CH₃

#### 1-(3-Fluoro-4-hydroxyphenyl)-1-octanone

$$\begin{array}{cccc} C_{14}H_{19}FO_2 & \mbox{mol. wt. } 238.30 \\ OH & Synthesis \\ \hline F & -Refer to: [1549]. \\ \hline Methyl ether & [137866-03-6] \\ C_{15}H_{21}FO_2 & \mbox{mol. wt. } 252.33 \\ CO(CH_2)_6CH_3 & -Refer to: [3041]. \end{array}$$

#### 1-(5-Fluoro-2-hydroxyphenyl)-1-octanone

 $\begin{array}{cccc} [784-64-5] & C_{14}H_{19}FO_2 & \text{mol. wt. } 238.30 \\ OH & Synthesis \\ + & CO(CH_2)_6CH_3 & -Preparation by Fries rearrangement of 4-fluorophenyl caprylate [1641] with aluminium chloride at 130° for 2 h \\ (95 \%) [1549]. \\ F & \text{m.p. } 47^\circ [1549]. \end{array}$ 

**2,4-Dinitrophenylhydrazone** [2728-92-9] C₂₀H₂₃FN₄O₅ mol. wt. 418.42

m.p. 141° [1549].

 $NO_2$ 

#### 1-(4-Hydroxy-3-nitrophenyl)-1-octanone

[70079-26-4] C₁₄H₁₉NO₄ mol. wt. 265.31 OH Synthesis

-Obtained by treatment of 4-octanoylphenol with concentrated nitric acid in concentrated sulfuric acid at 0° for 1 h 15 min [1222].

## CO(CH₂)₆CH₃ m.p. 51–52° [1222].

#### 1-(3,5-Dichloro-2,4-dihydroxy-6-methoxyphenyl)-1-octanone

[118222-72-3]	$C_{15}H_{20}Cl_2O_4$	mol. wt. 335.23
$\begin{array}{c} OH \\ Cl \\ HO \\ Cl \\ Cl \\ Cl \\ OCH_3 \\ \end{array}$	Synthesis -Obtained by reaction of chlorine 6-methoxyoctanophenone in wat ¹ H NMR [2012], MS [2012].	

BIOLOGICAL ACTIVITY: Differentiation-inducing factor from the slime mould *Dictyostelium discoideum* [2012].

# **1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-1-octanone** (*DIF-1*) (+2)

[118191-34-7]  $C_{15}H_{20}Cl_2O_4$  mol. wt. 335.23 OH  $Cl + CO(CH_2)_6CH_3$   $CH_3O + Cl$   $CH_3O + Cl$   $Cl + CO(CH_2)_6CH_3$   $CH_3O + Cl$   $CH_3O + Cl$   $Cl + CO(CH_2)_6CH_3$   $CH_3O + Cl$   $CH_3O + Cl$   $Cl + CO(CH_2)_6CH_3$   $Cl + CO(CH_2)_6CH_3$  $Cl + CO(CH_2)_6CH$ 

-Also refer to: [1772, 1773, 2341].

yellow amorphous solid [1129]; ¹H NMR [2012], MS [1129, 2012].

BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129]; Differentiation-inducing factor from the slime mould *Dictyostelium discoideum* [2012].

#### 2-Hydroxy-5-octanoylbenzoic acid

 $\begin{array}{cccc} [78418-01-6] & C_{15}H_{20}O_4 & \text{mol. wt. } 264.32 \\ OH & Syntheses \\ -Obtained by saponification of the methyl ester (93 \%) [689]. \\ -Also refer to: [80, 174, 196, 320, 974, 1844, 1855, 2439, 2582]. \end{array}$ 

CO(CH₂)₆CH₃

```
m.p. 117° [921], 113–114° [689]; <sup>1</sup>H NMR [921], IR [921], MS [921].
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BIOLOGICAL ACTIVITY: Antibacterial [1855]; Deodorant [1855].

Methyl ester [78417-96-6]  $C_{16}H_{22}O_4$  mol. wt. 278.35

-Obtained by reaction of octanoyl chloride with methyl salicylate in the presence of aluminium chloride in carbon disulfide first at  $5-10^{\circ}$ , then at r.t. for 12 h (85 %) [689].

m.p. 57–58° [689].

#### 1-(2,6-Dihydroxy-4-methylphenyl)-1,7-octanedione

3,5-Dihydroxy-4-(7-oxo-octanoyl)toluene

[30414-67-6]	$C_{15}H_{20}O_4$	mol. wt. 264.32
CH ₃ CO(CH ₂ ) ₅ COCH ₃	Synthesis -Obtained by reaction of with 3,5-dihydroxytoluene polyphosphoric acid at 60 ^o [2184].	in the presence of

pale yellow needles [2184]; m.p. 139.5–140.5° [2184]; ¹H NMR [2184], IR [2184], UV [2184].

Dimethyl ether	[30414-68-7]	$C_{17}H_{24}O_{4}$	mol. wt. 292.37
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-Obtained by reaction of dimethyl sulfate with the title ketone in the presence of potassium carbonate in refluxing acetone for 3 h (98 %) [2184].

oil [2184]; b.p. 100° (bath)/ 10⁻³ Torr [2184]; ¹H NMR [2184], IR [2184], UV [2184].

#### 1-(3-Chloro-2-hydroxy-5-methylphenyl)-1-octanone

[74604-16-3]	$C_{15}H_{21}ClO_2$	mol. wt. 268.78
CI CI CH ₃ CH ₃ CH ₃	Synthesis -Obtained by Fries 4-methyl-phenyl octan pale yellow-brown pris m.p. 35° [2520]; IR [	

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Oxime[74604-08-3]C_{15}H_{22}CINO_2mol. wt. 283.80
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m.p. 101° [2520]; IR [2520], UV [2520].

USE: Solvent extraction of copper (II) [2520].

1-(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)-1-octanone (*DIF-3*) (+2)

Syntheses

$C_{15}H_{21}ClO_4$	mol. wt. 300.78
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Cl CO(CH₂)₆CH CH₃O OH

CO(CH₂)₆CH₃ -Preparation by adding a solution of sulfuryl chloride (1.5 equiv.) in ethanol to a solution of 2,6-dihydroxy-4-methoxyoctanophenone in chloroform at r.t.; then, the solution was stirred for 1 h at r.t. [1129].

-Also refer to: [1772].

[861889-90-9]

colourless amorphous solid [1129]; MS [1129].

BIOLOGICAL ACTIVITY: Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

#### 1-(2-Fluoro-4-hydroxyphenyl)-2-methyl-1-octanone

[127928-54-5]	$C_{15}H_{21}FO_2$	mol. wt. 252.33
$\bigcup_{\substack{F \\ CO - CH - C_6H_{13}}}^{OH}$	Synthesis -Refer to: [1322]. <b>Methyl ether</b> (+) [127928-64-7] C ₁₆ H ₂₃ FO ₂ -Refer to: [1322].	mol. wt. 266.36

#### 1-(3-Fluoro-4-hydroxyphenyl)-2-methyl-1-octanone

[127928-53-4] (+)	$C_{15}H_{21}FO_2$	mol. wt. 252.33
OH F	Synthesis -Refer to: [1322].	
$\begin{array}{c} \begin{array}{c} & CH_3 \\ & I \\ CO - CH - C_6 H_{13} \end{array}$	Methyl ether (+) [127928-56-7] $C_{16}H_{23}FO_2$ -Refer to: [1322].	mol. wt. 266.36

#### 1-(2-Hydroxy-5-methyl-3-nitrophenyl)-1-octanone

[74604-14-1]	$C_{15}H_{21}NO_4$	mol. wt. 279.33
NO ₂ CO(CH ₂ ) ₆ CH ₃	Synthesis -Obtained by tro 5-methylphenyl)-1-o nitric acid in acetic a m.p. 68° [2520]; IR	

**Oxime**[74604-07-2] $C_{15}H_{22}N_2O_4$ mol. wt. 294.35

m.p.  $105^{\circ}$  [2520]; IR [2520], UV [2520].

USE: Solvent extraction of copper (II) [2520].

#### 1-(2-Hydroxy-3-methylphenyl)-1-octanone

[108667-53-4]	$C_{15}H_{22}O_2$	mol. wt. 234.34
CH ₃ CO(CH ₂ ) ₆ CH ₃		rearrangement of o-cresyl ium chloride at $160-180^{\circ}$ for .

b.p.₁₃ 176–182° [1644].

#### 1-(2-Hydroxy-4-methylphenyl)-1-octanone

[108666-97-3]	C ₁₅	$H_{22}O_2$	mol. wt. 234.34
CH ₃ CO(C	caprylate	l by Fries rearrang with aluminium cl at 120–148° for 30 m	hloride at $140-150^{\circ}$
b.p. ₁ 129–131°	[906], b.p. ₉ 173–175	° [1644].	
Phenylhydrazone	[112744-63-5	] C ₂₁ H ₂₈ N ₂ O	mol. wt. 324.47
m.p. 64–66° [1	644].		
Oxime	[84498-20-4]	C ₁₅ H ₂₃ NO ₂	mol. wt. 249.35
	er from aq. soln. [114 n. of metals [1142].	43].	
Oxime (E)	[113962-76-8]	$C_{15}H_{23}NO_2$	mol. wt. 249.35
-Extn. by, of cupric cation [230].			
Oxime, nickel co	mplex	[108111-23-5]	
-Refer to: [2284].			

USE: Photog. magenta dye image stabilizer [2284].

#### 1-(2-Hydroxy-5-methylphenyl)-1-octanone

[36946-07-3]	$C_{15}H_{22}O_2$	mol. wt. 234.34
OH CO(CH ₂ ) ₆ CH ₃ CH ₃	Syntheses -Obtained by Fries rearrangement with aluminium chloride, *at 120° for 10 min (70 %) [1644]; *in tetrachloroethane at 120° [2520]; *without solvent at 130–150° [2520]; -Also obtained by reaction of oc p-cresol in the presence of aluminium chloride at 110–120° for 8 h (58 %)	tanoyl chloride with m chloride in ethylene

-Also refer to: [1763, 2647].

**N.B.**: Changes of volatiles in soy sauce-stewed pork during cold storage and reheating [1895].

m.p. 36° [1644, 2647], 35.5° [2520], 34–34.5° [1769]; IR [2520], UV [2520].

USE: Detn. of, by potentiometric titration in methylpyrrolidinone [1763].

**Oxime** [51528-14-4] C₁₅H₂₃NO₂ mol. wt. 249.35

-Refer to: [2520, 3445].

m.p. 88.5-89.5° [888], 86° [2520]; IR [2520], UV [2520].

USE: Extraction of copper, kinetics and mechanism of, [2521, 3198, 3199]; Solvent extraction of copper (II) [1769, 2520].

Oxime (*E*) [103582-39-4] C₁₅H₂₃NO₂ mol. wt. 249.35 85–86° [1769]; ¹H NMR [1769], ¹³C NMR [1769], IR [1769], UV [1769], MS [1921].

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2,4-Dinitrophenylhydrazone [127699-72-3] C_{21}H_{26}N_4O_5 mol. wt. 414.46
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m.p. 146–147° [1769].

#### 1-(4-Hydroxy-2-methylphenyl)-1-octanone

	$C_{15}H_{22}O_2$	mol. wt. 234.34
ОН	Synthesis -Refer to: [2503].	
CO(CH ₂ ) ₆ CH ₃	<b>Phenyl ether</b> [791615-79-7] C ₂₁ H ₂₆ O ₂	mol. wt. 310.44

-Obtained by adding a mixture of m-phenoxytoluene and octanoyl chloride to a suspension of aluminium chloride in methylene chloride at  $0^{\circ}$ , then the mixture stirred for 1.5–2 h at 3–5° (59 %) [2503].

b.p.₃ 228–229° [2503]; ¹H NMR [2503], IR [2503], MS [2503].

#### 1-(4-Hydroxy-3-methylphenyl)-1-octanone

[95102-41-3]	$C_{15}H_{22}O_2$	mol. wt. 234.34
CO(CH ₂ ) ₆ CH ₃	Syntheses -Obtained by Fries rearrangement of o-cress aluminium chloride at 160–180° for 30 min ( -Also refer to: [1595, 2704]. m.p. 91° [2704], 70–73° [1644].	

USE: As colour developer [2704]; Colour developer, for thermal recording materials [1595].

Methyl ether [810661-48-4] C₁₆H₂₄O₂ mol. wt. 248.37

-Preparation by direct acylation of 2-methylanisole with octanoic acid in the presence of 6 mol% of  $AIPW_{12}O_{40}$  (aluminium dodecatungstophosphate) as catalyst,

*at 120° for 4 h (85 %) [1009];

*in the presence of trifluoroacetic anhydride (TFAA) for 0.34 h (93 %) [1009].

colourless viscous oil [1009]; b.p.₃₂ 218° [1009]; ¹H NMR [1009], ¹³C NMR [1009], IR [1009], MS [1009]; TLC [1009]; GC [1009].

#### 1-(4-Hydroxyphenyl)-2-methyl-1-octanone

USE: In preparation of polymeric ferroelectric liquid crystals [3025]; Liquid crystal siloxanes and liquid crystal compn. [1419].

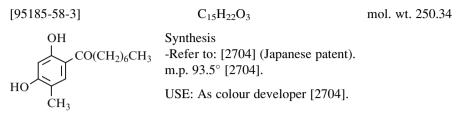
Methyl ether (+) [120837-13-0]  $C_{16}H_{24}O_2$  mol. wt. 248.37 -Refer to: [1320–1322, 1324].

#### 1-(2,4-Dihydroxy-3-methylphenyl)-1-octanone

[95149-08-9]	$C_{15}H_{22}O_3$	mol. wt. 250.34
CH ₃ HO	Syntheses -Refer to: [1515, 1595, 2704]. m.p. 81° [2704], 75.8–77° [1515]; ¹ H NMR [1515], ¹³ C NMR [1515],	MS [1515].

USE: As colour developer [2704]; Colour developer, for thermal recording materials [1595].

#### 1-(2,4-Dihydroxy-5-methylphenyl)-1-octanone



#### 1-(2,4-Dihydroxy-6-methylphenyl)-1-octanone

 $[30414-65-5] C_{15}H_{22}O_3 mol. wt. 250.34$ OH OH CO(CH₂)₆CH₃ OBtained by reaction of octanoyl chloride with orcinol in the presence of aluminium chloride in nitrobenzene at 0°, followed by hydrolysis next day (26.5 %) [2184].

This compound being heated at  $106^{\circ}$  (bath)/6 x  $10^{-5}$  Torr gave a waxy sublimate [2184].

C15H22O3

plates [2184]; m.p. 90–91° [2184]; ¹H NMR [2184], IR [2184], UV [2184].

#### 1-(2,5-Dihydroxy-4-methylphenyl)-1-octanone

[21182-61-6] OH CH₃ CO(CH₂)₆CH₃

Synthesis -Obtained by treatment of 2-hydroxy-5-methoxy-4-methyl-octanophenone with aluminium chloride

in boiling carbon disulfide for 34 h (63 %) [2370].

yellow plates [2370]; m.p. 103–104° [2370]; IR [2370].

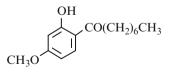
**Syntheses** 

#### 1-(2-Hydroxy-4-methoxyphenyl)-1-octanone

[143286-91-3]

C₁₅H₂₂O₃ mol. wt. 250.34

mol. wt. 250.34



-Obtained by reaction of methyl bromide with 1-(2,4-dihydroxyphenyl)-1-octanone in the presence of potassium carbonate in refluxing acetone for 20 h [284].

-Also refer to: [1787].

m.p. 30–34° [284].

mol. wt. 250.34

-Obtained by reaction of hydroxylamine hydrochloride with 1-(2-hydroxy-4-methoxyphenyl)-1-octanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

C₁₅H₂₂O₃

m.p. 49–53° [284].

#### 1-(2-Hydroxy-5-methoxyphenyl)-1-octanone

OH

OCH₃

Syntheses

CO(CH₂)₆CH₃ -Obtained by reaction of caprylic acid with p-methoxyphenol in the presence of boron trifluoride in tetrachloroethane for 4 h. The reaction mixture was allowed to stand overnight at r.t. and was then heated on a steam bath for about 3 h (55 %) [142].

-Also obtained by Fries rearrangement of 4-methoxyphenyl caprylate (b.p.₁  $137-140^{\circ}$ ) with boron trifluoride in *sym*-tetrachloroethane for 6 h, following 5 h of heating on a steam bath (33 %) [142].

-Also obtained by action of octanoyl chloride with quinol dimethyl ether in the presence of aluminium chloride in carbon disulfide for 2 h, then at r.t. for 1 h and refluxed 10 h on the steam bath (83 %) [770], (58 %) [2370]. -Also refer to: [1907, 2541].

yellow plates [2370]; b.p._{2–4} 188–189° [2541]; m.p. 45° [770, 2541], 44–45° [2370], 42.5–44° [142, 1907]; IR [2370].

**Oxime** [140943-14-2] C₁₅H₂₃NO₃ mol. wt. 265.35

-Refer to: [285].

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

2,4-Dinitrophenylhydrazone	$C_{21}H_{26}N_4O_6$	mol. wt. 430.46
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m.p. 134° [770].

#### 1-(2,4-Dihydroxyphenyl)-2-methyl-1-octanone

[134925-05-6]	$C_{15}H_{22}O_3$	mol. wt. 250.34
OH CH ₃ CO-CH-(CH ₂ ) ₅ CH ₃	Synthesis	
	<b>Isomer</b> (S) $C_{15}H_{22}O_3$	mol. wt. 250.34
HO		

-Obtained by reaction of (S)-(+)-2-methyloctanoic acid with resorcinol in the presence of zinc chloride at  $150^{\circ}$  for 30 min (46.2 %) [1715]. -Refer to: [1715, 1716, 1719–1721].

orange viscous liquid [1715];  $(\alpha)_D^{20} = 15.2^{\circ}$  (chloroform) [1715]; ¹H NMR [1715], IR [1715].

#### 1-(2,5-Dihydroxy-4-methoxyphenyl)-1-octanone

#### 1-(2,6-Dihydroxy-4-methoxyphenyl)-1-octanone

[861889-77-2]  $C_{15}H_{22}O_4$ 

Synthesis

CO(CH₂)₆CH₃ -Preparation by reaction of octanoyl chloride with phloroglucinol monomethyl ether in the presence of aluminium chloride in methylene chloride at r.t. [1129].

BIOLOGICAL ACTIVITY: DIF-1 derivs. for treating diabetes and obesity [1773]; Structural requirements of Dictyostelium differentiation-inducing factors for their stalk-cell-inducing activity in Dictyostelium cells and antiproliferative activity in K562 human leukemic cells [1129].

#### 1-[2,4,6-Trihydroxy-3-methylphenyl]-1-octanone

[74478-10-7]	$C_{15}H_{22}O_4$	mol. wt. 266.34
CO(CH ₂ ) ₆ CH ₃	Syntheses -Obtained by reaction of 3-methyl-phloroglucinol ( -Also refer to: [2111].	of caprylic nitrile with Hoesch reaction) [1608].

m.p. 135° [1608].

OH

CH₃O

BIOLOGICAL ACTIVITY: Fungicidal [2111].

797

mol. wt. 266.34

#### 1-(2,4-Dimethoxy-6-hydroxyphenyl)-1,3,5,7-octanetetraone

[76631-04-4]	$C_{16}H_{18}O_7$	mol. wt. 322.31
CH ₃ O	COCH ₂ COCH ₂ COCH ₂ COCH ₃ OCH ₃	Synthesis -Preparation by acylation of trianion of 2,4,6-heptanetrione with methyl 2,4-dimethoxy- 6-hydroxybenzoate (48 %) [2699].

orange-yellow crystals [2699]; m.p. 59–64° [2699]; ¹H NMR [2699], MS [2699].

#### [4,6-Dihydroxy-5-(1-oxooctyl)phenyl]-1,3-dicarboxylic acid

	$C_{16}H_{20}O_7$		mol. wt. 324.33
CO(CH ₂ ) ₆ CH ₃ HO HO ₂ C CO ₂ H	Synthesis -Refer to: [862]. <b>Dimethyl ester</b> C ₁₈ H ₂₄ O ₇	[13937-26-3]	mol. wt. 352.38
$HO_2C$ · $CO_2H$	018112407		mon. wt. 552.50

-Preparation by treating resorcinol-4,6-dicarboxylic acid dimethyl ester with caprylyl chloride in the presence of aluminium chloride (75 %) [862].

m.p. 63-65° [862].

#### 1-(4-Ethyl-2-hydroxyphenyl)-1-octanone

CO(CH₂)₆CH₃ -Obtained by Fries rearrangement of 3-ethylphenyl n-caprylate (1 equiv.),

> *in the presence of aluminium chloride (1.3 equiv.) in nitrobenzene at 25° for 6 h (84 %) [2801];

> *in the presence of aluminium chloride (2.8 M), first in refluxing carbon disulfide for 2 h, then for 2 h at 130° after solvent elimination (84 %) [2801].

b.p.28 200° [2801].

#### Methyl ether

C₁₇H₂₆O₂ mol. wt. 262.39

-Obtained by reaction of dimethyl sulfate with 1-(4-ethyl-2-hydroxyphenyl)-1-octanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating on the water bath for 5 h (79 %) [2801].

b.p.35 182° [2801].

2,4-Dinitrophenylhydrazone	$C_{22}H_{28}N_4O_5$	mol. wt. 428.49
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m.p. 136° [2801].

#### 1-(5-Ethyl-2-hydroxyphenyl)-1-octanone

	01012402	
$\bigcup_{C_2H_5}^{OH} CO(CH_2)_6CH_3$	Synthesis -Obtained by Fries rearrangement caprylate with aluminium chloride (80 %) [2800]. b.p. ₁₁ 184° [2800].	• • •

**2,4-Dinitrophenylhydrazone**  $C_{22}H_{28}N_4O_5$  mol. wt. 428.49

 $C_{16}H_{24}O_{2}$ 

m.p. 122° [2800].

#### 1-(2-Hydroxy-4,5-dimethylphenyl)-1-octanone

 2)₆CH₃ -Obtained by Fries rearrangement of 3,4-dimethylphenyl caprylate with aluminium chloride at 110° without solvent (75 %) [3117].
 b.p.₈ 236° [3117].

C22H28N4O5

2,4-Dinitrophenylhydrazone

m.p. 169° [3117].

CH₃

CH

#### 1-(2-Hydroxy-4,6-dimethylphenyl)-1-octanone

[101271-65-2]	$\mathrm{C_{16}H_{24}O_{2}}$		mol. wt. 248	.37	
CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃		plphenyl ence of	l caprylate aluminiun	rearrangement (1 equiv.), n chloride (1.3 equ n (82 %) [2801];	of iv.)

*in the presence of aluminium chloride (2.8 M), first in refluxing carbon disulfide for 2 h, then for 2 h at  $130^{\circ}$  after solvent elimination (80 %) [2801].

-Also obtained by reaction of caprylic acid with 3,5-xylenol in the presence of boron trifluoride at  $70^{\circ}$  for 2 h (91 %) [1685].

b.p.₃ 180° [2801], b.p.₁₅ 199–199.5° [1685]; m.p. 22° [1685].

mol. wt. 248.37

mol. wt. 428.49

2,4-Dinitrophenylhydrazone	$C_{22}H_{28}N_4O_5$	mol. wt. 428.49
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m.p. 180° [2801].

Methyl ether	$C_{17}H_{26}O_2$	mol. wt. 262.39
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-Obtained by reaction of dimethyl sulfate with 1-(2-hydroxy-4,6-dimethylphenyl)-1-octanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating on the water bath for 5 h (73 %) [2801].

b.p.40 200° [2801].

#### 1-(4-Hydroxy-2,3-dimethylphenyl)-1-octanone

 $\begin{array}{ccccccc} [95102-20-8] & C_{16}H_{24}O_2 & \text{mol. wt. } 248.37 \\ OH & Syntheses \\ & & -Refer to: [1595, 2704]. \\ & & & m.p. \; 80^{\circ} \; [2704]. \\ & & & CO(CH_2)_6CH_3 \end{array}$ 

USE: As colour developer [2704]; In preparation of thermographic recording material [1595].

#### 1-(4-Hydroxy-2,5-dimethylphenyl)-1-octanone

 $\begin{array}{cccc} [95185-67-4] & C_{16}H_{24}O_2 & \mbox{mol. wt. } 248.37 \\ OH & Synthesis \\ CH_3 & -Refer to: [2704] (Japanese patent). \\ m.p. 100^{\circ} [2704]. \\ CO(CH_2)_6CH_3 & \mbox{USE: As colour developer [2704].} \end{array}$ 

#### 1-(4-Hydroxyphenyl)-3,7-dimethyl-1-octanone

#### N-Diethylaminoethyl ether [14392-80-4] C₂₂H₃₇NO₂ mol. wt. 347.54

-A solution of 1-(4-hydroxyphenyl)-3,7-dimethyl-1-octanone in ethanol was added to a solution of sodium in ethanol; the mixture was refluxed 1 h, cooled, and a solution of ClCH₂CH₂N(C₂H₅)₂ added (80 %) [414, 415].

b.p._{0.005} 156–166° [414, 415];  $n_D^{21} = 1.5058$  [414, 415].

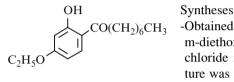
Fumarate of the N-diethylaminoethyl C₂₂H₃₇NO₂, C₄H₄O₄ mol. wt. 463.61 ether

m.p. 76–78° [414, 415].

#### 1-(4-Ethoxy-2-hydroxyphenyl)-1-octanone

[22198-47-6]

C16H24O3 mol. wt. 264.36



.CO(CH₂)₆CH₃ -Obtained by reaction of octanoyl chloride with m-diethoxybenzene in the presence of aluminium chloride in dichloroethane at 0°. Then, the mixture was stirred for 1 h at 10°, and then for 6 h at  $20-25^{\circ}$ .

The temperature was then raised to  $80^{\circ}$  and stirring was continued at that temperature for 5 h (64.5 %) [1194].

-Also refer to: [3469].

m.p. 54.5° [1194, 3469]; UV [1194, 3469].

USE: UV absorber [1194]; Light-stabilizer of polymeric materials [1194].

#### 1-(5-Ethoxy-2-hydroxyphenyl)-1-octanone

[140943-33-5]	$C_{16}H_{24}O_3$	mol. wt. 264.36
$\bigcup_{\substack{I \\ OC_2H_5}}^{OH} CO(CH_2)_6CH_3$	Synthesis -Refer to: [285]. <b>Oxime</b> [140943-20-0] C ₁₆ H ₂₅ NO ₃	mol. wt. 279.38

-Refer to: [285].

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

#### 1-(5-Ethyl-2,4-dihydroxyphenyl)-1-octanone

[95185-59-4] 
$$C_{16}H_{24}O_3$$
 mol. wt. 264.36  
OH Synthesis  
HO  $C_{2}H_5$  -Refer to: [2704].

#### 1-(2-Hydroxy-5-methoxy-4-methylphenyl)-1-octanone

[21182-60-5]	$C_{16}H_{24}O_3$	mol. wt. 264.36
OH CO(CH ₂ ) ₆ CH ₃		octanoyl chloride with the presence of aluminium
CH ₃ OCH ₃	•	on disulfide for 10 h on the

pale yellow needles [2370]; m.p. 57–59° [2370]; IR [2370].

#### 3,7-Dimethyl-1-(2,4,6-trihydroxyphenyl)-1-octanone

[198878-73-8]	$C_{16}H_{24}O_4$			mol. wt. 2	280.36
HO OH COCH ₂ OH	-CH-(CH ₂ ) ₃ CH(CH ₃ ) ₂ I CH ₃	Syntheses -Obtained 3,7-dimeth with phlor sence of nitrobenzer	yl-octar roglucii alumini	nol in the ium chlori	loride pre- de in

-Also refer to: [2683, 2685].

pale yellow oil [3166]; ¹H NMR [2683], MS [2683]; TLC [3166].

BIOLOGICAL ACTIVITY: As preventive and therapeutic agent for bone and cartilage diseases [2685].

#### 1-(2-Hydroxy-4,6-dimethoxyphenyl)-1-octanone

[1142936-18-2]	$C_{16}H_{24}O_4$	mol. wt. 280.36
CH ₃ O ^{OH} CO(CH ₂ ) ₆ CH ₃ CH ₃ O ^{OCH} 3	1-(2,4,6-trihydroxypher presence of potassium	of dimethyl sulfate with nyl)-1-octanone in the n carbonate in acetone at nitrogen (34 %) [2786].

m.p. 73–74° [2786]; ¹H NMR [2786], ¹³C NMR [2786], MS [2786].

#### 1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-octanone

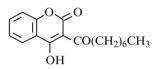
[134081-95-1]	$C_{16}H_{24}O_5$	mol. wt. 296.36
CH ₃ O CH ₃ O OCH ₃ OH CO(CH ₂ ) ₆ CH ₃	Synthesis -Preparation by treatmer 3,4-dimethoxy-2-(4-meth phenyl]-1-octanone with p refluxing methanol for 1–3	ylphenylsulfonyloxy) otassium carbonate in

m.p. 93–94.5° [1353]; ¹H NMR [1353].

#### 4-Hydroxy-3-(1-oxooctyl)-2H-1-benzopyran-2-one

[36953-90-9]

 $C_{17}H_{20}O_4$ mol. wt. 288.34



Syntheses -Obtained by reaction of octanoyl chloride with CO(CH₂)₆CH₃ 4-hydroxy-coumarin in pyridine containing one drop of piperidine for 12 h on a water bath (68 %) [3174].

-Also refer to: [3144].

m.p. 104–105° [3174].

#### 1-(8-Hydroxy-5-quinolinyl)-1-octanone

[110593-82-3]

C₁₇H₂₁NO₂

mol. wt. 271.36

mol. wt. 287.36



Synthesis

-Obtained by reaction of octanoyl chloride with 8-hydroxyquinoline in the presence of aluminium chloride in nitrobenzene at 80-85° for 15-16 h (39 %) [1725].

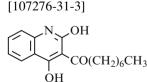
CO(CH₂)₆CH₃

m.p. 63.5–64.2° [1725]; ¹H NMR [1725], IR [1725].

Synthesis

USE: Ion flotation with, of gallium [1725].

#### 1-(2,4-Dihydroxy-3-quinolinyl)-1-octanone



-Obtained by reaction of octanoyl chloride with  $CO(CH_2)_6CH_3$  2,4-dihydroxyquinoline in the presence of aluminium chloride in nitrobenzene on the steam bath for 3 h (17 %) [3123].

m.p. 168–169° [3123]; UV [3123].

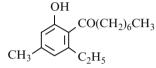
BIOLOGICAL ACTIVITY: Antibacterial properties (Staphylococcus aureus and Escherichia coli) [3123].

C₁₇H₂₁NO₃

#### 1-(3,4-Dihydro-5,7-dihydroxy-2H-1-benzopyran-6-yl)-1-octanone

#### 1-(2-Ethyl-6-hydroxy-4-methylphenyl)-1-octanone

$$C_{17}H_{26}O_2$$
 mol. wt. 262.39



Syntheses
-Preparation by Fries rearrangement of 3-ethyl-
5-methyl-phenyl caprylate with aluminium chloride,
*without solvent at 130° for 2 h (77 %) [2802];
*in nitrobenzene at $25^{\circ}$ for 6 h (79 %) [2802].

b.p.6 220° [2802].

#### Methyl ether

C₁₈H₂₈O₂ mol. wt. 276.42

-Obtained by methylation of the above ketone in the usual way (76 %) [2802].

b.p.₃₀ 205° [2802].

#### 1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-octanone

[134081-64-4]	$C_{17}H_{26}O_5$	mol. wt. 310.39
OH CH ₃ O CH ₃ O CH ₃ O OCH ₃	Syntheses -Obtained by partial select crude 2,3,4,6-tetramethox aluminium chloride in a 1–2 h (82 %) [1353]. -Also refer to: [1351].	cyoctanophenone with

m.p. 49–50° [1353]; ¹H NMR [1353].

**p-Toluenesulfonic ester** [134081-79-1] C₂₄H₃₂O₇S mol. wt. 464.58

-Obtained by reaction of p-toluenesulfonyl chloride with 2-hydroxy-3,4,6-trimethoxy-octanophenone in the presence of potassium carbonate in refluxing acetone for 6 to 14 h (93 %) [1353].

m.p. 83–85° [1353]; ¹H NMR [1353].

#### Methyl ether C18H28O5 mol. wt. 324.42

-Obtained by reaction of octanoyl chloride with 1,2,3,5-tetramethoxybenzene in the presence of aluminium chloride in ethyl ether at  $0^{\circ}$  for 30 min [1353].

#### 1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-octanone

[134081-71-3]	$C_{17}H_{26}O_5$	mol. wt. 310.39
CH ₃ O OH CO(CH ₂ ) ₆ CH ₃ OCH ₃	2,3,4-trimethoxyoctanop dium on charcoal in eth	on of (6-phenylmethoxy)- henone over 10 % palla- yl acetate/methanol (1:1) gen ceased (88 %) [1353].

m.p. 40–42° [1353]; ¹H NMR [1353].

### 1-(6,7-Dihydro-4-hydroxy-6-methylfuro[2,3-f]-1,3-benzodioxol-8-yl)-1-octanone

	$C_{18}H_{24}O_5$	mol. wt. 320.39
OH OH	Synthesis -Refer to: [2179].	
	Methyl ether Octanoyl furapiole	
$\downarrow$ $\uparrow$ $CO(CH_2)_6CH_3$	[82652-36-6] C ₁₉ H ₂₆ O ₅	mol. wt. 334.41

-Obtained by reaction of octanoyl chloride with furapiole in the presence of fused zinc chloride in methylene chloride for 3–4 h in an ice bath (70 %) [2179].

colourless plates; m.p. 55° [2179]; ¹H NMR [2179], IR [2179].

USE: Synergistic insecticidal activity of, with pyrethrum [2179].

#### 1-(6,7-Dihydroxy-5-propyl-1,3-benzodioxol-4-yl)-1-octanone

	$C_{18}H_{26}O_5$	mol.	wt. 322.40
OH OH OH $C_3H_7$	[82652-28-6]	octanoyl dihydrodillapiole	wt 350.46
$O \xrightarrow{C_3H_7} C_0(CH_2)_6CH_3$	[82652-28-6] C ₂₀ H ₃₀ O ₅	mol.	wt. 350

-Obtained by reaction of octanoyl chloride with dihydrodillapiole in the presence of fused zinc chloride in methylene chloride for 3–4 h in an ice bath (70 %) [2179].

colourless solid [2179]; ¹H NMR [2179], IR [2179].

USE: Synergistic insecticidal activity of, with pyrethrum [2179].

#### 1-[3-Chloro-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-octanone

[108300-00-1]	$C_{18}H_{27}ClO_2$	mol. wt. 310.86
$\begin{array}{c} OH\\ Cl & \downarrow \\ & \downarrow \\ & \downarrow \\ C(CH_3)_3 \end{array}$	Synthesis -Obtained by Fries rearrange butyl-phenyl caprylate with 110° (72 %) [3119]. b.p. ₁₀ 170° [3119].	ement of 2-chloro-4-tert- aluminium chloride at

**2,4-Dinitrophenylhydrazone** [102955-19-1] C₂₄H₃₁ClN₄O₅ mol. wt. 490.99

m.p. 127° [3119].

CO(CH₂)₆CH₃

### 1-[3-(1,1-Dimethylethyl)-4-hydroxyphenyl]-1-octanone

[95102-32-2]  $C_{18}H_{28}O_2$ mol. wt. 276.42 Synthesis OH  $C(CH_3)_3$  -Refer to: [2704].

#### 1-[2-Hydroxy-3-methyl-6-(1-methylethyl)phenyl]-1-octanone

	$C_{18}H_{28}O_2$	mol. wt. 276.42
CH ₃ CH ₃ CO(CH ₂ ) ₆ CH ₃ CH(CH ₃ ) ₂	Synthesis -Obtained by Fr caprylate with (60 %) [2798].	ies rearrangement of carvacryl aluminium chloride at 120°
b.p. ₁₀ 255° [2798].		

**2,4-Dinitrophenylhydrazone**  $C_{24}H_{32}N_4O_5$  mol. wt. 456.54

m.p. 127° [2798].

#### 1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-octanone

[778641-04-6]	$C_{18}H_{28}O_2$	mol. wt. 276.42
(CH ₃ ) ₂ CH CO(CH ₂ ) ₆ CH ₃ CH ₃	Synthesis -Obtained by Fries caprylate with alu (81 %) [2803].	rearrangement of thymyl minium chloride at 120°

b.p.₂ 185° [2803].

#### 2,4-Dinitrophenylhydrazone

$$C_{24}H_{32}N_4O_5$$

m.p. 185° [2803].

#### 1-[4-Hvdroxy-2-methyl-5-(1-methylethyl)phenyl]-1-octanone

 $(CH_3)_2CH$ CH₂ CO(CH₂)₆CH₃

-Obtained (XXVIII) by treatment of 4-methoxy-2-methyl-5-isopropyloctanophenone (IX) with boiling pyridinium chloride (205-215°) for 4.5 h (18 %) [2660].

-Also obtained by Fries rearrangement of thymyl caprylate with aluminium chloride in nitrobenzene for 16 h at 25° (84 %) [2647].

-Also refer to: [2704].

b.p.9 217-220° [2647], b.p.14 235° [2660]; m.p. 81-82° [2647], 80° [2660].

USE: Colour developer, for thermal recording materials [1595].

#### C19H30O2 Methyl ether (IX) mol. wt. 290.45

-Obtained by reaction of caprylyl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (61 %) [2660].

b.p.₁₃ 210° [2660];  $n_D^{23} = 1.5120$  [2660].

#### 1-[6-Hydroxy-2-methyl-3-(1-methylethyl)phenyl]-1-octanone

C18H28O2 mol. wt. 276.42 **Synthesis** OH  $CO(CH_2)_6CH_3$  -Refer to: [2664]. Methyl ether [102020-37-1]  $C_{19}H_{30}O_2$ mol. wt. 290.45 CH(CH₃)₂

-Obtained by reaction of octanoyl chloride with p-thymol in the presence of aluminium chloride in carbon disulfide for 24 h at r.t. (79 %) [2664].

pale yellow oil [2664]; b.p.₁₉ 212° [2664]; IR [2664];  $n_D^{24} = 1.5160$  [2664].

mol. wt. 456.54

mol. wt. 304.43

### 1-(4-Butoxy-2-hydroxyphenyl)-1-octanone

$$\begin{array}{cccc} [24294-76-6] & C_{18}H_{28}O_3 & \text{mol. wt. } 292.42 \\ & OH & \\ & C_{0}(CH_2)_6CH_3 & Obtained by reaction of octanoyl chloride with m-dibutoxybenzene in the presence of aluminium chloride in dichloroethane at 0°. Then, the mixture was stirred for 1 h at 10°, and then for 6 h at 20-25°. \\ \end{array}$$

The temperature was then raised to  $80^{\circ}$  and stirring was continued at that temperature for 5 h (77 %) [1194, 1195].

m.p. 33.5–34° [1194, 1195]; UV [1194, 1195].

USE: UV absorber [1194]; Light-stabilizer of polymeric materials [1194].

## 1-(5-Butoxy-2-hydroxyphenyl)-1-octanone

$$\begin{array}{cccc} [140943-38-0] & C_{18}H_{28}O_3 & \text{mol. wt. } 292.42 \\ OH & Synthesis \\ -Refer to: [285]. \\ Oxime & [140943-24-4] \\ C_{18}H_{29}NO_3 & \text{mol. wt. } 307.43 \end{array}$$

-Refer to: [285].

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

## 1-[2,5-Dihydroxy-3,4-bis(methylthio)phenyl]-3,7-dimethyl-1-octanone

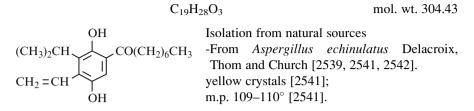
$C_{18}H_{28}O_3S_2$	mol. wt. 356.55
$\begin{array}{c} OH \\ CH_3S \\ H_3S \\ CH_3S \\ OH \end{array} \begin{array}{c} OH \\ CH_2 - CH - (CH_2)_3 CH(CH_3)_2 \\ CH_3 \\ CH_3 \end{array}$	$\begin{array}{llllllllllllllllllllllllllllllllllll$

### 1-[2,5-Dihydroxy-3-(1-ethenyl)-4-(1-methylethyl)phenyl]-1-octanone

C₁₉H₂₈O₃

OH  $CH_2=CH$   $(CH_3)_2CH$ OH OH OH OH  $CO(CH_2)_6CH_3$  OH OH Isolation from natural sources-From Aspergillus echinulatus Delacroix,Thom and Church [2539, 2541, 2542].yellow crystals [2541];m.p. 109–110° [2541].

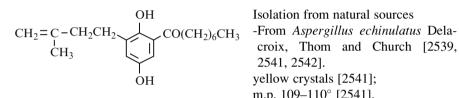
### 1-[2,5-Dihydroxy-4-(1-ethenyl)-3-(1-methylethyl)phenyl]-1-octanone



## 1-[2,5-Dihydroxy-3-(3-methyl-3-butenyl)phenyl]-1-octanone

 $C_{19}H_{28}O_3$ 

mol. wt. 304.43



Isolation from natural sources yellow crystals [2541]; m.p. 109–110° [2541].

## 1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-octanone

2-octanoyl-4-(3-methylbuten-2-yl)phloroglucinol (11) [1026].

[85602-21-7]

C19H28O4

mol. wt. 320.43

$$CH_{3}-C=CHCH_{2}$$

Syntheses -Obtained by adding cuprous chloride,

a saturated aqueous solution of sodium carbonate and prenyl bromide to a solution of phloro-octanophenone

in ethyl ether at r.t., then to keep the mixture for 12 h at this temperature [1026]. -Also prepared by adding prenyl bromide to a suspension of the sodium salt of phlorooctanophenone in benzene, then the mixture obtained was refluxed for 3 h [1026].

-Also obtained by reaction of prenyl chloride with phlorooctanophenone in an alkaline two-phase (aqueous ether) system and catalyzed by CuCl [838, 3193].

m.p. 140–142° [3193]; ¹³C NMR [1026, 3193], IR [1026].

BIOLOGICAL ACTIVITY: Bactericidal and fungicidal [1026, 3193]; Antimicrobial [3193].

mol. wt. 306.45

mol. wt. 486.57

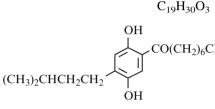
#### 2-Butyl-1-(2,5-dihydroxy-4-methylphenyl)-1-octanone

$$[357172-28-2] C_{19}H_{30}O_{3} mol. wt. 306.45$$

$$OH Synthesis -Refer to: [2352].$$

$$CH_{3} OH C_{4}H_{9} OH$$

## 1-[2,5-Dihydroxy-4-(3-methylbutyl)phenyl]-1-octanone



Synthesis -Obtained by treatment of 2-hydroxy-5-methoxy-4-isoamyloctanophenone with aluminium bromide [161, 770].

pale yellow solid [770]; m.p. 105–106° [161].

2,4-Dinitrophenylhydrazone

m.p. 130–131° [161].

## 1-(2,5-Dihydroxy-4-pentylphenyl)-1-octanone

C. H. O.

$$\begin{array}{c} C_{19}H_{30}O_{3} \\ OH \\ CH_{3}(CH_{2})_{4} \\ OH \end{array} \begin{array}{c} OH \\ OH \\ OH \end{array} \begin{array}{c} Synthesis \\ -Obtained by demethylation of 2-hydroxy- 5-methoxy-4-amyloctophenone with aluminium bromide in benzene (almost quantitatively yield) [770]. \end{array}$$

pale yellow needles [770]; m.p. 94° [770].

#### C25H34N4O6 2,4-Dinitrophenylhydrazone mol. wt. 486.57

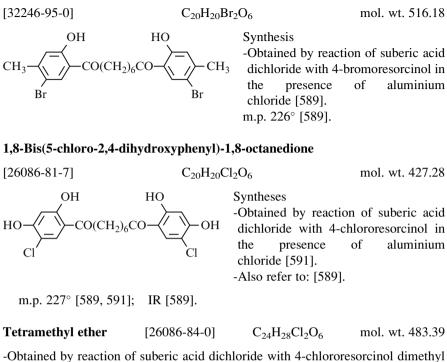
purple needles [770]; m.p. 112° [770].

## 1-(2-Hydroxy-5-pentyloxyphenyl)-1-octanone

	$C_{19}H_{30}O_3$	mol. wt. 306.45
OH	Synthesis	
CO(CH ₂ ) ₆ CH ₃	-Refer to: [770]. b.p. _{1.5} 190–195° [770].	
	2,4-Dinitrophenylhydrazone	
OC ₅ H ₁₁	$C_{25}H_{34}N_4O_6$	mol. wt. 486.57
m.p. 121° [770].		

C25H34N4O6

#### 1,8-Bis(5-bromo-2,4-dihydroxyphenyl)-1,8-octanedione



-Obtained by reaction of suberic acid dichloride with 4-chlororesorcinol dimethyl ether in the presence of aluminium chloride [591]. -Also refer to: [589].

m.p. 186° [589, 591].

## 1,8-Bis(4-hydroxyphenyl)-1,8-octanedione

[22811-90-1]	$C_{20}H_{22}$	O ₄	mol. wt. 326.39
но-Со(С	Н2)6СО-ОН	Syntheses -Refer to: [584, 1337,	2693].
m.p. 218–219° [	1337], 200° [584].		
Diacetate	[102957-05-1]	$C_{24}H_{26}O_{6}$	mol. wt. 410.47
m.p. 119–120° [1337].			
Dimethyl ether	[4280-50-6]	$C_{22}H_{26}O_4$	mol. wt. 354.45
01/ 11		11 11 14 1 1	.1 6

-Obtained by reaction of suberic acid dichloride with anisole in the presence of aluminium chloride without solvent at <40° (87 %) [905]. -Also refer to: [584, 2693 (70 %)].

m.p. 133° [584], 129–130° [905], 128.8–129.2° [2693].

mol. wt. 358.39

mol. wt. 414.50

mol. wt. 358.39

526.54

## Di-2,4-dinitrophenylhydrazone of the dimethyl ether

$$[22811-95-6] C_{34}H_{34}N_8O_{10} mtext{mol. wt. 714.69}$$

m.p. 223.7–224.7° [2693], 200° [584].

CO(CH₂)₆CO-

## 1,8-Bis(2,4-dihydroxyphenyl)-1,8-octanedione

$$\begin{array}{ccc} [26086-74-8] & C_{20}H_{22}O_6 \\ OH & HO & Syntheses \end{array}$$

-Obtained by reaction of suberic acid dichloride with resorcinol in the presence of aluminium chloride [591].

-Also obtained by reaction of suberic acid with resorcinol in the presence of zinc chloride at 140° for 5 h (55 %) [445].

-OH

-Also refer to: [445, 589, 2606].

m.p. 189° [589, 591], 187° [445], 186–187° [2606]; IR [589].

**Tetramethyl ether** 

-Refer to: [589].

m.p. 140° [589]; IR [589].

### Tetraacetate

$$C_{28}H_{30}O_{10}$$
 mol. wt.

C24H30O6

-Obtained by reaction of acetic anhydride with the titled diketone [445].

C20H22O6

[32246-82-5]

m.p. 135° [445].

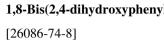
## 1,8-Bis(3,4-dihydroxyphenyl)-1,8-octanedione

HO OH Synthesis -Refer to: [1014]. OH Tetramethyl ether [32435-18-0] CO(CH₂)₆CO C24H30O6 mol. wt. 414.50

-Obtained by reaction of suberic acid dichloride with pyrocatechol dimethyl ether in the presence of aluminium chloride [591].

-Also obtained by hydrogenating of its oxime in acetic acid in the presence of 10%Pd/C in a Parr apparatus at r.t. and 45-50 psig for 12-36 h (63 %) [1014]. -Also refer to: [589].

m.p. 143° [589, 591], 141–143° [1014]; ¹H NMR [2342], ¹³C NMR [2342], IR [589].



HO

Dioxime of the tetramethyl ether [50766-30-8]  $C_{24}H_{32}N_2O_6$  mol. wt. 444.53

m.p. 140–143° [1014].

## Di-2,4-dinitrophenylhydrazone of the tetramethyl ether

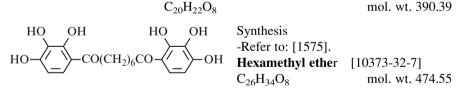
[32246-93-8]	$C_{36}H_{38}N_8O_{12}$	
m.p. 230° [589].		
Dimethylenedioxy	$C_{22}H_{22}O_{6}$	mol. wt. 382.41
1,8-Bis(3,4-methylenedioxypher	nyl)-1,8-octanedione	
-Refer to: [2693 (36 %)].		
m.p. 180.3–181.3° [2693].		
Di-2,4-dinitrophenylhydrazon of the dimethylenedioxy	$c_{34}H_{30}N_8O_{12}$	mol. wt. 742.66

m.p. 280.4-281.4° [2693]

## 1,8-Bis(3,5-dihydroxyphenyl)-1,8-octanedione

$$\begin{array}{ccc} C_{20}H_{22}O_6 & \text{mol. wt. 358.39} \\ HO & OH & \text{Synthesis} \\ & -\text{Refer to: [1316].} \\ HO & OH & \text{m.p. 185-188^{\circ} [1316].} \\ \end{array}$$

## 1,8-Bis(2,3,4-trihydroxyphenyl)-1,8-octanedione



-Obtained by reaction of suberic acid dichloride with pyrogallol trimethyl ether in the presence of aluminium chloride [591].

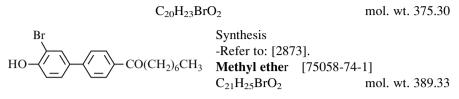
-Also obtained by reaction of dimethyl sulfate with 1,8-bis(2-hydroxy-3,4-dimethoxyphenyl)-1,8-octanedione the presence of 30 % sodium hydroxide (65–90 %) [1574].

-Also refer to: [1575].

m.p. 160° [591], 101° [1574, 1575].

N.B.: One the reported melting point is obviously wrong.

## 1-(3'-Bromo-4'-hydroxy[1,1'-biphenyl]-4-yl)-1-octanone



-Refer to: [2873].

### 1-(3'-Chloro-4'-hydroxy[1,1'-biphenyl]-4-yl)-1-octanone

$C_{20}H_{23}Cl$	0 ₂	mol. wt. 330.85
HO $  -$ CO(CH ₂ ) ₆ CH ₃	Syntheses -Refer to: [2115, 2116]. <b>Methyl ether</b> [65687-21 $C_{21}H_{25}CIO_2$	-0] mol. wt. 344.88

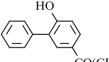
-Refer to: [2115, 2116].

## 1-(2-Hydroxy[1,1'-biphenyl]-3-yl)-1-octanone

### 1-(2-Hydroxy[1,1'-biphenyl]-5-yl)-1-octanone

[95102-22-0]

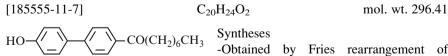
C₂₀H₂₄O₂ mol. wt. 296.41



Syntheses -Refer to: [2006, 2704] (Japanese patents). m.p. 86° [2704].

 $CO(CH_2)_6CH_3$  USE: As colour developer [2006, 2704].

#### 1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-1-octanone



-Obtained by Fries rearrangement of 4-octanoyl-oxybiphenyl with aluminium chloride in nitrobenzene, first at  $20^{\circ}$  for 12 h, then at  $60^{\circ}$  for 1 h [522].

-Also refer to: [1568, 1923].

colourless silky needles [522]; m.p. 123° [522].

**Methyl ether** [56116-80-4] C₂₁H₂₆O₂ mol. wt. 310.44

-Obtained from octanoyl chloride and 4-methoxybiphenyl [522]. -Also refer to: [1923].

smooth spangles [522]; b.p.₁₅ 272–275° [522]; m.p. 120° [522].

## Various ethers (10)

-Preparations and liquid crystalline properties of, [847].

Ethyl ether	[56116-89-3]	$C_{22}H_{28}O_2$	mol. wt. 324.46
Propyl ether	[56116-97-3]	$C_{23}H_{30}O_2$	mol. wt. 338.49
Butyl ether	[56117-05-6]	$C_{24}H_{32}O_2$	mol. wt. 352.52
Pentyl ether	[56117-14-7]	$C_{25}H_{34}O_2$	mol. wt. 366.54
Hexyl ether	[56117-23-8]	$C_{26}H_{36}O_2$	mol. wt. 380.57
Heptyl ether	[56117-32-9]	$C_{27}H_{38}O_2$	mol. wt. 394.60
Octyl ether	[56117-40-9]	$C_{28}H_{40}O_2$	mol. wt. 408.62
-Refer to: [1661].			
Nonyl ether	[56117-49-8]	$C_{29}H_{42}O_2$	mol. wt. 422.65
Decyl ether	[56117-58-9]	$C_{30}H_{44}O_2$	mol. wt. 436.68
Dodecyl ether	[56117-67-0]	$C_{32}H_{48}O_2$	mol. wt. 464.73

## 1-(2-Hydroxy-5-methoxy-3-pentylphenyl)-1-octanone

[873416-42-3]	$C_{20}H_{32}O_3$	mol. wt. 320.47
CH ₃ (CH ₂ ) ₄ OCH ₃ OCH ₃	4-methoxy-2-a	Fries rearrangement of mylphenyloctanoate with alu- ide at 180° for 12 h under %) [770].

discoloured oil [770]; b.p._{0.1} 180–190° [770].

## 2,4-Dinitrophenylhydrazone

C₂₆H₃₆N₄O₆ mo

mol. wt. 500.60

m.p. 103° [770].

## 1-[2-Hydroxy-5-methoxy-4-(3-methylbutyl)phenyl]-1-octanone

[717103-49-6]	$C_{20}H_{32}O_3$	mol. wt. 320.47
$i-C_5H_{11}$ OH OCH ₃ CO(CH ₂ ) ₆ CH ₃	Synthesis -Obtained by reaction of oc 2,5-dimethoxyisoamylbenz aluminium chloride [161] (70 %) [770].	ene in the presence of

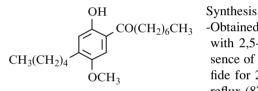
dark oil [770]; b.p._{0.4} 143–144° [161], b.p.₁₋₂ 170–174° [161].

#### 2,4-Dinitrophenylhydrazone C₂₆H₃₆N₄O₆ mol. wt. 500.60

leaves [161]; m.p. 146° [770], 140–142° [161].

## 1-(2-Hydroxy-5-methoxy-4-pentylphenyl)-1-octanone

C₂₆H₃₆N₄O₆



CO(CH₂)₆CH₃ -Obtained by reaction of octanoyl chloride with 2,5-dimethoxyamylbenzene in the pre-sence of aluminium chloride in carbon disulfide for 2 h at  $0^{\circ}$ , then 1 h at r.t. and 8 h at reflux (87 %) [770].

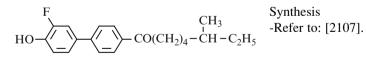
pale yellow needles [770]; m.p.  $42^{\circ}$  [770].

2,4-Dinitrophenylhydrazone

m.p. 117° [770].

## 1-(3'-Fluoro-4'-hydroxy[1,1'-biphenyl]-4-yl)-6-methyl-1-octanone $C_{21}H_{25}FO_2$

mol. wt. 328.43



**Decyl ether** (S) [112780-64-0]  $C_{31}H_{45}FO_2$ mol. wt. 468.70 USE: Liq.-crystal compns. contg., for display devices [2107].

**Dodecyl ether** (S) [112780-65-1] C₃₃H₄₀FO₂ mol. wt. 496.75 USE: Liq.-crystal compns. contg., for display devices [2107].

mol. wt. 500.60

#### 1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-2-methyl-1-octanone

USE: For liquid crystal compns. for display devices [1323].

Methyl ether (+) C22H28O2 mol. wt. 324.46 [141681-77-8] -Refer to: [3403].

IR [1457].

## 1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-6-methyl-1-octanone

$$\begin{array}{c} C_{21}H_{26}O_2 & \text{mol. wt. 310.44} \\ CH_3 & \text{Synthesis} \\ HO - \swarrow - CO(CH_2)_4 - \overset{1}{C}H - C_2H_5 & -\text{Refer to: [2205].} \end{array}$$

Methyl ether	[117235-07-1]	$C_{22}H_{28}O_2$	mol. wt. 324.46

-Preparation from p-methoxybiphenyl and 6-methyloctanoyl chloride [2205].

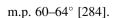
## 1-(3,4'-Dihydroxy[1,1'-biphenyl]-4-yl)-2-methyl-1-octanone

[133407-00-8] (S) mol. wt. 326.44  $C_{21}H_{26}O_3$ Synthesis OH  $-CO-CH-C_6H_{13}$ -Refer to: [1718]. HO CH₂

## 1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1-octanone

[143287-05-2]	$C_{21}H_{26}O_3$	mol. wt. 326.44
OH	Synthesis -Obtained by reac	tion of benzyl chloride with

Obtained by reaction of benzyl chloride with 2,4-dihydroxycaprylylphenone in the presence of potassium carbonate in refluxing acetone for 20 h [284].



C₆H₅CH₂



-Obtained by reaction of hydroxylamine hydrochloride with 1-[2-hydroxy-4-(phenylmethoxy)-phenyl]-1-octanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 62–66° [284]; ¹H NMR [284].

## 3,7-Dimethyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-octanone

[198878-80-7] 
$$C_{21}H_{32}O_4$$
 mol. wt. 348.48  
 $CH_3-C=CHCH_2$   $OH$   $COCH_2-CH-(CH_2)_3CH(CH_3)_2$  -Refer to: [2685].  
 $CH_3$   $HO$   $OH$   $CH_3$ 

BIOLOGICAL ACTIVITY: As preventive and therapeutic agent for bone and cartilage diseases [2685].

## 1,8-Bis(4-hydroxy-2-methylphenyl)-1,8-octanedione

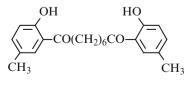
 $\begin{array}{c} C_{22}H_{24}Cl_{2}O_{4}\\ \end{array}$   $\begin{array}{c} OH \\ CH_{3} \\ CH_{3} \\ Cl \end{array} \xrightarrow{OH} CO(CH_{2})_{6}CO \\ Cl \end{array} \xrightarrow{Cl} CH_{3} \\ \begin{array}{c} Synthesis \\ -Refer to: [584] \\ m.p. \ 115^{\circ} \ [584]. \end{array}$ 

## 1-[4-Hydroxy-3-[[[3-(trifluoromethyl)phenyl]amino]methyl]phenyl]-1-octanone

 $[107076-65-3] C_{22}H_{26}F_3NO_2$ mol. wt. 393.45 OH CF₃ Synthesis -Refer to: [1823]. USE: Antiplaque dentifrices contg. [1823]. CO(CH₂)₆CH₃

## 1,8-Bis(2-hydroxy-5-methylphenyl)-1,8-octanedione

C₂₂H₂₆O₄ mol. wt. 354.45



[13282-26-3]

Syntheses -Obtained by Fries rearrangement of di(4-methylphenyl) suberate with aluminium chloride, *without solvent at 130° for 4 h (35 %) [3107]; *in refluxing chlorobenzene for 6 h (72 %)

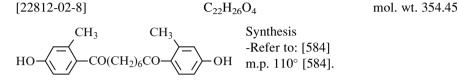
*in refluxing chlorobenzene for 6 h (72 %) [3107].

mol. wt. 423.34

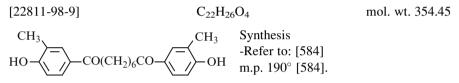
-Also refer to: [584].

m.p. 136–137° [3107], 110° [584]. **N.B.**: One of the reported melting point is obviously wrong. IR [3107].

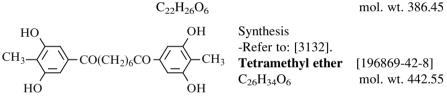
## 1,8-Bis(4-hydroxy-2-methylphenyl)-1,8-octanedione



## 1,8-Bis(4-hydroxy-3-methylphenyl)-1,8-octanedione



## 1,8-Bis(3,5-dihydroxy-4-methylphenyl)-1,8-octanedione



-Synthesis from 3,5-dimethoxy-4-methylbenzaldehyde (70 %) [3132].

¹H NMR [3132], ¹³C NMR [3132]. Colourless solid [3132];

## 1,8-Bis(2-hydroxy-4-methoxyphenyl)-1,8-octanedione

[26086-77-1]

C22H26O6

mol. wt. 386.45



-Also refer to: [589].

m.p. 155° [589, 591].

Syntheses

-Obtained by reaction of suberic acid dichloride with 3-methoxyphenol in the presence of aluminium chloride [591].

## 1,8-Bis(2-hydroxy-5-methoxyphenyl)-1,8-octanedione

1,0-Dis(2-injul oxy-5-incluoxy pilenyi)-1,0-octainculoite			
[10491-15-3]	C ₂₂ H	I ₂₆ O ₆	mol. wt. 386.45
OH CH ₃ O	HO H ₂ ) ₆ CO – CO OCH ₃	Synthesis -Obtained by reaction dichloride with p-o in the presence chloride [1575].	
m.p. 130° [1575]			
<b>Dimethyl ether</b> m.p. 116° [1575]	[10491-14-2]	$C_{24}H_{30}O_6$	mol. wt. 414.50
Diacetate	[10365-35-2]	$C_{26}H_{30}O_8$	mol. wt. 470.52
-Obtained by reaction of acetic anhydride (4 vol) with the title ketone in the presence of 1–2 drops concentrated sulfuric acid, the mixture warmed 5 min [1575]. m.p. 107° [1575].			
1-(3-Hydroxy-4'-methoxy[1,1'-biphenyl]-4-yl)-2-methyl-1-octanone			
[133406-99-2] (S)	$C_2$	$_{2}H_{28}O_{3}$	mol. wt. 340.46
СН ₃ О-	$OH$ $-CO-CH-C_6H_{12}$ $CH_3$	Synthesis -Refer to: [1718].	
1-(2-Hexyl-6-hydroxy-5-benzofuranyl)-1-octanone			
	$C_{22}H_{32}O_3$		mol. wt. 344.49
HO CH ₃ (CH ₂ ) ₆ CO	1,5	thesis tained by treatment of 2 diacetoxybenzene with THF/MeOH/H ₂ O at 80°	NaOH (6 equiv.)

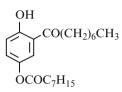
¹H NMR [1875], ¹³C NMR [1875], IR [1875], MS [1875].

#### 1,1'-(4,6-Dihydroxy-1,3-phenylene)bis-1-octanone

[760989-23-9]	$C_{22}H_{34}O_4$	mol. wt. 362.51
HO CO(CH ₂ ) ₆ CH ₃	Synthesis -Obtained by reaction of octanoic resorcinol (1 mol) in the presence of Bronsted acid catalyst [1077].	
CO(CH ₂ ) ₆ CH ₃	USE: For preparation of cosmetic a	active [1077].

## 1-(2-Hydroxy-5-octanoyloxyphenyl)-1-octanone

$$[21182-58-1]$$
 C₂₂H₃₄O₄ mol. wt. 362.51



Synthesis -Obtained by action of octanoyl chloride with quinol

dimethyl ether in the presence of aluminium chloride in refluxing carbon disulfide for 10 h (6 %) [2370].

colourless needles [2370]; m.p. 58-59° [2370]; IR [2370].

#### 1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-octanone

mol. wt. 378.51 [3118-46-5]  $C_{22}H_{34}O_5$ 

Syntheses

OH CH₃(CH₂)₆CO HC

.CO(CH₂)₆CH₃ -Obtained by reaction of caprylic acid with phloroglucinol in the presence of boron trifluoride etherate [3019], at 100° for 2 h (50-75 %) [338].

-Also obtained by reaction of octanoic anhydride with phloroglucinol in the presence of boron trifluoride etherate on heating for 4 h [457]. -Also refer to: [457, 644, 2911].

m.p. 93–95° [2911], 93–94° [457]; ¹H NMR [3019], ¹³C NMR [3019], IR [3019], UV [3019].

BIOLOGICAL ACTIVITY: As a new class of GPR40 (FFAR1) agonists [338]; Antagonist both thromboxane A2 and Leukotriene D4 [3019]; Antiviral activity towards the replication of vesicular stomatitis virus in HEL cell cultures [644]; Anthelmintic [457].

## 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-octanone

BIOLOGICAL ACTIVITY: High larvicidal activity in test on mosquito larvas [2810].

Acetate [30392-06-4] C₂₄H₃₈O₃ mol. wt. 374.56

-Obtained by acetylation of 3,5-di-tert-butyl-4-hydroxyoctanophenone [2145]. -Also refer to: [228].

b.p.₁₀ 234–236° [228]; ¹H NMR [2145], IR [2145].

## 1-(2-Hydroxy-5-octylphenyl)-1-octanone

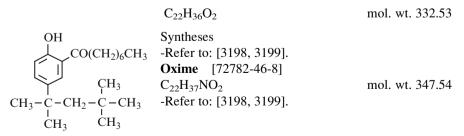
 $\begin{array}{cccc} [74604-21-0] & C_{22}H_{36}O_2 & \mbox{mol. wt. } 332.53 \\ OH & Synthesis \\ -Obtained by Fries rearrangement of 4-octylphenyl \\ octanoate (b.p._{0.02} 168^\circ) [2520]. \\ b.p._{0.02} 170^\circ [2520]; \mbox{ IR } [2520], \mbox{UV } [2520]. \\ \end{array}$ 

 Oxime
 [74604-11-8]
  $C_{22}H_{37}NO_2$  mol. wt. 347.54

 waxy needles [2520];
 m.p. 59° [2520];
 IR [2520], UV [2520].

USE: Solvent extraction of copper (II) [2520].

## 1-[2-Hydroxy-5-(1,1,3,3-tetramethylbutyl)phenyl]-1-octanone



USE: Extraction. by, of copper, kinetics and mechanism of, [3198, 3199].

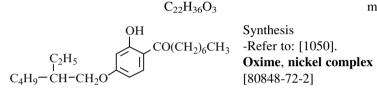
#### 1-(2,5-Dihydroxy-4-octylphenyl)-1-octanone

[63134-27-0] 
$$C_{22}H_{36}O_3$$
 mol. wt. 348.53  
OH  $CO(CH_2)_6CH_3$  Obtained by reaction of caprylic acid with 2-octylhydroquinone in the presence of boron trifluoride in 1,2-dichloroethane at 40–45° for 1.25 h. The mixture was allowed to stand overnight (70 %) [142].

-Also refer to: [1907].

m.p. 82–83° [142, 1907].

## 1-[4-[(2-Ethylhexyl)oxy]-2-hydroxyphenyl]-1-octanone



USE: Photog. stabilizer compns. contg. phenol derivs. and, for magenta images [1050].

## 1-[2-Hydroxy-4-[(1-methylheptyl)oxy]phenyl]-1-octanone

$$[127789-29-1] C_{22}H_{36}O_{3} mol. wt. 348.53$$

$$OH CO(CH_{2})_{6}CH_{3} -Refer to: [3345].$$

$$Oxime (E) [127789-31-5]$$

$$C_{22}H_{37}NO_{3} mol. wt. 363.54$$

-Refer to: [3345].

[127789-34-8] C₂₂H₃₇NO₃ mol. wt. 363.54 Oxime (Z)

-Refer to: [3345].

## 1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-1-octanone

[134082-03-4]	$C_{23}H_{30}O_5$	mol. wt. 386.49
ОН	Synthesis	
CH ₃ O CO(CH ₂ ) ₆ CH ₃	-Obtained by elimination group in 2-position by	
CH ₃ O OCH ₂ C ₆ H ₅	benzyloxy-3,4-dimethoxyp	henyl)-1-octanone with
	concentrated hydrochloric	acid and acetic acid at
	r.t. for 2–3 h (84 %) [1353	].

m.p. 85.5–86° [1353]; ¹H NMR [1353].

mol. wt. 348.53

## 1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]-1-octanone

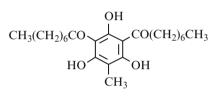
[134081-87-1]	$C_{23}H_{30}O_7S$	mol. wt. 450.55
$CH_{3}O \xrightarrow{OH} CO(CH_{2})_{6}CH_{3}$ $CH_{3}O \xrightarrow{I} OSO_{2}C_{6}H_{4}CH_{3}(p)$	Synthesis -Obtained by treatment 3,4,6-trimethoxyphenyl)-1-0 25 % aluminium bromide r.t. for 2–3 h (84 %) [1353]	octanone with in acetonitrile at

m.p. 63–64.5° [1353]; ¹H NMR [1353].

## 1,1'-(2,4,6-Trihydroxy-5-methyl-1,3-phenylene)bis-1-octanone

[3118-45-4]

C₂₃H₃₆O₅ mol. wt. 392.54



CO(CH₂)₆CH₃ -Obtained by reaction of octanoic anhydride with 2-methylphloroglucinol in the presence of boron trifluoride etherate on heating for 4 h [457]. -Also refer to: [457, 600, 2911].

BIOLOGICAL ACTIVITY: For treatment of immune dysfunction associated with human immuno deficiency virus infection [600]; Anthelmintic [457].

Syntheses

## 1-(2-Hydroxy-5-nonylphenyl)-1-octanone

	$C_{23}H_{38}O_2$	mol. wt. 346.55
$\bigcup_{C_9H_{19}}^{OH} CO(CH_2)_6CH_3$	octanoate with alumin	rearrangement of 4-nonylphenyl nium chloride [3445]. 143, 1145, 1146, 1507].

-Also refer to: [3445].

USE: Extn. by, of copper from aq. soln. [1143]; Prepn. of, for extn. of metals [1142]; Copper extn. by, [1146]; Extn. by, of copper, cobalt and nickel from sulfate solns [1507]; Extn. by, of non-ferrous metals [1145].

m.p. 102–104° [457, 2911].

#### 1-(2-Hydroxy-5-methoxy-3-octylphenyl)-1-octanone

[102898-66-8]	$C_{23}H_{38}O_3$	mol. wt. 362.55
---------------	-------------------	-----------------

 $CH_3(CH_2)_7$   $CO(CH_2)_6CH_3$  $OCH_3$ 

Syntheses

-Obtained by reaction of octanoic acid with 4-methoxy-2-octylphenol in the presence of boron trifluoride in tetrachloroethane for 6 h. The reaction mixture was allowed to stand overnight at r.t. and was then heated on a steam bath for about 5 h (43 %) [142].

-Also obtained by Fries rearrangement of 4-methoxy-2-octylphenyl caprylate (b.p.  $_{1}$  170–180°) with boron trifluoride in *sym*-tetrachloroethane for 6 h, following 5 h of heating on a steam bath (59 %) [142].

-Also refer to: [1907].

b.p.₁ 195–205° [142, 1907]; m.p. 31–32° [142], 30–31° [1907].

## 1-(2-Hydroxy-5-methoxy-4-octylphenyl)-1-octanone

[21182-66-1]

 $C_{23}H_{38}O_3$ 

mol. wt. 362.55

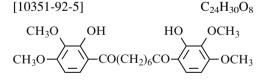
mol. wt. 446.50

Synthesis

 $_{\rm CO(CH_2)_6CH_3}$  -Obtained by action of octanoyl chloride with 2-octylhydroquinone dimethyl ether in the presence of aluminium chloride in refluxing carbon disulfide for 10 h on the steam bath (68 %) [2370].

yellow plates [2370]; m.p. 53–54° [2370]; IR [2370].

## 1,8-Bis(2-hydroxy-3,4-dimethoxyphenyl)-1,8-octanedione



Syntheses -Obtained by reaction of suberic acid dichloride with pyrogallol trimethyl ether in the presence of aluminium chloride in tetrachloroethane [1574].

-Also refer to: [589, 1575].

m.p. 163° [1574, 1575], 160° [589]; IR [589].

Dioxime	[32246-91-6]	$C_{24}H_{32}N_2O_8$	mol. wt. 476.53

m.p. 195° [589].

CH₃(CH₂)₇ CO(CH₂)₆C

#### 1,1'-(4-Hydroxy-2,6-dimethoxy-1,3-phenylene)bis-1-octanone

[1142936-30-8] 
$$C_{24}H_{38}O_5$$
 mol. wt. 406.56  
OH Synthesis  
-Refer to: [2786].  
 $CH_{3O} - OCH_3$   
 $CO(CH_2)_6CH_3$   
 $CO(CH_2)_6CH_3$ 

## 3-(4-Hydroxyphenyl)-4-[4-hydroxy-3-(1-oxooctyl)phenyl]hexane (3-n-Butyrylhexestrol)

C₂₆H₃₆O₃

C7H15CO HO-

## **Dimethyl ether**

C₂₈H₄₀O₃

mol. wt. 424.62

mol. wt. 396.57

-Obtained by reaction of octanoyl chloride with hexestrol dimethyl ether in the presence of aluminium chloride in nitrobenzene for 3 h at r.t. [510].

light colourless leaflets [510]; b.p.₁₅ about 300° [510]; m.p. 67° [510].

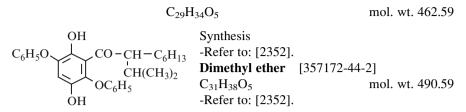
## 1-(4,4'-Dihydroxy[1,1'-biphenyl]-3,3'-diyl)-1-octanone

*in the presence of sodium chloride at  $140^{\circ}$ . The reaction is carried out by adding the ester to the melt, rapidly increasing the temperature to 200° and maintaining it there for 2 min before quick cooling [2091];

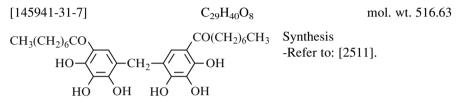
*in refluxing chlorobenzene for 24 h (88 %) [2377].

m.p. 88.5–90° [2377]; IR [2377].

## 1-(2,5-Dihydroxy-3,6-diphenoxyphenyl)-2-(1-methylethyl)-1-octanone



## 1,1'-[Methylenebis(4,5,6-trihydroxy-3,1-phenylene)]bis-1-octanone



## 1,1'-(6,6'-Dihydroxy-5,5'-dimethoxy[1,1'-biphenyl]-3,3'-diyl)bis-1-octanone

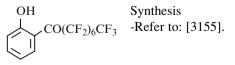
[157687-62-2]	$C_{30}H_{42}O_{6}$	mol. wt. 498.66
C ₇ H ₁₅ CO COC ₇ H	I ₁₅ Synthesis -Refer to: [1428].	
$CH_{3}O$ OH HO OCH ₃	BIOLOGICAL ACTIVITY gen scavenger, for therapeu	•

## 2 Aromatic Hydroxyketones Derived from Various Halogenooctanoic Acids

## 2.1 Unsubstituted Hydroxyketones

## 1-(2-Hydroxyphenyl)-2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-pentadecafluoro-1-octanone

 $[176515-55-2] \qquad \qquad C_{14}H_5F_{15}O_2 \qquad \qquad \text{mol. wt. } 490.27$ 



## 1-(2-4-Dihydroxyphenyl)-2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-pentadecafluoro-1-octanone

[105932-66-9]	$C_{14}H_5F_{15}O_3$	mol. wt. 506.16
OH CO(CF ₂ ) ₆ CF ₃	Synthesis -Refer to: [2171].	

## 2-Bromo-1-(4-hydroxyphenyl)-1-octanone

	$C_{14}H_{19}BrO_2$	mol. wt. 299.21
ОН	Synthesis	
	-Refer to: [3472].	
	Methyl ether	
$\mathbf{i}$	$C_{15}H_{21}BrO_2$	mol. wt. 313.23
COCHBr(CH ₂ ) ₅ CH ₃	-Refer to: [3472].	

## 8-Bromo-1-(4-hydroxyphenyl)-1-octanone

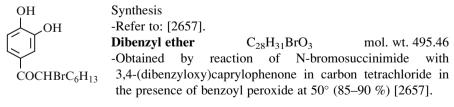
	$C_{14}H_{19}BrO_2$	mol. wt. 299.21
ОН	Synthesis -Refer to: [572].	
CO(CH ₂ ) ₆ CH ₂ Br	<b>Methyl ether</b> [224775-35-3] C ₁₅ H ₂₁ BrO ₂	mol. wt. 313.23

-Obtained by Friedel-Crafts acylation of anisole with 8-bromooctanoyl chloride in the presence of aluminium chloride in dichloromethane at  $-10^{\circ}$  for 1–2 h under nitrogen (40 %) [572].

m.p. 45–48° [572]; ¹H NMR [572].

## 2-Bromo-1-(3,4-dihydroxyphenyl)-1-octanone

$$C_{14}H_{19}BrO_3$$
 mol. wt. 315.20



m.p. 90° [2657].

## 3-Chloro-1-(4-hydroxyphenyl)-1-octanone

¹H NMR [1747, 1748], IR [1747, 1748], UV [1747, 1748].

## 2.2 Substituted Hydroxyketones

## 1-(2,4-Dihydroxy-3-methylphenyl)-2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-pentadecafluoro-1-octanone

[65240-06-4]	$C_{15}H_7F_{15}O_3$	mol. wt. 520.19
OH CH ₃ CO(CF ₂ ) ₆ CF ₃	Synthesis -Refer to: [450].	
но	BIOLOGICAL ACTIVITY: Antimi	crobial [450].

## 8-Bromo-1-(2-hydroxy-5-methoxyphenyl)-1-octanone

[173055-38-4] 
$$C_{15}H_{21}BrO_3$$
 mol. wt. 329.23  
OH Synthesis  
CO(CH₂)₆CH₂Br -Refer to: [2623].  
OCH₃

## 8-Bromo-1-(2-hydroxy-4,5-dimethoxyphenyl)-1-octanone

[173055-44-2] 
$$C_{16}H_{23}BrO_4$$
 mol. wt. 359.26  
OH Synthesis  
 $CH_{3}O + CO(CH_2)_6CH_2Br$  -Refer to: [2623].

#### 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-8-bromo-1-octanone

[158869-45-5] mol. wt. 411.42 C₂₂H₃₅BrO₂ Synthesis OH -Refer to: [1689].

C(CH₃)₃ CO(CH₂)₆CH₂Br

## **3** Aromatic Hydroxyketones Derived from 8-Oxooctanoic Acids

#### Unsubstituted Hydroxyketones 3.1

## 8-(4-Hydroxyphenyl)-8-oxo-1-octanoic acid

[22811-89-8]	$\mathrm{C}_{14}\mathrm{H}_{18}\mathrm{O}_{4}$	mol. wt. 250.29
HO CO(CH ₂ ) ₆ CO ₂ H	Synthesis -Refer to: [584]. m.p. 129° [584].	

## 8-(2,4-Dihydroxyphenyl)-8-oxo-1-octanoic acid

[32246-77-8] C14H18O5 mol. wt. 266.29 OH

HO

Syntheses  $CO(CH_2)_6CO_2H$  -Obtained by reaction of suberic acid with resorcinol in the presence of zinc chloride at  $140^{\circ}$  for 5 h (40 %) [445]. -Also refer to: [589, 3370].

m.p. 145° [445],

b.p._{0.5} 260–264° [445]; m.p. 84° [445].

Methyl ester

C15H20O5

mol. wt. 280.32

(CH₃)₃C

### 8-(3,4-Dihydroxyphenyl)-8-oxo-1-octanoic acid

	$C_{14}H_{18}O_5$	mol. wt. 266.29
OH	Synthesis	
Он	-Refer to: [1013].	
	<b>Dimethyl ether</b> [32246-94-9]	
$\mathbf{i}$	$C_{16}H_{22}O_5$	mol. wt. 294.35
CO(CH ₂ ) ₆ CO ₂ H	-Obtained by heating of its ethyl ester below	with 10 % aque-
	ous NaOH on the steam bath for 1.5 h (88	%) [1013].

-Also refer to: [589].

white solid [1013]; m.p. 94–95° [1013], 85° [589]; IR [1013].

## 2,4-Dinitrophenylhydrazone of the dimethyl ether

[32340-79-7]	$C_{22}H_{26}N_4O_8$	mol. wt. 474.47
m.p. 236° [589].		

Ethyl ester of the dimethyl ether [57641-19-7] C₁₈H₂₆O₅ mol. wt. 322.40

-Obtained by reaction of 8-chloro-8-oxooctanoic acid ethyl ester with 1,2-dimethoxybenzene in the presence of aluminium chloride in 1,1,2,2-tetrachloroethane first at  $0-3^{\circ}$  for 2.5 h, and overnight to  $2^{\circ}$  (59 %) [1013].

white solid [1013]; b.p._{0.05} 192–193° [1013]; m.p. 40–43° [1013]; IR [1013]; GLC [1013].

## 3.2 Substituted Hydroxyketones

#### 8-(2,4-Dihydroxy-5-bromophenyl)-8-oxo-1-octanoic acid

[32246-17-6]	$C_{14}H_{17}BrO_5$	mol. wt. 345.19
HO Br	Synthesis -Refer to: [589]. m.p. 161° [589].	

## 8-(2-Hydroxy-5-chlorophenyl)-8-oxo-1-octanoic acid

mol. wt. 284.74 [22812-06-2] C14H17ClO4 OH **Synthesis** -Refer to: [584]. CO(CH₂)₆CO₂H m.p. 132° [584].

## 8-(2,4-Dihydroxy-5-chlorophenyl)-8-oxo-1-octanoic acid

[32246-86-9] C14H17ClO5 mol. wt. 300.74 OH Synthesis .CO(CH₂)₆CO₂H -Refer to: [589]. m.p. 141° [589]. H( C1

## 8-(2-Hydroxy-5-chloro-4-methylphenyl)-8-oxo-1-octanoic acid

C15H19ClO4 mol. wt. 298.77 OН **Synthesis** CO(CH₂)₆CO₂H -Refer to: [584]. m.p. 161° [584]. CH

## 8-(2-Hydroxy-4-methylphenyl)-8-oxo-1-octanoic acid

[22812-09-5] C15H20O4 mol. wt. 264.32 Synthesis OH CO(CH₂)₆CO₂H -Refer to: [584]. m.p. 122° [584]; UV [1827]. CH₃

## 8-(4-Hydroxy-2-methylphenyl)-8-oxo-1-octanoic acid

[22812-00-6]  $C_{15}H_{20}O_{4}$ mol. wt. 264.32 Synthesis CH₃  $CO(CH_2)_6CO_2H$  -Refer to: [584]. m.p. 95° [584].

## 8-(4-Hydroxy-3-methylphenyl)-8-oxo-1-octanoic acid

[22811-96-7]	$C_{15}H_{20}O_4$	mol. wt. 264.32
CH ₃ HO	Synthesis -Refer to: [584]. m.p. 102° [584].	

# **Chapter 7** Nonanones

#### 1 Aromatic Hydroxyketones Derived from Nonanoic Acids

## 1.1 Unsubstituted Hydroxyketones

## 1-(2-Hydroxyphenyl)-1-nonanone

[22362-60-3]

C15H22O2

mol. wt. 234.34

Syntheses CO(CH₂)₇CH₃ -Phenol was energetically mixed with finely divided aluminium chloride (1 mol) and heated to  $100^{\circ}$  (1 mol of hydrochloric acid is evolved); 1 mol. of n-pelargonoyl chloride was then added and heated to  $125-130^{\circ}$  for 1 h (55 %) [2700].

-Also obtained by stirring a solution of salicylaldehyde, 1-octene,  $RhCl(PPh_3)_3$ , acetonitrile and sodium acetate in methylene chloride at r.t. for 8 h under an argon atmosphere (82 %) [1434].

colourless oil [1434]; b.p.₁₀ 180° [2700]; m.p. 18.4° [2700]; ¹H NMR [1434] (Sadtler standard N° 38630 M); IR [1434] (Sadtler standard N° 65679 K), UV [1996], MS [1434];  $n_{\rm D}^{25.5} = 1.5139$  [2700].

$C_{15}H_{23}NO_2$	mol. wt. 249.35
	$C_{15}H_{23}NO_2$

USE: Blocking agent, binders containing blocked PAPI and phenolic resins for wood composites [2077].

## 1-(3-Hydroxyphenyl)-1-nonanone

[859995-51-0]	$C_{15}H_{22}O_2$	mol. wt. 234.34
OH CO(CH ₂ ) ₇ CH ₃	Syntheses -Obtained by treatment of its methyl chloride in benzene by heating on (80 %) [966]. -Also refer to: [967].	

m.p. 54° [966, 967].

## **2,4-Dinitrophenylhydrazone** $C_{21}H_{26}N_4O_5$ mol. wt. 414.46

m.p. 101° [967].

**Methyl ether** [172264-98-1] C₁₆H₂₄O₂ mol. wt. 248.37

-Obtained by condensation of dodecylmagnesium bromide with m-methoxybenzamide in ethyl ether. The reaction was carried out at  $60^{\circ}$  for 48 h under hydrogen atmosphere (60-70 %) [966].

-Also refer to: [967, 1213, 1626].

b.p.₁₄ 170° [966, 967]; ¹H NMR [1626], ¹³C NMR [1626];  $n_D^{37} = 1.5070$  [966].

## 2,4-Dinitrophenylhydrazone of the methyl ether C22H28N4O5 mol. wt. 428.49

m.p. 90° [967].

 $CO(CH_2)_7CH_3$ 

## 1-(4-Hydroxyphenyl)-1-nonanone

[14392-69-9]	$C_{15}H_{22}O_2$	mol. wt. 234.34
ОН	Syntheses	
人	-Phenol was energetically mixed with f	inely divided aluminium

-Phenol was energetically mixed with finely divided aluminium chloride (1 mol) and heated to  $100^{\circ}$  (1 mol of hydrochloric acid is evolved); 1 mol. of n-pelargonoyl chloride was then added and heated to  $125-130^{\circ}$  for 1 h (35 %) [2700].

-Also obtained by reaction of nonanoyl chloride with phenol in the presence of aluminium chloride,

*in methylene chloride [64] for 14 h at r.t. (33 %) [1910];

*in nitrobenzene at r.t. overnight (70 %) [1769].

-Also obtained by Fries rearrangement of phenyl nonanoate with aluminium chloride in nitrobenzene at  $38^{\circ}$  for 2 days (60 %) [414].

-Also refer to: [62, 368, 442, 1730, 2658, 3100, 3457].

Isolation from natural sources

-From a cell suspension culture of Humulus lupulus cv Wye Northdown [602].

viscous oil [602]; b.p.₄ 195–199° [1769], b.p.₁₀ 232° [2700];
white solid [1910];
m.p. 57.2–57.6° [1910], 55.5–56.5° [1730], 55° (Sadtler standard N° 65675 K), 54.5° [2700], 50–52° [414], 42–46° [1769];
¹H NMR [602, 1910] (Sadtler standard N° 38626 M),
¹³C NMR [602, 1910];
IR [602, 1910] (Sadtler standard N° 65675 K), UV [1995],
MS [602, 1910]; TLC [1910]; GC [1910].

BIOLOGICAL ACTIVITY: Inhibition of  $17-\beta$  hydroxysteroid dehydrogenase 3 [1910]; Binary classification models for endocrine disrupter effects mediated through the estrogen receptor [2640].

**Methyl ether** [52754-68-4] C₁₆H₂₄O₂ mol. wt. 248.37

-Obtained from 1-nonene and  $(p-MeO-C_6H_4)I^+PhBF_4^-$  (69 %) [1592].

-Also obtained by electrolyzing in an undivided cell a DMF solution containing n-octyl iodide and 4-iodoanisole, iron pentacarbonyl and a catalytic amount of a nickel 2,2'-bipyridine complex (35 %) [894].

-Also obtained by reaction of 1-(4-methoxyphenyl)-4-iodo-1-butanone with dipentylzinc in the presence of Ni(acac)₂ in THF/NMP and acetophenone at  $-35^{\circ}$  (71 %) [1122].

-Obtained from 4-methoxybenzaldehyde and 1-octene (93 %) [3211].

-Also refer to: [1121, 1200, 1202, 2901, 3211, 3250].

m.p. 43° [2901]; ¹H NMR [894, 3211], ¹³C NMR [894, 3211], IR [894], MS [894, 3211].

USE: Photolysis of, energy transfer in, [3250].

Phenylhydrazone of the methyl ether		$C_{22}H_{30}N_2O$	mol. wt. 338.49
m.p. 48° [2901].			
4-Bromophenyl ether	[74261-29-3]	$C_{21}H_{25}BrO_2$	mol. wt. 389.33

m.p. 79° [866].

USE: Prepn. nonmesogenic [865].

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

[862666-37-3]  $C_{15}H_{22}O_3$  mol. wt. 250.34 OH HO CO(CH₂)₇CH₃ -Obtained by deprotection of its dimethyl ether with boron tribromide in methylene chloride at 0°. Then, the mixture was stirred overnight at r.t. (80 %) [82].

brown solid [82]; m.p. 41° [82]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82].

USE: Reactant for preparation of lithium gallium/titanium ketone complexes [82].

**Dimethyl ether** [862666-31-7] C₁₇H₂₆O₃ mol. wt. 278.39

-Obtained by treatment of 1-(2,3-dimethoxyphenyl)-1-nonanol in acetone with potassium dichromate in dilute sulfuric acid (Jones reagent) at r.t. for 3 h (61 %) [82].

colourless oil [82]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82].

## 1-(2,4-Dihydroxyphenyl)-1-nonanone

b.p.₁₂ 245–248° [893]; m.p. 73° [2704], 65° [2114], 50° [2673]. **N.B.**: There is a large disparity between the various melting points. ¹H NMR [3242].

USE: As colour developer [2704].

BIOLOGICAL ACTIVITY: Antifungal [2112, 2114].

**2,4-Dinitrophenylhydrazone** [95282-26-1] C₂₁H₂₆N₄O₆ mol. wt. 430.46 m.p. 145° [2673].

### 1-(2,5-Dihydroxyphenyl)-1-nonanone

$$\begin{array}{c} C_{15}H_{22}O_3 \\ OH \\ CO(CH_2)_7CH_3 \\ OH \\ OH \\ \end{array} \begin{array}{c} \text{Synthesis} \\ -\text{Refer to: [2874].} \\ \text{Dimethyl ether} \\ C_{17}H_{26}O_3 \\ 0H \\ \end{array} \begin{array}{c} \text{mol. wt. 250.34} \\ \text{mol. wt. 278.39} \\ \text{mol. wt. 278.39} \\ \end{array}$$

-Obtained by reaction of pelargonic acid chloride with hydroquinone dimethyl ether in the presence of aluminium chloride in carbon disulfide first at  $0^\circ$ , kept overnight, heated to a boil [2874].

b.p.3 180-182° [2874].

#### 1-(3,4-Dihydroxyphenyl)-1-nonanone

4-Nonanoylcatechol

$$C_{15}H_{22}O_3$$
 mol. wt. 250.34

Synthesis

OH -Obtained by Fries rearrangement of pyrocatechol nonanoate (1 mol) with aluminium chloride (2 mol) in the presence of pyrocatechol (1 mol) in nitrobenzene at  $80-100^{\circ}$  for 2 h (CH₂)₇CH₃ (40 %) [1283]. m.p. 92–93° [1283].

## **Dimethyl ether**

-Refer to: [1347].

¹H NMR [1347], ¹³C NMR [1347], IR [1347], UV [1347], MS [1347].

## 1-(3,5-Dihydroxyphenyl)-1-nonanone

$$C_{15}H_{22}O_3$$
 mol. wt. 250.34

Isolation from natural sources -From the roots and stems of *Ardisia virens* Kurz (Myrsinaceae) [607].

H₂)₇CH₃ -From the roots of Ardisia cornudentata Mez. [607].

colourless needles [607], m.p. 99–100° [607], ¹H NMR [607], ¹³C NMR [607], IR [607], UV [607], MS [607].

mol. wt. 278.39

$$C_{17}H_{26}O_3$$

## **Dimethyl ether**

 $C_{17}H_{26}O_3$ 

mol. wt. 278.39

-Obtained by reaction of n-octylmagnesium bromide with 3,5-dimethoxybenzamide (76 %) [30].

-Preparation in the usual way [25 (77 %), 2990].

b.p._{0.03} 150–156° [30], b.p.₁ 180° [25]; m.p. 35.5–36° [30].

## 1-(2,4,6-Trihydroxyphenyl)-1-nonanone

(Phlorononaphenone) [1026]

 $[74478-11-8] C_{15}H_{22}O_4 mol. wt. 266.34$ 

Syntheses
 -Obtained by reaction of nonanoyl chloride with phloroglucinol in the presence of aluminium chloride in nitrobenzene and carbon disulfide mixture (57 %) [2113].

-Also refer to: [1026, 2111, 2781].

pale yellow needles [2113]; m.p. 82° [2781], 109° [2113]; **N.B.**: One of the reported melting point is obviously wrong. IR [2113], UV [2113].

BIOLOGICAL ACTIVITY: Antifungal [2111, 2113].

## **Trimethyl ether**

 $C_{18}H_{28}O_4$ 

mol. wt. 308.42

mol. wt. 266.34

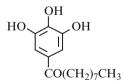
-Refer to: [16]; m.p. 34° [16].

## 1-(3,4,5-Trihydroxyphenyl)-1-nonanone

[100079-26-3]

C₁₅H₂₂O₄ Syntheses

-Refer to: [1969, 3248].

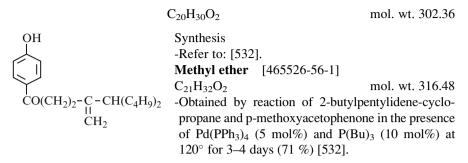


BIOLOGICAL ACTIVITY: As platelet aggregation inhibitor and antiallergic agent [3248].

## 1-(4-Hydroxyphenyl)-2-methyl-1-nonanone

[120837-31-2] [120837-03-8] (+)	$C_{16}H_{24}O_2$	mol. wt. 248.37
OH	Syntheses -Refer to: [1321, 1322, 2277]. <b>Methyl ether</b> [179037-21-9]	
CO-CH-(CH ₂ ) ₆ CH ₃	C ₁₇ H ₂₆ O ₂	mol. wt. 262.39

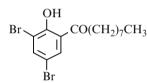
#### 5-Butyl-1-(4-hydroxyphenyl)-4-methylene-1-nonanone



#### Substituted Hydroxyketones 1.2

#### 1-(3,5-Dibromo-2-hydroxyphenyl)-1-nonanone

$$C_{15}H_{20}Br_2O_2$$
 mol. wt. 392.13



Synthesis  $CO(CH_2)_7CH_3 \quad -\text{Refer to: [2002].}$ m.p. 55° (Sadtler standard N° 65681 K); ¹H NMR (Sadtler standard N° 38632 M); IR (Sadtler standard N° 65681 K).

### 1-(3,5-Dibromo-4-hydroxyphenyl)-1-nonanone

 $C_{15}H_{20}Br_2O_2$ mol. wt. 392.13



Synthesis -Refer to: [2002]. m.p.  $38^{\circ}$  (Sadtler standard N° 65677 K); ¹H NMR (Sadtler standard N° 38628 M);  $CO(CH_2)_7CH_3$  IR (Sadtler standard N° 65677 K).

#### 1-(3,5-Dibromo-2,4-dihydroxyphenyl)-1-nonanone

[76092-86-9]	$C_{15}H_{20}Br_2O_3$	mol. wt. 408.13
OH Br CO(CH ₂ ) ₇ CH ₃	Synthesis -Refer to: [2112].	
HOBr	BIOLOGICAL ACTIVITY: contg., [2112]	Antifungal compns.

## 1-(4-Chloro-2-hydroxyphenyl)-1-nonanone

## 1-(5-Chloro-2-hydroxyphenyl)-1-nonanone

## 1-(3-Fluoro-4-hydroxyphenyl)-1-nonanone

	$C_{15}H_{21}FO_2$	mol. wt. 252.33
OH	Synthesis	
∠~F	-Refer to: [2363].	
	Methyl ether [136936-71-5]	
Ý	$C_{16}H_{23}FO_2$	mol. wt. 266.36
CO(CH ₂ ) ₇ CH ₃		

-Preparation by Friedel-Crafts acylation of 2-fluoroanisole with nonanoyl chloride (75 %) [2363].

## 1-(5-Fluoro-2-hydroxyphenyl)-1-nonanone

 $\begin{array}{cccc} [1396756\text{-}56\text{-}1] & C_{15}H_{21}FO_2 & \text{mol. wt. } 252.33 \\ \\ OH & Synthesis \\ -Refer \ to: \ [3242]. \\ colourless \ crystal \ [3242]; & \text{m.p. } 40\text{-}42^\circ \ [3242]; \\ H \ NMR \ [3242], \ ^{13}C \ NMR \ [3242], \ MS \ [3242]. \end{array}$ 

## 1-(5-Iodo-2-hydroxyphenyl)-1-nonanone

[1396756-58-3]	$C_{15}H_{21}IO_2$	mol. wt. 360.24
OH CO(CH ₂ ) ₇ CH ₃	Synthesis -Refer to: [3242]. colourless crystal [3242]; m.p. 52–54° ¹ H NMR [3242], ¹³ C NMR [3242], MS	[3242]; [3242].

## 1-(3,5-Dichloro-2,4-dihydroxy-6-methoxyphenyl)-1-nonanone

[118191-35-8]	$C_{16}H_{22}Cl_2O_4$	mol. wt. 349.25
$\begin{array}{c} OH\\ Cl\\ HO\\ Cl\\ Cl\\ Cl\\ Cl\\ Cl\\ Cl\\ Cl\\ Cl\\ Cl\\ Cl$	Synthesis -Obtained by reaction of chlor 6-methoxynonanophenone in ¹ H NMR [2012], MS [2012].	

BIOLOGICAL ACTIVITY: Differentiation-inducing factor from the slime mould Dictyostelium discoideum [2012].

## 1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-1-nonanone

[118191-36-9]	$C_{16}H_{22}C$	$1_{2}O_{4}$		mol. wt. 3	49.25
$Cl \rightarrow CO(CH_2)_7CH_3$ $CH_3O \rightarrow Cl OH$ $OH$ $OH$ $OH$ $OH$	Synthesis -Obtained 2,6-dihydro water [201 ¹ H NMR [2	2].		chlorine bhenone	with in

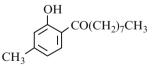
BIOLOGICAL ACTIVITY: Differentiation-inducing factor from the slime mould Dictyostelium discoideum [2012].

## 2,4,6-Trihydroxy-3-nonanoylbenzaldehyde

(	$C_{16}H_{22}O_5$	mol. wt. 294.35
CO(CH ₂ ) ₇ CH ₃	Synthesis -Refer to: [3408].	
		• • • • • • • • • • • • • • • • • • • •

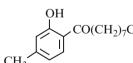
**BIOLOGICAL ACTIVITY: Effects on transpiration** and stomatal closure [3408].

## 1-(2-Hydroxy-4-methylphenyl)-1-nonanone



Synthesis -Preparation by Fries rearrangement of m-cresyl pelargonate with aluminium chloride at 120-140° for 10-20 min (75 %) [243].

b.p.4 175-177° [243].



CHC

HO

## 1-(2-Hydroxy-5-methylphenyl)-1-nonanone

[75487-43-3] 
$$C_{16}H_{24}O_2$$
 mol. wt. 248.37  
OH Syntheses  
CO(CH₂)₇CH₃ -Refer to: [1355, 1356].

## 1-(2-Hydroxy-6-methylphenyl)-1-nonanone

 $\begin{array}{cccc} [1396756\text{-}55\text{-}0] & C_{16}H_{24}O_2 & \text{mol. wt. } 248.37 \\ & OH & Synthesis \\ & CO(CH_2)_7CH_3 & -\text{Refer to: } [3242]. \\ & Colourless \ crystal \ [3242]; & ^1H \ \text{NMR} \ [3242], \\ & ^{13}C \ \text{NMR} \ [3242]. \end{array}$ 

## 1-(3,5-Dihydroxy-4-methylphenyl)-1-nonanone

	$C_{16}H_{24}O_3$	mol. wt. 264.36
HO CO(CH ₂ ) ₇ CH ₃	Synthesis -Refer to: [3132]. <b>Dimethyl ether</b> [196869-48-4] C ₁₈ H ₂₈ O ₃	mol. wt. 292.42

-Refer to: [3132 (80 %)].

colourless crystalline solid [3132]; ¹H NMR [3132], ¹³C NMR [3132].

USE: Preparation of striatol and related compounds [3132].

## 1-(2-Hydroxy-4-methoxyphenyl)-1-nonanone

[43221-42-7]	$C_{16}H_{24}O_3$	mol. wt. 264.36
CH ₃ O ^{OH} CO(CH ₂ ) ₇ CH ₃	Synthesis -Refer to: [3092, 3242]. m.p. 48.5–49° [3092]; ¹ H NMR [3242], ¹³ C NMR [32	42], MS [3242].

USE: Light stabilizer, for polyolefins, [3092].

### 1-(4-Hydroxy-3-methoxyphenyl)-1-nonanone

[143378-82-9]  $C_{16}H_{24}O_3$  mol. wt. 264.36 OH Syntheses -Refer to: [1427, 1429]. CO(CH₂)₇CH₃

### 1-(2,4,6-Trihydroxy-3-methylphenyl)-1-nonanone

	$C_{16}H_{24}O_4$	mol. wt. 280.36
CH ₃ CO(CH ₂ ) ₇ CH ₃	Syntheses -Refer to: [1026, 1501, 1502].	
ностран	BIOLOGICAL ACTIVITY: Anti 1501, 1502].	microbial [1026,

### 1-(4-Hydroxy-2,5-dimethylphenyl)-1-nonanone

 $[95102-38-8] \qquad C_{17}H_{26}O_2 \qquad \text{mol. wt. } 262.39$   $OH \qquad Syntheses \\ -Refer to: [1595, 2704] (Japanese patents).$ 

 $CH_3$  USE: In preparation of thermographic recording material [1595].

### 1-(4-Ethoxy-2-hydroxyphenyl)-1-nonanone

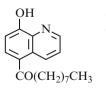
C ₁	$_{7}H_{26}O_{3}$	mol. wt. 278.39
C2H5O	Synthesis -Refer to: [1835]. <b>Oxime</b> [33488-76-5] C ₁₇ H ₂₇ NO ₃	mol. wt. 293.41

USE: Hydrogen bonding, spectra in relation to, [1835].

### 1-(8-Hydroxy-5-quinolinyl)-1-nonanone

[79111-56-1]

$$C_{18}H_{23}NO_2$$
 mol. wt. 285.39



Syntheses -Obtained by reaction of nonanoyl chloride with 8-hydroxyquinoline in the presence of aluminium chloride in nitrobenzene or 1,2-dichloroethane first at  $10-30^{\circ}$ , then at  $70-80^{\circ}$  for 20 h [2261].

-Also refer to: [938, 2638, 3108, 3173].

m.p. 66–67° [938], 64–65° [2261], 62–63° [3173]; ¹H NMR [3173], ¹³C NMR [2261, 2638], IR [3173], MS [2261].

USE: For extraction of gallium [3172]; Amebicidal action [3108]

### **Hydrochloride** [79111-55-0] C₁₈H₂₃NO₂, HCl mol. wt. 321.85

-Refer to: [3173].

m.p. 163-164° [3173].

### **Hydrobromide** $C_{18}H_{23}NO_2$ , HBr mol. wt. 366.30

-Obtained by reaction of pelargonyl chloride with 8-quinolinol in the presence of aluminium chloride in nitrobenzene first at  $75^{\circ}$  for 16 h, then at r.t. for 20 h. Then, the acetone solution was saturated with hydrogen bromide (17 %) [938].

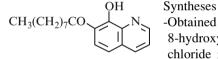
m.p. 229-231° (d) [938].

### 1-(8-Hydroxy-7-quinolinyl)-1-nonanone

[79111-54-9]

 $C_{18}H_{23}NO_2$ 

mol. wt. 285.39



-Obtained by reaction of nonanoyl chloride with 8-hydroxyquinoline in the presence of aluminium chloride in nitrobenzene or 1,2-dichloroethane first at 10–30°, then at 70–80° for 20 h [2261]. -Also refer to: [2638, 3171, 3173].

m.p. 64° [2261], 63–64° [3173]; ¹H NMR [3173], ¹³C NMR [2638], IR [3173], MS [2261].

	Hydrazone	O mol. wt. 299.	$C_{18}H_{25}N_{3}O$	[88559-43-7]	Hydrazone
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m.p. 98–101° [3171].

### 1-(4-Hydroxy-3-methyl-2-quinolinyl)-1-nonanone

[331749-02-1]	$C_{19}H_{25}NO_2$	mo	ol. wt. 299.41
CO(CH ₂ ) ₇ CH ₃	Synthesis -Refer to: [2086].		
$\sim$ $\gamma$ CH ₃	<b>BIOLOGICAL ACTIVITY:</b>	In vitro	antibacterial

BIOLOGICAL ACTIVITY: *In vitro* antibacterial activity against Helicobacter pylori [2086].

Acetate [331749-03-2] C₂₁H₂₇NO₃ mol. wt. 341.45

-Refer to: [279].

USE: Preparation of antibacterial quinoline derivatives for inhibition of Helicobacter pylori [2086].

### 1-(8-Hydroxy-2-methyl-5-quinolinyl)-1-nonanone

 $\label{eq:constraint} [217815-22-0] \qquad \qquad \text{C}_{19}\text{H}_{25}\text{NO}_2 \qquad \qquad \text{mol. wt. } 299.41$ 

Synthesis

-Obtained by reaction of nonanoyl chloride with 8-hydroxyquinaldine in the presence of aluminium chloride in nitrobenzene or 1,2-dichloroethane first at  $10-30^{\circ}$ , then at  $70-80^{\circ}$  for 20 h [2261].

m.p. 72° [2261]; ¹H NMR [2261], ¹³C NMR [2261], MS [2261].

### 1-(8-Hydroxy-2-methyl-7-quinolinyl)-1-nonanone

[217815-25-3]

CO(CH₂)₇CH₃

OH

CH₃

-Obtained by reaction of nonanoyl chloride with 8-hydroxyquinaldine in the presence of aluminium chloride in nitrobenzene or 1,2-dichloroethane first at  $10-30^{\circ}$ , then at  $70-80^{\circ}$  for 20 h [2261].

m.p. 58–60° [2261]; ¹H NMR [2261], ¹³C NMR [2261], MS [2261].

**Synthesis** 

### 1-(5,8-Dihydroxy-4-methyl-2-quinolinyl)-1-nonanone

C ₁₉ H ₂₅ NO ₃			mol. wt. 315.41
OH N CO(CH ₂ ) ₇ CH ₃	Synthesis -Refer to: [2266]. <b>Dimethyl ether</b> $[C_{21}H_{29}NO_3]$	67188-50-5]	mol. wt. 343.47

-Obtained by reaction of n-octylmagnesium bromide with 2-cyano-5,7-dimethoxy-4-methyl-quinoline in tetrahydrofuran (65 %) [2266].

m.p. 76–79° [2266].

#### 1-(6,7-Dihydroxy-5-propyl-1,3-benzodioxol-4-yl)-1-nonanone

 $\begin{array}{cccc} C_{19}H_{28}O_5 & \text{mol. wt. 336.43} \\ OH & Synthesis \\ OH & -Refer to: [2179]. \\ \hline Dimethyl ether & (Pelargonyl dihydrodillapiole) \\ C_3H_7 & [82652-29-7] & C_{21}H_{32}O_5 & \text{mol. wt. 364.48} \\ \hline CO(CH_2)_7CH_3 & -Refer to: [2179]. \\ \hline Dimethyl ether & (Pelargonyl dihydrodillapiole) \\ \hline C_2 H_3 = 0 \\ \hline C_3 H_7 & -Refer to: [2179]. \\ \hline Dimethyl ether & (Pelargonyl dihydrodillapiole) \\ \hline C_3 H_7 & -Refer to: [2179]. \\ \hline Dimethyl ether & (Pelargonyl dihydrodillapiole) \\ \hline C_3 H_7 & -Refer to: [2179]. \\ \hline Dimethyl ether & (Pelargonyl dihydrodillapiole) \\ \hline C_3 H_7 & -Refer to: [2179]. \\ \hline Dimethyl ether & (Pelargonyl dihydrodillapiole) \\ \hline C_3 H_7 & -Refer to: [2179]. \\ \hline Dimethyl ether & (Pelargonyl dihydrodillapiole) \\ \hline C_3 H_7 & -Refer to: [2179]. \\ \hline Dimethyl ether & (Pelargonyl dihydrodillapiole) \\ \hline C_3 H_7 & -Refer to: [2179]. \\ \hline Dimethyl ether & (Pelargonyl dihydrodillapiole) \\ \hline C_3 H_7 & -Refer to: [2179]. \\ \hline Dimethyl ether & (Pelargonyl dihydrodillapiole) \\ \hline C_3 H_7 & -Refer to: [2179]. \\ \hline Dimethyl ether & (Pelargonyl dihydrodillapiole) \\ \hline C_3 H_7 & -Refer to: [2179]. \\ \hline Dimethyl ether & (Pelargonyl dihydrodillapiole) \\ \hline C_3 H_7 & -Refer to: [2179]. \\ \hline C_3 H_7 & -Refer to: [2170]. \\ \hline C_3 H_7 & -Refer to: [2170]. \\ \hline C_3 H_7 & -Refer to: [2170]. \\ \hline C_3 H_7 & -Refer to:$ 

-Obtained by reaction of pelargonyl chloride with dihydrodillapiole in the presence of fused zinc chloride in methylene chloride for 3–4 h in an ice bath (70 %) [2179].

colourless solid [2179]; ¹H NMR [2179], IR [2179].

USE: Synergistic insecticidal activity of, with pyrethrum [2179].

### 1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-nonanone

 $\begin{array}{ccc} C_{19}H_{30}O_2 & \text{mol. wt. } 290.45 \\ OH & Synthesis \\ -Obtained (\textbf{XXIX}) by treatment of 1-[4-methoxy-2-methyl-5-(1-methylethyl)phenyl]-1-nonanone \\ (\textbf{X}) with boiling pyridinium chloride (205–215°) for \\ CO(CH_2)_7CH_3 & 5.5 h (7 \%) [2660]. \end{array}$ 

b.p.₁₇ 248–252° [2660]; m.p. 62° [2660].

### Methyl ether (X)

-Obtained by reaction of pelargonyl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (47 %) [2660].

C20H32O2

b.p.₁₄ 222° [2660];  $n_D^{23} = 1.5155$  [2660].

### 1-(4-Butoxy-2-hydroxyphenyl)-1-nonanone

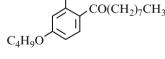
 $C_{19}H_{30}O_3$ 

mol. wt. 306.45

mol. wt. 304.47

Synthesis

CO(CH₂)₇CH₃ -Obtained by reaction of butyl bromide with 1-(2,4-dihydroxyphenyl)-1-nonanone in the presence of potassium carbonate in refluxing acetone for 20 h [284].



m.p. 33-34° [284].

-Obtained by reaction of hydroxylamine hydrochloride with 1-(4-butoxy-2-hydroxyphenyl)-1-nonanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 43–45° [284].

### 1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-nonanone

2-Nonanoyl-4-(3-methylbuten-2-yl)phloroglucinol (10) [1026]

[85602-20-6]

C20H30O4

mol. wt. 334.46

	OF	I
$CH_2 - C = CH$		CO(CH ₂ ) ₇ CH ₃
СП3	HO	∿он

Syntheses

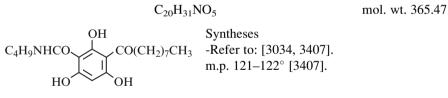
-Obtained by adding cuprous chloride, a saturated aqueous solution of sodium carbonate and prenyl bromide to a solution of phlorononanophenone in ethyl ether at r.t., then to keep the mixture for 12 h at this temperature [1026].

-Also prepared by adding prenyl bromide to a suspension of the sodium salt of phlorononanophenone in benzene, then the mixture obtained was refluxed for 3 h [1026]. -Also refer to: [3193].

¹³C NMR [1026], IR [1026].

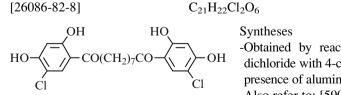
BIOLOGICAL ACTIVITY: Bactericidal and fungicidal activity of [3193].

### 1-[3-(Butylaminocarbonyl)-2,4,6-trihydroxyphenyl]-1-nonanone



BIOLOGICAL ACTIVITY: Cytotoxicity [3034]; Inhibition of Epstein-Barr virus early antigen activation [3034].

### 1,9-Bis(5-chloro-2,4-dihydroxyphenyl)-1,9-nonanedione



m.p. 157° [590, 591]; IR [590].

mol. wt. 441.31

-Obtained by reaction of azelaic acid dichloride with 4-chlororesorcinol in the presence of aluminium chloride [591]. -Also refer to: [590].

Dioxime [37166-91-9]  $C_{21}H_{24}Cl_2N_2O_6$ mol. wt. 471.34

m.p. 165–166° [590].

**Tetramethyl ether** [26086-85-1] C25H30Cl2O6 mol. wt. 497.42

-Obtained by reaction of azelaic acid dichloride with 4-chlororesorcinol dimethyl ether in the presence of aluminium chloride [591]. -Also refer to: [590].

m.p. 147° [590, 591]: IR [590].

### **Dioxime of the tetramethyl ether** [37166-94-2] C₂₅H₃₂Cl₂N₂O₆ mol. wt. 527.45

m.p. 140° [590].

### 1,9-Bis(4-hydroxyphenyl)-1,9-nonanedione

[2533-62-2] C21H24O4 mol. wt. 340.42 Syntheses HO-_____OCO(CH₂)₇CO-____OH -Obtained by reaction of azelaic acid dichloride (azeloyl chloride) with phenol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for

1 h, then at r.t. for 4 h [585].

-Also refer to: [1337].

rectangular plates [585]; m.p. 146.5–147° [1337], 142° [585].

**Di-2,4-dinitrophenylhydrazone** [2533-60-0]  $C_{33}H_{32}N_8O_{10}$  mol. wt. 700.67 m.p. 240° [585].

Diacetate	[2533-61-1]	$C_{25}H_{28}O_{6}$	mol. wt. 424.49
m.p. 100.5–1	.02° [1337].		

Dimethyl ether	[2525-89-5]	$C_{23}H_{28}O_4$	mol. wt. 368.47
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-Obtained by reaction of azelaic acid dichloride with anisole in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

m.p. 99° [585].

### Di-2,4-dinitrophenylhydrazone of the dimethyl ether

[24336-93-4] C35H36N8O10 mol. wt. 728.72 m.p. 130° [585].

### 1,9-Bis(2,4-dihydroxyphenyl)-1,9-nonanedione

[7640-25-7] C₂₁H₂₄O₆ mol. wt. 372.42 OH HO Syntheses

$$HO \longrightarrow CO(CH_2)_7CO \longrightarrow OH$$

-Obtained by adding resorcinol into the dinitrile of azelaic acid and hydrogen chloride in ethyl ether in the presence of zinc chloride.

Then, the diketimine dichloride obtained was hydrolyzed by boiling water (79 %) [2674].

-Also obtained by reaction of azelaic acid with resorcinol in the presence of zinc chloride at  $140^{\circ}$  for 5 h (60 %) [445].

-Also obtained by reaction of azelaic acid dichloride with resorcinol in the presence of aluminium chloride [591].

-Also refer to: [445, 590, 1735, 2504].

yellow crystals [445]; m.p. 141–143° [445], 116° [590], 115° [591], 114° [2504, 2674]; **N.B.**: One of the reported melting points is obviously wrong. IR [590], UV [2504].

**N.B.**: Apparently the m.p.  $141^{\circ}$  was erroneously overstated [2674]. This diketone obtained according to the method in [445] had a m.p. of  $114^{\circ}$ ; a mixed m.p. with the diketone synthetized according to Hoesh, showed no depression [2674].

**Dioxime** [37166-86-2]  $C_{21}H_{26}N_2O_6$  mol. wt. 402.44

m.p. 217° [590].

**Di-2,4-dinitrophenylhydrazone** [124141-66-8] C₃₃H₃₂N₈O₁₂ mol. wt. 732.66

m.p. 275° [2674].

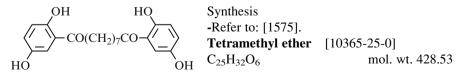
**Tetramethyl ether** [37166-89-5] C₂₅H₃₂O₆ mol. wt. 428.53

-Refer to: [590].

m.p. 96° [590]; IR [590].

### 1,9-Bis(2,5-dihydroxyphenyl)-1,9-nonanedione

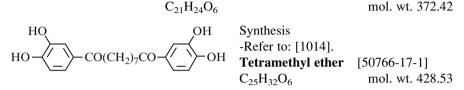
C₂₁H₂₄O₆ mol. wt. 372.42



-Obtained by reaction of azelaic acid dichloride (1 mol) with hydroquinone dimethyl ether (2.5 mol) in the presence of aluminium chloride (1 mol) (73 %) [1575].

m.p. 42° [1575].

### 1,9-Bis(3,4-dihydroxyphenyl)-1,9-nonanedione



-Obtained by reaction of azelaic acid dichloride with pyrocatechol dimethyl ether in the presence of aluminium chloride [591].

-Also obtained by hydrogenating of its oxime in acetic acid in the presence of 10 % Pd/C in a Parr apparatus at r.t. and 45–50 psig for 12–36 h (66 %) [1014]. -Also refer to: [1012, 2342, 3364].

m.p. 102–103° [3059], 102° [591], 100–101° [3363], 98–100° [1014]; ¹H NMR [2342], ¹³C NMR [2342], IR [3364].

### Dioxime of the tetramethyl ether [50766-31-9] $C_{25}H_{34}N_2O_6$ mol. wt. 458.56

m.p. 111–113° [1012], 108–110° [1014].

### 1,9-Bis(2,3,4-trihydroxyphenyl)-1,9-nonanedione

 $\begin{array}{ccc} C_{21}H_{24}O_8 & \text{mol. wt. 404.41} \\ HO & OH & HO & OH \\ HO & - & - OH & CO(CH_2)_7CO & - OH \\ HO & - & - OH & CO(CH_2)_7CO & - OH \\ Hexamethyl ether & [10475-18-0] \\ C_{27}H_{36}O_8 & \text{mol. wt. 488.58} \end{array}$ 

-Obtained by reaction of azelaic acid dichloride with pyrogallol trimethyl ether in the presence of aluminium chloride [591].

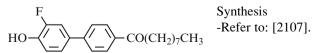
-Also obtained by reaction of dimethyl sulfate with 1,9-bis(2-hydroxy-3,4-dimethoxy-phenyl)-1,9-nonanedione the presence of 30 % sodium hydroxide (65–90 %) [1574]. -Also refer to: [1575].

m.p. 117° [591], 74° [1574, 1575]. **N.B.**: One the reported melting point is obviously wrong.

### 1-(3'-Fluoro-4'-hydroxy[1,1'-biphenyl]-4-yl)-nonanone

$$C_{21}H_{25}FO_2$$

mol. wt. 328.43



**3-Methylpentyl ether** (S) [112780-51-5]  $C_{27}H_{37}FO_2$  mol. wt. 413.26 USE: Liq.-crystal compns. contg., for display devices [2107].

**4-Methylhexyl ether** (S) [112815-87-9]  $C_{28}H_{39}FO_2$  mol. wt. 426.62 USE: Liq.-crystal compns. contg., for display devices [2107].

**5-Methylheptyl ether** (S) [112780-52-6]  $C_{29}H_{41}FO_2$  mol. wt. 440.64 USE: Liq.-crystal compns. contg., for display devices [2107].

**6-Methyloctyl ether** (S) [112780-53-7]  $C_{30}H_{43}FO_2$  mol. wt. 454.67 USE: Liq.-crystal compns. contg., for display devices [2107].

### 1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-1-nonanone

Acetate [121586-49-0] C₂₃H₂₈O₃ mol. wt. 352.47

-Refer to: [1647].

### Various ethers (11)

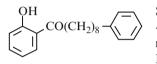
-Preparations and liquid crystalline properties of, [847].

Methyl ether	[56116-81-5]	$C_{22}H_{28}O_2$	mol. wt. 324.46
-Also refer to: [1923].			
Ethyl ether	[56116-90-6]	$C_{23}H_{30}O_2$	mol. wt. 338.49

Ethyl Cher		C231130C2	1101. wt. 550.47		
Propyl ether	[56116-98-4]	$C_{24}H_{32}O_2$	mol. wt. 352.52		
Butyl ether	[56117-06-7]	$C_{25}H_{34}O_2$	mol. wt. 366.54		
Pentyl ether	[56117-15-8]	$C_{26}H_{36}O_2$	mol. wt. 380.57		
Hexyl ether	[56117-24-9]	$C_{27}H_{38}O_2$	mol. wt. 394.60		
-Also refer to: [1233] (Japanese patent).					
Heptyl ether	[56189-90-3]	$C_{28}H_{40}O_2$	mol. wt. 408.62		
Octyl ether	[56117-41-0]	$C_{29}H_{42}O_2$	mol. wt. 422.65		

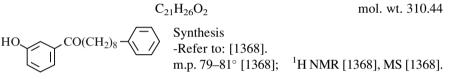
Nonyl ether	[56117-50-1]	$C_{30}H_{44}O_2$	mol. wt. 436.68
Decyl ether	[56117-59-0]	$C_{31}H_{46}O_2$	mol. wt. 450.71
Dodecyl ether	[56117-68-1]	$C_{33}H_{50}O_2$	mol. wt. 478.76

### 1-(2-Hydroxyphenyl)-9-phenyl-1-nonanone



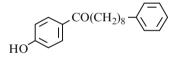
Synthesis -Refer to: [1368]. m.p. 47–48° [1368]; ¹H NMR [1368], IR [1368], MS [1368].

### 1-(3-Hydroxyphenyl)-9-phenyl-1-nonanone



### 1-(4-Hydroxyphenyl)-9-phenyl-1-nonanone

 $C_{21}H_{26}O_2$  mol. wt. 310.44



Synthesis -Refer to: [1368]. m.p. 45–47° [1368]; ¹H NMR [1368], IR [1368], MS [1368].

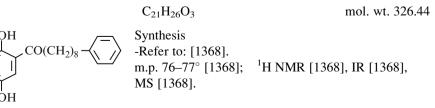
### 1-(2,4-Dihydroxyphenyl)-9-phenyl-1-nonanone

 $C_{21}H_{26}O_3$  mol. wt. 326.44



Synthesis -Refer to: [1368]. m.p. 49–50° [1368]; ¹H NMR [1368], IR [1368], MS [1368].

### 1-(2,5-Dihydroxyphenyl)-9-phenyl-1-nonanone



#### 1-(2,6-Dihydroxyphenyl)-9-phenyl-1-nonanone

(Malabaricone A)

-From the fruit rind of Myristica malabarica (Myristicaceae) [2528].

bright yellow crystals [1786]; crystals [719]; m.p. 81–82° [2528], 80–82° [719], 80–81° [1786]; ¹H NMR [2528], ¹³C NMR [1786, 2528], IR [2528], UV [2528], MS [2528].

#### 1-(2,6-Dihydroxyphenyl)-9-(4-hydroxyphenyl)-1-nonanone

(Malabaricone B)

 $[63335-24-0] \qquad C_{21}H_{26}O_4 \qquad \text{mol. wt. 342.44}$   $(OH) \qquad OH \qquad Isolation from natural sources -From stem bark of Myristica dactyloides (Myristicaceae) [1786]. -From seed kernel of Myristica dactyloides (Myristicaceae) [719].$ 

-From the fruit rind of Myristica malabarica (Myristicaceae) [2528].

pale yellow crystals [719]; m.p. 105–106° [1786], 102° [2528], 100–102° [719]; ¹H NMR [2528], ¹³C NMR [1786], IR [2528], UV [2528], MS [2528].

Trimethyl ether

[114226-21-0] C₂₄H₃₂O₄

mol. wt. 384.52

-Obtained by treatment of 1-(2-hydroxy-6-methoxyphenyl)-9-(4-hydroxyphenyl)-1-nonanone or 1-(2,6-dihydroxyphenyl)-9-(4-hydroxyphenyl)-1-nonanone with diazomethane in ethyl ether [1786].

¹H NMR [1786], IR [1786], MS [1786].

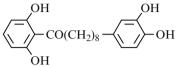
### 1-(2,4,6-Trihydroxyphenyl)-9-phenyl-1-nonanone

 $\begin{array}{c} C_{21}H_{26}O_4 & \text{mol. wt. 342.44} \\ & & \\ OH & \\ CO(CH_2)_8 & \\ OH &$ 

### 1-(2,6-Dihydroxyphenyl)-9-(3,4-dihydroxyphenyl)-1-nonanone

(Malabaricone C)

[63335-25-1] C21H26O5 mol. wt. 358.43



Isolation from natural sources -From mace, aril of Myristica fragrans HOUTT (Myristicaceae) [2231]. -From seed kernel of Myristica dactyloides (Myristicaceae) [719].

-From the fruit rind of Myristica malabarica (Myristicaceae) [2528].

pale yellow prisms [2231]; yellow crystals [719]: pale yellow crystals [2528]; m.p. 123–124° [2231, 2528], 122–124° [719]: ¹H NMR [2231, 2528], ¹³C NMR [2231, 2528], IR [2231, 2528], UV [2231, 2528], MS [2231, 2528]; TLC [2231].

BIOLOGICAL ACTIVITY: Nematocidal [2231].

**Tetramethyl ether** C25H34O5 mol. wt. 414.54

¹H NMR [1308], IR [1308], MS [1308].

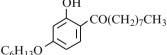
Tetraacetate C29H34O9

-Obtained by acetylation of malabaricone C with acetic anhydride in the presence of pyridine [2231].

oil [2231]; ¹H NMR [2231], MS [2231].

### 1-(4-Hexyloxy-2-hydroxyphenyl)-1-nonanone

[143286-93-5] C21H34O3 mol. wt. 334.50



Synthesis							
-Obtained	by	reaction	of	hexyl	bron	nide	with
1-(2,4-dib	nydr	oxyphenyl	l)-1	-nonan	one	in	the
presence	of	potassium	Са	arbonat	e in	reflu	ıxing
acetone for	or 2	0 h [284].					

m.p. 38–40° [284].

Oxime [143286-62-8] C₂₁H₃₅NO₃ mol. wt. 349.51

-Obtained hydroxylamine by reaction of hydrochloride with 1-(2,4-hydroxyphenyl)-1-nonanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 44–48° [284]; ¹H NMR [284].

mol. wt. 526.58

### 9-(1,3-Benzodioxol-5-yl)-1-(2,6-dihydroxyphenyl)-1-nonanone

(Malabaricone D)

 $[63335-26-2] \qquad C_{22}H_{26}O_5 \qquad \text{mol. wt. 370.45}$   $[63335-26-2] \qquad OH \qquad From stem bark of Myristica dactyloides (Myristicaceae) [1786]. -From seed kernel of Myristica dactyloides (Myristicaceae) [719].$ 

-From the fruit rind of Myristica malabarica (Myristicaceae) [2528].

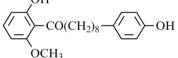
pale yellow crystals [719, 1786]; m.p. 90–91° [2528], 89–91° [719], 86–87° [1786]; ¹H NMR [2528], ¹³C NMR [1786], IR [2528], UV [2528], MS [2528].

**Dimethyl ether** [114226-19-6]  $C_{24}H_{30}O_5$  mol. wt. 398.50

-Obtained by treatment of the title ketone with diazomethane in ethyl ether [1786].

¹H NMR [1786], IR [1786], MS [1786].

### 1-(2-Hydroxy-6-methoxyphenyl)-9-(4-hydroxyphenyl)-1-nonanone



Isolation from natural sources -From stem bark of Myristica dactyloides (Myristicaceae) [1786]. pale yellow crystals [1786]; m.p. 65–66° [1786];

¹H NMR [1786], ¹³C NMR [1786], IR [1786], MS [1786].

### 1-[2,4,6-Trihydroxy-3-(phenylmethyl)phenyl]-1-nonanone

2-Nonanoyl-4-benzylphloroglucinol (24) [1026]

[85602-33-1]

C₆H₅CH₂·

C22H28O4

mol. wt. 356.46

Syntheses

-CO(CH₂)₇CH₃ -Obtained by adding cuprous chloride, a saturated aqueous solution of sodium carbonate OH and benzyl chloride to a solution of phlorononanophenone in ethyl ether at r.t., then to keep the mixture for 12 h at this temperature [1026].

-Also obtained by adding benzyl chloride to a two-phase mixture consisting of phlorononanophenone in diethyl ether and saturated aqueous sodium carbonate. A catalytic amount of CuCl was added and the mixture was stirred or shaken vigorously for 3 h at r.t. (48 %) [838].

-Also obtained by reaction of benzyl chloride with phlorononanophenone in an alkaline two-phase (aqueous-ether) system and catalyzed by CuCl [838, 3193]. -Also refer to: [839].

m.p. 131–132° [838]; ¹H NMR [1026], ¹³C NMR [838, 1026], IR [838, 1026], MS [838].

BIOLOGICAL ACTIVITY: Bactericidal, and fungicidal [1026, 3193].

### 1-(2,6-Dihydroxyphenyl)-9-(4-hydroxy-3-methoxyphenyl)-1-nonanone

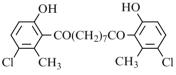
[113201-69-7]  $C_{22}H_{28}O_5$  mol. wt. 372.46 OH OCH₃ Isolation from natural sources -From seed kernel of *Myristica dactyloides* (Myristicaceae) [719]. White needles [719]; m.p. 109–111° [719];

¹H NMR [719], ¹³C NMR [719], IR [719], UV [719], MS [719].

### 1,9-Bis(3-chloro-6-hydroxy-2-methylphenyl)-1,9-nonanedione

C23H26Cl2O4

mol. wt. 437.36



Synthesis

-Obtained by reaction of azelaic acid dichloride (azeloyl chloride) with 4-chloro-3-methylphenol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

m.p. 105° [585].

### Di-2,4-dinitrophenylhydrazone

m.p. 200° [585].

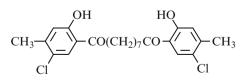
### 1,9-Bis(5-chloro-2-hydroxy-4-methylphenyl)-1,9-nonanedione

[25715-26-8]



mol. wt. 437.36

mol. wt. 797.61



Synthesis

C₃₅H₃₄Cl₂N₈O₁₀

-Obtained by reaction of azelaic acid dichloride (azeloyl chloride) with 4-chloro-3-methylphenol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

m.p. 105° [585].

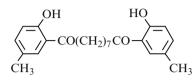
### Di-2,4-dinitrophenylhydrazone [25715-27-9] C₃₅H₃₄Cl₂N₈O₁₀ mol. wt. 797.61

m.p. 200° [585].

### 1,9-Bis(2-hydroxy-5-methylphenyl)-1,9-nonanedione

[113796-20-6]

C₂₃H₂₈O₄ Syntheses mol. wt. 368.47



-Obtained by Fries rearrangement of di (4-methyl-phenyl) azelate with aluminium chloride in refluxing chlorobenzene for 6 h (58 %) [3107].

-Also obtained by reaction of azelaic acid dichloride (azeloyl chloride) with p-cresol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

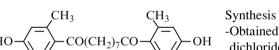
m.p. 79–80° [3107], 71° [585]; IR [3107].

### Di-2,4-dinitrophenylhydrazone

m.p. 84° [585].

### 1,9-Bis(4-hydroxy-2-methylphenyl)-1,9-nonanedione

[24336-97-8]



C35H36N8O10

mol. wt. 368.47

mol. wt. 728.72

-Obtained by reaction of azelaic acid dichloride (azeloyl chloride) with m-cresol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

m.p. 100° [585].

# $\label{eq:24339-82-0} \textbf{Di-2,4-dinitrophenylhydrazone} \quad [24339-82-0] \quad C_{35}H_{36}N_8O_{10} \quad \text{mol. wt. 728.72}$

C23H28O4

m.p. 250° [585].

### **Dimethyl ether**

 $C_{25}H_{32}O_4$  mol. wt. 396.53

-Obtained by methylation of 1,9-Bis(4-hydroxy-2-methylphenyl)-1,9-nonanedione [585].

m.p. 120° [585].

### 1,9-Bis(4-hydroxy-3-methylphenyl)-1,9-nonanedione

[24336-94-5]	C ₂₃ H ₂₈ O	4	mol. wt. 368.47
$HO \longrightarrow CO(CH_2)_7CO \longrightarrow$	СН3 5	⁴ Synthesis Obtained by reaction dichloride (azeloyl o-cresol in the presen chloride in nitrobenz for 1 h, then at r.t. for	a of azelaic acid chloride) with ice of aluminium ene first at 0–5°
needles [585]; m.p. 139°			
<b>Di-2,4-dinitrophenylhydrazo</b> m.p. 210° [585].	one (	$C_{35}H_{36}N_8O_{10}$	mol. wt. 728.72
Dimethyl ether [2433	36-96-7]	$C_{25}H_{32}O_4$	mol. wt. 396.53
-Obtained by reaction of azela presence of aluminium chlori 4 h [585].			•
prismatic rods [585]; m.p	. 140° [585].		
Di-2,4-dinitrophenylhydrazo of the dimethyl ether	one C	$C_{37}H_{40}N_8O_{10}$	mol. wt. 756.77

m.p. 200° [585].

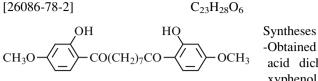
### 9-(1,3-Benzodioxol-5-yl)-1-(2-hydroxy-6-methoxyphenyl)-1-nonanone

[114226-25-4]	$C_{23}H_{28}O_5$	mol. wt. 384.47
$r = \langle r = \langle r \rangle$	Isolation from natural sou- From stem bark of (Myristicaceae) [1786]. Synthesis	Myristica dactyloides
01. 1 1		

-Obtained by treatment of 1-(2,6-dihydroxyphenyl)-9-(3,4-dihydroxyphenyl)-1-nonanone with diazomethane in ethyl ether [1786].

m.p. 51–52° [1786]; ¹H NMR [1786], ¹³C NMR [1786], IR [1786], MS [1786].

#### 1,9-Bis(2-hydroxy-4-methoxyphenyl)-1,9-nonanedione



-Obtained by reaction of azelaic acid dichloride with 3-methoxyphenol in the presence of aluminium chloride [591].

-Also refer to: [590].

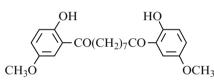
m.p. 89° [590, 591].

**Di-2,4-dinitrophenylhydrazone** [37402-33-8] C₃₅H₃₆N₈O₁₂ mol. wt. 760.72 m.p. 151° [590].

 $C_{23}H_{28}O_{6}$ 

### 1,9-Bis(2-hydroxy-5-methoxyphenyl)-1,9-nonanedione

[10365-30-7]



mol. wt. 400.47

Synthesis

-Obtained by reaction of azelaic acid dichloride with p-dimethoxybenzene in the presence of aluminium chloride [1575].

m.p. 73° [1575].

Diacetate	[10365-36-3]	$C_{27}H_{32}O_8$	mol. wt. 484.55
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-Obtained by reaction of acetic anhydride (4 vol) with the title ketone in the presence of 1-2 drops concentrated sulfuric acid, the mixture warmed 5 min [1575].

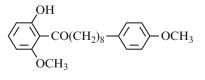
m.p. 82° [1575].

### 1-(2-Hydroxy-6-methoxyphenyl)-9-(4-methoxyphenyl)-1-nonanone

[114226-20-9]

 $C_{23}H_{30}O_4$ 

mol. wt. 370.49



Synthesis

-Obtained by treatment of 1-(2-hydroxy-6-methoxyphenyl)-9-(4-hydroxyphenyl)-1-nonanone or 1-(2,6-dihydroxyphenyl)-9-(4-hydroxyphenyl)-1-nonanone with diazomethane in ethyl ether [1786].

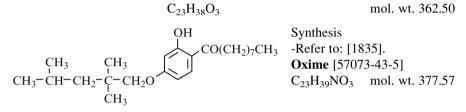
¹H NMR [1786], IR [1786], MS [1786].

mol. wt. 400.47

### 1-[2-Hydroxy-5-(2,2,4-trimethylpentyl)phenyl]-1-nonanone

	$C_{23}H_{38}O_2$	mol. wt. 346.50
OH CO(CH ₂ ) ₇ CH ₃	Synthesis -Refer to: [1835]. <b>Oxime</b> [57073-45-7]	
CH ₃ CH ₃	$C_{23}H_{39}NO_2$	mol. wt. 361.56
CH ₂ -C-CH ₂ -CH-CH ₃ CH ₃	USE: Hydrogen bonding, to, [1835].	spectra in relation

### 1-[2-Hydroxy-4-[(2,2,4-trimethylpentyl)oxy]phenyl]-1-nonanone



USE: Hydrogen bonding, spectra in relation to, [1835].

### 1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-nonanone

[3118-42-1] C24H38O5  $\begin{array}{c} OH \\ CH_3(CH_2)_7CO \\ HO \\ OH \\ \end{array} \\ \begin{array}{c} OH \\ CO(CH_2)_7CH_3 \\ HO \\ OH \\ \end{array} \\ \begin{array}{c} Syntheses \\ Obtained by reaction of nonanoic anhydride \\ with phloroglucinol in the presence of boron \\ trifluoride etherate on head in the second seco$ 

mol. wt. 406.56

mol. wt. 460.52

-Also refer to: [457, 644, 2911].

m.p. 84–87° [457, 2911].

BIOLOGICAL ACTIVITY: Antiviral activity towards the replication of vesicular stomatitis virus in HEL cell cultures [644]; Anthelmintic [457].

### 1,9-Bis(2-hydroxy-3,4-dimethoxyphenyl)-1,9-nonanedione

[10483-68-8] C₂₅H₃₂O₈  $\begin{array}{c|c} CH_{3}O & OH & HO & OCH_{3} \\ CH_{3}O & & \\ \hline \\ CH_{3}O & & \\ \hline \\ \end{array} \\ \begin{array}{c|c} CO(CH_{2})_{7}CO & & \\ \hline \\ CO(CH_{2})_{7}CO & & \\ \hline \\ \hline \\ CH_{3}O & & \\ \hline \\ \hline \\ CH_{3}O & & \\ \hline \\ \hline \\ CH_{3}O & & \\ \hline \\ CH_{$ 

-Also refer to: [590, 1575].

m.p. 117° [590]; 115° [1574, 1575].

trimethyl ether in the presence of aluminium chloride in tetrachloroethane [1574].

### $\label{eq:2.1} \textbf{Di-2,4-dinitrophenylhydrazone} \quad [37166-96-4] \quad C_{37}H_{40}N_8O_{14} \quad \text{mol. wt. } 820.77$

m.p. 175° [590].

### 1,1'-(2,4,6-Trihydroxy-5-methyl-1,3-phenylene)bis-1-nonanone

[2999-12-4]	C ₂₅ I	$H_{40}O_5$	mol. wt. 420.59
CH ₃ (CH ₂ ) ₇ CO HO	OH CO(CH ₂ ) ₇ CH ₃ OH CH ₃	Syntheses -Obtained by reaction of dride with 2-methylphlo presence of boron triflu heating for 4 h [457]. -Also refer to: [457, 600,	oroglucinol in the oride etherate on

m.p. 100–102° [457, 2911].

BIOLOGICAL ACTIVITY: For treatment of immune dysfunction associated with human immuno deficiency virus infection [600]; Anthelmintic [457].

### 1,1'-(4,4'-Dihydroxy[1,1'-biphenyl]-3,3'-diyl)-1-nonanone

[104295-23-0]	$C_{30}H_{42}$	$_{2}O_{4}$	mol. wt. 466.66
CH ₃ (CH ₂ ) ₇ CO HO	CO(CH ₂ ) ₇ CH ₃	Synthesis -Preparation by Fries 4,4'-biphenyl dipelar minium chloride in a benzene for 24 h (72)	gonate with alu- refluxing chloro-

m.p. 72–73° [2377]; IR [2377].

## 2 Aromatic Hydroxyketone Derived from 9-Bromononanoic Acid

### 9-Bromo-1-(2-hydroxy-5-methoxyphenyl)-1-nonanone

[173055-40-8]	$C_{16}H_{23}BrO_3$	mol. wt. 343.26
OH CO(CH ₂ ) ₇ CH ₂ Br OCH ₃	Synthesis -Refer to: [2623].	

### 3 Aromatic Hydroxyketones Derived from 9-Oxononanoic Acids

### 3.1 Unsubstituted Hydroxyketones

### 9-(3,4-Dihydroxyphenyl)-9-oxo-1-nonanoic acid

	$C_{15}H_{20}O_5$	mol. wt. 280.32
он Дон	Synthesis -Refer to: [1013].	
	<b>Dimethyl ether</b> [57640-96-7]	1 ( 200 27
CO(CH ₂ ) ₇ CO ₂ H	C ₁₇ H ₂₄ O ₅	mol. wt. 308.37

-Obtained by heating of its ethyl ester below with 10 % aqueous NaOH on the steam bath for 1.5 h (79 %) [1013]. -Also refer to: [3363, 3364].

m.p. 93–94° [1013], 92–93.5° [3363]; IR [1013, 3364].

### 2,4-Dinitrophenylhydrazone of the dimethyl ether C23H28N4O8 mol. wt. 488.50

m.p. 100-102° [3363].

Ethyl ester of the dimethyl ether [57641-20-0] C₁₉H₂₈O₅ mol. wt. 336.43

-Obtained by reaction of 9-chloro-9-oxononanoic acid ethyl ester with 1,2-dimethoxybenzene in the presence of aluminium chloride in 1,1,2,2-tetrachloroethane first at  $0-3^{\circ}$  for 2.5 h, then overnight to  $2^{\circ}$  (68 %) [1013].

b.p._{0.1} 195–197° [1013]; IR [1013]. GLC [1013].

### Methyl ester of the methylenedioxy [72674-91-0] C₁₇H₂₂O₅ mol. wt. 306.36

-Obtained by reaction of azelayl chloride monomethyl ester with methylene-1,2-dioxybenzene in the presence of stannic chloride in 1,1,2,2-tetrachloroethane for 15 h at r.t. (47 %) [2333].

m.p. 52–53° [2333]; ¹H NMR [2333], IR [2333], UV [2333], MS [2333].

#### 3.2 Substituted Hydroxyketones

#### 9-(5-Chloro-2-hydroxyphenyl)-9-oxo-1-nonanoic acid

[24381-67-7] C15H19ClO4 mol. wt. 298.77 **Synthesis** OH

-Obtained by reaction of azelaic acid dichloride (azeloyl CO(CH₂)₇CO₂H chloride) with p-chlorophenol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

m.p. 85° [585].

### **2,4-Dinitrophenylhydrazone** [24339-87-5] C₂₁H₂₃ClN₄O₇ mol. wt. 478.89

m.p. 150° [585].

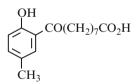
### 9-(5-Chloro-2,4-dihydroxyphenyl)-9-oxo-1-nonanoic acid

[37166-92-0] C15H19ClO5 mol. wt. 314.77 Synthesis CO(CH₂)₇CO₂H -Refer to: [590]. m.p. 126° [590]. H

### 9-(2-Hydroxy-5-methylphenyl)-

Synthesis

.... (



-Obtained by reaction of azelaic acid dichloride (azeloyl chloride) with p-cresol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

m.p. 80° [585].

2,4-Dinitrophenylhydrazone

m.p. 110° [585].

mol. wt. 458.47

C22H26N4O7

$$C_{16}H_{22}O_4$$
 mol. wt. 278.35

## Chapter 8 Decanones

### 1 Aromatic Hydroxyketones Derived from Decanoic Acids

### 1.1 Unsubstituted Hydroxyketones

### 1-(4-Hydroxyphenyl)-1,4-decanedione

	$C_{16}H_{22}O_3$		mol. wt. 262.35
ОН Ц	Synthesis -Refer to: [295	3]	
	Methyl ether		
$\Upsilon$ CO(CH ₂ ) ₂ COC ₆ H ₁₃	$C_{17}H_{24}O_3$		mol. wt. 276.38

-Obtained from heptanal and 1-(4-methoxyphenyl)-2-propen-1-one (60 %) [2953].

b.p._{0.1} 170° [2953]; m.p. 48° [2953]; ¹H NMR [2953], IR [2953].

### 1-(2,4-Dihydroxyphenyl)-1,4-decanedione

$$\begin{array}{c} C_{16}H_{22}O_4 & \text{mol. wt. } 278.35\\ OH & Synthesis\\ -Refer to: [2953].\\ \textbf{Dimethyl ether} & [67756-21-2]\\ C_{18}H_{26}O_4 & \text{mol. wt. } 306.40\end{array}$$

-Obtained from heptanal and 1-(2,4-dimethoxyphenyl)-2-propen-1-one (60 %) [2953].

b.p._{0.4} 187° [2953]; m.p. 44–47° [2953], ¹H NMR [2953], IR [2953].

### 1-(3,4-Dihydroxyphenyl)-1,4-decanedione

 $\begin{array}{ccc} C_{16}H_{22}O_4 & \mbox{mol. wt. } 278.35 \\ \mbox{Synthesis} & & \\ \mbox{-Refer to: } [2953]. \\ \mbox{Dimethyl ether} & [67756-24-5] \\ C_{18}H_{26}O_4 & \mbox{mol. wt. } 306.40 \end{array}$ 

COCH₂CH₂COC₆H₁₃

-Obtained from heptanal and 1-(3,4-dimethoxyphenyl)-2-propen-1-one (57 %) [2953].

b.p._{0.06} 175° [2953]; m.p. 55° [2953], ¹H NMR [2953], IR [2953].

### 1-(2-Hydroxyphenyl)-1-decanone

 $[14353-76-5] C_{16}H_{24}O_2 mol. wt. 248.37$   $OH OC(CH_2)_8CH_3 Syntheses -Obtained by reaction of decanoyl chloride with phenol in the presence of aluminium chloride, *in nitrobenzene at 70° for 3 h (21 %) [2549]; *in carbon disulfide at 47° for 5.5 h (44 %) [2549]; *in tetrachloroethane at 55–60° for 6 h [2669].$ 

-Also obtained by Fries rearrangement of phenyl decanoate with aluminium chloride, *in tetrachloroethane [3169];

*first in refluxing carbon disulfide for 2 h, then at  $136-140^{\circ}$  for 4 h, after solvent elimination (46.3 %) [13].

-Also refer to: [1985, 2035, 2658].

b.p._{0.3} 130–133° [2035], b.p.₆ 174° [13]; prisms [13]; m.p. 35–35.5° [2549], 35° [2669, 3169], 33.5–34.5° [13]; IR [13], UV [1996]; TLC [1994].

2,4-Dinitrop	henylhydrazone	$C_{22}H_{28}N_4O_5$	mol. wt. 428.49
m.p. 111-	-112° [2549], 111° [3169]	, 84–85° [524].	
Oxime	[439948-79-5]	C ₁₆ H ₂₅ NO ₂	mol. wt. 263.38
Defer to [7			

-Refer to: [2077].

OH

OH

### **2,3-Epoxypropyl ether** [18110-28-6] C₁₉H₂₈O₃ mol. wt. 304.43

-Obtained by reaction of epichlorohydrin with o-decanoylphenol (0.1 mol) in the presence of 30 % sodium hydroxide between 40 and 50° (61 %) [2669].

b.p.0.5 220-230° [2669].

### 1-(3-Hydroxyphenyl)-1-decanone

-Refer to: [1984, 1985].

b.p.₁₅ 128–130° [1984]; IR [1984].

### 1-(4-Hydroxyphenyl)-1-decanone

[14353-77-6]	$C_{16}H_{24}O_2$	mol. wt. 248.37
ОН	Syntheses	
$\land$	-Obtained by reaction of decanoyl chloride	with phenol in the
	presence of aluminium chloride,	
Ý	*in nitrobenzene at $70^{\circ}$ for 3 h (74 %) [2549]	;
$CO(CH_2)_8CH_3$	*in carbon disulfide at $47^{\circ}$ for 5.5 h (52 %) [2	2549];

*in methylene chloride for 1 h at 0°, then at r.t. overnight (39–41 %) [114, 1977] or for 14 h at r.t. (14 %) [1910];

*in tetrachloroethane at  $55-60^{\circ}$  for 6 h [2669].

-Also obtained by Fries rearrangement of phenyl decanoate with aluminium chloride,

*at 110–120° for 1 h [3336];

*in tetrachloroethane [3169];

*in nitrobenzene (49 %) [2947];

*first in refluxing carbon disulfide for 2 h, then at  $136-140^{\circ}$  for 4 h, after solvent elimination (32.2 %) [13];

*in the presence of aluminium chloride in refluxing carbon disulfide for 5 h. Then, the mixture was kept at  $80-90^{\circ}$  for 2 h and at  $140-150^{\circ}$  for 1 h after solvent elimination [1222].

-Also obtained by Fries rearrangement of phenyl decanoate with boron trifluoride at  $30-35^{\circ}$  for 20 h (53.3 %) [1938].

-Also obtained by reaction of decanoic acid with phenol in the presence of boron trifluoride at 20–25° for 19.5 h (60 %) [142]. -Also refer to: [62, 1116, 1743, 2658, 3423].

b.p._{0.5} 160–200° [3336], b.p.₆ 224° [13], b.p.₁ 225–228° [2947];
white solid [1910]; rhombic plates [3336]; short needles [13];
m.p. 67.2–67.8° [1910], 64–65° [3336], 63.5–64° [2549], 63–64° [1938], 63° [2669], 62.5–63.5° [142], 62–63° [13], 61–62° [3169], 60–61° [1977], 59° [1743];
¹H NMR [114, 1910], ¹³C NMR [114, 1910], IR [13, 1910, 1977], UV [1995], MS [1910];
TLC [1910, 1994]; GC [1910].

USE: Preparation of 2-hydroxy-3,5-dialkylbenzenesulfonate anionic surfactant for defoaming agent [3423]; Reactant in smectic C liquid crystal [1299].

BIOLOGICAL ACTIVITY: Inhibition of 17-β-hydroxysteroid dehydrogenase 3 [1910]; Antimicrobial [1743]; Antifungal [1743].

2,4-Dinitrophenylhydrazone	[17744-45-5]	$C_{22}H_{28}N_4O_5$	mol. wt. 428.49
m.p. 148–148.5° [2549], 148	° [3169], 111–11	2° [2549].	

iso-Nicotinylhydrazone [102701-26-8] C₂₂H₂₉N₃O₂ mol. wt. 367.49 m.p. 203° [520, 521].

USE: In chemotherapy of leprosy [520, 521].

Semicarbazone	[14353-85-6]	$C_{17}H_{27}N_3O_2$	mol. wt. 305.42
Refer to: [1743].			

Semicarbazone dihydrate	C ₁₇ H ₂₇ N ₃ O ₂ , 2 H ₂ O	mol. wt. 341.46
-------------------------	------------------------------------------------------------------------------------	-----------------

m.p. 130° [1743].

Phosphate	(93 %) [114];		¹ H NMR [114].	
Methyl ether	[101741-01-9]	$C_{17}H_{26}O_2$	mol. wt. 262.39	

-Obtained by acylation of anisole with decanoic acid,

*over a range of solid-acid catalysts under conventional heating and microwave stimulation [422];

*in the presence of a catalytic amount of p-toluenesulfonic acid and graphite for 3 h at  $90^{\circ}$  (92 %) [2709];

- *on the surface of graphite in the presence of methanesulfonic acid at  $80^{\circ}$  for 20 min (90 %) [1369];
- *in the presence of silica-supported phosphotungstic acid and Ce-exchanged zeolite catalysts for anisole acylation [1092].
- -Also obtained by reaction of decanoic acid with anisole over three large pore zeolites-beta (BEA), faujasite (FAU) and mordenite (MOR) [3246].
- -Also obtained by treatment of anisole with decanoic anhydride or decanoyl chloride in the presence of sulfated zirconia [867].
- -Also refer to: [2016].
  - m.p. 49–50.5° [2016], 110° [2709];
  - **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [1369, 2709], IR [422, 1369, 2709].

### iso-Nicotinylhydrazone of the methyl ether

[102753-19-5]	$C_{23}H_{31}N_3O_2$	mol. wt. 381.52
m.p. 126° [520, 521].		

### 2,4-Dinitrophenylhydrazone of the methyl ether $C_{23}H_{30}N_4O_5$ mol. wt. 442.52

m.p. 114-115° [2016].

2-Acetoxyethyl ether	[82944-57-8]	$C_{20}H_{30}O_4$	mol. wt. 334.46
-Refer to: [1929].			
<b>2-Hydroxyethyl ether</b> -Refer to: [1929].	[82684-67-1]	$C_{18}H_{28}O_3$	mol. wt. 292.42
2-Chloroethyl ether	[666836-96-0]	$C_{18}H_{27}ClO_2$	mol. wt. 310.86

-Obtained by reaction of decanoyl chloride with 2-chloroethoxybenzene in the presence of aluminium chloride in carbon disulfide for 4 h at  $50^{\circ}$  (51 %) [476].

m.p. 74.5° [476].

**N-Dimethylaminoethyl ether** [666836-97-1]  $C_{20}H_{33}NO_2$  mol. wt. 319.49 -Obtained by reaction of 4-(2-chloroethoxy)decanophenone with dimethylamine in a sealed tube for 2 h at 150° [476].

free base: b.p. $_{0.05}$  199° [476]; m.p. 40–41° [476]. hydrochloride: m.p. 180° (88.5 %) [476].

### **2,3-Epoxypropyl ether** [18211-87-5] C₁₉H₂₈O₃ mol. wt. 304.43

-Obtained by reaction of epichlorohydrin (0.1 mol) with p-decanoylphenol (0.1 mol) in the presence of 30 % sodium hydroxide between 40 and 50° (53 %) [2669].

m.p. 112° [2669].

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

Syntheses

### 1-(2,4-Dihydroxyphenyl)-1-decanone

[24313-95-9]

C₁₆H₂₄O₃ mol. wt. 264.36

HO CO(CH₂)₈CH₃

-Obtained by reaction of capric acid chloride with resorcinol in the presence of KU-2x8 cation-exchange resin [3145].

-Also obtained by reaction of decanoyl chloride with resorcinol (59 %) [3146] in the presence of aluminium chloride in 1,2-dichloroethane for 5 h at  $65^{\circ}$  [284].

-Also refer to: [76, 489, 490, 893, 1194, 1743, 2112, 2114, 2228, 2673, 2864, 3125].

b.p.₁ 180° [1743], b.p._{2.5} 205–206° [3125], b.p.₁₀ 240–245° [893]; m.p. 78° [2114], 74.2–75° [3125], 74–75° [3145], 69° [2673], 65° [1743]; IR [1194, 1195, 3145].

BIOLOGICAL ACTIVITY: Antimicrobial [1743]; Antifungal [1743, 2112, 2114]; Aldose reductase of eye lens inhibition by [2228]; Bactericidal action of, [76].

Hemihydrate	$C_{16}H_{24}O_3, 0.5 H_2O$	mol. wt. 273.37
m.p. 67–68° [3125].		

### **2,4-Dinitrophenylhydrazone** [95809-40-8] C₂₂H₂₈N₄O₆ mol. wt. 444.49

m.p. 153° [2673].

**Dimethyl ether** [430425-42-6] C₁₈H₂₈O₃ mol. wt. 292.42

-Obtained by reaction of decanoyl chloride with resorcinol dimethyl ether in the presence of HY-zeolite. Before reaction, the zeolites were activated at  $500^{\circ}$  for 5 h in nitrogen flow [352].

### 1-(2,5-Dihydroxyphenyl)-1-decanone

$$\begin{array}{cccc} [7337-50-0] & C_{16}H_{24}O_3 & \text{mol. wt. } 264.36 \\ \\ OH & Syntheses \\ -Refer to: [1743] (Japanese paper). \\ -Also refer to: [1691, 1950]. \\ b.p._2 & 170-172^{\circ} [1743]; & \text{m.p. } 63^{\circ} [1743]. \end{array}$$

BIOLOGICAL ACTIVITY: Antimicrobial [1743]; Antifungal [1743].

-Obtained by acylation of hydroquinone dimethyl ether in the presence of stannic chloride in benzene (30 %) [1040].

-Also obtained by reaction of decanoic acid chloride with hydroquinone dimethyl ether,

*in the presence of aluminium chloride in carbon disulfide first at  $0^{\circ}$ , kept overnight, heated to a boil [2874];

*in the presence of HY-zeolite. Before reaction, the zeolites were activated at  $500^{\circ}$  for 5 h in nitrogen flow [352].

-Also refer to: [1691].

b.p.₁₂ 230° [1040]; m.p. 38° [2874]; 
$$n_D^{25} = 1.5075$$
 [1040].

USE: Radioprotective activity of, [1040].

### 1-(2,6-Dihydroxyphenyl)-1-decanone

[185301-37-5]		С	$_{16}H_{24}O_{3}$		m	ol. wt. 264.36
OH			natural source			
CO(CH ₂ ) ₈ CH ₃	-Of	the	andromeda	lace	bug	Stephanitis
	rhode	odendrii	[2334].			
≫` _{OH}	-In th	e wood	d of Knema	austrosia	mensis	W. J. J. O.

-In the wood of *Knema austrosiamensis* W. J. J. O. DeWilde (Myristaceae) [1133].

### 1-(3,4-Dihydroxyphenyl)-1-decanone

Syntheses

 $\label{eq:constraint} [2754-54-3] \qquad \qquad \text{C}_{16}\text{H}_{24}\text{O}_{3} \qquad \qquad \text{mol. wt. 264.36}$ 

OH OH CO(CH₂)₈C

-Obtained by Fries rearrangement of pyrocatechol decanoate with aluminium chloride in the presence of pyrocatechol at  $135-140^{\circ}$  for 5 h (15 %) [283].

 $\dot{CO}(CH_2)_8CH_3$  -Also obtained by reaction of decanoic acid with catechol in the presence of boron trifluoride at 80° for 2 h (19 %) [2954].

-Also refer to: [1809].

b.p.₂ 236–240° [283]; m.p. 102–104° [2954], 96–98° [283]; ¹H NMR [283, 2954], ¹³C NMR [2954], MS [2954].

BIOLOGICAL ACTIVITY: Protective agent against the lethal effects of X rays [1809].

<b>Dimethyl ether</b> [109558-46-5]	$C_{18}H_{28}O_3$	mol. wt. 292.42
-------------------------------------	-------------------	-----------------

-Preparation by Friedel-Crafts acylation of veratrole [3056].

-Also obtained by reaction of decanoyl chloride with veratrole in the presence of zinc chloride in refluxing carbon disulfide,

*for 6 h and then left overnight (43 %) [2420];

*for 4 h and then left overnight (68 %) [2836].

-Also obtained by reaction of decanoyl chloride with veratrole in the presence of HY-zeolite. Before reaction, the zeolites were activated at  $500^{\circ}$  for 5 h in nitrogen flow [352].

-Also refer to: [2366, 2836, 3056].

white needles [2420, 2836]; b.p.₂ 198–200° [2420], b.p.₆ 205–208° [2836]; m.p. 62° [2420, 2836], 60–61° [3056].

### 2,4-Dinitrophenylhydrazone of the dimethyl ether

[113927-78-9]	$C_{24}H_{32}N_4O_6$	mol. wt. 472.54	
m.p. 90–91° [2420].			
Semicarbazone of the dimethyl ether	[102709-19-3]	$C_{19}H_{31}N_3O_3$	mol. wt. 349.47
white prisms [2836];	m.p. 80° [2836].		
Dibenzyl ether	$C_{30}H_{36}O_3$		mol. wt. 444.61
-Refer to: [2657].			
m.p. 65° [2657].			

#### 1-(2,3,4-Trihydroxyphenyl)-1-decanone

 $\begin{array}{ccc} [1154-72-9] & C_{16}H_{24}O_4 & \mbox{mol. wt. } 280.36 \\ & OH & \\ HO & CO(CH_2)_8CH_3 & -Obtained by reaction of decanoic acid with pyrogal$ lol in the presence of zinc chloride at 140–145° for $4 h (75–80 %) [506]. \\ & -Also refer to: [1101, 1810, 1883]. \end{array}$ 

b.p.₁₃ 254–256° [506]; m.p. 78–79° [506].

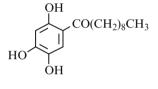
BIOLOGICAL ACTIVITY: Protection against whole-body irradiation [1810]; Antitumor [1883]; Protection from O-poisoning [1101].

C₁₆H₂₄O₄

#### 1-(2,4,5-Trihydroxyphenyl)-1-decanone

[108978-55-8]

Syntheses



-Obtained by reaction of decanoyl chloride with 1,2,4-trihydroxybenzene in the presence of aluminium chloride in nitrobenzene at r.t. several hrs, and heated 0.5 h at  $65^{\circ}$  [290].

-Also refer to: [1101, 1708].

m.p. 108–111° [290].

USE: Antioxidant [1708]; Antioxidant in fats and oils [290]; Protection from O-poisoning [1101].

**BIOLOGICAL ACTIVITY: Toxicity** [1708].

### 1-(2,4,6-Trihydroxyphenyl)-1-decanone

[6048-89-1]

OH

Syntheses

 $C_{16}H_{24}O_{4}$ 

CO(CH2)8CH3-Obtained by reaction of decanoyl chloride with phloro-<br/>glucinol in the presence of aluminium chloride in nitro-<br/>benzene and carbon disulfide mixture (46 %) [2113].

-Also obtained by reaction of caprinonitrile with phloroglucinol (Hoesch reaction) [1441].

-Preparation (23 %) [3202] using to the process [3201]. -Also refer to: [1441, 2111, 2433]. mol. wt. 280.36

mol. wt. 280.36

Isolation from natural sources

-In the wood of *Knema austrosiamensis* W. J. J. O. DeWilde (Myristaceae) [1133]. -From the Myristaceae family [3111].

white crystals [3202]; m.p. 133° [2113], 127° [3202], 109–110° [1441]; **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [3202], ¹³C NMR [3202], IR [3202], UV [3202], MS [3202]; TLC [3202].

BIOLOGICAL ACTIVITY: Antifungal [2111, 2113]; Antibiotic [3202]; Antibacterial [3202].

### 1-(4-Hydroxyphenyl)-2-methyl-1-decanone (S)

[120837-01-6] (+)	$C_{17}H_{26}O_2$	mol. wt. 262.39
OH CO-CH-(CH ₂ ) ₇ CH ₃	Syntheses -Refer to: [1063, 1064, 1321]. Acetate (S) [152430-07-4] C ₁₉ H ₂₈ O ₃ -Refer to: [1063, 1064].	mol. wt. 304.43

### 1-(2-Hydroxyphenyl)-2-methyl-5-methylene-1-decanone

[526208-17-3]	$C_{18}H_{26}O_2$	mol. wt. 274.40
$\bigcup_{i=1}^{OH} \begin{array}{c} CO-CH-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-(CH_2)_2-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-(CH_2)_2-C-$	acylation and 2-per in the pro equiv.) i	by intermolecular hydro- between salicylaldehyde htyl-1,5-hexadiene (6 equiv.) esence of RhCl(PPh ₃ ) ₃ (0.2 n methylene chloride for . (53 %) [1435].

-Also refer to: [3066].

¹H NMR [1435].

 $CO-CH-(CH_2)_7CH_3$  $C_3H_7$ 

### 1-(4-Hydroxyphenyl)-2-propyl-1-decanone (S)

 $\begin{array}{cccc} [120837-05-0] \ (+) & C_{19}H_{30}O_2 & \text{mol. wt. } 290.45 \\ \\ OH & Syntheses \\ -Refer \ to: \ [1318, \ 1321]. \end{array}$ 

### 1-(2,5-Dihydroxyphenyl)-2-hexyl-1-decanone

### 2-Hexyl-1-(2,4,5-trihydroxyphenyl)-1-decanone

 $\begin{array}{cccc} [103449 \hbox{-} 10 \hbox{-} 1] & C_{22}H_{36}O_4 & \mbox{mol. wt. 364.53} \\ & OH & C_6H_{13} & Synthesis \\ & -Refer to: [2326]. \\ & USE: Colour photog. paper contg. antistaining agent from, [2326]. \end{array}$ 

### 1-(2,5-Dihydroxyphenyl)-2-octyl-1-decanone

 $[375172-14-6] C_{24}H_{40}O_3$ mol. wt. 376.58 OH Synthesis -Refer to: [2352]. CO-CH-C_8H_{17} -Refer to: [2352].

### 1.2 Substituted Hydroxyketones

### 1-(3-Bromo-5-fluoro-4-hydroxyphenyl)-1-decanone

[402-81-3]	$C_{16}H_{22}BrFO_2$	mol. wt. 345.25
$F$ $H$ $Br$ $CO(CH_2)_8CH_3$	Synthesis -Obtained by reaction of bromine with 3 decanophenone in acetic acid [516]. m.p. 68° [516].	3-fluoro-4-hydroxy-

### 1-(3,5-Dibromo-4-hydroxyphenyl)-1-decanone

[20683-53-8]	$C_{16}H_{22}Br_2O_2$	mol. wt. 406.16
Br Br CO(CH ₂ ) ₈ CH ₃	Synthesis -Obtained by reaction of decanophenone in aqueous acc m.p. 54° [516].	

### 1-(3,5-Dichloro-2-hydroxyphenyl)-1-decanone

	$C_{16}H_{22}Cl_2O_2$	mol. wt. 317.25
ŎН	Synthesis	
CO(CH ₂ ) ₈ CH ₃	-Obtained by Fries rea	rrangement of 2,4-dichlorophenyl
	caprate with aluminit	
Ý	m.p. 53.5° [3170]; U	UV [3170].

### 1-(3-Fluoro-4-hydroxy-5-iodophenyl)-1-decanone

[402-82-4]  $C_{16}H_{22}FIO_2$  mol. wt. 392.25 OH Synthesis I  $\downarrow$   $\downarrow$   $\downarrow$  F -Obtained by reaction of iodine with 3-fluoro-4-hydroxy-

F -Obtained by reaction of iodine with 3-fluoro-4-hydroxydecanophenone in ethanol in the presence of yellow mercuric oxide [519]. CO(CH₂)₈CH₃ fine colourless needles [519];

m.p. 73° [519].

### 1-(2,3-Difluoro-4-hydroxyphenyl)-1-decanone

[144292-58-0]	$C_{16}H_{22}F_2O_2$	mol. wt. 284.35
OH F	Syntheses -Refer to: [2720, 2975].	
F CO(CH ₂ ) ₈ CH ₃	<b>Benzyl ether</b> [144292-57-9] $C_{23}H_{28}F_2O_2$ -Refer to: [2975].	mol. wt. 374.47

### 1-(5-Bromo-2,3,4-trihydroxyphenyl)-1-decanone

	$C_{16}H_{23}BrO_4$	mol. wt. 359.26
HO HO HO HO HO HO HO HO HO	Synthesis -Obtained by reaction of oylpyrogallol in acetic ac m.p. 86–87° [506].	of bromine with 4-decan- bid [506].

#### 1-(3-Chloro-2-hydroxyphenyl)-1-decanone

	$C_{16}H_{23}ClO_2$	mol. wt. 282.81
ОН	Synthesis	
$CO(CH_2)_8CH_3$	-Obtained by Fries rearra	angement of 2-chlorophenyl
	caprate with aluminium	chloride [3170].
$\sim$	b.p. _{0.4} 146–147° [3170];	m.p. 42–43° [3170];
	UV [3170].	

### 1-(3-Chloro-4-hydroxyphenyl)-1-decanone

 $C_{16}H_{23}ClO_2$ mol. wt. 282.81 Synthesis -Obtained by Fries rearrangement of 2-chlorophenyl caprate with aluminium chloride [3170]. m.p. 53–54° [3170]; UV [3170].

### 1-(4-Chloro-2-hydroxyphenyl)-1-decanone

[666836-99-3]	$C_{16}H_{23}ClO_2$	mol. wt. 282.81
CI CO(CH ₂ ) ₈ CH ₃	Syntheses -Preparation by Fries rearrangement caprate with aluminium chloride, *without solvent at 130° for 2 h (52 *in nitrobenzene at 25° for 6 h (79 °	%) [2802];

b.p.40 230° [2802].

CO(CH₂)₈CH₃

### Methyl ether

 $C_{17}H_{25}ClO_2$ -Obtained by methylation of the above ketone in the usual way (85 %) [2802].

b.p.29 150° [2802].

### **2,4-Dinitrophenylhydrazone** $C_{22}H_{27}ClN_4O_5$ mol. wt. 462.93

m.p. 126° [2802].

### 1-(5-Chloro-2-hydroxyphenyl)-1-decanone

[98813-29-7]	$C_{16}H_{23}ClO_2$	mol. wt. 282.81
OH CO(CH ₂ ) ₈ CH ₃	Syntheses -Obtained by Fries rearrangement of caprate with aluminium chloride [3170] -Also obtained by reaction of decand 4-chlorophenol in the presence of alu (49.6 %) [2680].	byl chloride with

mol. wt. 296.84

-Also refer to: [148].

b.p.₁ 158–160° [2680]; m.p. 69–70° [3170], 68–69° [2680]; UV [3170].

USE: Retard photodegradation of high-d polyethylene in air [148].

### 1-(4-Chloro-2,5-dihydroxyphenyl)-1-decanone

	$C_{16}H_{23}ClO_3$		mol. wt. 298.81
OH CO(CH ₂ ) ₈ CH ₃ OH	Synthesis -Refer to: [418]. <b>Dimethyl ether</b> $C_{18}H_{27}CIO_3$ -Refer to: [418].	[434340-29-1]	mol. wt. 326.86

### 1-(3-Fluoro-4-hydroxyphenyl)-1-decanone

[403-08-7]	$C_{16}H_{23}FO_2$	mol. wt. 266.36
OH F CO(CH ₂ ) ₈ CH ₃	Synthesis -Obtained by refluxing 3-fluor pyridinium chloride for 15 mi b.p. ₂₀ 233–234° [516]; m.p. (	

Methyl ether	[455-77-6]	$C_{17}H_{25}FO_2$	mol. wt. 280.38
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-Obtained by Friedel-Crafts acylation of 2-fluoroanisole with decanoyl chloride in the presence of aluminium chloride in carbon disulfide (75–85 %) [516].

b.p.₂₀ 232–233° [516]; m.p. 72° [516].

### 1-(5-Fluoro-2-hydroxyphenyl)-1-decanone

 $\begin{array}{cccc} [392-02-9] & C_{16}H_{23}FO_2 & \mbox{mol. wt. } 266.36 \\ & OH & Synthesis \\ -Obtained by Fries rearrangement of 4-fluorophenyl caprate with aluminium chloride at 130° for 2 h \\ (55 \%) [1549]. \\ & b.p._5 270° [1549]. \end{array}$ 

**2,4-Dinitrophenylhydrazone** [2546-82-9] C₂₂H₂₇FN₄O₅ mol. wt. 446.48 m.p. 98° [1549].

### 1-(4-Hydroxy-3-nitrophenyl)-1-decanone

 $\begin{array}{cccc} [70079-27-5] & C_{16}H_{23}NO_4 & \mbox{mol. wt. } 293.36 \\ OH & Synthesis \\ -Obtained by treatment of 4-decanoylphenol with concentrated nitric acid in concentrated sulfuric acid at 0° for 1 h 15 min [1222]. \\ CO(CH_2)_8CH_3 & \mbox{m.p. } 63.5-64.5^{\circ} \ [1222]. \end{array}$ 

### 1-(3,4-Dihydroxy-5-nitrophenyl)-1-decanone

### 2-Hydroxy-5-decanoylbenzoic acid

5-n-decanoylsalicylic acid

[78418-02-7]

 $C_{17}H_{24}O_4$ 

mol. wt. 292.37



Syntheses -Obtained by saponification of the methyl ester [2522], (91 %) [689] with 15 % aqueous sodium hydroxide at reflux for 20 h (53 %) [782].

-Also obtained from methyl 2-(n-decyloxy)benzoate by a Fries rearrangement in carbon disulfide (38 % yield) followed by basic hydrolysis of the resulting methyl ester in 89 % yield [783].

-Also refer to: [849, 1743, 1844, 1855, 1857, 2439, 2477, 2643, 2884–2886, 3129, 3130].

m.p. 120.5–121.5° [2522], 120–122° [782], 118–119° [689], 117° [1743]; ¹H NMR [782], IR [782].

USE pattern: Refer to: [2885].

BIOLOGICAL ACTIVITY: Antimicrobial [1743]; Antifungal [1743].

**Methyl ester** [78417-97-7] C₁₈H₂₆O₄ mol. wt. 306.40

-Obtained by reaction of decanoyl chloride with methyl salicylate in the presence of aluminium chloride in carbon disulfide first at  $5-10^{\circ}$ , then at r.t. for 12 h (86 %) [689].

-Also obtained by Fries rearrangement of methyl 2-(n-decanoyloxy)benzoate [2522] with aluminium chloride in refluxing carbon disulfide for 2 h, then at 90– $100^{\circ}$  for 30 min after solvent elimination (67 %) [782].

b.p._{1.5} 180–190° [2522]; m.p. 66.5–67.5° [2522], 65–66° [782], 63–64° [689]; ¹H NMR [782], IR [782].

# 2,4-Dihydroxy-5-decanoylbenzoic acid

 $\begin{array}{c} C_{17}H_{24}O_5 \\ OH \\ + O(CH_2)_8CH_3 \\ HO \\ CO_2H \end{array} \qquad \begin{array}{c} CO(CH_2)_8CH_3 \\ -Refer to: [1743] (Japanese paper). \\ m.p. \ 110^{\circ} \ [1743]. \end{array}$ 

# 1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-decanone

C	$C_{17}H_{25}ClO_2$	mol. wt. 296.84
CO(CH ₂ ) ₈ CH ₃	Synthesis -Refer to: [3138]. Fluorescence spectral data [3138].	

# 1-(2-Hydroxy-3-methylphenyl)-1-decanone

[109251-96-9]	$C_{17}H_{26}O_2$	mol. wt. 262.39
CH ₃ CO(CH ₂ ) ₈ CH ₃	Synthesis -Obtained by Fries rearran with aluminium chloride (28 %) [1644].	agement of o-cresyl caprate e at 160–180° for 30 min
b.p. ₈ 190–193° [1644].		
2,4-Dinitrophenylhydrazon	$C_{23}H_{30}N_4O_5$	mol. wt. 442.52
m.p. 96–98° [1644].		
1 (2 Hardmann 4 an athalach ar	J) 1 Jacomono	

# 1-(2-Hydroxy-4-methylphenyl)-1-decanone

 $CH_3$   $CH_3$   $CO(CH_2)_8CH_3$   $CH_3$   $CH_3$ 

[

Synthesis -Preparation by Fries rearrangement of 3-methylphenyl caprate with aluminium chloride without solvent at 140–150° [906]. b.p._{1.5} 148–149° [906]; m.p. 32° [906].

# 1-(2-Hydroxy-5-methylphenyl)-1-decanone

-Also refer to: [142, 148, 490, 1921].

b.p._{0.7} 144–148° [489], b.p.₁ 145° [142], b.p.₆₀ 180–181° [1769], b.p._{6.5} 182–186° [1644];

 ${}^{1}\text{H NMR [489], IR [489];} \quad n_{D}^{25} = 1.5110 \text{ [142]}.$ 

USE: Retard photodegradation of high-d polyethylene in air [148].

**Oxime** [71491-29-7] C₁₇H₂₇NO₂ mol. wt. 277.41

-Refer to: [123, 1768, 1769, 2706, 3321].

m.p. 92–93.5° [1769]; ¹H NMR [1769], ¹³C NMR [1769], IR [1769], UV [1769], MS [1921].

USE: Solvent extraction of copper (II) [1769, 2520]; Hydroxyoxime adsorption at octane-toluene/water interfaces [3321].

**Oxime** (*E*) [103582-40-7]  $C_{17}H_{27}NO_2$  mol. wt. 277.41

-Refer to: [124, 1769, 1921, 2600].

m.p. 92–93.5° [1769]; ¹H NMR [1769], ¹³C NMR [1769], IR [1769], UV [1769], MS [1921].

USE: Copper (II) complexing and extracting ability from aqueous solutions [2600].

**2,4-Dinitrophenylhydrazone** [115098-09-4] C₂₃H₃₀N₄O₅ mol. wt. 442.52 m.p. 139–141° [1644], 99–100° [1769].

#### Methyl ether

$$C_{18}H_{28}O_2$$
 mol. wt. 276.42

¹H NMR [879], ¹³C NMR [879].

**Oxime of the methyl ether** [101396-10-5]  $C_{18}H_{29}NO_2$  mol. wt. 291.43 USE: Palladium extn. and purifn. with [3191].

# 1-(4-Hydroxy-2-methylphenyl)-1-decanone

[1263095-96-0] 
$$C_{17}H_{26}O_2$$
 mol. wt. 262.39  
CH₃ Synthesis  
CO(CH₂)₈CH₃ -Refer to: [2189].

# 1-(4-Hydroxy-3-methylphenyl)-1-decanone

[109250-85-3]	$C_{17}H_{26}O_2$	mol. wt. 262.39
CO(CH ₂ ) ₈ CH ₃ HO	Synthesis -Refer to: [142]. m.p. 72.5–74° [142].	

# 1-(2-Hydroxy-4-methoxyphenyl)-1-decanone

[393519-46-5]	$C_{17}H_{26}O_3$	mol. wt. 278.39
CH ₃ O ^{OH} CO(CH ₂ ) ₈ CH ₃	Synthesis -Obtained by treatment silyloxy)-4-methoxy-2'-oc 129–131°) with ( <i>n</i> -Bu) ₄ 45 min (79 %) [1467].	ctylacetophenone (m.p.

m.p. 151–153° [1467]; ¹H NMR [1467], ¹³C NMR [1467], IR [1467], MS [1467].

# 1-(2-Hydroxy-5-methoxyphenyl)-1-decanone

OCH₃

[80427-37-8]	37-8]		$C_{17}H_{26}O_3$	mol. wt. 278.39	
	_				

Synthesis .CO(CH₂)₈CH₃ -Obtained by Fries rearrangement of 4-methoxyphenyl decanoate with titanium tetrachloride for 1 h at 120° (60–74 %) [2000]. I₃ m.p. 39° [2000]; IR [2000], UV [2000].

#### 1-(3-Hydroxy-4-methoxyphenyl)-1-decanone

[66476-00-4]		$C_{17}H_{26}O_3$	mol. wt. 278.39
CH ₃ O	CO(CH ₂ ) ₈ CH ₃	chloride with 2-me presence of stannic	eps: First, reaction of decanoyl ethoxyphenyl decanoate in the chloride in nitromethane at 20° n, the m-ketoester obtained was [1999, 2000].

m.p. 58° [1999, 2000]; ¹H NMR (Sadtler standard N° 28213M) [2000], IR (Sadtler standard N° 55285) [1999, 2000], UV [1999, 2000].

Decanoate	[66475-97-6]	$C_{27}H_{44}O_4$	mol. wt. 432.65
m.p. 39° [199	9, 2000];		

IR (Sadtler standard N° 57342) [1999, 2000], UV [1999, 2000].

# 1-(4-Hydroxy-3-methoxyphenyl)-1-decanone



Syntheses -Obtained by Fries rearrangement of 2-methoxyphenyl decanoate (1 mol) in the presence of various catalysts* (2 mol) in nitromethane at 20° for 24 h, *stannic chloride (69 %) [1999]; *aluminium chloride (65 %) [1999];

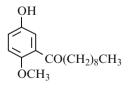
*titanium tetrachloride (62 %) [1999]; *antimony pentachloride (28 %) [1999].

#### 1-(5-Hydroxy-2-methoxyphenyl)-1-decanone

[80427-32-3]

 $C_{17}H_{26}O_3$ 

mol. wt. 278.39

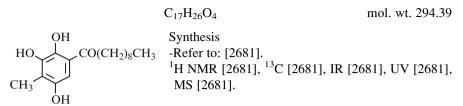


-Preparation by treatment of 3-decanoyl-4-methoxyphenyl decanoate with potassium hydroxide, itself obtained by acylation of 4-methoxyphenyl decanoate with decanoyl chloride in the presence of stannic chloride [2000]. -Also refer to: [2002].

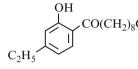
m.p. 56° [2000], 55° (Sadtler standard N° 62646K);
¹H NMR (Sadtler standard N° 35278M), [2000];
IR (Sadtler standard N° 62646K), [2000], UV [2000].

Syntheses

#### 1-(2,3,5-Trihydroxy-4-methylphenyl)-1-decanone



#### 1-(4-Ethyl-2-hydroxyphenyl)-1-decanone



Syntheses  $CO(CH_2)_8CH_3$  -Obtained by Fries rearrangement of 3-ethylphenyl n-caprate (1 equiv.), *in the presence of aluminium chloride (1.3 equiv.)

in nitrobenzene at  $25^{\circ}$  for 6 h (86 %) [2801];

*in the presence of aluminium chloride (2.8 M), first in refluxing carbon disulfide for 2 h, then for 2 h at 130° after solvent elimination (83 %) [2801].

b.p.30 200° [2801].

# 2,4-Dinitrophenylhydrazone

m.p. 108° [2801].

#### Methyl ether

-Obtained by reaction of dimethyl sulfate with 1-(4-ethyl-2-hydroxyphenyl)-1-decanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating on the water bath for 5 h (83 %) [2801].

b.p.₃₆ 165° [2801].

# 1-(5-Ethyl-2-hydroxyphenyl)-1-decanone

$$C_{18}H_{28}O_2 \qquad \text{mol. wt. } 276.42$$
  
Synthesis  
$$CO(CH_2)_8CH_3 \qquad -\text{Obtained by Fries rearrangement of 4-ethylphenyl caprate with aluminium chloride at 100° for 2 h (76 %) [2800].}$$
  
b.p.₁₁ 182° [2800].

# **2,4-Dinitrophenylhydrazone** $C_{24}H_{32}N_4O_5$

mol. wt. 456.54

m.p. 115° [2800].

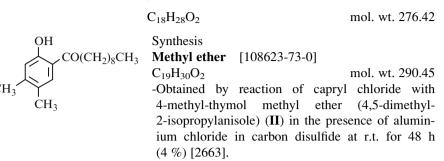
 $C_{19}H_{30}O_{2}$ 

 $C_{24}H_{32}N_4O_5$ 

mol. wt. 290.45

mol. wt. 456.54

#### 1-(2-Hydroxy-4,5-dimethylphenyl)-1-decanone

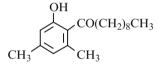


**N.B.**: There is an isopropyl group elimination [2663].

oil yellow [2663]; b.p._{0.2} 228–231° [2663];  $n_D^{24} = 1.4992$  [2663].

## 1-(2-Hvdroxy-4,6-dimethylphenyl)-1-decanone

$$[101873-69-2] C_{18}H_{28}O_2 mol. wt. 276.42$$



Syntheses CO(CH₂)₈CH₃ -Obtained by Fries rearrangement of 3,5-dimethylphenyl n-caprate (1 equiv.), CH₃ *in the presence of aluminium chloride (1.3 equiv.) in nitrobenzene at 25° for 6 h (75 %) [2801];

*in the presence of aluminium chloride (2.8 M), first in refluxing carbon disulfide for 2 h, then for 2 h at 130° after solvent elimination (70 %) [2801].

-Also obtained by reaction of capric acid with 3,5-xylenol in the presence of boron trifluoride at 70° for 2 h (86 %) [1685].

 $C_{24}H_{32}N_4O_5$ 

b.p.₁ 182–183° [1685], b.p.₂ 190° [2801]; m.p. 33° [1685].

#### 2,4-Dinitrophenylhydrazone

m.p. 195° [2801].

#### Methyl ether

C19H30O2 mol. wt. 290.45

mol. wt. 456.54

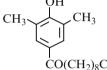
-Obtained by reaction of dimethyl sulfate with 1-(2-hydroxy-4,6-dimethylphenyl)-1-decanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating on the water bath for 5 h (75 %) [2801].

b.p.39 220° [2801].

#### 1-(4-Hydroxy-2,5-dimethylphenyl)-1-decanone

# 1-(4-hydroxy-3,5-dimethylphenyl)-1-decanone

$$[29665-56-3] C_{18}H_{28}O_2 mol. wt. 276.42$$



Synthesis -Obtained by Fries rearrangement of 2,6-dimethylphenyl decanoate in the presence of aluminium chloride in nitrobenzene [718], according to [380]. O(CH₂)₈CH₃ m.p. 48–49.5° [718].

# 1-(4-Ethoxy-2-hydroxyphenyl)-1-decanone

[20825-28-9] C18H28O3 mol. wt. 292.42

 CO(CH₂)₈CH₃
 Synthesis
 Obtained by reaction of decanoyl chloride with m-diethoxy-benzene in the presence of aluminium chloride in dichloroethane at 0°. Then, the mixture was stirred for 1 h at  $10^\circ$ , and then for 6 h at  $20-25^\circ$ .

The temperature was then raised to 80° and stirring was continued at that temperature for 5 h (75 %) [1194, 1195].

m.p. 48.5° [1194, 1195]; UV [1194, 1195].

USE: UV absorber [1194]; Light-stabilizer of polymeric materials [1194].

#### **Copper complex** [66468-51-7].

-Refer to: [3181].

C2H5

## 1-(5-Ethoxy-2-hydroxyphenyl)-1-decanone

[140943-34-6]	$C_{18}H_{28}O_3$	mol. wt. 292.42
OH CO(CH ₂ ) ₈ CH ₃	Synthesis -Refer to: [285]. <b>Oxime</b> [141027-87-4]	
OC ₂ H ₅	C ₁₈ H ₂₉ NO ₃	mol. wt. 307.43

-Refer to: [285].

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

#### 1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-decanone

[134081-96-2]	$C_{18}H_{28}O_5$	mol. wt. 324.42
CH ₃ O OH CO(CH ₂ ) ₈ CH ₃ OH OCH ₃	3,4-dimethoxy-2-(4-methoxy-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4-methox)-2-(4	ment of 1-[6-hydroxy- hylphenylsulfonyloxy)phe- potassium carbonate in -3 h (83 %) [1353].
	NT (D. 14050)	

m.p. 71–72.5° [1353]; ¹H NMR [1353].

# 1-(5-Chloro-8-hydroxy-7-quinolinyl)-1-decanone

[88559-39-1]

C₁₉H₂₄ClNO₂ mol. wt. 333.86

CH₃(CH₂)₈CO

**Synthesis** -Obtained by Friedel-Crafts acylation of 5-chloro-8-hydroxyquinoline with decanoyl chloride in the presence of aluminium chloride in nitrobenzene for 15 to 24 h (10 %) [3171].

m.p. 68–69° [3171]; IR [3171].

Hydrazone [88559-45-9]  $C_{19}H_{26}ClN_{3}O$ mol. wt. 347.89

 $C_{19}H_{24}O_{4}$ 

m.p. 119–120° [3171].

# 4-Hydroxy-3-(1-oxodecyl)-2H-1-benzopyran-2-one

[20924-70-3]

Syntheses

of piperidine for 12 h on a water bath (62 %) [3174].

-Also refer to: [3143, 3144].

m.p. 108° [3174].

# 1-(8-Hydroxy-5-quinolinyl)-1-decanone

[88559-37-9]

C10H25NO2

mol. wt. 299.41

mol. wt. 316.40



**Synthesis** 

-Obtained by Friedel-Crafts acylation of 8-hydroxyquinoline in the presence of aluminium chloride in nitrobenzene for 15-24 h (20-25 %) [3171]. m.p. 60–61° [3171]; IR [3171].

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#### 1-(8-Hydroxy-7-quinolinyl)-1-decanone

[88559-44-8]  $C_{19}H_{27}N_3O$ 

C₁₉H₂₅NO₃

Hydrazone [885 m.p. 100–103° [3171].

# 1-(2,4-Dihydroxy-3-quinolinyl)-1-decanone

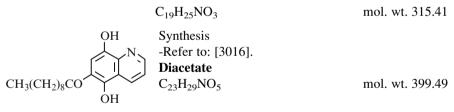
[94432-99-2]

 $CO(CH_2)_8CH_3$  -Obtained by reaction of decanoyl chloride with 2,4-dihydroxyquinoline in the presence of aluminium chloride in nitrobenzene on the steam bath for 3 h (10 %) [3123].

m.p. 127–130° [3123]; UV [3123].

BIOLOGICAL ACTIVITY: Antibacterial properties (Staphylococcus aureus and Escherichia coli) [3123].

#### 1-(5,8-Dihydroxy-6-quinolinyl)-1-decanone



-Obtained by irradiation of an 6-halo-5,8-quinolinequinone and capraldehyde (decanal) mixture in acetic acid [3016].

# 1-(5,8-Dihydroxy-7-quinolinyl)-1-decanone

-Obtained by irradiation of an 7-halo-5,8-quinolinequinone and capraldehyde (decanal) mixture in acetic acid [3016].

Synthesis

mol. wt. 315.41

mol. wt. 313.44

#### 1-(2-Ethyl-6-hydroxy-4-methylphenyl)-1-decanone

$C_{19}H_{30}O_2$				mo	ol. w	t. 290.45
CH ₃ CH ₃ CH ₃ CH ₃ CO(CH ₂ ) ₈ CH ₃ C ₂ H ₅	5-methylphe	nyl c	aprate	rearrangement with aluminium of for 2 h (77 %)	chlo	ride,
	*in nitrobenz	zene	at 25° :	for 6 h (79 %) [	2802	2].

m.p. 40° [2802].

#### Methyl ether

 $C_{20}H_{32}O_2$  mol. wt. 304.47

-Obtained by methylation of the above ketone in the usual way (77 %) [2802].

b.p.27 230° [2802].

**2,4-Dinitrophenylhydrazone**  $C_{25}H_{34}N_4O_5$  mol. wt. 470.57

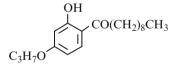
m.p. 115° [2802]

# 1-(2,4-Dihydroxy-3-propylphenyl)-1-decanone

[172932-00-2]	$C_{19}H_{30}O_3$	mol. wt. 306.45
C ₃ H ₇ HO CO(CH ₂ ) ₈ CH ₃	Synthesis -Refer to: [2388].	

#### 1-(2-Hydroxy-4-propoxyphenyl)-1-decanone

[143286-94-6]  $C_{19}H_{30}O_3$  mol. wt. 306.45



CO(CH₂)₈CH₃ -Obtained by reaction of propyl bromide with 1-(2,4-dihydroxyphenyl)-1-decanone in the presence of potassium carbonate in refluxing acetone for 20 h [284].

m.p. 38-40° [284].

<b>Oxime</b> [143286-63-9] $C_{19}H_{31}NO_3$ mol.
----------------------------------------------------

-Obtained by reaction of hydroxylamine hydrochloride with 1-(2-hydroxy-4-propoxyphenyl)-1-decanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 72–74.5° [284]; ¹H NMR [284].

#### 1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-decanone

[134081-65-5]	$C_{19}H_{30}O_5$	mol. wt. 338.44
CH ₃ O CH ₃ O CH ₃ O OCH ₃	of crude 2,3,4,6-tetrame	selective demethylation ethoxydecanophenone with cetonitrile at $50^{\circ}$ for 1–2 h
m.p. 47.5–48.5° [1353];	¹ H NMR [1353].	

p-Toluenesulfonic ether [134081-80-4] C₂₆H₃₆O₇S mol. wt. 492.63

-Obtained by reaction of p-toluenesulfonyl chloride with 2-hydroxy-3,4,6-trimethoxy-decanophenone in the presence of potassium carbonate in refluxing acetone for 6-14 h (83 %) [1353].

m.p. 84–85° [1353]; ¹H NMR [1353].

#### Methyl ether C20H32O5 mol. wt. 352.47

-Obtained by reaction of decanoyl chloride with 1,2,3,5-tetramethoxybenzene in the presence of aluminium chloride in ethyl ether at  $0^{\circ}$  for 30 min [1353].

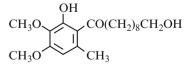
## 1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-decanone

[134081-72-4]	$C_{19}H_{30}O_5$	mol. wt. 338.44
CH ₃ O OCH ₃ O OCH ₃ O OCH ₃ O OCH ₃ O	dium on charcoal in eth	on of (6-phenylmethoxy)- henone over 10 % palla- yl acetate/methanol (1:1) gen ceased (93 %) [1353].

m.p. 31–31.8° [1353]; ¹H NMR [1353].

#### 10-Hydroxy-1-(2-hydroxy-3,4-dimethoxy-6-methylphenyl)-1-decanone

[104966-97-4]	$C_{19}H_{30}O_5$	mol. wt. 338.44
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Synthesis CO(CH₂)₈CH₂OH -Obtained by treatment of its 10-acetyl ester with sodium hydroxide in methanol for 2 h at r.t. (79 %) [1147].

colourless needles [1147]; m.p. 67° [1147]; ¹H NMR [1147], IR [1147], MS [1147].

**10-Acetyl ester** [104966-92-9] C₂₁H₃₂O₆ mol. wt. 380.48

-Obtained by Friedel-Crafts reaction of 10-acetoxydecanoyl chloride with 3,4,5-trimethoxy-toluene in 1,2-dichloroethane,

*in the presence of aluminium chloride (2.1 mol) at  $20^{\circ}$  for 72 h (79 %) [1147]; *in the presence of zinc chloride (2.1 mol) at  $10^{\circ}$  for 72 h (41 %) [1147].

colourless oil [1147]; ¹H NMR [1147], IR [1147], MS [1147].

Methyl ether of the 10-acetyl ester [104967-07-9] C₂₂H₃₄O₆ mol. wt. 394.51

-Obtained by Friedel-Crafts reaction of 10-acetoxydecanoyl chloride with 3,4,5-trimethoxytoluene in 1,2-dichloroethane in the presence of aluminium chloride (1.1 mol) at  $20^{\circ}$  for 72 h (43 %) [1147].

colourless oil [1147]; ¹H NMR [1147], IR [1147], MS [1147].

**Synthesis** 

# 1-(8-Hydroxy-2-methyl-5-quinolinyl)-1-decanone

$$[217815-23-1] C_{20}H_{27}NO_2 mtext{mol. wt. } 313.44$$



-Obtained by reaction of decanoyl chloride with 8-hydroxyquinaldine in the presence of aluminium chloride in nitrobenzene or 1,2-dichloroethane first at  $10-30^{\circ}$ , then at  $70-80^{\circ}$  for 20 h [2261].

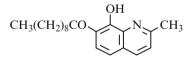
m.p. 63° [2261]; ¹H NMR [2261], ¹³C NMR [2261], IR [2261].

# 1-(8-Hydroxy-2-methyl-7-quinolinyl)-1-decanone

[217815-26-4]

 $\mathrm{C}_{20}\mathrm{H}_{27}\mathrm{NO}_2$ 

mol. wt. 313.44



Synthesis

-Obtained by reaction of decanoyl chloride with 8-hydroxyquinaldine in the presence of aluminium chloride in nitrobenzene or 1,2-dichloroethane first at  $10-30^{\circ}$ , then at  $70-80^{\circ}$  for 20 h [2261].

m.p. 63° [2261]; ¹³C NMR [2261].

#### 1-(4,7-Dihydroxy-2,3-dimethyl-6-benzofuranyl)-1-decanone

USE: Prepn. and radioprotective activity of, [1040].

**Dimethyl ether** [42782-77-4] C₂₂H₃₂O₄ mol. wt. 360.49

-Obtained by acylation of 4,7-dimethoxy-2,3-dimethylbenzofuran in the presence of stannic chloride in benzene (73 %) [1040].

m.p. 58.5° [1040]; LD₅₀ [1040].

USE: Prepn. and radioprotective activity of, [1040].

#### 1-(6,7-Dihydro-4-hydroxy-6-methylfuro[2,3-f]-1,3-benzodioxol-8-yl)-1-decanone

 $\begin{array}{ccc} C_{20}H_{28}O_5 & \text{mol. wt. 348.44} \\ OH & Synthesis \\ O & CH_3 & -Refer to: [2179]. \\ \textbf{Methyl ether} & [82652-37-7] \\ C_{21}H_{30}O_5 & \text{mol. wt. 362.47} \\ CO(CH_2)_8CH_3 & \text{Decanoyl furapiole} \end{array}$ 

-Obtained by reaction of decanoyl chloride with furapiole in the presence of fused zinc chloride in methylene chloride for 3–4 h in an ice bath (70 %) [2179].

colourless plates; m.p. 53° [2179]; ¹H NMR [2179], IR [2179].

USE: Synergistic insecticidal activity of, with pyrethrum [2179].

#### 1-(6,7-Dihydroxy-5-propyl-1,3-benzodioxol-4-yl)-1-decanone

	$C_{20}H_{30}O_5$	mol. wt. 350.46
ОН	Synthesis	
0 OH	-Refer to: [2179].	
	<b>Dimethyl ether</b> [82652-30-0]	
$O \sim C_3 H_7$	$C_{22}H_{34}O_5$	mol. wt. 378.51
CO(CH ₂ ) ₈ CH ₃	Decanoyl dihydrodillapiole	

-Obtained by reaction of decanoyl chloride with dihydrodillapiole in the presence of fused zinc chloride in methylene chloride for 3–4 h in an ice bath (70 %) [2179].

colourless solid [2179]; ¹H NMR [2179], IR [2179].

USE: Synergistic insecticidal activity of, with pyrethrum [2179].

## 10-(Acetyloxy)-1-(2,3-dihydroxy-4-methoxy-6-methylphenyl)-1-decanone

[104967-09-1]	$C_{20}H_{30}O_6$	mol. wt. 366.45
HO HO CH ₃ O CH ₃ O CH ₃ O CH ₃ CH ₂	of 10-acet 3,4,5-trime ethane in chloride (1	by Friedel-Crafts reaction toxy-decanoyl chloride with thoxytoluene in 1,2-dichloro- the presence of aluminium 3.1 mol) at $20^{\circ}$ for 72 h al of two isomers) [1147].

colourless oil [1147]; ¹H NMR [1147], IR [1147], MS [1147].

and

# 10-(Acetyloxy)-1-(2,4-dihydroxy-3-methoxy-6-methylphenyl)-1-decanone

[104967-08-0]	$C_{20}H_{30}O_{6}$	mol. wt. 366.45
CH ₃ OH HOCO(CH ₂ ) ₈ CH ₂ CH ₃	3,4,5-trime ethane in t	by Friedel-Crafts reaction etoxy-decanoyl chloride with thoxytoluene in 1,2-dichloro- he presence of aluminium chlo- ol) at $20^{\circ}$ for 72 h (35 %) (total of rs) [1147].

colourless oil [1147]; ¹H NMR [1147], IR [1147], MS [1147].

# 1-[3-Chloro-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-decanone

[102370-52-5]	$C_{20}H_{31}ClO_2$	mol. wt. 338.92
CI CO(CH ₂ ) ₈ CH ₃ -C	(nthesis Obtained by Fries rearrangement o utylphenyl caprate with aluminium 58 %) [3119]. p. ₁₃ 162° [3119].	

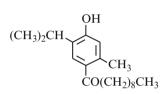
**2,4-Dinitrophenylhydrazone** [102946-84-9] C₂₆H₃₅ClN₄O₅ mol. wt. 519.04 m.p. 185° [3119].

## 1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-decanone

[60170-85-6]	$C_{20}H_{32}O_2$	mol. wt. 304.47
OH CO(CH ₂ ) ₈ CH ₃	Synthesis -Obtained by photo-Fries butylphenyl decanoate [148].	rearrangement of 4-tert-
$\int_{C(CH_3)_3}$	USE: Retard photodegradatio air [148].	n of high-d polyethylene in

# 1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-decanone

C20H32O2



Synthesis -Refer to: [2660]. Methyl ether (XI) C₂₁H₃₄O₂ mol. wt. 318.50 -Obtained by reaction of capryl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (50 %) [2660].

b.p.₁₄ 230–231° [2660];  $n_D^{24} = 1.5125$  [2660].

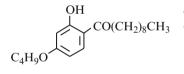
# 1-[4-(Butyloxy)-2-hydroxyphenyl]-1-decanone

[24313-96-0]

C₂₀H₃₂O₃

mol. wt. 320.47

mol. wt. 304.47



CO(CH₂)₈CH₃ -Obtained by reaction of decanoyl chloride with m-dibutoxybenzene in the process. chloride in dichloroethane at  $0^{\circ}$ . Then, the mixture was stirred for 1 h at 10°, and then for 6 h at 20–25°. The temperature was then raised to  $80^{\circ}$ and stirring was continued at that temperature for 5 h [1194].

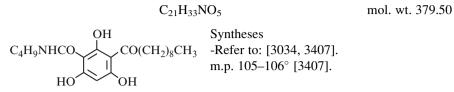
IR [1194, 1195].

# 1-[5-(1,1-Dimethylethyl)-2,4-dihydroxyphenyl]-1-decanone

	$C_{20}H_{32}O_3$	mol. wt. 320.47
OH CO(CH ₂ ) ₈ CH ₃	Syntheses -Refer to: [1051, 3089]. <b>Oxime, nickel complex</b>	[82322-05-2]

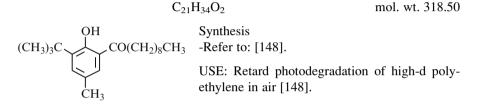
USE: Colour photog. stabilizer [1051]; Singlet oxygen quencher, laser recording materials contg. cyanine dye and, [3089].

#### 1-[3-(Butylaminocarbonyl)-2,4,6-trihydroxyphenyl]-1-decanone



BIOLOGICAL ACTIVITY: Cytotoxicity [3034]; Inhibition of Epstein-Barr virus early antigen activation [3034].

### 1-[3-(1,1-Dimethylethyl)-2-hydroxy-5-methylphenyl]-1-decanone



# 1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methylphenyl]-1-decanone

C ₂₁	$H_{34}O_2$	mol. wt. 318.50
ОН	Syntheses	
$(CH_3)_3C$ $CO(CH_2)_8CH_3$	-Obtained by Fries rearrangem	ent of 2-tert-butyl-
	5-methylphenyl decanoate,	
CH ₃	*in the presence of alumini	um chloride (1.5
	equiv.) in nitrobenzene at 2	5° for 6 h (74 %)
	[3118];	
	11 11 (2 1 ) 1100 0 2	

*in the presence of aluminium chloride (3 equiv.) at 110° for 2 h (74 %) [3118].

b.p.₈ 180° [3117, 3118].

# 2,4-Dinitrophenylhydrazone C₂₇H₃₈N₄O₅

$$C_{27}H_{38}N_4O_5$$
 mol. wt. 498.62

m.p. 113° [3117, 3118].

#### 1-[2-Hydroxy-5,6-dimethyl-3-(1-methylethyl)phenyl]-1-decanone

[102456-07-5]	$C_{21}H_{34}O_2$	mol. wt. 318.50
(CH ₃ ) ₂ CH (CH ₃ ) ₂ CH (CH ₃ ) ₂ CH (CH ₃ ) ₂ CH ₃ (CH ₃ ) ₂ CH	Synthesis -Obtained ( <b>XXXVII</b> ) by chloride with 4-methylt (4,5-dimethyl-2-isopropy presence of aluminium disulfide at r.t. for 48 h (	hymol methyl ether l-anisole) ( <b>II</b> ) in the chloride in carbon

yellow oil [2663]; b.p._{0.6} 170° [2663]; m.p. 0° [2663].

## 1-[2,4,6-Trihydroxy-3-(3-methylbutyl)phenyl]-1-decanone

# 1-(2,4,6-Trihydroxy-3-pentylphenyl)-1-decanone

 $\begin{array}{ccc} [74478-12-9] & C_{21}H_{34}O_4 & \text{mol. wt. 350.50} \\ & OH & Synthesis \\ C_5H_{11} & CO(CH_2)_8CH_3 & -\text{Refer to: [2111].} \\ & \text{BIOLOGICAL ACTIVITY: Fungicidal [2111].} \end{array}$ 

## 1,10-Bis(5-bromo-2,4-dihydroxyphenyl)-1,10-decanedione

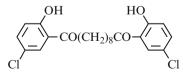
[37174-78-0]  $C_{22}H_{24}Br_2O_6$ mol. wt. 544.24 **Synthesis** -OH -Refer to: [590]. HO CO(CH₂)₈CO m.p. 210° [590]. C22H26Br2N2O6 Dioxime [37402-01-0] mol. wt. 574.27 m.p. 110° [590]. [37174-79-1]  $C_{26}H_{32}Br_2O_6$ **Tetramethyl ether** mol. wt. 600.34 m.p. 173° [590].

Dioxime of the tetramethyl ether [37174-80-4]  $C_{26}H_{34}Br_2N_2O_6$  mol. wt. 630.37

m.p.  $143^{\circ}$  [590].

# 1,10-Bis-(5-chloro-2-hydroxyphenyl)-1,10-decanedione

C₂₂H₂₄Cl₂O₄ mol. wt. 423.34



m.p. 176° [3235].

Synthesis -Obtained by Fries rearrangement of bis-4-chlorophenyl sebacate with aluminium chloride at  $150^{\circ}$  for 1 h [3235].

898

**Dimethyl ether** [10400-56-3]

$$C_{24}H_{28}Cl_2O_4$$
 mol. wt. 451.39

-Refer to: [1575].

m.p. 125° [1575].

# 1,10-Bis(5-chloro-2,4-dihydroxyphenyl)-1,10-decanedione

[26195-11-9]	$C_{22}H_{24}$	$Cl_2O_6$	mol. wt. 455.33
HO CO(C	HO CH ₂ ) ₈ CO — OH Cl	acid dichloride with	
m.p. 202° [590,	, 591].		
<b>Dioxime</b> m.p. 171–172°	[37401-99-3] [590].	$C_{22}H_{26}Cl_2N_2O_6\\$	mol. wt. 485.36
Tetramethyl ethe	r [26086-86-2]	$C_{26}H_{32}Cl_2O_6$	mol. wt. 511.44

-Obtained by reaction of sebacic acid dichloride with 4-chlororesorcinol dimethyl ether in the presence of aluminium chloride [591]. -Also refer to: [590].

m.p. 157° [590, 591].

# Di-2,4-dinitrophenylhydrazone of the tetramethyl ether

 $[37174-76-8] \qquad \qquad C_{38}H_{40}Cl_2N_8O_{12} \qquad \qquad \text{mol. wt. } 871.69$ 

m.p. 187° [590].

# 1,10-Bis(2,4-dihydroxy-3-nitrophenyl)-1,10-decanedione

[113752-04-8]	$C_{22}H_{24}$	$N_2O_{10}$	mol. wt. 476.44
NO ₂ OH HO-CO(CH ₂	$HO NO_2$ HO OH	Synthesis -Refer to: [1337]. m.p. 227° [1337].	

#### 1,10-Bis(2-hydroxyphenyl)-1,10-decanedione

[10401-05-5] 
$$C_{22}H_{26}O_4$$
 mol. wt. 354.45  
OH HO Syntheses  
-Obtained by Fries rearrangement of phenyl sebacate  
with aluminium chloride (15–20 %) [1576] in  
tetrachloroethane for 4 h at 50–60° (21 %) [3207].

Also refer to: [15/5].

m.p. 103° [1575, 1576], 101–102° [3207]; UV [3207].

C22H28N2O4 Oxime [102758-43-0] mol. wt. 384.48

**Syntheses** 

m.p. 145° [3207].

# 1,10-Bis(3-hydroxyphenyl)-1,10-decanedione

[10365-55-6]



C22H26O4

mol. wt. 354.45

-Obtained by diazotization of 1,10-bis(3-aminophenyl)-1,10-decanedione (40-50 %) [1576].

-Also refer to: [1575].

m.p. 164° [1575, 1576].

# 1,10-Bis(4-hydroxyphenyl)-1,10-decanedione

[2533-59-7] C22H26O4 mol. wt. 354.45 Syntheses  $\rightarrow$  CO(CH₂)₈CO  $\checkmark$ Ŋ_OH HO--Obtained by Fries rearrangement of phenyl sebacate with aluminium chloride in tetrachloroethane for 4 h at

50-60° (40 %) [3207].

-Obtained by reaction of sebacic acid dichloride (sebasoyl chloride) with phenol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

-Also refer to: [445, 1337].

m.p. 212° [1337], 197° [585], 196° [445], 136° [3207].

# Dioxime

# C22H28N2O4

mol. wt. 384.48

m.p. 127–129° [1337].

# **Di-2.4-dinitrophenylhydrazone** [2630-99-1] $C_{34}H_{34}N_8O_{10}$ mol. wt. 714.69

m.p. 224° [585].

Diacetate	[2619-46-7]	$C_{26}H_{30}O_{6}$	mol. wt. 438.52
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m.p. 117–118° [1337], 117° [445].

**Dimethyl ether** [2525-88-4] C₂₄H₃₀O₄ mol. wt. 382.50

-Obtained by reaction of sebacic acid with anisole in the presence of boron trifluoride at  $75-80^{\circ}$  for  $45 \min(>60 \%)$  [2597].

-Also obtained by reaction of sebacic acid dichloride with anisole [585] in the presence of aluminium chloride (80–90 %) [1576], (82 %) [905].

-Also obtained by methylation of 1,10-bis(4-hydroxyphenyl)-1,10-decanedione [585]. -Also refer to: [1337, 1575, 2596 (90 %)].

m.p. 130° [585], 120–121° [1337, 2597], 120° [1575, 1576], 119–119.5° [2596], 118.5–119.5° [905].

# Di-2,4-dinitrophenylhydrazone of the dimethyl ether

[24339-81-9]	$C_{36}H_{38}N_8O_{10}$	mol. wt. 742.75.
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m.p. 142° [585].

# 1,10-Bis(2,4-dihydroxyphenyl)-1,10-decanedione

1,8-Di(2,4-dihydroxy-1-benzoyl)octane (X) [3207]

[3088-14-0]	$C_{22}H_{2}$	mol. wt. 386.45
HO HO HO	он со-Сон он	Syntheses -Obtained by reaction of sebacic acid with resorcinol, *in the presence of zinc chloride for 3–5 h at 140° (85 %) or for 3–5 h at 110–115° (31 %) [445];

*in the presence of p-toluenesulfonic acid in xylene at  $170-180^{\circ}$  for 12 h (30 %) [3207];

*in the presence of boron trifluoride in anisole at  $75-80^{\circ}$  for 45 min (65 %) [2597]. -Also obtained by adding resorcinol into the dinitrile of sebacic acid and hydrogen chloride in ethyl ether in the presence of zinc chloride. Then, the diketimine dichlorhydrate obtained was hydrolyzed by boiling water (90 %) [2674].

-Also obtained by reaction of sebacic acid dichloride with resorcinol in the presence of aluminium chloride [591].

-Also refer to: [381, 590, 1735, 2504, 2607, 3468].

m.p. 174° [2597], 172° [2504, 2674], 171° [590, 591], 168° [381, 445, 2607, 3207]; UV [2504].

# Monohydrate

 $C_{22}H_{26}O_6, H_2O$ 

mol. wt. 404.46

-Refer to: [2597].

# 2,4-Dinitrophenylhydrazone $C_{28}H_{30}N_4O_9$ mol. wt. 566.57 m.p. 68° [2674]. **Di-2,4-dinitrophenylhydrazone** [37166-99-7] C₃₄H₃₄N₈O₁₂ mol. wt. 746.69 -Refer to: [2597]. m.p. 268° [2674], 266° [590]. Tetraacetate [13379-59-4] $C_{30}H_{34}O_{10}$ mol. wt. 554.59 -Obtained by treatment of this diketone with boiling an excess of acetic anhydride for some min [2597]. -Also refer to: [445, 2607]. m.p. 108–109° [445], 108° [2607], 105° [2597]. **Tetramethyl ether** [37167-02-5] C₂₆H₃₄O₆ mol. wt. 442.55 -Refer to: [590, 1337]. m.p. 128° [590], 127° [1337]. Di-2,4-dinitrophenylhydrazone of the tetramethyl ether [37402-34-9] C₃₈H₄₂N₈O₁₂ mol. wt. 802.80 m.p. 137° [590].

1,10-Bis(2,5-dihydroxyphenyl)-1,10-decanedione

$C_{22}H_{26}O_{6}$		mol. wt. 386.45
$ \begin{array}{c} OH & HO \\ \hline \\ -CO(CH_2)_8CO & \hline \\ HO & OH \end{array} $	Synthesis -Refer to: [2331]. <b>Tetramethyl ether</b> $C_{26}H_{34}O_6$	[10365-10-3] mol. wt. 442.55

-Preparation from hydroquinone dimethyl ether and sebacoyl chloride [2331]. -Obtained by reaction of sebacic acid dichloride (1 mol) with hydroquinone dimethyl ether (2.5 mol) in the presence of aluminium chloride (1 mol) (88 %) [1575].

-Also refer to: [1575].

needles [2331]; m.p.  $103^{\circ}$  [1575], 99–100° [2331].

#### 1,10-Bis(3,4-dihydroxyphenyl)-1,10-decanedione

$$C_{22}H_{26}O_6$$
  
HO OH  
HO CO(CH₂)₈CO OH

Synthesis -Obtained by reaction of sebacic acid with pyrocatechol in the presence of boron trifluoride in anisole at 75–80°

for 45 min [2597].

-Obtained by reaction of sebacic acid dichloride with pyrocatechol dimethyl ether in the presence of aluminium chloride [591]. -Also refer to: [2342, 3060].

m.p. 106–107° [3060], 103° [591]; ¹H NMR [2342], ¹³C NMR [2342].

#### 1,10-Bis(3,5-dihydroxyphenyl)-1,10-decanedione

 $\begin{array}{c} C_{22}H_{26}O_{6} & \text{mol. wt. 386.45} \\ HO & OH & \text{Synthesis} \\ -CO(CH_{2})_{8}CO & -CO(CH_{2})_{$ 

#### 1,10-Bis(2,3,4-trihydroxyphenyl)-1,10-decanedione

[13178-17-1]	$C_{22}H_{20}$	₆ O ₈ mol. wt. 418.44
HO OH HO CO(CH ₂ ) ₈ C	но он	Synthesis -Obtained by reaction of sebacic acid with pyrogallol in the presence of boron trifluoride in anisole at 75–80° for 45 min (46 %) [2597].

m.p. 182–183° [2597].

# **Di-2,4-dinitrophenylhydrazone** $C_{34}H_{34}N_8O_{14}$ mol. wt. 778.69

Refer to: [2597].

#### Hexaacetate

 $C_{34}H_{38}O_{14}$  mol. wt. 670.67

-Obtained by reaction of acetic anhydride with the title ketone [2597].

mol. wt. 386.45

#### Hexamethyl ether [10373-33-8] C28H38O8 mol. wt. 502.61

-Obtained by reaction of sebacic acid dichloride with pyrogallol trimethyl ether in the presence of aluminium chloride [591].

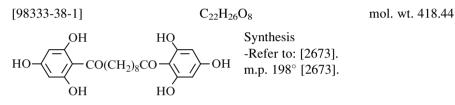
-Also obtained by reaction of dimethyl sulfate with 1,10-bis(2-hydroxy-3.4-dimethoxyphenyl)-1,10-decanedione the presence of 30 % sodium hydroxide (65-90 %) [1574].

-Also refer to: [1575].

m.p. 126° [591], 77° [1574, 1575].

**N.B.**: One the reported melting point is obviously wrong.

# 1,10-Bis(2,4,6-trihydroxyphenyl)-1,10-decanedione

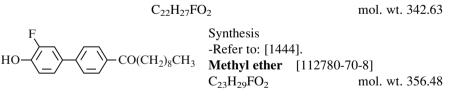


# 1-(3'-Bromo-4'-hydroxy[1,1'-biphenyl]-4-yl)-1-decanone

$C_{22}H_{27}BrO_2$		mol. wt. 403.36
Br HO CO(CH ₂ ) ₈ CH ₃	Synthesis -Refer to: [1444]. <b>6-Methyloctyl ether</b> (S) $C_{31}H_{45}BrO_2$	[112780-54-8] mol. wt. 529.60

-Refer to: [1444].

# 1-(3'-Fluoro-4'-hydroxy[1,1'-biphenyl]-4-yl)-1-decanone



-Refer to: [1444].

# 1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-1-decanone

[93249-83-3] C22H28O2 mol. wt. 324.46 CO(CH₂)₈CH₃ Syntheses -Refer to: [68, 1647, 2995, 3075]. HO

USE: In preparation of liquid crystals [1647].

Acetate [93249-88-8]	$C_{24}H_{30}O_3$	mol. wt. 366.49
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-Refer to: [1647, 3075].

USE: In preparation of liquid crystals [1647].

# Various ethers (11)

-Preparations and liquid crystalline properties of, [847].

Methyl ether	[56116-82-6]	$C_{23}H_{30}O_2$	mol. wt. 338.49
Ethyl ether	[56116-91-7]	$C_{24}H_{32}O_2$	mol. wt. 352.52
Propyl ether	[56116-99-5]	$C_{25}H_{34}O_2$	mol. wt. 366.54
Butyl ether	[56117-07-8]	$C_{26}H_{36}O_2$	mol. wt. 380.57
Pentyl ether	[56117-16-9]	$C_{27}H_{38}O_2$	mol. wt. 394.60
Hexyl ether	[56117-25-0]	$C_{28}H_{40}O_2$	mol. wt. 408.62
Heptyl ether	[56117-33-0]	$C_{29}H_{42}O_2$	mol. wt. 422.65
Octyl ether	[56117-42-1]	$C_{30}H_{44}O_2$	mol. wt. 436.68
Nonyl ether	[56117-51-2]	$C_{31}H_{46}O_2$	mol. wt. 450.71
Decyl ether	[56117-60-3]	$C_{32}H_{48}O_2$	mol. wt. 464.73
<b>Dodecyl ether</b>	[56117-69-2]	$C_{34}H_{52}O_2$	mol. wt. 492.19

# 1-(4,4'-Dihydroxy[1,1'-biphenyl]-3-yl)-1-decanone

[36756-42-0]	$C_{22}H_{28}O_3$	mol. wt. 340.46
HO-CO(CH ₂ ) ₈ CH ₃	Synthesis -Preparation by Fries 4,4'-biphenyl dicaprate chloride in refluxing chlor (19 %) [2091].	with aluminium

m.p. 90–91° [2091].

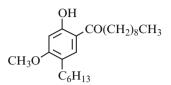
# 2-Hexyl-1-(6-hydroxy-1,3-benzodioxol-5-yl)-1-decanone

[103449-09-8]	$C_{23}H_{34}O_4$	mol. wt. 374.52
O OH	Synthesis	

USE: In prepn. of antistaining agent for colour photog. paper [2326].

#### 1-(5-Hexyl-2-hydroxy-4-methoxyphenyl)-1-decanone

[143287-08-5]	$C_{23}H_{38}O_3$	mol. wt. 362.55
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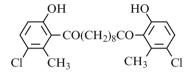
Synthesis -Obtained (**12c**) by reaction of bromomethane with 1-(2,4-dihydroxy-5-hexylphenyl)-1-decanone inthe presence of potassium carbonate in refluxingacetone for 20 h [284].m.p. 49–50° [284].

# 1,10-Bis(3-chloro-6-hydroxy-2-methylphenyl)-1,10-decanedione

[25715-28-0]

 $C_{24}H_{28}Cl_2O_4$ 

mol. wt. 451.39



Synthesis

-Obtained by reaction of sebacic acid dichloride (sebasoyl chloride) with 4-chloro-3-methylphenol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

needles [585].

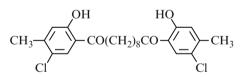
**Di-2,4-dinitrophenylhydrazone** [25779-68-4] C₃₆H₃₆Cl₂N₈O₁₀ mol. wt. 811.64

m.p. 193° [585].

#### 1,10-Bis(5-chloro-2-hydroxy-4-methylphenyl)-1,10-decanedione

 $C_{24}H_{28}Cl_2O_4$ 

mol. wt. 451.39



Synthesis -Obtained by reaction of sebacic acid dichloride (sebasoyl chloride) with 4-chloro-3-methylphenol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

needles [585].

# Di-2,4-dinitrophenylhydrazone

$$C_{36}H_{36}Cl_2N_8O_{10}$$
 mol. wt. 811.64

m.p. 193° [585].

#### 1,10-Bis(2-hydroxy-3-methylphenyl)-1,10-decanedione

[10483-69-9]		$C_{24}H_{30}O_4$	mol. wt. 382.50
$CH_3 OH$ $CO(CH_2)_8CO$	HO CH ₃	Syntheses -Obtained by Fries rear sebacate with aluminiu [1576] in tetrachloroeth (13 %) [3207].	m chloride (15–20 %)

-Also refer to: [1575].

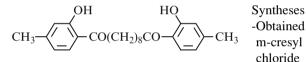
m.p. 107° [1575, 1576], 106° [3207]; UV [3207]. **Dioxime** [115097-69-3]  $C_{24}H_{32}N_2O_4$  mol. wt. 412.53 m.p. 170° [184].

#### 1,10-Bis(2-hydroxy-4-methylphenyl)-1,10-decanedione

[10400-43-8]

 $C_{24}H_{30}O_4$ 

mol. wt. 382.50



-Obtained by Fries rearrangement of m-cresyl sebacate with aluminium chloride (70–80 %) [1576] in tetrachloroethane for 4 h at 50–60° (53 %) [3207].

-Also refer to: [1575].

m.p. 121° [1575, 1576], 120.5° [3207]; UV [3207].

## 1,10-Bis(2-hydroxy-5-methylphenyl)-1,10-decanedione

[13282-28-5]  $C_{24}H_{30}O_4$  mol. wt. 382.50 OH HO Syntheses -Obtained by Fries rearrangement of di-pcresyl sebacate with aluminium chloride, *in refluxing chlorobenzene for 6 h (79 %) [3107];

*in tetrachloroethane for 4 h at  $50-60^{\circ}$  (62 %) [3207].

-Also obtained by reaction of sebacic acid dichloride (sebasoyl chloride) with p-cresol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

m.p. 126–127° [3107], 126° [585], 110.5° [3207]; IR [3107], UV [3207]. **Dioxime** [115096-86-1] C₂₄H₃₂N₂O₄ mol. wt. 412.53

m.p. 178° [3207].

# $\label{eq:24340-07-6} \textbf{Di-2,4-dinitrophenylhydrazone} \quad [24340-07-6] \quad C_{36}H_{38}N_8O_{10} \quad \text{mol. wt. 742.75}$

m.p. 200° [585].

**Dimethyl ether** [10400-51-8] C₂₆H₃₄O₄ mol. wt. 410.55

-Obtained by reaction of sebacic acid dichloride with 4-methylanisole in the presence of aluminium chloride (50–70 %) [1576]. -Also refer to: [1575].

m.p. 88° [1575, 1576].

# 1,10-Bis(4-hydroxy-2-methylphenyl)-1,10-decanedione

[24340-00-9]  $C_{24}H_{30}O_4$  mol. wt. 382.50 HO -  $CH_3$   $CH_3$   $CH_3$  Synthesis -Obtained by reaction of sebacic acid dichloride (sebasoyl chloride) with

dichloride (sebasoyl chloride) with m-cresol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

needles [585].

**Di-2,4-dinitrophenylhydrazone** [24340-01-0] C₃₆H₃₈N₈O₁₀ mol. wt. 742.75

m.p. 190° [585].

Dimethyl ether	[24340-02-1]	$C_{26}H_{34}O_{4}$	mol. wt. 410.55
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-Obtained by reaction of dimethyl sulfate with 1,10-bis(4-hydroxy-2-methylphenyl)-1,10-decanedione in the presence of alkali [585].

-Also obtained by reaction of sebacic acid dichloride with m-cresol methyl ether in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

m.p. 110° [585].

# Di-2,4-dinitrophenylhydrazone of the dimethyl ether

 $\begin{array}{ll} \mbox{[24340-03-2]} & C_{38}H_{42}N_8O_{10} & \mbox{mol. wt. 770.80} \\ \mbox{m.p. 150}^\circ \mbox{[585]}. \end{array}$ 

#### 1,10-Bis(4-hydroxy-3-methylphenyl)-1,10-decanedione

-Obtained by Fries rearrangement of di-o-cresyl sebacate with aluminium chloride in tetrachloroethane for 4 h at  $50-60^{\circ}$  (47 %) [3207].

-Also obtained by reaction of sebacic acid with o-cresol in the presence of boron trifluoride in chlorobenzene, first at  $60^{\circ}$ , then at  $80^{\circ}$  for 90 min (55 %) [2597].

-Also obtained by reaction of sebacic acid dichloride (sebasoyl chloride) with o-cresol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

# **Di-2,4-dinitrophenylhydrazone** $C_{36}H_{38}N_8O_{10}$ mol. wt. 742.75

m.p. 152° [585].

**Dimethyl ether** [24339-99-9] C₂₆H₃₄O₄ mol. wt. 410.55

-Obtained by reaction of dimethyl sulfate with 1,10-bis(4-hydroxy-3-methylphenyl)-1,10-decanedione in the presence of alkali [585].

-Also obtained by reaction of sebacic acid dichloride with o-cresol methyl ether [585].

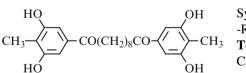
m.p. 131° [585].

**Di-2,4-dinitrophenylhydrazone**  $C_{38}H_{42}N_8O_{10}$  mol. wt. 770.80 of the dimethyl ether

m.p. 207° [585].

#### 1,10-Bis(3,5-dihydroxy-4-methylphenyl)-1,10-decanedione

C24H30O6



 Synthesis

 -Refer to: [3132].

 Tetramethyl ether
 [196869-44-0]

 C₂₈H₃₈O₆
 mol. wt. 470.61

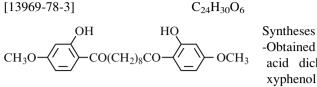
mol. wt. 414.50

-Synthesis from 3,5-dimethoxy-4-methylbenzaldehyde (75 %) [3132].

Colourless crystalline solid [3132]; ¹H NMR [3132], ¹³C NMR [3132].

mol. wt. 414.50

#### 1,10-Bis(2-hydroxy-4-methoxyphenyl)-1,10-decanedione



Syntheses -Obtained by reaction of sebacic

acid dichloride with 3-methoxyphenol in the presence of aluminium chloride [590, 591].

m.p. 122° [590, 591].

 $\textbf{Di-2,4-dinitrophenylhydrazone} \quad [37167-01-4] \quad C_{36}H_{38}N_8O_{12} \quad \text{mol. wt. 774.74}$ 

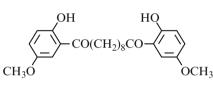
m.p. 119° [590].

# 1,10-Bis(2-hydroxy-5-methoxyphenyl)-1,10-decanedione

[10365-31-8]

 $C_{24}H_{30}O_{6}$ 

mol. wt. 414.50



Syntheses -Obtained by reaction of sebacic acid dichloride with p-dimethoxybenzene in the presence of aluminium chloride [1575].

-Also refer to: [1575] (Japanese paper).

m.p. 109° [1575].

Diacetate	[10365-37-4]	C ₂₈ H ₃₄ O ₈	mol. wt. 498.57
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-Obtained by reaction of acetic anhydride (4 vol) with the title ketone in the presence of 1-2 drops concentrated sulfuric acid, the mixture warmed 5 min [1575].

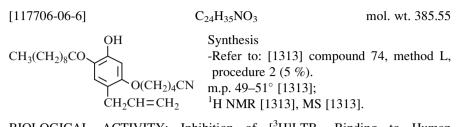
-Also refer to: [1575].

m.p. 77° [1575].

# 1-[5-Chloro-3-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-decanone

C ₂₄ H	$_{31}ClO_2$	mol. wt. 386.96
$(CH_3)_3C$ $\leftarrow$ $CO(CH_2)_8CH_3$	Synthesis -Refer to: [148].	
CI	USE: Retard photodegradation ethylene in air [148].	of high-d poly-

# 5-[5-Hydroxy-4-(1-oxodecyl)-2-(2-propenyl)phenoxy]pentanenitrile



BIOLOGICAL ACTIVITY: Inhibition of [³H]LTB₄ Binding to Human PMN [1313].

# 1-[3,5-Bis(1,1-dimethylethyl)-2-hydroxyphenyl]-1-decanone

C ₂₄	$H_{40}O_2$	mol. wt. 360.58
$(CH_3)_3C$ $(CH_2)_8CH_3$	Synthesis -Refer to: [148].	
C(CH ₃ ) ₃	USE: Retard photodegradation ethylene in air [148].	of high-d poly-

# 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-decanone

	$C_{24}H_{40}O_2$	mol. wt. 360.58
OH	Synthesis	
$(CH_3)_3C$ $C(CH_3)_3$	-Refer to: [1408].	
	<b>O-Methyloxime</b> [169888-15-7]	
$\mathbf{Y}$	$C_{25}H_{43}NO_2$	mol. wt. 389.61
ĊO(CH ₂ ) ₈ CH ₃	-Refer to: [1408].	

# 1-[2-Hydroxy-5-(1,1,3,3-tetramethylbutyl)phenyl]-1-decanone

[94899-68-0]	$C_{24}H_{40}O_2$	mol. wt. 360.58
$CH_{3}-C-CH_{2}-C-CH_{3}$	Synthesis -Refer to: [3361]. <b>Oxime</b> [94613-09-9] $C_{24}H_{41}NO_2$ IR [3362].	mol. wt. 375.60

USE: As copper extg. agent, [3361, 3362].

Methyl ether	[94899-67-9]	$C_{25}H_{42}O_2$	mol. wt. 374.61

-Refer to: [3361].

**Oxime of the methyl ether** [94613-10-2] C₂₅H₄₃NO₂ mol. wt. 389.61

IR [3362].

USE: As copper extg. agent, [3361, 3362].

# 1-[4-[(2-Ethylhexyl)oxy]-2-hydroxyphenyl]-1-decanone

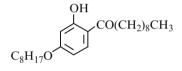
OH  $C_2H_5$   $C_4H_9$ -CH-CH₂O  $CO(CH_2)_8CH_3$   $CO(CH_2)_8CH_3$  $CO(CH_2)_8CH$ 

USE: Photog. stabilizer compns. contg. phenol derivs. and, for magenta images [1050].

# 1-[2-Hydroxy-4-(octyloxy)phenyl]-1-decanone

 $[143286-95-7] C_{24}H_{40}O_3 mol. wt. 376.58$ 

Synthesis



m.p. 45-47° [284].

Oxime	[143286-64-0]	$C_{24}H_{41}NO_3$	mol. wt. 391.59
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for 20 h [284].

-Obtained by reaction of hydroxylamine hydrochloride with 1-(4-octyloxy-2-hydroxyphenyl)-1-decanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 53–57° [284].

# 1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-1-decanone

[134082-04-5]	$C_{25}H_{34}O_5$	mol. wt. 414.54
ОН	Synthesis	

	OH	
CH ₃ O	$\nearrow$	CO(CH ₂ ) ₈ CH ₃
СН ₃ 0		OCH ₂ C ₆ H ₅

-Obtained by elimination of one phenylmethyl group in 2-position by treatment of 1-(2,6-dibenzyloxy-3,4-dimethoxyphenyl)-1-decanone with concentrated hydrochloric acid and acetic acid at r.t. for 2–3 h (80 %) [1353].

-Obtained by reaction of octyl bromide with

1-(2,4-dihydroxyphenyl)-1-decanone in the pre-

sence of potassium carbonate in refluxing acetone

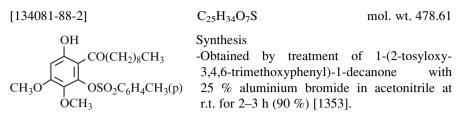
m.p. 83–83.5° [1353]; ¹H NMR [1353].

 $C_{24}H_{40}O_3$ 

mol. wt. 376.58

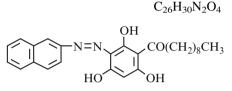
. .

#### 1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]-1-decanone



m.p. 59–60° [1353]; ¹H NMR [1353].

## 3-[(2-Naphthylazo)-2,4,6-trihydroxyphenyl]-1-decanone



 $\begin{array}{ccc} & & Synthesis \\ \hline & CO(CH_2)_8CH_3 & -Obtained & by & coupling & diazotized \\ \hline & & 2\text{-naphthyl-amine} & with & 2,4,6\text{-tri-} \\ OH & & hydroxyphenyl nonyl ketone [872]. \end{array}$ 

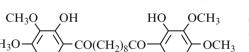
#### 1,10-Bis(4-hydroxy-2,6-dimethylphenyl)-1,10-decanedione

 $[107259-37-0] C_{26}H_{34}O_4$ mol. wt. 410.55 HO  $\leftarrow CO(CH_2)_8CO \leftarrow CH_3$  Synthesis -Refer to: [875]. m.p. 128–130° [875].

 $C_{26}H_{34}O_8$ 

#### 1,10-Bis(2-hydroxy-3,4-dimethoxyphenyl)-1,10-decanedione

[10351-93-6]



mol. wt. 474.55

mol. wt. 434.54

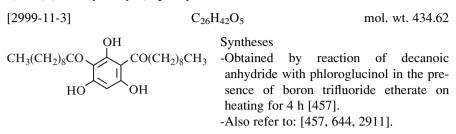
Syntheses

-Obtained by reaction of sebacic acid dichloride with pyrogallol trimethyl ether in the presence of aluminium chloride [3058] in tetrachloroethane [1574].

-Also refer to: [590, 1575].

m.p. 126° [590], 125–126.5° [3058], 125° [1574, 1575].

#### 1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-decanone



m.p. 80-82° [457, 2911].

BIOLOGICAL ACTIVITY: Antiviral activity towards the replication of vesicular stomatitis virus in HEL cell cultures [644]; Anthelmintic [457].

# 1-(4-Butoxy-5-hexyl-2-hydroxyphenyl)-1-decanone

[143287-09-6]	$C_{26}H_{44}O_3$	mol. wt. 404.63
OH C ₄ H ₉ O C ₆ H ₁₃	H ₂ ) ₈ CH ₃ Synthesis -Obtained ( <b>12d</b> ) by reaction 1-(2,4-dihydroxy-5-hexyl the presence of potassiun acetone for 20 h [284]. m.p. 38° [284].	phenyl)-1-decanone in

Oxime	[143287-10-9]	C ₂₆ H ₄₅ NO ₃	mol. wt. 419.65
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m.p. 48-50° [284].

### 1-(2-Decyl-4,5-dihydroxyphenyl)-1-decanone

	$C_{26}H_{44}O_3$		mol. wt. 404.63
ОН НО	Synthesis -Refer to: [2366].		
C ₁₀ H ₂₁	<b>Dimethyl ether</b> $C_{28}H_{48}O_3$	[919800-80-9]	mol. wt. 432.69
CO(CH ₂ ) ₈ CH ₃			

-Refer to: [2366].

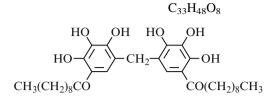
¹H NMR [2366].

USE: Supramolecular fibers and microbelts from a phthalhydrazide derivative of crown ether with alkyl chains [2366].

# 1,1'-(2,4,6-Trihydroxy-5-methyl-1,3-phenylene)bis-1-decanone

mol. wt. 448.64 [4807-43-6] C₂₇H₄₄O₅ OH **Syntheses** CO(CH₂)₈CH₃ -Obtained by reaction of decanoic anhydride CH₃(CH₂)₈CO with 2-methylphloroglucinol in the presence of boron trifluoride etherate on HO heating for 4 h [457]. -Also refer to: [457, 2911]. m.p. 99–101° [457, 2911]. **BIOLOGICAL ACTIVITY: Anthelmintic [457].** 1,10-Bis(2-hydroxy-3,4,5-trimethylphenyl)-1,10-decanedione [84978-22-3] C28H38O4 mol. wt. 438.61 HO CH₃ Synthesis CH₂ OH -Refer to: [2325]. -CH₃ CH₃ CO(CH₂)₈CO m.p. 135–142° [2325]; ¹H NMR [2325], IR [2325]. CH₃ 1,10-Bis(2-hydroxy-3,4,6-trimethylphenyl)-1,10-decanedione [84978-16-5] C28H38O4 mol. wt. 438.61 Synthesis CH₃ OH HO CH₃ -Refer to: [2325]. -CH₃ m.p. 125–129.5° [2325]; ¹H NMR CH₂ CO(CH₂)₈CO [2325], IR [2325]. . CH3  $CH_3$ 3-[(4-Octylphenylazo)-2,4,6-trihydroxyphenyl]-1-decanone mol. wt. 496.69  $C_{30}H_{44}N_2O_4$ Synthesis CO(CH₂)₈CH₃ -Obtained bv coupling p-octylazobenzene with 2,4,6-trihydroxyphenyl nonyl ketone [872]. 1,1'-(4,4'-Dihydroxy[1,1'-biphenyl]-3,3'-diyl)-1-decanone [36677-71-1] C32H46O4 mol. wt. 494.71 CH₃(CH₂)₈CC CO(CH₂)₈CH₃ Synthesis -Preparation by Fries rearrangement of HO OH 4,4'-biphenyl dicaprate with aluminium chloride in refluxing chlorobenzene for 24 h (75 %) [2091].

## 1,1'-[Methylenebis(2,3,4-trihydroxy-5,1-phenylene)]bis-1-decanone



mol. wt. 572.74

mol. wt. 552.80

Synthesis -Obtained by treatment of 4-decanoyl-pyrogallol with 35 % aqueous solution of formaldehyde in refluxing ethanol for 10 min [506].

m.p. 147–148° [506].

# 3-[(4-Dodecylphenylazo)-2,4,6-trihydroxyphenyl]-1-decanone

3'-(p-Dodecylphenylazo)-2',4',6'-trihydroxydecanophenone

 $C_{34}H_{52}N_2O_4$   $C_{12}H_{25}$   $\longrightarrow$  N=N  $CO(CH_2)_8CH_3$ HO OH

Syntheses	
-Obtained by coupling	diazotized
dodecylaniline with	2,4,6-tri-
hydroxy-phenyl	nonyl
ketone [872].	
-Also refer to: [2433].	

USE: As water-repellent dye for wood [2433].

# 2 Aromatic Hydroxyketones Derived from Various Halogenodecanoic Acids

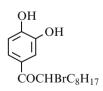
# 2.1 Unsubstituted Hydroxyketones

# 2-Bromo-1-(3,4-dihydroxyphenyl)-1-decanone

Synthesis

$$C_{16}H_{23}BrO_3$$

mol. wt. 343.26

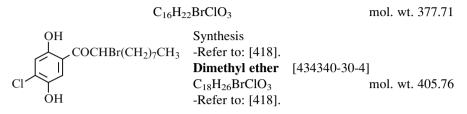


-Refer to: [2657]. **Dibenzyl ether**  $C_{30}H_{35}BrO_3$  mol. wt. 523.51 -Obtained by reaction of N-bromosuccinimide with 3,4-(dibenzyloxy)capriphenone in carbon tetrachloride in the presence of benzoyl peroxide at 50° (85–90 %) [2657].

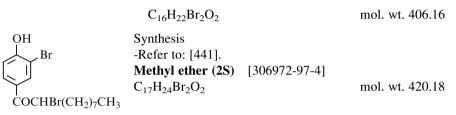
m.p. 90° [2657].

# 2.2 Substituted Hydroxyketones

# 2-Bromo-1-(4-chloro-2,5-dihydroxyphenyl)-1-decanone



# 2-Bromo-1-(3-bromo-4-hydroxyphenyl)-1-decanone



-Preparation of nonracemic  $\alpha$ -bromoketones by stereoselective bromination of aryl alkyl tartrate acetals followed by hydrolysis (43 %, >98 % ee) [441].

m.p. 58–59° [441]; ¹H NMR [441], ¹³C NMR [441], IR [441], MS [441].

# 10-Bromo-1-[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-decanone

C ₂₀	$C_{20}H_{31}BrO_2$		
(CH ₃ ) ₂ CH	Synthesis -Refer to: [220].		
	<b>Methyl ether</b> [72236-95-4]		
CH ₃	$C_{21}H_{33}BrO_2$	mol. wt. 397.39	
$CO(CH_2)_8CH_2Br$			

-Obtained by reaction of 10-bromodecanoyl chloride with thymol methyl ether in the presence of aluminium chloride in methylene chloride at r.t. (90 %) [220].

 $n_{\rm D}^{20} = 1.5262$  [220].

BIOLOGICAL ACTIVITY: Amebicidal and bactericidal and molluscicidal activity of, [220].

mol. wt. 278.35

#### **Aromatic Hydroxyketones Derived** 3 from 10-Oxodecanoic Acids

#### 3.1 Unsubstituted Hydroxyketones

# 10-(2-Hvdroxvphenvl)-10-oxo-1-decanoic acid

$$\begin{array}{cccc} [101253-68-3] & C_{16}H_{22}O_4 & \text{mol. wt. } 278.35 \\ OH & Syntheses \\ \hline & CO(CH_2)_8CO_2H & -Refer to: [3122, 3135]. \\ \hline & m.p. \ 91^\circ \ [3122], \ 84^\circ \ [3135]. \end{array}$$

# 10-(4-Hydroxyphenyl)-10-oxo-1-decanoic acid

[24339-95-5]

Syntheses

C16H22O4

 $HO - CO(CH_2)_8 CO_2 H$ -Obtained by reaction of sebacic acid dichloride (sebasoyl chloride) with phenol in the presence of aluminium chloride in nitrobenzene first at  $0-5^{\circ}$  for 1 h, then at r.t. for 4 h [585].

-Also refer to: [445, 1131, 2392, 3135].

m.p. 114° [585], 113° [3135], 110° [445]; ¹H NMR [1131].

BIOLOGICAL ACTIVITY: Refer to: [1131].

#### 2,4-Dinitrophenylhydrazone [24339-96-6] C₂₂H₂₆N₄O₇ mol. wt. 458.47

m.p. 160° [585].

Methyl ether	[24339-93-3]	$C_{17}H_{24}O_4$	mol. wt. 292.37
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-Obtained by reaction of  $\omega$ -carbethoxypelargonoyl chloride with anisole in the presence of aluminium chloride in tetrachloroethane at  $0^{\circ}$  for 3–4 h (96 %) [2392]. -Also obtained by reaction of sebacic anhydride with anisole in the presence of aluminium chloride in a mixture of nitrobenzene and tetrachloroethane first at  $0^{\circ}$ , then at  $<5^{\circ}$  for 3 days (51 %) [2367].

-Also obtained by reaction of sebacic acid dichloride with anisole [585].

colourless needles [2367]; m.p. 130° [585], 101.5–102° [2392], 98–98.5° [2367]. **N.B.**: One of the reported melting point is obviously wrong.

# 2,4-Dinitrophenylhydrazone of the methyl ether

[24339-80-8]	$C_{23}H_{28}N_4O_7$	mol. wt. 472.50
m.p. 79° [585].		

# Ethyl ether $C_{18}H_{26}O_4$ mol. wt. 306.40

-Obtained by reaction of  $\omega$ -carbethoxypelargonoyl chloride with phenetole in the presence of aluminium chloride in tetrachloroethane at 0° for 3–4 h (90 %) [2392].

m.p. 101–101.5° [2392].

# 10-(2,4-Dihydroxyphenyl)-10-oxo-1-decanoic acid

[858189-64-7]	$C_{16}H_{22}O_5$	mol. wt. 294.35
HO HO HO	with resorcinol (Hoesch -Also obtained by react	of sebacic acid dinitrile reaction) (90 %) [445]. ion of sebacic acid with we of zinc chloride at $140^{\circ}$

-Also refer to: [1056, 2606, 2607].

m.p. 122° [445, 2606], 121.5–122.5° [1056], 119° [2607].

**BIOLOGICAL ACTIVITY:** Antibacterial [1056].

Methyl ester	[854909-05-0]	$C_{17}H_{24}O_5$	mol. wt. 308.37
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-Refer to: [445].

b.p._{0.3} 245° [445].

Ethyl ester	[859994-77-7]	$C_{18}H_{26}O_5$	mol. wt. 322.40

-Preparation by adding the acid chloride of ethyl hydrogen azelate to a mixture of resorcinol and aluminium chloride in tetrachlorethane over 1 h. Then, the mixture was stirred 24 h at r.t. (51 %) [489].

-Also refer to: [1056 (60 %)].

b.p._{0.3} 220–225° [1056], b.p._{0.5} 222–226° [489], b.p._{0.5} 233–234° [1056]; m.p. 47° [1056]; ¹H NMR [489], IR [489].

BIOLOGICAL ACTIVITY: Antibacterial [1056].

# 10-(2,5-Dihydroxyphenyl)-10-oxo-1-decanoic acid

 Ethyl ester
 [858189-97-6]
  $C_{18}H_{26}O_5$  mol. wt. 322.40

 -Refer to:
 [1056] (40 %).

 $b.p._{0.5} \ 210^\circ \ [1056]; \quad m.p. \ 45^\circ \ [1056].$ 

# 3.2 Substituted Hydroxyketones

# 10-(5-Chloro-2-hydroxyphenyl)-10-oxo-1-decanoic acid

[24340-04-3]	$C_{16}H_{21}ClO_4$	mol. wt. 312.79
OH -CO(CH ₂ ) ₈ CO ₂ H Cl	Synthesis -Obtained by reaction of sebaci (sebasoyl chloride) with p-chloro sence of aluminium chloride in m $0-5^{\circ}$ for 1 h, then at r.t. for 4 h [58]	phenol in the pre- trobenzene first at
m.p. 134° [585].		

**2,4-Dinitrophenylhydrazone** [24340-05-4] C₂₂H₂₅ClN₄O₇ mol. wt. 492.92 m.p. 198° [585].

# 10-(2-Hydroxy-5-methylphenyl)-10-oxo-1-decanoic acid

	$C_{17}H_{24}O_4$	mol. wt. 292.37
CO(CH ₂ ) ₈ CO ₂ H	Synthesis -Refer to: [1827]. UV [1827].	

OH

#### 10-(4-Hydroxy-3-methylphenyl)-10-oxo-1-decanoic acid

[24339-89-7]	$C_{17}H_{24}O_4$	mol. wt. 292.37
HO $\leftarrow$ CH ₃ $\leftarrow$ CO(CH ₂ ) ₈ CO ₂ H	Synthesis -Obtained by reaction of sebaci (sebasoyl chloride) with o-cresc of aluminium chloride in nitre $0-5^{\circ}$ for 1 h, then at r.t. for 4 h	ol in the presence obenzene first at
m.p. 83° [585].		
BIOLOGICAL ACTIVITY: R	efer to: [2770].	

**2,4-Dinitrophenylhydrazone** [24339-88-6] C₂₃H₂₈N₄O₇ mol. wt. 472.50

m.p. 105° [585].

#### Methyl ether C18H26O4 mol. wt. 306.40

-Obtained by reaction of dimethyl sulfate with 10-(4-hydroxy-3-methylphenyl)-10-oxo-1-decanoic acid in the presence of alkali [585].

m.p. 100° [585].

# 10-(2-Hydroxy-5-methoxyphenyl)-10-oxo-1-decanoic acid

	$C_{17}H_{24}O_5$	mol. wt. 308.37
OH CO(CH ₂ ) ₈ COOH	Synthesis -Refer to: [1056] (Japanese paper). m.p. 65–66° [1056].	

# Ethyl ester

C₁₉H₂₈O₅

mol. wt. 336.43

-Refer to: [1056 20 %)].

b.p._{0.5} 199–206° [1056].

**BIOLOGICAL ACTIVITY:** Antibacterial [1056].

#### 10-(4-Methyl-2,3,5-trihydroxyphenyl)-10-oxo-1-decanone

(	$C_{17}H_{24}O_6$	mol. wt. 324.37
HO HO HO HO HO HO HO HO HO HO HO HO HO H	Synthesis -Refer to: [2681].	
CH ₃ OH	¹³ C NMR [2681], MS [2681].	¹ H NMR [2681], IR [2681], UV [2681],

# 10-(2-Hydroxy-3,4,5-trimethylphenyl)-10-oxo-1-decanone

[84978-21-2]	$C_{19}H_{28}O_4$	mol. wt. 320.43
$CH_{3} \xrightarrow[CH_{3}]{} CO(CH_{2})_{8}CO_{2}H$ $CH_{3} \xrightarrow[CH_{3}]{} CH_{3}$	Synthesis -Refer to: [2325]. m.p. 97–98° [2325];	¹ H NMR [2325], IR [2325].

# 10-(2-Hydroxy-3,4,6-trimethylphenyl)-10-oxo-1-decanone

[58185-77-6]	$C_{19}H_{28}O_4$	mol. wt. 320.43
CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃ CH ₃	Synthesis -Refer to: [2149, 2325]. m.p. 97–100° [2149, 2325]; 2325], IR [2149, 2325].	¹ H NMR [2149,

# Chapter 9 Undecanones

# 1 Aromatic Hydroxyketones Derived from Undecanoic Acids

# 1.1 Unsubstituted Hydroxyketones

# 1-(2-Hydroxyphenyl)-1-undecanone

	$C_{17}H_{26}O_2$	mol. wt. 262.39
OH CO(CH ₂ ) ₉ CH ₃	Synthesis -Refer to: [2077].	
	<b>Oxime</b> [439948-80-8] C ₁₇ H ₂₇ NO ₂	mol. wt. 277.41

# 1-(3-Hydroxyphenyl)-1-undecanone

	$C_{17}H_{26}O_2$	mol. wt. 262.39
OH I	Synthesis	
	-Refer to: [928]. <b>Methyl ether</b> [72424-10-3]	
CO(CH ₂ ) ₉ CH ₃		mol. wt. 276.42

-The ethereal Grignard reagent solution from n-decyl bromide, magnesium and ether was treated with cadmium chloride and the cooled mixture refluxed (2 h) with 3-methoxybenzoyl chloride in ether (41 %) [928]. -Also refer to: [2893].

oil [928]; ¹H NMR [928], IR [928].

# 1-(4-Hydroxyphenyl)-1-undecanone

[137034-61-8] C₁₇H₂₆O₂ mol. wt. 262.39 OH Syntheses

CO(CH₂)₉CH

-Obtained by reaction of undecanoyl chloride with phenol in the presence of aluminium chloride in methylene chloride for 14 h at r.t. (43 %) [1910].

 $CO(CH_2)_9CH_3$  -Also obtained by Fries rearrangement of phenyl undecanoate with boron trifluoride at 45–50° for 6 h (70.6 %) [1938].

-Also refer to: [2698].

light brown solid [1910]; m.p. 61.5–62° [1938], 53.7–54.2° [1910]; ¹H NMR [1910], ¹³C NMR [1910], IR [1910], MS [1910]; TLC [1910]; GC [1910].

BIOLOGICAL ACTIVITY: Inhibition of 17β-hydroxysteroid dehydrogenase 3 [1910]; Polyoxyethylene ether, as emulsifier for pesticides, [2698].

**Methyl ether** [69657-36-9] C₁₈H₂₈O₂ mol. wt. 276.42

-Obtained by Friedel-Crafts acylation of anisole with tert-butyl undecanoate in the presence of indium tribromide using dimethylchlorosilane (1r) (52 %) [2287].

-Also obtained by reaction of undecanoic acid with anisole in the presence of  $HSiMe_2Cl$  and  $InCl_3$  in 1,2-dichloroethane, first 1 h at r.t., then heated at 80° for 4 h (73 %) [218].

-Also obtained by reaction of 4-methoxybenzaldehyde with 1-decene (90–95 %) [1918].

-Also refer to: [1963, 2255].

m.p. 51–52° [1918], 49° [1963], 44–46° [218]; ¹H NMR [218, 1918, 2287], ¹³C NMR [218, 1918, 2287]; IR [218, 1918], MS [218].

# 2-Chloroethyl ether

 $C_{19}H_{29}ClO_2$ 

mol. wt. 324.89

-Obtained by reaction of undecanoyl chloride with 2-chloroethoxybenzene in the presence of aluminium chloride in carbon disulfide for 4 h at  $50^{\circ}$  (45 %) [476].

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m.p. 73-74° [476].
```

# **N-Dimethylaminoethyl ether** $C_{21}H_{35}NO_2$ mol. wt. 333.51

-Obtained by reaction of 4-(2-chloroethoxy)undecanophenone with dimethylamine in a sealed tube for 2 h at 150° [476].

free base: b.p._{0.005} 185° [476]; m.p. 57–58° [476]. hydrochloride (92 %): m.p. 183° [476]. Benzyldimethylethylammonium chloride ether

m.p. 105–106° [476].

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

HO CO(CH₂)₉CH₃

-Obtained by deprotection of its dimethyl ether with boron tribromide in methylene chloride at 0°. Then, the mixture was stirred overnight at r.t. (86 %) [82].

C₂₈H₄₂NO₂*Cl

brown solid [82]; m.p. 52° [82]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82].

USE: Reactant for preparation of lithium gallium/titanium ketone complexes [82].

**Dimethyl ether** [862666-32-8] C₁₉H₃₀O₃ mol. wt. 306.45

-Obtained by treatment of 1-(2,3-dimethoxyphenyl)-1-undecanol in acetone with potassium dichromate in dilute sulfuric acid (Jones reagent) at r.t. for 3 h (40 %) [82].

colourless oil [82]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82].

# 1-(2,4-Dihydroxyphenyl)-1-undecanone

[19810-04-9]

HC

OH

Syntheses

-Obtained by reaction of undecanoic acid with resorcinol in the presence of zinc chloride for 2 h at 160° (35 %) [3442].

-Also obtained by the Hoesch method (62 %) [2673]. -Also obtained by reaction of undecanoic acid with resorcinol in the presence of concentrated sulfuric acid in refluxing xylene for 24 h (17.4 %) [2543]. -Also refer to: [893, 2114, 2673].

C17H26O3

b.p.₁₁ 255–260° [893]; m.p. 80° [2114], 78.3–79.4° [2543], 72° [2673], 68–70° [3442]; ¹H NMR [2543], IR [2543], UV [2543].

BIOLOGICAL ACTIVITY: Antifungal [2114].

# **2,4-Dinitrophenylhydrazone** [95958-93-3] C₂₃H₃₀N₄O₆ mol. wt. 458.51

m.p. 158° [2673].

mol. wt. 460.10

mol. wt. 278.39

#### 

-Obtained by treatment of 1-(2,4-dihydroxyphenyl)-1-undecanone with 3,5-dinitrobenzoyl chloride under standard conditions [2543].

m.p. 94.8–96.5° [2543]; IR [2543].

4-Undecanoate	[19810-03-8]	$C_{28}H_{46}O_4$	mol. wt. 446.67
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-Obtained by reaction of undecanoic acid with resorcinol in the presence of concentrated sulfuric acid in refluxing xylene for 24 h (1.8 %) [2543].

m.p. 55.2–55.7° [2543]; IR [2543], UV [2543].

# 1-(2,5-Dihydroxyphenyl)-1-undecanone

	$C_{17}H_{26}O_3$	mol. wt. 278.39
CO(CH ₂ ) ₉ CH ₃	Syntheses -Obtained by reaction of undecanoyl of quinone dimethyl ether in the presence ride [156] in carbon disulfide [159]. m.p. 73.5–74.5° [156, 159].	•

Dibenzoate

 $C_{31}H_{34}O_5$ 

mol. wt. 486.61

m.p. 93–94.5° [156, 159].

# 1-(2,6-Dihydroxyphenyl)-1-undecanone

[85298-88-0]  $C_{17}H_{26}O_3$  mol. wt. 278.39 OH Syntheses CO(CH₂)₉CH₃ -Refer to: [1047, 1621]. OH

USE: Photog. black coupler [1621]; Photog. materials contg. aminobenzylideneaniline deriv. developing agent precursor and, [1047]; Multilayer colour photog. film assembly contg. organometallic stabilizer and primary amine developer and, for superior image formation and storage stability [1049].

**Dimethyl ether** [85298-95-9]  $C_{19}H_{30}O_3$  mol. wt. 306.44

-Refer to: [1621].

# 1-(3,4-Dihydroxyphenyl)-1-undecanone

(4-Undecanoylcatechol)

OH Synthesis OH -Obtained by treatment of a pyrocatechol and undecanoic acid mixture with zinc chloride at 135–140° for 2 h (15 %) [1283]. m.p.  $105^{\circ}$  [1283].

C31H38O3

USE: Bromination of, [2657].

# 1-(3,5-Dihydroxyphenyl)-1-undecanone

[85298-90-4]

OH

**Dibenzyl ether** 

C₁₇H₂₆O₃ mol. wt. 278.39 Syntheses

HO CO(CH₂)₉CH₃

**Dimethyl ether** [41497-33-0] C₁₉H₃₀O₃ mol. wt. 306.45

USE: Photog. black coupler [1621].

-Refer to: [543, 1621].

-Refer to: [543].

m.p. 43–43.5° [543]; ¹H NMR [543], IR [543].

**Synthesis** 

# 1-[2,3,4-Trihydroxyphenyl]-1-undecanone

(4-Undecanoylpyrogallol)

C ₁₇ H ₂₆ O ₄ me	ol. wt. 294.39
---------------------------------------------------	----------------

HO HO HO

-Obtained by reaction of undecanoic acid with pyrogallol in the presence of zinc chloride (Nencki reaction) at  $135-140^{\circ}$  for 2 h (35 %) [1283].

m.p. 76–77° [1283].

mol. wt. 458.64

mol. wt. 294.39

## 1-(2,4,6-Trihydroxyphenyl)-1-undecanone

[74478-14-1]  $C_{17}H_{26}O_4$  mol. wt. 294.39 OH OH OH OH OH CO(CH₂)₉CH₃ Syntheses -Obtained by reaction of undecanoyl chloride with phloroglucinol in the presence of aluminium chloride in nitrobenzene and carbon disulfide mixture (49 %) [2113].

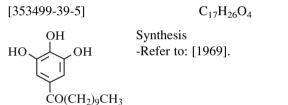
-Also obtained by reaction of undecanonitrile with phloroglucinol (Hoesch reaction) [1441].

-Also refer to: [2111].

m.p. 117–118° [1441], 114° [2113].

BIOLOGICAL ACTIVITY: Antifungal [2111, 2113].

# 1-(3,4,5-Trihydroxyphenyl)-1-undecanone



# 1-(3,4-Dihydroxyphenyl)-11-hydroxy-1-undecanone

	$C_{17}H_{26}O_4$	mol. wt. 294.39
OH OH	Synthesis -Refer to: [2749]. <b>11-Acetate</b> [22421-08-5]	
CO(CH ₂ ) ₉ CH ₂ OH	C ₁₉ H ₂₈ O ₅ -Obtained by reaction of sodium 4-(11-bromoundecanoyl)pyrocatechine in acid for 14 h (81 %) [2749].	

m.p. 108.5-110.5° [2749].

# 1-(2-Hydroxyphenyl)-6-methylene-1-undecanone

[526208-18-4]	$C_{18}H_{26}O_2$	mol. wt. 274.40
$\bigcup_{i=1}^{OH} CO(CH_2)_4 - C - (CH_2)_4 CH_3$	Syntheses -Obtained by intermolecular between salicylaldehyde 1,5-hexadiene (6 equiv.) in RhCl(PPh ₃ ) ₃ (0.2 equiv.) in r for 72 h at r.t. (8 %) [1435].	and 2-pentyl- the presence of

-Also refer to: [3066].

¹H NMR [1435].

# 1.2 Substituted Hydroxyketones

# 1-(3,5-Dichloro-2,6-dihydroxyphenyl)-1-undecanone

 $[85298-94-8] C_{17}H_{24}Cl_2O_3 mol. wt. 347.28$  OH Synthesis  $Cl + CO(CH_2)_9CH_3 -Refer to: [1621].$  USE: Photog. black coupler [1621].

# 1-(3-Chloro-2,6-dihydroxyphenyl)-1-undecanone

[85298-89-1]	C ₁₇ H ₂₅ ClO ₃	mol. wt. 312.83
	Synthesis -Refer to: [1621]. USE: Photog. black coupler [1621].	

# 1-(5-Chloro-2,4-dihydroxyphenyl)-1-undecanone

[19809-99-5]	C ₁₇ H ₂₅ ClO ₃	mol. wt. 312.83
HO Cl CO(CH ₂ ) ₉ CH ₃	Synthesis -Obtained by reaction of 4-chloro-resorcinol in the sulfuric acid in reflux (4,6 %) [2543]. m.p. 98–98.5° [2543]; ¹ H UV [2543].	presence of concentrated ing xylene for 24 h

**2,4-Dinitrophenylhydrazone** [19810-00-5] C₂₃H₂₉ClN₄O₆ mol. wt. 492.96

-Refer to: [2543].

m.p. 167.5–168.1° [2543].

4-Undecanoate	[19809-98-4]	$C_{28}H_{45}ClO_4$	mol. wt. 481.12
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-Obtained by reaction of undecanoic acid with 4-chlororesorcinol in the presence of concentrated sulfuric acid in refluxing xylene for 24 h (1,2 %) [2543].

m.p. 58.2–59.3° [2543]; IR [2543], UV [2543].

# 1-(4-Hydroxy-3-nitrophenyl)-1-undecanone

 $\begin{array}{cccc} [141124-94-9] & C_{17}H_{25}NO_4 & \text{mol. wt. } 307.39 \\ OH & Synthesis \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\$ 

CO(CH₂)₉CH₃

# 1-(3-Amino-4-hydroxyphenyl)-1-undecanone

[141124-96-1]	$C_{17}H_{27}NO_2$	mol. wt. 277.41
OH NH ₂	Syntheses -Refer to: [1478, 3046].	
CO(CH ₂ ) ₉ CH ₃	USE: Liquid crystal compound and its contair composition used in liquid crystal display [147]	<b>U</b> 1 <b>I</b>

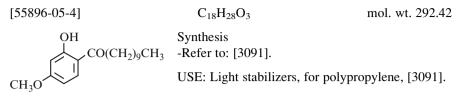
# 2,4,6-Trihydroxy-3-undecanoylbenzaldehyde

	$C_{18}H_{26}O_5$	mol. wt. 322.40
CHO CHO CO(CH ₂ ) ₉ CH ₃	Synthesis -Refer to: [3408].	
ностран	BIOLOGICAL ACTIVITY: Effect and stomatal closure [3408].	ts on transpiration
1 [2 (Chloromothyl) 4 hydr	overhandl 1 undesenans	

# 1-[3-(Chloromethyl)-4-hydroxyphenyl]-1-undecanone

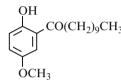
	$C_{18}H_{27}ClO_2$	mol. wt. 310.86
OH	Synthesis -Refer to: [2255].	
	<b>Methyl ether</b> [69657-35-8]	1 4 224 00
Т CO(CH ₂ ) ₉ CH ₃	$C_{19}H_{29}ClO_2$ -Refer to: [2255].	mol. wt. 324.89

#### 1-(2-Hydroxy-4-methoxyphenyl)-1-undecanone



# 1-(2-Hydroxy-5-methoxyphenyl)-1-undecanone

$$C_{18}H_{28}O_3$$
 mol. wt. 292.42  
Syntheses



CO(CH₂)₉CH₃ -Obtained by reaction of undecanoyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride [156] in carbon disulfide [159]. m.p. 47.5–48.5° [156].

mol. wt. 472.54

 $C_{24}H_{32}N_4O_6$ 

2,4-Dinitrophenylhydrazone

m.p. 125–127° [156, 159].

# 1-[2,3,4-Trihydroxy-5-(1-methylethyl)phenyl]-1-undecanone

[877877-92-4]	$C_{20}H_{32}O_4$	mol. wt. 336.47
HO CO(CH ₂ ) ₉ CH ₃	Synthesis -Refer to: [3267].	
HO CH(CH ₃ ) ₂	BIOLOGICAL ACTIVITY: antiapoptotic Bcl-2 [3267].	As inhibitors of

## 11-Hydroxy-1-(2-hydroxy-3,4-dimethoxy-6-methylphenyl)-1-undecanone

[77712-08-4]	$C_{20}H_{32}O_5$ mol. wt. 352	.47
CH ₃ O CH ₃ O CH ₃ O CH ₃ O CH ₃ O CH ₃ O CH ₃ O	Synthesis -Obtained by treatment of its 11-acetyl es with sodium hydroxide in methanol for 2 h r.t. (81 %) [1147].	ster 1 at

colourless needles [1147]; m.p. 81° [1147]; ¹H NMR [1147], IR [1147], MS [1147].

11-Acetyl ester	[77712-07-3]	$C_{22}H_{34}O_{6}$	mol. wt. 394.51
<b>H Heee J Heee J</b>	1,,,12,0, 51	022113400	11101

-Obtained by Friedel-Crafts reaction of 11-acetoxyundecanoyl chloride with 3,4,5-trimethoxy-toluene in 1,2-dichloroethane in the presence of aluminium chloride (2.1 mol) at 20° for 72 h [1147].

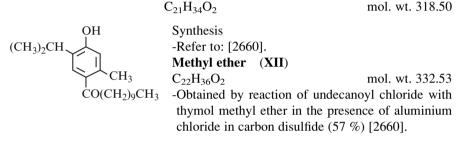
colourless oil [1147]; ¹H NMR [1147], IR [1147], MS [1147].

# 1-[3-(Propylaminocarbonyl)-2,4,6-trihydroxyphenyl]-1-undecanone

mol. wt. 379.50 C₂₁H₃₃NO₅ OH Synthesis  $C_{3}H_{7}NHCO$  CO(CH₂)₉CH₃ -Refer to: [3034].  $m.p. 106-107^{\circ}$  [3034]; ¹H NMR [3034], IR [3034].

BIOLOGICAL ACTIVITY: Cytotoxicity [3034]; Inhibition of Epstein-Barr virus early antigen activation [3034].

# 1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-undecanone



b.p.₁₅ 234–236° [2660];  $n_D^{23} = 1.5125$  [2660].

# 1-[3-(Butylaminocarbonyl)-2,4,6-trihydroxyphenyl]-1-undecanone

$$\begin{array}{c} C_{22}H_{35}NO_5 & \text{mol. wt. 393.52} \\ OH & Syntheses \\ C_4H_9NHCO & CO(CH_2)_9CH_3 & -Refer to: [3034, 3407]. \\ HO & OH & \text{m.p. 110-111^{\circ} [3407].} \end{array}$$

BIOLOGICAL ACTIVITY: Cytotoxicity [3034]; Inhibition of Epstein-Barr virus early antigen activation [3034].

# 1-(2,6-Dihydroxyphenyl)-11-phenyl-1-undecanone

$$C_{23}H_{30}O_3$$

mol. wt. 354.49

-From Horsfieldia glabra warb [2489];

-From seeds of Virola peruviana [570];

-From ripening fruits of Virola sebifera Aubl. [1905].

m.p. 76–77° [1368], 69–71° [1905]; ¹H NMR [1368, 1905, 2336], ¹³C NMR [1368, 1622], IR [1368, 1905, 2336], UV [1905], MS [1368, 1905, 2336].

BIOLOGICAL ACTIVITY: Nematocidal activity against second-stage larvae of *Toxocara canis* [1368].

# 1-(2,4,6-Trihydroxyphenyl)-11-phenyl-1-undecanone

 $[129684-20-4] C_{23}H_{30}O_4 mol. wt. 370.49$  $OH CO(CH_2)_{10} OH CO($ 

# 1-[2,3,4-Trihydroxy-5-(phenylmethyl)phenyl]-1-undecanone

 $[877877-98-0] C_{24}H_{32}O_4 mol. wt. 384.52$ OH HO CO(CH₂)₉CH₃ -Refer to: [3267]. ¹H NMR [3267], ¹³C NMR [3267].

BIOLOGICAL ACTIVITY: As inhibitors of antiapoptotic Bcl-2 [3267].

# 1-[3-(Hexylaminocarbonyl)-2,4,6-trihydroxyphenyl]-1-undecanone

C₂₄H₃₉NO₅

 $\begin{array}{c} OH \\ C_{6}H_{13}NHCO \\ HO \\ HO \\ OH \\ HO \\ OH \\ CO(CH_{2})_{9}CH_{3} \\ -Refer to: [3034]. \\ m.p. 102-103^{\circ} [3034]; \\ IR [3034]. \\ \end{array}$ 

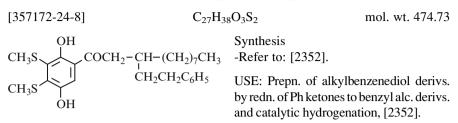
BIOLOGICAL ACTIVITY: Cytotoxicity [3034]; Inhibition of Epstein-Barr virus early antigen activation [3034].

# 1-[3-(Octylaminocarbonyl)-2,4,6-trihydroxyphenyl]-1-undecanone

BIOLOGICAL ACTIVITY: Cytotoxicity [3034]; Inhibition of Epstein-Barr virus early antigen activation [3034].

mol. wt. 421.58

# 1-[2,5-Dihydroxy-3,4-bis(methylthio)phenyl]-3-(2-phenylethyl)-1-undecanone



# 1-[2,3,4-Trihydroxy-5-(1-phenylundecyl)phenyl]-1-undecanone

 $\begin{array}{cccc} [877878-00-7] & C_{34}H_{52}O_4 & \mbox{mol. wt. 524.78} \\ & OH & Synthesis \\ HO & CO(CH_2)_9CH_3 & -Refer to: [3267]. \\ & HO & BIOLOGICAL & ACTIVITY: & As & inhibitors & of \\ & antiapoptotic & Bcl-2 & [3267]. \end{array}$ 

# 2 Aromatic Hydroxyketones Derived from 11-Bromoundecanoic Acids

# 2.1 Unsubstituted Hydroxyketones

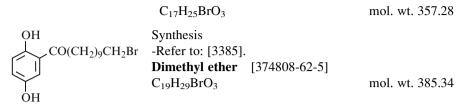
# 11-Bromo-1-(4-hydroxyphenyl)-1-undecanone

 $C_{17}H_{25}BrO_2$  mol. wt. 341.29 Synthesis -Refer to: [2709]. **Methyl ether** [867213-74-9]

 $\begin{array}{c} & C_{18}H_{27}BrO_2 \\ CO(CH_2)_9CH_2Br \end{array} \qquad \qquad \mbox{mol. wt. } 355.31 \\ -Obtained by acylation of anisole with 11-bromo-undecanoic acid in the presence of a catalytic amount of p-toluenesulfonic acid and graphite for 4 h at 90° (84 %) [2709]. \end{array}$ 

m.p. 190° [2709]; ¹H NMR [2709], ¹³C NMR [2709], IR [2709], MS [2709].

#### 11-Bromo-1-(2,5-dihydroxyphenyl)-1-undecanone



USE: Linking agents for fluorescent labelling of nanocrystals or quantum dots [2653].

# 11-Bromo-1-(3,4-dihydroxyphenyl)-1-undecanone

[22421-07-4]	$C_{17}H_{25}B$	rO ₃	mol. wt. 357.29
OH OH CO(CH ₂ ) ₉ CH ₂ Br	pyrocatechine in the for 2 h 30 min (70 %		
Diacetate	[34767-67-4]	$\mathrm{C}_{21}\mathrm{H}_{29}\mathrm{BrO}_5$	mol. wt. 441.36
-Refer to: [2749].			

m.p. 73–75° [2748].

# 2.2 Substituted Hydroxyketones

#### 11-Bromo-1-(2-hydroxy-4-methylphenyl)-1-undecanone

[218784-30-6]	$C_{18}H_{27}BrO_2$	mol. wt. 355.32
CO(CH ₂ ) ₉ CH ₂ Br	Syntheses -Obtained by direct acylation 11-bromo-undecanoic acid in AMA, a mixture of alumina an acid, at 140° for 5 min (80 %)	the presence of d methanesulfonic

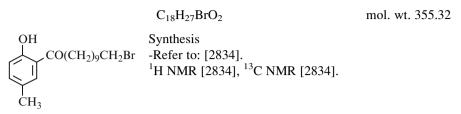
-Also obtained by Fries rearrangement of m-cresyl 11-bromoundecanoate in the presence of AMA, a mixture of alumina and methanesulfonic acid, at  $160^{\circ}$  for 30 min (80 %) [2833].

white crystals [2833]; m.p. 62° [2833]; ¹H NMR [2833], IR [2833], UV [2833], MS [2833].

mol. wt. 411.38

# 11-Bromo-1-(2-hydroxy-5-methylphenyl)-1-undecanone

2-(11-Bromo-1-undecanoyl)-5-methylphenol



## 11-Bromo-1-(4-hydroxy-3-methylphenyl)-1-undecanone

	$C_{18}H_{27}BrO_2$	mol. wt. 355.27
OH CH ₃	Synthesis -Refer to: [218]. <b>Methyl ether</b> [927911-89-5]	
CO(CH ₂ ) ₉ CH ₂ Br	$C_{19}H_{29}BrO_2$	mol. wt. 369.34

-Obtained by reaction of 11-bromoundecanoic acid with 2-methylanisole in the presence of HSiMe₂Cl and InCl₃ (or InBr₃) in 1,2-dichloroethane, first 1 h at r.t., then heated at  $80^{\circ}$  for 4 h (71 %) (or 74 %) [218].

m.p. 35-37° [218]; ¹H NMR [218], ¹³C NMR [218], IR [218], MS [218].

# 11-Bromo-1-(4-hydroxy-3-methoxyphenyl)-1-undecanone

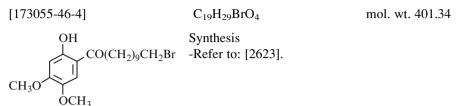
[13149-44-5]

[13149-43-4]	$C_{18}H_{27}BrO_3$	mol. wt. 371.32
OH OCH ₃ CO(CH ₂ ) ₉ CH ₂ Br	Synthesis -Obtained by reaction of 11-brom guaiacol in the presence of boron then at 70° for 2.5 h (44.9 %) [2750] m.p. 68–69° with softening at 63° [27	trifluoride first at $40^{\circ}$ , ].

 $C_{21}H_{31}BrO_3$ Allyl ether -Obtained by reaction of allyl bromide with the title ketone in the presence of

potassium carbonate in refluxing acetone for 9 h [2750]. m.p.  $68-70^{\circ}$  with sintering at  $65^{\circ}$  [2750].

#### 11-Bromo-1-(2-hydroxy-4,5-dimethoxyphenyl)-1-undecanone



#### Aromatic Hydroxyketone Derived 3 from 11-Oxoundecanoic Acid

# 11-(2-Hydroxyphenyl)-11-oxo-1-undecanoic acid

$$C_{17}H_{24}O_4$$

mol. wt. 292.38



Synthesis CO(CH₂)₉CO₂H -Refer to: [1131]. m.p. 85–87° [1131]; ¹H NMR [1131].

# Chapter 10 Dodecanones

# 1 Aromatic Hydroxyketones Derived from Dodecanoic Acids

# 1.1 Unsubstituted Hydroxyketones

# 1-(2-Hydroxyphenyl)-1-dodecanone

[2589-83-5]

 $C_{18}H_{28}O_2$ 

mol. wt. 276.42

OH  $CO(CH_2)_{10}CH_3$  Syntheses -Preparation by reaction of lauric acid with phenol, *in the presence of melted zinc chloride for 6 h (65–70 %) [2398];

*in the presence of the Japanese acid clay at 190° for 2 h [1785].

-Also obtained by Fries rearrangement of phenyl laurate [12],

*with aluminium chloride at  $70^{\circ}$  for 10 h (28 %) [2550], at 150° for 1 h [293] or in tetrachloroethane [3169].

*in the presence of aluminium chloride in refluxing carbon disulfide for 5 h. Then, the mixture was kept at  $80-90^{\circ}$  for 2 h and at  $140-150^{\circ}$  for 1 h after solvent elimination (33 %) [1222];

*in the presence of aluminium chloride in nitrobenzene [718] according to [380];

*in the presence of aluminium chloride in nitrobenzene for 15 h at  $50^{\circ}$  (10 %) [378];

*in the presence of titanium tetrachloride at high temperature [718] according to [1762].

-Also obtained by reaction of lauroyl chloride with phenol in the presence of aluminium chloride,

*in tetrachloroethane for 6 h at 55–60° (33 %) [2548];

*in nitrobenzene for 3 h at 70° (21 %) [2549];

*in carbon disulfide for 5.5 h at  $47^{\circ}$  (41 %) [2549].

-Also refer to: [12, 77, 110, 718, 873, 1230, 1271, 1456, 1985, 2316, 3122].

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R. Martin, J.-P. Buisson, Aromatic Hydroxyketones: Preparation & Physical Properties, DOI 10.1007/978-3-319-14185-5_10

b.p._{1.6} 180–204° [293]; m.p. 45–46.5° [718], 44–45.5° [2548], 44–45° [12, 3169], 44° [2398], 43.8–44.6° [293]; IR [12]; TLC [1456].

USE: Textile rot proofing by, [873].

BIOLOGICAL ACTIVITY: Effect on bacteria (*staphylococcus aureus and tricophyton asteroides*) [3122].

**Oxime** [20803-93-4] C₁₈H₂₉NO₂ mol. wt. 291.43

-Refer to: [12, 110].

m.p. 75–76° [12]; IR [12].

USE: In extn. of copper and nickel from sulfate solns. [110].

# Oxime, nickel complex

USE: Quenchers for singlet oxygen, [3483].

Semicarbazone	[20803-53-6]	$C_{19}H_{31}N_3O_2$	mol. wt. 333.47
m.p. 139–145° (d	) [12]; IR [12].		

2,4-Dinitrophenylhydrazone	$C_{24}H_{32}N_4O_5$	mol. wt. 456.54
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m.p. 92–93° [2548], 91° [3169], 89–89.2° [293].

Methyl ether	$C_{19}H_{30}O_2$	mol. wt. 290.45
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-Obtained by reaction of dimethyl sulfate with the above ketone in the presence of potassium carbonate in boiling acetone for 6 h [2398].

colourless liquid [2398]; b.p.₅₀ 110° [2398]. ¹H NMR [1596], ¹³C NMR [1596], MS [1596].

2,3-Epoxypropyl ether	[18110-29-7]	$C_{21}H_{32}O_3$	mol. wt. 332.48
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-Obtained by reaction of epichlorohydrin with o-dodecanoylphenol (0.1 mol) in the presence of 30 % sodium hydroxide between 40 and 50° (62 %) [2669].

b.p._{0.7} 235–240° [2669].

[55917-79-8]

# 1-(3-Hydroxyphenyl)-1-dodecanone

[63442-86-4]	$C_{18}H_{28}$	$O_2$	mol. wt. 276.42
OH CO(CH ₂ ) ₁₀ CH ₃	Syntheses -Refer to: [1984, 1 m.p. 40° [1984];	-	
Methyl ether	[63442-83-1]	$C_{19}H_{30}O_2$	mol. wt. 290.45
-Refer to: [1984, 1985	, 2725].		

b.p.₁₀ 132–135° [1984]; IR [1984].

# 1-(4-Hydroxyphenyl)-1-dodecanone

[2589-74-4]

OH

 $C_{18}H_{28}O_2$ 

mol. wt. 276.42

Syntheses

-Preparation by reaction of lauric acid with phenol,

*in the presence of boron trifluoride for 2 h at 70° (84 %) [1685] or for 3 h at 80–90° (68.6 %) [1938];

 $CO(CH_2)_{10}CH_3$  *in the presence of the Japanese acid clay at 190° for 2 h [1785].

-Also obtained by Fries rearrangement of phenyl laurate with aluminium chloride, (65 %) [414],

*for 10 h at 70° (46 %) [2550] or for 1 h at 150° [293, 2163];

*in tetrachloroethane [3169];

- *in nitrobenzene (67 %) [2947], at 38° for 48 h (65 %) [414, 415] or at r.t. overnight (56 %) [1769];
- *in the presence of aluminium chloride in nitrobenzene [718] according to [380];

*in the presence of aluminium chloride in nitrobenzene for 15 h at  $50^{\circ}$  (60 %) [378];

*in the presence of aluminium chloride in refluxing carbon disulfide for 5 h. Then, the mixture was kept at 80–90° for 2 h and at  $140-150^{\circ}$  for 1 h after solvent elimination (42 %) [1222].

-Also obtained by demethylation of its methyl ether [2398].

-Also obtained by reaction of lauroyl chloride with phenol in the presence of aluminium chloride,

*in tetrachlorethane for 6 h at  $55-60^{\circ}$  (25 %) [2548];

*in nitrobenzene at 70° for 3 h (73 %) [2549];

*in carbon disulfide at  $47^{\circ}$  for 5.5 h (55 %) [2549].

-Also obtained by reaction of dodecanoyl chloride with phenol in the presence of aluminium chloride in methylene chloride for 14 h at r.t. (28 %) [1910].

-Also obtained by heating 2-hydroxy-5-(1-oxododecyl)benzoic acid in quinoline in the presence of copper for 1 h at  $210^{\circ}$  (96 %) [448].

-Also obtained by reaction of lauric acid with phenol in the presence of activated acid clay catalyst at  $190^{\circ}$  for 2 h [3277].

-Also refer to: [12, 293, 378, 379, 513, 1271, 1299, 1456, 1863, 1908, 1985, 2035, 2316, 2697, 2945, 3109, 3122, 3371, 3424].

b.p._{0.2} 195° [448], b.p._{0.3} 198° [2035], b.p.₁₀ 250–257° [2947],
b.p.₁₅ 275° [2163]; b.p.₁₅ 277° [293];
off-white solid [1910];
m.p. 72° [2398], 71.2–71.6° [1863], 71–72° [2548], 71–71.5° [3277], 71° [12, 1685, 3169],
70.5–71° [293], 70–72° [1938], 70–71° [378, 2163, 2549], 70° [414, 415],
69.6–70.4° [1910], 68–70° [1769], 62° [3424], 61–62° [379];
¹H NMR [1910, 3424], ¹³C NMR [1910], IR [12, 1910], MS [1910]; TLC [1456].

USE: Activator for peroxygen bleach in laundry detergent for mud-soiled clothing [3371]; Ether with polyoxyethylene glycol, as herbicide [2697]; Foaming improvement of N-acylamino alkanesulfonate detergents by, [2945]; Textile rot proofing by, [873].

BIOLOGICAL ACTIVITY: Inhibition of 17-β-hydroxysteroid dehydrogenase 3 [1908, 1910]; Effect on bacteria (*staphylococcus aureus and tricophyton asteroides*) [3122].

2,4-Dinitrophen	ylhydrazone	$C_{24}H_{32}N_4O_5$	mol. wt. 456.54
m.p. 151–152	° [293], 150.5° [3169], 1	50–151° [378, 2548]	, 144–144.5° [448].
Oxime	[20803-52-5]	C ₁₈ H ₂₉ NO ₂	mol. wt. 291.43
m.p. 77–79° [	12]; IR [12].		
Semicarbazone	[20803-54-7]	$C_{19}H_{31}N_3O_2$	mol. wt. 333.47
m.p. 143–143	.6° [293], 140–141° [12]	; IR [12].	
Acetate	C ₂₀ H ₃₀ O ₃	3	mol. wt. 318.46

-Preparation by reaction of acetic anhydride with the title ketone in the presence of sulfuric acid [378].

m.p. 68° [378].

# Benzoate

C₂₅H₃₂O₃ mol. wt. 380.53

-Obtained from Schotten-Baumann reaction [293, 378, 448].

m.p. 109–110° [448], 109–109.8° [293], 108° [378].

# Laurate [122214-67-9] C₃₀H₅₀O₃ mol. wt. 458.73

-Obtained by reaction of lauric acid with phenol in the presence of activated acid clay catalyst at  $190^{\circ}$  for 2 h [3277].

m.p. 76.5-77.5° [3277].

# **Methyl ether** [63829-20-9] C₁₉H₃₀O₂ mol. wt. 290.45

-Obtained by reaction of lauroyl chloride with anisole,

*in the presence of aluminium chloride (90 %) [2398];

*in the presence of iodine (50 %) [378];

*under microwaves (90 W; 2 min x 4; 410°) in the presence of graphite (74 %) [1830].

-Also obtained by reaction of lauric acid with anisole,

*in the presence of polyvalent-metal salts of dodecatungstophosphate as effective heterogeneous catalysts for Friedel-Crafts acylation [2870];

*in the presence of montmorillonite-enwrapped titanium as a solid acid catalyst for 48 h at 165° (97 %) [937];

*over zeolite catalysts through Friedel-Crafts acylation [3353].

-Also obtained by reaction of dimethyl sulfate with the title ketone in the presence of alkali [448], (quantitative yield) [378].

-Also obtained by reaction of dodecanoic acid with anisole in the presence of  $Cs_{2.5}H_{0.5}PW_{12}O_{40}$  at 110° for 5 h (25 %) [1636].

-Also obtained by reaction of dodecanoic acid with anisole in the presence of  $HNTf_2$  in refluxing toluene for 36 h, using a Dean-Stark apparatus (84 %) [1648]. -Also refer to: [175, 378, 416, 1009, 1480, 1963, 2016, 2035, 2252, 2641, 3221].

white powder [937]; plates [2398, 2548]; m.p. 62.5° [1963], 58.5° [378], 57–59° [2548], 57° [2398], 55.5–56.5° [2016], 55–57.5° [3221], 40–45° [448]; ¹H NMR [937], ¹³C NMR [937].

# 2,4-Dinitrophenylhydrazone of the methyl ether C25H34N4O5 mol. wt. 470.57

m.p. 108–109° [2016].

# **2-Chloroethyl ether** $C_{20}H_{31}ClO_2$ mol. wt. 338.92

-Obtained by reaction of dodecanoyl chloride with 2-chloroethoxybenzene in the presence of aluminium chloride in carbon disulfide for 4 h at  $50^{\circ}$  (55 %) [476].

shining leaflets [476]; m.p. 72.5° [476].

# **3-Bromopropyl ether** $C_{21}H_{33}BrO_2$ mol. wt. 397.39

-Obtained by reaction of dodecanoyl chloride with 3-bromopropoxybenzene in the presence of aluminium chloride in carbon disulfide for 4 h at  $50^{\circ}$  (45 %) [476].

m.p. 39–40° [476].

#### 4-Bromobutyl ether

-Obtained by reaction of dodecanoyl chloride with 4-bromobutoxybenzene in the presence of aluminium chloride in carbon disulfide for 4 h at  $50^{\circ}$  (33 %) [476].

m.p. 57-58° [476].

**2-Hydroxyethyl ether** 
$$C_{29}H_{32}O_3$$
 mol. wt. 320.47

USE: Foaming improvement of N-acylamino alkanesulfonate detergents by, [2945].

-Obtained by reaction of 4-(2-chloroethoxy)dodecanophenone with dimethylamine in a sealed tube for 2 h at  $150^{\circ}$  (88.5 %) [476].

free base: b.p. $_{0.01}$  193–197° [476]; m.p. 45–46° [476]. hydrochloride: m.p. 173–175° [476].

**N-Dimethylaminopropyl ether** C₂₃H₃₉NO₂ mol. wt. 361.57

-Obtained by reaction of 4-(3-bromopropoxy)dodecanophenone with dimethylamine in a sealed tube for 2 h at  $150^{\circ}$  (89 %) [476].

free base: b.p. $_{0.1}$  206° [476]; m.p. 31–32° [476]. hydrochloride: m.p. 186° [476].

-Obtained by reaction of 4-(4-bromobutoxy)dodecanophenone with dimethylamine in a sealed tube for 2 h at  $150^{\circ}$  (92 %) [476].

free base: b.p._{0.005} 202° [476]. hydrochloride: m.p. 179° [476].

**N-Diethylaminoethyl ether** [14392-83-7] C₂₄H₄₁NO₂ mol. wt. 375.60

-Obtained by treatment of 4-hydroxydodecanophenone sodium salt in ethanol with diethylaminoethyl chloride in refluxing toluene for 5 h (71 %) [414], (68 %) [415].

free base: b.p._{0.002} 210–220° [414, 415];  $n_D^{21} = 1.5036$  [414, 415]. hydrochloride: m.p. 107–108° [513].

**2,3-Epoxypropyl ether** [18110-30-0] C₂₁H₃₂O₃ mol. wt. 332.48

-Obtained by reaction of epichlorohydrin with p-dodecanoylphenol (0.1 mol) in the presence of 30 % sodium hydroxide between 40 and 50° (59 %) [2669].

m.p. 105° [2669].

C22H35BrO2

#### 1-(2,4-Dihydroxyphenyl)-1-dodecanone

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[25632-60-4]  $C_{18}H_{28}O_3$  mol. wt. 292.42 OH  $CO(CH_2)_{10}CH_3$  -Preparation by reaction of lauric acid with resorcinol, *in the presence of boron trifluoride for 2 h at 90° (79 %) [2312]; *in the presence of zinc chloride at 150° [2517] or at 125–135° [893].

-Also prepared by reaction of lauronitrile with resorcinol (Hoesch reaction) (20 %) [2928].

-Also obtained by Fries rearrangement of resorcinol dilaurate with aluminium chloride in nitrobenzene for 8 h at  $40-50^{\circ}$  (24 %) [379].

-Also refer to: [284, 379, 598, 788, 859, 1655, 1700, 2273, 2509, 2790, 2842, 2973, 3077, 3125, 3221].

b.p.₈ 260–265° [1700], b.p.₇ 243° [3125], b.p._{6–7} 237–239° [893, 2842]; m.p. 84–85.5° [893, 2842], 84° [2312], 82.5–83.5° [3125], 82–83° [379], 81–82° [2273], 79–80° [1700], 77–78° [3221].

USE: Synthesis of substituted polyaniline monomer [788]; Hair conditioning and/or colouring compositions [598]; In prepn. of photog. black coupler [2509]; Polyamide fibers modified with, transparency of, [2790].

4-Nitrophenylhydrazone		$C_{24}H_{33}N_3O_4$	mol. wt. 427.54
m.p. 86–87° [85	9].		
2,4-Dinitrophenyl	•	$C_{24}H_{32}N_4O_6$	mol. wt. 472.54
m.p. 165° [379]			
Diacetate	[251463-52-2]	$C_{22}H_{32}O_5$	mol. wt. 376.49
-Obtained by reaction of acetic anhydride with the title ketone in the presence of pyridine (>80 %) [2517].			
white solid [2517]; m.p. 50–51° [2517]; ¹ H NMR [2517], ¹³ C NMR [2517], IR [2517], UV [2517], MS [2517].			
Monohydrate m.p. 78–79° [31	10 2	₈ O ₃ , H ₂ O	mol. wt. 310.43
<b>Dimethyl ether</b> m.p. 46° [16].	C ₂	₀ H ₃₂ O ₃	mol. wt. 320.47

mol. wt. 292.42

# **4-(2-Propenyl) ether** $C_{21}H_{32}O_3$ mol. wt. 332.48

USE: Protection against actinic radiations [2959].

4-(2-Chloro-2-propenyl) ether	$C_{21}H_{31}ClO_3$	mol. wt. 366.93
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C₁₈H₂₈O₃

m.p. 57.2–58.3 $^{\circ}$  [2959]; UV [2959].

USE: Protection against actinic radiations [2959].

# 1-(2,5-Dihydroxyphenyl)-1-dodecanone

[4693-30-5]

OH

Syntheses

 $CO(CH_2)_{10}CH_3$  -Obtained by reaction of lauroyl chloride with hydroquinone in the presence of aluminium chloride in nitrobenzene at r.t. overnight, then heated on a water bath for 3 h [1442].

-Also obtained by Fries rearrangement of hydroquinone dilaurate with aluminium chloride at  $150-160^{\circ}$  for 5 h [1442].

- -Also obtained by reaction of dodecanoyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride [156].
- -Also obtained by treatment of hydroquinone and dodecanoic acid mixture in carbon tetrachloride below  $60^{\circ}$  with boron trifluoride, kept overnight at r.t., then heated 5 h at  $90-95^{\circ}$  (58 %) [3204].
- -Also obtained by treatment of its dimethyl ether with hydrobromic acid in acetic acid at  $0^{\circ}$ . The mixture was heated on a stand bath for 6 h under reflux [1442].
- -Also obtained by deketalization of 2-dodecanoyl-3-hydroxycyclohex-2-en-1-one with concentrated sulfuric acid in refluxing acetone for 160 min and stand overnight at r.t. [2335].

-Also obtained by reaction of lauroyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride in carbon disulfide [159].

-Also refer to: [394, 3204].

light yellow material [2335]; yellow needles [159]; m.p. 101° [1442, 3204], 99° [156, 159]; ¹H NMR [2335], MS [2335]; TLC [2335]; GLC [2335].

USE: Effect on photographic fogging in multilayered colour films, [394].

# **Oxime** $C_{18}H_{29}NO_3$ mol. wt. 307.43

USE: Effect on photographic fogging in multilayered colour films [394].

# **Dimethyl ether** [98314-46-6] C₂₀H₃₂O₃ mol. wt. 320.47

-Obtained by reaction of lauroyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride [2859],

*in nitrobenzene at r.t. overnight, the mixture was heated on a water bath for 3 h [1442];

*in tetrachloroethane (76 %) [714].

-Also refer to: [714, 2151].

b.p._{0.2} 175–178° [714], b.p._{1.2} 180–185° [1442]; m.p. 27.5° [714]; ¹H NMR [2859], MS [2859].

# **Diethyl ether** $C_{22}H_{36}O_3$ mol. wt. 348.53

-Obtained by reaction of lauroyl chloride with hydroquinone diethyl ether in the presence of aluminium chloride in tetrachloroethane (65 %) [714].

b.p._{0.34} 180–190° [714]; m.p. 34–35° [714].

2,4-Dinitrophenylhydrazone of the diethyl ether C28H40N4O6 mol. wt. 528.65

bright red crystals [714]; m.p. 77.5° [714].

Di-dodecyl ether	[72047-09-7]	$C_{43}H_{78}O_3$	mol. wt. 643.09
-Refer to: [3021–3023].			

-Refer to: [3021 - 3023].

# 1-(2,6-Dihydroxyphenyl)-1-dodecanone

 $[125009-82-7] C_{18}H_{28}O_3 mol. wt. 292.42$ 

OH  $CO(CH_2)_{10}CH_3$  Syntheses OB OH OH Syntheses Obtained by refluxing a solution of 2-dodecanoyl-<math>3-hydroxycyclohex-2-en-1-one, Hg(OAc)₂ and NaOAc in acetic acid under nitrogen for 6.5 h (66 %) [2334].

-Obtaining from 1,3-cyclohexanedione (54 %) [2336].

-Also obtained from methyl 2,6-dimethoxybenzoate [2336].

-Also obtained in three steps, starting from the enolate of 5-hydroxycyclohexane-1,3-dione [3439].

-Also refer to: [2249, 2418].

Isolation from natural sources

-From the exocrine secretions of lace bugs [1563].

-From the exocrine secretions of andromeda lace bug *Stephanitis takeyai* (Hemiptera: Tingidae) [2334].

-A component of the setal exudate of immature andromeda lace bugs (*Stephanitis takeyai* Drake and Maa) [2334, 2336].

-Of the setal exudate of nymphs of the andromeda lace bug, *Stephanitis takeyai* (Hemiptera: Tingidae) [2334].

-From Stephanitis rhododendrii [2334].

-In the wood of *Knema austrosiamensis* W. J. J. O. DeWilde (Myristaceae) [1133] or in Ground wood of this same plant [1134].

-From Horsfieldia irya seeds [3314].

m.p. 86–88° [2334, 2336]; ¹H NMR [1133, 2334, 2336], IR [2336], UV [2334], MS [1133, 2334, 2336].

USE: Nickel-chelating agent [3080].

BIOLOGICAL ACTIVITY: This compound was equal to aspirin in inhibition of PGE₂ formation with fat body preparations of the American cockroach (*Periplaneta americana*) [1563]; This compound inhibited prostaglandin synthase in two *in vitro systems* [1563]; Inhibition of soybean urease by cyclic  $\beta$ -triketones and fluoride ions [3080].

# 1-(3,4-Dihydroxyphenyl)-1-dodecanone

(4-Dodecanoylcatechol)

[1158-20-9]  $C_{18}H_{28}O_3$  mol. wt. 292.42 OH Syntheses -Obtained by treatment of a pyrocatechol and lauric acid mixture,

*with zinc chloride at 135–140° for 2 h (20 %) [1283];

 $CO(CH_2)_{10}CH_3$  *with zinc chloride and phosphorous oxychloride at 70° for 90 min (83.2 %) [2812].

-Also obtained by reaction of dodecanoyl chloride with pyrocatechol in the presence of aluminium chloride in nitrobenzene, first at  $5-7^{\circ}$  for 2 h, then at r.t. for 2–3 days (62 %) [1726].

-Also refer to: [283, 985, 3056].

m.p. 105–106° [2812], 97–98° [1283]; ¹H NMR [1726], UV [985].

USE: Antioxidant [2812].

BIOLOGICAL ACTIVITY: Protective agent against the lethal effects of X-rays [1809].

**2,4-Dinitrophenylhydrazone** [97153-67-8] C₂₄H₃₂N₄O₆ mol. wt. 472.54

bright red needles [2812]; m.p. 216° [2812].

**Dimethyl ether** [96966-45-9] C₂₀H₃₂O₃ mol. wt. 320.47

-Preparation by Friedel-Crafts acylation of veratrole [3056].

-Also obtained by reaction of dodecanoyl chloride with veratrole in the presence of zinc chloride in refluxing carbon disulfide for 6 h and then left overnight (55 %) [2420].

-Also refer to: [1167, 1960, 1963, 3365].

white needles [2420]; b.p.₁ 200° [2420]; m.p. 70–71° [2420], 68–69° [1960, 1963], 57° [3056]; ¹H NMR [1167], ¹³C NMR [1167], IR [3365].

# 2,4-Dinitrophenylhydrazone of the dimethyl ether

[102947-41-1]	$C_{26}H_{36}N_4O_6$	mol. wt. 500.60

m.p. 80–81° [2420].

# 1-(3,5-Dihydroxyphenyl)-1-dodecanone

[1250871-16-9]	$C_{18}H_{28}O_3$	mol. wt. 292.42
HO CO(CH ₂ ) ₁₀ CH ₃	Synthesis -Refer to: [2903]. m.p. 101–103° [2903]; ¹ H NMR [2903], ¹³ C NMR [2903]	, MS [2903].

**Dimethyl ether** [55049-56-4] C₂₀H₃₂O₃

-Refer to: [1899, 2903].

m.p. 52–53° [2903]; ¹H NMR [2903], ¹³C NMR [2903], MS [2903].

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

[133839-66-4]		$C_{18}H_{28}O_3$	mol. wt. 292.42
OH	OH	Synthesis	
COCH2-	-CH-(CH ₂ ) ₈ CH ₃	-Refer to: [3151].	

# 12-Hydroxy-1-(3-hydroxyphenyl)-1-dodecanone

C ₁₈	$_{3}H_{28}O_{3}$	mol. wt. 292.42
HO CO(CH ₂ ) ₁₀ CH ₂ OH	Synthesis	
	-Obtained by treatment of its tert-	outvl dimethylsilyl

Obtained by treatment of its tert-butyl dimethylsilyl ether with tetrabutylammonium fluoride in tetrahydrofuran at  $0^{\circ}$  for 0.5 h (99 %) [1596].

mol. wt. 320.47

¹H NMR [1596], ¹³C NMR [1596], MS [1596].

mol. wt. 308.42

## 12-Hydroxy-1-(4-hydroxyphenyl)-1-dodecanone

$C_{13}$	₃ H ₂₈ O ₃	mol. wt. 292.42
HO CO(CH ₂ ) ₁₀ CH ₂ OH	Synthesis -Obtained by treatment dimethylsilyl ether with fluoride in tetrahydrofura (99 %) [1596].	tetrabutylammonium

¹H NMR [1596], ¹³C NMR [1596], MS [1596].

## 1-(2,3,4-Trihydroxyphenyl)-1-dodecanone

[15251-74-8]

HO

HO

OH

CO(CH₂)₁₀CH₃ -Preparation by reaction of lauric acid with pyrogallol,

*in the presence of boron trifluoride,

-in trichloroethylene or carbon tetrachloride for 2–3 h between 65 and 85° (95–98 %) [503];

-without solvent at  $55^{\circ}$  for 1.5 h [3200];

*in the presence of zinc chloride at  $135-140^{\circ}$  for 2 h (30 %) [1283];

*in the presence of strongly acidic ion exchanger Amberlyst-15 at 120° for 24 h (38 %) [231].

-Also obtained by reaction of dodecanoyl chloride with pyrogallol,

*without catalyst at 150° for 2 h (5 %) [3200];

*in the presence of aluminium chloride in nitrobenzene, first at  $5-7^{\circ}$  for 2 h, then at r.t. for 2-3 days (31 %) [1726].

-Also refer to: [859, 2328, 3200].

m.p. 85–86° [3200], 78° [503], 76–77° [1283], 74–75° [859]; ¹H NMR [231, 1726], ¹³C NMR [231], UV [3200].

USE: Colour photographic material containing scavenger for oxidized colour developing agent [2328]; Pressure-sensitive copying paper coatings contg for examination papers with hidden texts [1982].

BIOLOGICAL ACTIVITY: Protection against whole-body irradiation [1810].

4-Nitrophenylhydrazone	$C_{24}H_{33}N_3O_5$	mol. wt. 443.54
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m.p. 182–183° [859].

2,4-Dinitrophenylhydrazone [14725-80-5] C₂₄H₃₂N₄O₇ mol. wt. 488.54 m.p. 215–216° (d) [3200]; UV [3200].

Syntheses

C18H28O4

#### 1-(2,4,5-Trihydroxyphenyl)-1-dodecanone

[109559-39-9] C18H28O4 mol. wt. 308.42 OH Syntheses CO(CH₂)₁₀CH₃ -Obtained by reaction of lauroyl chloride with 1,2,4-trihydroxybenzene in the presence of alu-HO minium chloride in nitrobenzene [291, 292]. ÔН -Also refer to: [1708].

m.p. 119–121° [291, 292].

USE: Antioxidant [1708]; Antioxidant for fats, oils and paraffin waxes [292]; Manuf. of, and oxidative stabilization of fats, oils and paraffin waxes by, [291].

**BIOLOGICAL ACTIVITY: Toxicity** [1708].

**Trimethyl ether** [56134-35-1]  $C_{21}H_{34}O_4$ mol. wt. 350.50

-Preparation by treatment of 2,4,5-trihydroxydodecanophenone with dimethyl sulfate in the presence of potassium carbonate in refluxing acetone for 4 h (88 %) [2752].

-Also refer to: [714].

m.p. 55–56° [2752], 53° [714].

#### 1-(2,4,6-Trihydroxyphenyl)-1-dodecanone

[6790-21-2]

Syntheses

 $C_{18}H_{28}O_4$ 

 $CO(CH_2)_{10}CH_3$  -Obtained by reaction of dodecanoyl chloride with phloroglucinol in the presence of boron trifluoride etherate, first at 0°, then at r.t. for 48 h under nitrogen [2786].

mol. wt. 308.42

- -Also obtained by reaction of dodecanoyl chloride with phloroglucinol in the presence of aluminium chloride [2364] in nitrobenzene and carbon disulfide mixture (58 %) [2113].
- -Also obtained by reaction of lauronitrile with phloroglucinol (Hoesch reaction) [1441].
- -Also obtained by reaction of dodecanoic acid with phloroglucinol in the presence of aluminium chloride and phosphorous oxychloride (40 %) [3202].
- -Also obtained by reaction of lauric anhydride with phloroglucinol in tetrahydrofuran in the presence of boron trifluoride etherate at r.t. under nitrogen, for 48 h (78 %) [1975].
- -Preparation (25 %) [3202] using to the process [3201].
- -Also refer to: [1184, 1694, 1974, 2433, 3202].

Isolation from natural sources

-From the fruits of *Knema glauca* (Myristicaceae) [2562]. -In the wood of *Knema austrosiamensis* W. J. J. O. DeWilde (Myristaceae) [1133] or in Ground wood of this same plant [1134]. -In the seeds of *Horsfieldia iryaghedi* WARB. also named *Myristica horsfieldia* or

*Myristica iryaghedhi* (Myristicaceae) [1184, 1694]. -In the bark, leaf and timber of *Horsfieldia iryaghedi* (Myristicaceae) [3111].

-In the seeds of *Myristica fragrans* Houttuyn (nutmegs) [1694].

White amorphous solid [2562], white crystals [3202]; colourless needles [3111], colourless crystalline solid [1184], colourless plates [1694], colourless solid [1975]; m.p. 134° [2113, 3202], 128–130° [1184], 126–127° [3111], 125–126° [1694], 94–96° [1441]. **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [1133, 1694, 1975, 2364, 2562, 3111, 3202], ¹³C NMR [1975, 2562, 3202], IR [1184, 1694, 2562, 3111, 3202], UV [1694, 2562, 3202], MS [1133, 1694, 1975, 2562, 3111, 3202]; TLC [1184, 1694, 1975, 3202]; GLC [1694].

BIOLOGICAL ACTIVITY: Simplified YM-26734 inhibitors of secreted phospholipase A₂ group IIA [2364]; Antituberculosis activity against the microbe Mycobacterium tuberculosis [2562]; Antiviral activity against herpes simplex virus type 1 [2562]; Antimycobacterial and antimalarial activities [2562]; Cytotoxicity [2562, 3202]; Antifungal [2113, 3202]; Antibacterial [3202].

Trimethyl ether[888026-39-9] $C_{21}H_{34}O_4$ mol. wt. 350.50

-Refer to: [16, 1974].

m.p. 40° [16]; ¹H NMR [1974], ¹³C NMR [1974].

**Triacetate** [40220-94-8] C₂₄H₃₄O₇ mol. wt. 434.53

-Obtained by reaction of acetic anhydride with 2,4,6-trihydroxydodecanophenone in the presence of pyridine at r.t. for 48 h [1694].

Amorphous [1694]; ¹H NMR [1694], IR [1694], MS [1694].

**Tris(phenylmethyl) ether** [850816-19-2] C₃₉H₄₆O₄ mol. wt. 578.79

-Preparation by reaction of benzyl bromide with 1-(2,4,6-trihydroxyphenyl)-1-dodecanone in DMF in the presence of potassium carbonate at  $40^{\circ}$  under a nitrogen atmosphere overnight (63 %) [1975].

USE: DNA polymerase  $\beta$ -inhibiting, and DNA-damaging activity of (+)-myristinin A [1975]; Stereoselective synthesis, abs. stereochem. [1975].

colourless oil [1975]; ¹H NMR [1975], ¹³C NMR [1975], MS [1975]; TLC [1975].

# 1-(3,4,5-Trihydroxyphenyl)-1-dodecanone

[40336-21-8]	$C_{18}H_{28}O_4$	mol. wt. 308.42
HO HO CO(CH ₂ ) ₁₀ CH ₃	Synthesis -Refer to: [1982]. USE: Pressure-sensitive copying examination papers with hidden	
Trimethyl ether	$C_{21}H_{34}O_4$	mol. wt. 350.50

m.p. 65° [160].

# **4-Nitrophenylhydrazone** $C_{27}H_{39}N_3O_5$ mol. wt. 485.62

m.p.  $96^{\circ}$  [160].

# 3-Amino-1-(4-hydroxyphenyl)-1-dodecanone

$$C_{18}H_{29}NO_2 \qquad \text{mol. wt. 291.43}$$

$$NH_2 \qquad Synthesis$$

$$CO-CH-(CH_2)_9CH_3 \quad -\text{Refer to: [2253].}$$

$$HO$$

Hydrochloride	[63424-82-8]	C ₁₈ H ₂₉ NO _{2, HCl}	mol. wt. 327.90
m.p. 182–183° [2	2253]; IR [2253].		

# 2-Ethyl-1-(4-hydroxyphenyl)-1-dodecanone

C₂₀H₃₂O₂ mol. wt. 304.47

OH	Synthesis		
$\checkmark$	-Refer to: [956	].	
	Methyl ether	[201791-66-4]	
$C_2H_5$	$C_{21}H_{34}O_2$		mol. wt. 318.50
CO-CH-C ₁₀ H ₂₁			

-Obtained from 4-methoxyphenyl cyclopropyl ketone and 1-iododecane (95 %) [956].

¹H NMR [956], ¹³C NMR [956], MS [956]; TLC [956].

# 1.2 Substituted Hydroxyketones

# 1-(3,5-Dibromo-4-hydroxyphenyl)-1-dodecanone

[1393654-90-4]	$C_{18}H_{26}Br_2O_2$	mol. wt. 434.21
Br CO(CH ₂ ) ₁₀ CH ₃ HO	Synthesis -Obtained by reaction of bromine in acetic acid with the hydroxydodecanone [2339].	
Br	BIOLOGICAL ACTIVITY: Enzymetion of, [2339].	e activity, inhibi-

# 1-(5-Bromo-2,4-dihydroxyphenyl)-1-dodecanone

$C_{18}H_{27}BrO_3$		mol. wt. 371.32
HO Br	Synthesis -Refer to: [859]. m.p. 84–85° [859].	

# 1-(5-Bromo-2,3,4-trihydroxyphenyl)-1-dodecanone

$C_{18}H_{27}BrO_4$		mol. wt. 387.31
HO HO Br $CO(CH_2)_{10}CH_3$	Synthesis -Refer to: [859]. m.p. 84–85° [859].	

# 1-(3-Chloro-2-hydroxyphenyl)-1-dodecanone

	$C_{18}H_{27}ClO_2$	mol. wt. 310.86
Cl CO(CH ₂ ) ₁₀ CH ₃	Synthesis -Refer to: [483]. <b>Oxime</b> [101002-23-7] C ₁₉ H ₂₈ CINO ₂	mol. wt. 325.87

USE: Topical preparations comprising at least one aryloxime and bisabolol [483]; As lipoxygenase inhibitor [3077].

#### 1-(4-Chloro-2-hydroxyphenyl)-1-dodecanone

[859310-32-0]	C ₁₈ H ₂₇ ClO	2		mol. wt. 310	.86
CI CO(CH ₂ ) ₁₀ CH ₃		laurat at 13	e with al 0° for 2		of
h = 240° [2802]					

b.p.₃₈ 240° [2802].

# **2,4-Dinitrophenylhydrazone** $C_{24}H_{31}ClN_4O_5$ mol. wt. 490.99

m.p. 118° [2802].

# Methyl ether $C_{19}H_{29}ClO_2$ mol. wt. 324.89

-Obtained by methylation of the above ketone in the usual way (77 %) [2802].

 $b.p._{31}$  150° [2802].

# 1-(5-Chloro-2-hydroxyphenyl)-1-dodecanone

[98813-30-9] C₁₈H₂₇ClO₂ mol. wt. 310.86

OH	Syntheses
CO(CH ₂ ) ₁₀ CH ₃	-Obtained by reaction of dodecanoyl chloride with
	4-chlorophenol in the presence of aluminium chloride
Ŷ	(67.8 %) [2680].
Ċl	-Also refer to: [483].

b.p.₁ 170–173° [2680]; m.p. 74–75° [2680].

Oxime	[101002-24-8]	$C_{18}H_{28}CINO_2$	mol. wt. 325.88
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USE: Topical preparations comprising at least one aryloxime and bisabolol [483]; As lipoxygenase inhibitor [3077].

# 1-(5-Fluoro-2-hydroxyphenyl)-1-dodecanone

$$\begin{array}{cccc} [1480-47-3] & C_{18}H_{27}FO_2 & \mbox{mol. wt. } 294.41 \\ OH & Synthesis \\ -Obtained by Fries rearrangement of p-fluorophenyl laurate with aluminium chloride for 2 h at 130° \\ (50 \%) [1549]. \\ b.p._{10} 320° [1549]. \end{array}$$

 $\label{eq:2.4.1} \textbf{2,4-Dinitrophenylhydrazone} \quad [2341-98-2] \quad C_{24}H_{31}FN_4O_5 \quad \text{mol. wt. } 474.53$ 

m.p. 105° [1549].

# 1-(4-Hydroxy-3-nitrophenyl)-1-dodecanone

$$\begin{array}{c} [70079\text{-}24\text{-}2] & C_{18}H_{27}NO_4 & \text{mol. wt. } 321.42 \\ OH & Syntheses \\ -Obtained by treatment of 4-dodecanoylphenol with concentrated nitric acid in concentrated sulfuric acid at 0° for 1 h 15 min (91 %) [1222]. \\ CO(CH_2)_{10}CH_3 & -Also refer to: [1221, 1223, 1226]. \\ \text{m.p. } 72\text{-}73^{\circ} [1222]; \ ^1\text{H NMR } [1222]. \end{array}$$

**N.B.**: Active 4-dodecanoyl-2-nitrophenyl esters of  $\beta$ -alanine,  $\beta$ -alanyl- $\beta$ -alanine and  $\beta$ -alanyl- $\beta$ -alanyl- $\beta$ -alanine were prepared, and tried to polymerize in various solvents [1223].

Methyl ether	[76752-90-4]	$C_{19}H_{29}NO_4$	mol. wt. 335.44

-Refer to: [1221].

# 2-Hydroxy-5-dodecanoylbenzoic acid

CO(CH₂)₁₀CH₃ (94 %) [689]. -Also refer to: [1844, 1855, 2439].

b.p._{0.5} 205–230° [448]; m.p. 121–122° [448], 119–120° [689].

USE: For the preparation of a cosmetic, dermatological or pharmaceutical composition [2885].

Oxime	$C_{19}H_{29}NO_4$	mol. wt. 335.44
m.p. 113–116° [448].		

**Thiosemicarbazone**  $C_{20}H_{31}N_3O_3S$  mol. wt. 393.55

m.p. 230–232° [448].

	Methyl ester	[78432-96-9]	$C_{20}H_{30}O_4$	mol. wt. 334.46
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-Obtained by Fries rearrangement of methyl 2-lauroyloxybenzoate (b.p. $_{0.5}$  200–205°; m.p. 23–27°) with aluminium chloride in nitrobenzene for 4 h at 60° (82 %) [448].

-Also obtained by reaction of dodecanoyl chloride with methyl salicylate in the presence of aluminium chloride in carbon disulfide first at  $5-10^{\circ}$ , then at r. t. for 12 h (82 %) [689].

oil [448]; m.p. 70–72° [689].

#### Ethyl ester

$$C_{21}H_{32}O_4$$
 mol. wt. 348.48

b.p._{0.5} 210–215° [448].

# 1-[(3-Chloromethyl)-4-hydroxyphenyl]-1-dodecanone

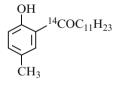
	C ₁₉ H ₂₉ ClO ₂	mol. wt. 324.89
OH	Synthesis -Refer to: [2252].	
CO(CH ₂ ) ₁₀ CH ₃	Methyl ether $C_{20}H_{31}ClO_2$ -Refer to: [2252].	mol. wt. 338.92
00(0112)100113		

m.p. 41-43° [2252].

# 1-(2-Hydroxy-5-methylphenyl)-¹⁴C-1-dodecanone

Synthesis

$$C_{18}^{14}CH_{30}O_2$$
 mol. wt. 292.44



¹⁴COC₁₁H₂₃ -It was prepared in 2 steps from p-cresol and  $CH_3(CH_2)_{10}$ ¹⁴COCl by Friedel-Crafts acylation in the presence of aluminium chloride at 40° to give intermediate ester, which underwent a Fries rearrangement in carbon disulfide (80 %) [474].

HPLC [474], TLC [474].

# 1-(2-Hydroxy-4-methylphenyl)-1-dodecanone

[72724-26-6]  $C_{19}H_{30}O_{2}$ mol. wt. 290.45 Syntheses OH CO(CH₂)₁₀CH₃ -Preparation by Fries rearrangement of 3-methylphenyl laurate with aluminium chloride CH without solvent at  $140-150^{\circ}$  [906]. -Preparation by one-step esterification-Fries rearrangement:

Obtained by heating a mixture of m-cresol and lauric acid with aluminium chloride at 180° for 3 h (esterification). Then, aluminium chloride was added and the mixture heated 2 h again at 180° (70 %) (Fries rearrangement) [1144]. -Also refer to: [17, 92, 1050, 2983, 3013].

m.p. 45–46° [906], 43–44° [3013]; GC [3012, 3013]; polarity [3013].

Nickel complex [80849-31-6].

-Refer to: [1050].

**Oxime** [40867-42-3]  $C_{19}H_{31}NO_2$  mol. wt. 305.46

-Refer to: [17, 2473, 3044].

USE: Copper extn. by, [3044]; Triplet state quenching by, kinetics of [17].

# Oxime, nickel complex [29666-10-2]

USE: Antifading agent from, for colour photothermog. image [1071]; Colour photog. material contg. antioxidant and magenta coupler and, with improved image stability [2283]; Photog. stabilizer compns. contg. phenol derivs. and, for magenta images [1050]; Singlet oxygen quenching by, [976]; Antioxidant light stabilizers, for acrylic graft polymers, [3484]; Light stabilizers, for polystyrene, [1251]; Light stabilizers, for titanium dioxide-pigmented polyolefins, [88]; Light stabilizers, for piperidinoanthraquinone dye in epoxy resin film, [86]; Light stabilizers, for polypropylene, mechanism of activity of, [87].

# Oxime, nickel complex [40690-25-3]

-Refer to: [451].

USE: Triplet state quenching by, kinetics of [17].

# Oxime, palladium complex [41894-24-0]

-Energy transfer to ligand-field states of, in aromatic hydrocarbon triplet state quenching [92].

# Oxime, cobalt alloys [60202-03-1]

-Catalyst, for oxidation of cycloalkanes to cycloalkanol-cycloalkanone mixtures [426].

 $C_{19}H_{30}O_{2}$ 

# 1-(2-Hydroxy-5-methylphenyl)-1-dodecanone

[75487-44-4]

Syntheses

mol. wt. 290.45

 $\begin{array}{c} OH \\ CO(CH_2)_{10}CH_3 \\ \end{array}$ 

-Obtained by Fries rearrangement of 4-methylphenyl dodecanoate,

*in the presence of aluminium chloride in nitrobenzene for 2 h at 120° [3434];

*under microwave irradiation for 8 min (89.5–93.4 %) [3434].

-Also obtained by reaction of dodecanoyl chloride with p-cresol in the presence of aluminium chloride [3431], (77 %) [2909] in ethylene chloride at  $110-120^{\circ}$  for 8 h (71 %) [1769].

-Also refer to: [972, 1355, 1356, 1827, 2078, 2079, 3077, 3432, 3433]. -Refer to: [1827, 2167].

Light yellow solid [2909]; b.p.₄ 186–188° [1769]; b.p.₃ 190° [2909]; m.p. 43–45° [2909], 37.5–39° [1769]; UV [1827], MS [1827].

USE: Surface activity of Schiff base surfactant [3433].

BIOLOGICAL ACTIVITY: Biotransformation of the lipoxygenase inhibitor (FLM 5011) [1827].

**Oxime** [50652-76-1] [103582-41-8] (E)  $C_{19}H_{31}NO_2$  mol. wt. 305.46 -Refer to: [110, 484–488, 1034, 1769, 1826, 1921, 1927, 2167, 2474, 2475, 2742, 3011, 3149, 3290].

m.p. 95–96.5° [1769]; ¹H NMR [1769, 1921], ¹³C NMR [1769], IR [1769, 1921], UV [1769, 1827, 1921], MS [1769, 1827, 1921].

USE: Compounds comprising active agents, natural products, and vitamins in combination with energy for regulation of mammalian keratinous tissue using skin and/or hair care actives [2078]; Skin and/or hair care compounds comprising Boswellia serrata tree gum and resin xymenynic acid and glycyrrhizinic acid for regulation of mammalian keratinous tissue [2079]; Protective effect of lipoxygenase inhibition during the early septic state [2119]; Topical preparations comprising at least one aryloxime and bisabolol [483]; Solvent extraction of copper (II) [1769, 2520]; In extn. of copper and nickel from sulfate solns. [110].

BIOLOGICAL ACTIVITY: Biotransformation of the lipoxygenase inhibitor (FLM 5011) [1827]; Cytotoxicity [1826]; As lipoxygenase inhibitor [3077]; Also refer to: [2167, 3149].

# Oxime, nickel complex [52672-75-0]

USE: Light stabilizer, for polypropylene, mechanism of action of, [3441].

# $\label{eq:2.4-Dinitrophenylhydrazone} \ensuremath{ [127699-73-4] C_{25}H_{34}N_4O_5 \ensuremath{ mol. wt. 470.57} \ensuremath{ km_{34}} \ensuremath{ mol. wt. 470.57} \ensuremath{ mol.$

m.p. 94–95° [1769].

# 1-(4-Hydroxy-2-methylphenyl)-1-dodecanone

	$C_{19}H_{30}O_2$	mol. wt. 290.45
Н	Synthesis	
	-Refer to: [2503].	
I	Phenyl ether [791615-80-0]	
CH ₃	$C_{25}H_{34}O_2$	mol. wt. 366.54
$O(CH_2)_{10}CH_3$		

-Obtained by adding a mixture of m-phenoxytoluene and dodecanoyl chloride to a suspension of aluminium chloride in methylene chloride at  $0^{\circ}$ , then the mixture stirred for 1.5–2 h at 3–5° (34 %) [2503].

b.p.₃ 240–245° [2503]; ¹H NMR [2503], IR [2503], MS [2503].

# 1-(4-Hydroxy-3-methylphenyl)-1-dodecanone

 $\begin{array}{cccc} [29665-55-2] & C_{19}H_{30}O_2 & \text{mol. wt. } 290.45 \\ & \\ OH & \\ CH_3 & \\ Obtained by Fries rearrangement of phenyl laurate in the presence of aluminium chloride in nitrobenzene [718], according to [380]. \end{array}$ 

 $CO(CH_2)_{10}CH_3$  m.p. 66–67° [718].

# 12-Hydroxy-1-(2-hydroxy-5-methylphenyl)-1-dodecanone

	$C_{19}H_{30}O_3$	mol. wt. 306.45
OH CO(CH ₂ ) ₁₀ CH ₂ OH CH ₃	Synthesis -Refer to: [1827]. UV [1827].	

BIOLOGICAL ACTIVITY: Metabolite of 2-hydroxy-5-methylaurophenone oxime in isolated rat hepatocytes.

#### 1-(2,4-Dihydroxy-5-methylphenyl)-1-dodecanone

 $[95102-14-0] C_{19}H_{30}O_{3} mol. wt. 306.45$   $OH CO(CH_{2})_{10}CH_{3} -Refer to: [1595, 2704].$  USE: Colour developer, for thermal recording materials [1595].

#### **Dimethyl ether**

-Obtained by reaction of dodecanoyl chloride with 4-methylresorcinol dimethyl ether in the presence of aluminium chloride in 1,2-dichloroethane first at  $0^{\circ}$ , then 2 h at r.t. (30 %) [11].

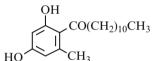
C21H34O3

m.p. 67–69° [11].

#### 1-(2,4-Dihydroxy-6-methylphenyl)-1-dodecanone

(4-Dodecanoylorcin)

$C_{19}H_{30}O_3$	mol. wt. 306.45
Synthesis	



 $CO(CH_2)_{10}CH_3$  -Obtained by reaction of dodecanoyl chloride with orcinol in the presence of aluminium chloride in nitrobenzene first 3 h at 0°, then at r.t. (20 %) [11].

m.p. 95–97° [11].

#### 1-(2,5-Dihydroxy-4-methylphenyl)-1-dodecanone

	$C_{19}H_{30}O_3$	mol. wt. 306.45
$OH$ $CO(CH_2)_{10}CH_3$	Syntheses -Refer to: [3021–3023].	
	Dihexadecyl ether [72046-93-6]	
CH ₃	$C_{51}H_{94}O_3$	mol. wt. 755.31
ОН	-Refer to: [3021–3023].	

USE: Colour photog. stabilizer compn. contg. [3023]; Photog. stabilizer compn. contg. hydroquinone deriv. and, [3021]; Photog. stabilizer compns. contg. for colour photog. materials [3022].

mol. wt. 334.50

#### 1-(2-Hydroxy-4-methoxyphenyl)-1-dodecanone

 $[143286-96-8] C_{19}H_{30}O_{3} mol. wt. 306.45$ OH CO(CH₂)₁₀CH₃ Syntheses -Obtained by reaction of methyl bromide with 1-(2,4-dihydroxyphenyl)-1-dodecanone in the presence of potassium carbonate in refluxing acetone for 20 h [284].

-Also refer to: [1787, 2273, 2959].

m.p. 50–51° [2273], 48–50° [284]; ¹H NMR [284].

USE: Protection against actinic radiations [2959].

Oxime	[143286-65-1]	$C_{19}H_{31}NO_3$	mol. wt. 321.46
Omne	145200 05 1	C 19113 1103	1101. Wt. 521.4

-Obtained by reaction of hydroxylamine hydrochloride with 1-(2-hydroxy-4-methoxyphenyl)-1-dodecanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 76.5–78.5° [284]; ¹H NMR [284].

#### 1-(2-Hydroxy-5-methoxyphenyl)-1-dodecanone

[56134-29-3]	$C_{19}H_{30}O_3$	mol. wt. 306.45
OH CO(CH ₂ ) ₁₀ CH ₃	Syntheses -Obtained by reaction of dodecanoyl of quinone dimethyl ether in the pres	•
OCH ₃	chloride [156] in carbon disulfide [15 -Also obtained by reaction of dodecan quinone monomethyl ether in the trifluoride at 60° for 15 h (49 %) [27	presence of boron

-Also obtained by reaction of lauroyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride in carbon disulfide [159].

-Also obtained by treatment of lauroylhydroquinone with diazomethane [159].

light yellow quartz-like crystals [159]; m.p. 43–44.5° [2752], 42–43° [156, 159].

-Refer to: [285].

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

#### 2,4-Dinitrophenylhydrazone

m.p. 121–124° [156, 159].

#### 1-(2,5-Dihydroxy-4-methoxyphenyl)-1-dodecanone

C25H34N4O6

 $O(CH_2)_{10}CH_3$ CH₂

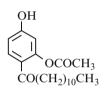
Synthesis					
-Obtained	by	deprotecti	on of	2-h	ydroxy-
5-levulinylo	oxy-4-i	nethoxydo	decanop	ohenoi	ne (m.p.
55–56°) w	vith a	sodium	sulfite	and	sodium
metabisulfite mixture in aqueous tetrahydrofuran,					
acetonitrile or ethanol at $40^{\circ}$ for 3 h (95 %) [2345].					

# 1-[2-(Acetyloxy)-4-hydroxyphenyl]-1-dodecanone

[251463-56-6]

C20H30O4

mol. wt. 334.46



Synthesis -Obtained by selective deacetylation of 2,4-diacetoxyphenyl undecyl ketone mediated by porcine pancreatic lipase (PPL) in THF at 42-45° for 24 h in the presence of n-butanol (45 %) [2517].

light yellow solid [2517]; m.p. 104° [2517]; ¹H NMR [2517], ¹³C NMR [2517], IR [2517], UV [2517], MS [2517].

# 1-(4-Ethyl-2-hydroxyphenyl)-1-dodecanone

$C_{20}H_{32}O_2$	mol. wt. 304.47
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O(CH₂)₁₀CH₃ -Obtained by Fries rearrangement of 3-ethylphenyl n-laurate (1 equiv.),

*in the presence of aluminium chloride (1.3 equiv.) in nitrobenzene at 25° for 6 h (75 %) [2801];

*in the presence of aluminium chloride (2.8 M), first in refluxing carbon disulfide for 2 h, then for 2 h at 130° after solvent elimination (73 %) [2801].

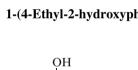
b.p.28 230° [2801].

#### 2,4-Dinitrophenylhydrazone

C₂₆H₃₆N₄O₅

mol. wt. 484.60

m.p. 90° [2801].



mol. wt. 486.57

### Methyl ether

 $C_{21}H_{34}O_2$ 

mol. wt. 318.50

mol. wt. 484.60

-Obtained by reaction of dimethyl sulfate with 1-(4-ethyl-2-hydroxyphenyl)-1-dodecanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating on the water bath for 5 h (82 %) [2801].

b.p.35 195° [2801].

# 1-(5-Ethyl-2-hydroxyphenyl)-1-dodecanone

	47
<ul> <li>CO(CH₂)₁₀CH₃</li> <li>Obtained by Fries rearrangement of 4-ethylphenyl laur with aluminium chloride at 100° for 2 h (67 %) [2800 b.p.₁₉ 210° [2800].</li> </ul>	

C₂₆H₃₆N₄O₅

### 2,4-Dinitrophenylhydrazone

m.p. 75° [2800].

# 1-(2-Hydroxy-3,5-dimethylphenyl)-1-dodecanone

	$C_{20}H_{32}O_2$			mol. wt.	304.47
CH ₃ CO(CH ₂ ) ₁₀ CH ₃	Synthesis -Refer to: [18	3311.			
	Oxime, nick	el comp			61
$\Gamma_{\rm CH_3}$	-Ultraviolet contg., [183	0	inhibitor,	spandex	fibers

# 1-(2-Hydroxy-4,6-dimethylphenyl)-1-dodecanone

[7282-05-5]	C ₂₀ H ₃₂ O	2		mol. wt. 304	1.47
OH CO(CH ₂ ) ₁₀ CH ₃	Syntheses -Obtained	by	Fries	rearrangement	of
	3,5-dimethylphenyl n-laurate (1 equiv.),				
CH ₃ CH ₃	*in the presence of aluminium chloride (1.3 equiv.)				
	in nitrobenzene at 25° for 6 h (67 %) [2801];				

*in the presence of aluminium chloride (2.8 M), first in refluxing carbon disulfide for 2 h, then for 2 h at  $130^{\circ}$  after solvent elimination (60 %) [2801].

-Also obtained by reaction of lauric acid with 3,5-dimethylphenol in the presence of boron trifluoride (56 %) [749].

light yellow oil [749], white crystals [749]; b.p.₁ 180–182° [749], b.p.₂ 200° [2801]; m.p. 42–44° [749].

OH

C₂H₅

4-Nitrophenylhydrazone	C ₂₆ H ₃₇ N ₃ O ₃	mol. wt. 439.60
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m.p. 190° [2801].

### Methyl ether

C₂₁H₃₄O₂ mol. wt. 318.50

-Obtained by reaction of dimethyl sulfate with 1-(2-hydroxy-4,6-dimethylphenyl)-1-dodecanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating on the water bath for 5 h (75 %) [2801].

b.p.35 220° [2801].

# 1-(4-Hydroxy-3,5-dimethylphenyl)-1-dodecanone

[23666-67-3]

C₂₀H₃₂O₂

Syntheses

-Obtained by Fries rearrangement of 2,6-dimethylphenyl laurate (*vic-m-Xylenyl dodecanoate*) (b.p.₁₂ 216–218°; m.p. 28–29°) in the presence of aluminium chloride O(CH₂)₁₀CH₃ (67 %) [184].

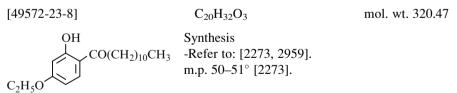
-Also obtained by reaction of dodecanoyl chloride with 2,6-dimethylphenol according to the method described previously [2871], (51 %) [119].

-Also obtained by reaction of lauroyl chloride with 2,6-dimethylphenol at 100° in the presence of aluminium chloride and heating at  $120-140^{\circ}$  for 1 h (58 %) [1832]. -Also obtained by reaction of dodecanoic acid with 2,6-dimethylphenol in the presence of boron trifluoride [1832].

-Also refer to: [1832].

b.p._{0.5} 175–185° [1832]; m.p. 52–53° [184], 52–52.5° [119], 50–51° [1832]; ¹H NMR [119], IR [119].

# 1-(4-Ethoxy-2-hydroxyphenyl)-1-dodecanone



USE: Protection against actinic radiations [2959].

965

mol. wt. 304.47

#### 1-(5-Ethoxy-2-hydroxyphenyl)-1-dodecanone

-Refer to: [285].

CH₂O

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

### 1-(2-Hydroxy-4-methoxy-5-methylphenyl)-1-dodecanone

[60488-53-1]	$C_{20}H_{32}O_3$	mol. wt. 320.47
CH ₃ O CH ₃ CO(CH ₂ ) ₁₀ CH ₃	1-(2,4-dimethox with boron trich	selective demethylation of y-5-methylphenyl)-1-dodecanone loride in methylene chloride first min at r.t. (83 %) [11].

### 1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1-dodecanone

 $[60488-57-5] C_{20}H_{32}O_3 mol. wt. 320.47$  OH Synthesis  $CO(CH_2)_{10}CH_3 -Preparation by partial methylation of$ 

1-(2,4-dihydroxy-6-methylphenyl)-1-dodecanone with methyl iodide in the presence of potassium carbonate in refluxing acetone for 8 h (75 %) [11]. m.p.  $58-59^{\circ}$  [11].

# 1-(2-Hydroxy-4,5-dimethoxyphenyl)-1-dodecanone

$$\begin{array}{c} [56134-34-0] \\ OH \\ CH_{3}O \end{array} \begin{array}{c} OH \\ OCH_{3} \end{array} \begin{array}{c} CO(CH_{2})_{10}CH_{3} \\ OCH_{3} \\ OCH_{3} \\ OCH_{3} \end{array} \begin{array}{c} CO(CH_{2})_{10}CH_{3} \\ OCH_{3} \\ OCH_{3} \\ OCH_{3} \end{array} \begin{array}{c} CO(CH_{2})_{10}CH_{3} \\ OCH_{3} \\ OCH_{3}$$

#### 1-(2-Hydroxy-4,6-dimethoxyphenyl)-1-dodecanone

$$[40000-60-0] C_{20}H_{32}O_4 mtext{mol. wt. 336.47} \\ OH \\ CO(CH_2)_{10}CH_3 mtext{CO(CH_2)_{10}CH_3} mtext{Syntheses} \\ -Obtained by reaction of dimethyl sulfate with 1-(2,4,6-trihydroxyphenyl)-1-dodecanone in the presence of potassium carbonate in acetone at 50–60° for 1.5 h under nitrogen (46 %) [2786]. \\ \end{tabular}$$

-Also obtained by methylation of dodecanoylphloroglucinol with diazomethane in ethanol at r. t. [1694].

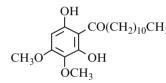
-Also refer to: [2786].

m.p. 80-83° [2786], 79-82° [1694]; ¹H NMR [2786], ¹³C NMR [1694, 2786], IR [1694], MS [1694, 2786].

#### 1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-dodecanone

$$[134081-97-3] C_{20}H_{32}O_5 mol. wt. 352.47$$

Synthesis



HSynthesis $CO(CH_2)_{10}CH_3$ -Preparation by treatment of 1-[6-hydroxy-<br/>3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phe-<br/>nyl]-1-dodecanone with potassium carbonate in<br/>refluxing methanol for 1–3 h (85 %) [1353]. refluxing methanol for 1-3 h (85 %) [1353].

m.p. 73–74° [1353]; ¹H NMR [1353].

#### 4-Hydroxy-3-(1-oxododecyl)-2H-1-benzopyran-2-one

C21H28O4 mol. wt. 344.45

Synthesis

-Obtained by reaction of dodecanoyl chloride with  $CO(CH_2)_{10}CH_3$  4-hydroxycoumarin in pyridine containing one drop of piperidine for 12 h on a water bath (57 %) [3174].

m.p. 110° [3174].

# 1-(8-Hydroxy-5-quinolinyl)-1-dodecanone

[101829-84-9]

 $C_{21}H_{29}NO_2$ 

mol. wt. 327.47



Syntheses

-Obtained by reaction of dodecanoyl chloride with 8-hydroxyquinoline in the presence of aluminium chloride [1336], *in nitrobenzene at  $80-85^{\circ}$  for 15-16 h (55 %) [1725];

*in nitrobenzene or 1,2-dichloroethane first at  $10-30^{\circ}$ , then at  $70-80^{\circ}$  for 20 h [2261].

-Also obtained by treatment of its hydrochloride with a sodium acetate solution [1946].

-Also refer to: [2464].

m.p. 66.8–68.5° [1725], 66–67° [2261], 45–46° [1336], 43° [1946]; **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [1725], IR [1725].

USE: Ion-flotation collector [1725]; Ion flotation with, of gallium [1725]; Good fungicide [1336]; Aluminium and gallium complexes, in extn., [2464]; Extn by, aluminium and gallium, [2464]; Extn. by Kelex 100 and, of aluminium and gallium, interface kinetics in relation to, [271].

# Hydrochloride

 $C_{21}H_{29}NO_2$ , HCl

mol. wt. 363.92

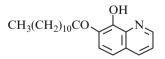
-Obtained by reaction of lauroyl chloride with 8-hydroxyquinoline in the presence of aluminium chloride in nitrobenzene for 12 h at 75–80° [1946].

# Methyl ether

-Obtained by treatment of 1-(8-hydroxy-5-quinolinyl)-1-dodecanone with dimethyl sulfate in benzene [1946].

# 1-(8-Hydroxy-7-quinolinyl)-1-dodecanone

[217815-28-6]



 $C_{21}H_{29}NO_2$ 

mol. wt. 327.47

Synthesis -Obtained by reaction of dodecanoyl chloride with 8-hydroxyquinoline in the presence of aluminium chloride in nitrobenzene or 1,2-dichloroethane first

at  $10-30^{\circ}$ , then at  $70-80^{\circ}$  for 20 h [2261].

m.p. 56–58° [2261]; ¹H NMR [2261].

# 1-(3-Acetyl-2,4,6-trihydroxy-5-methylphenyl)-1-dodecanone

[1092783-61-3]	$C_{21}H_{32}O_5$	mol. wt. 364.48
CH ₃ CO CO(CH ₂ ) ₁₀ CH ₃	Synthesis	of dodecanovl chloride

with 2,4,6-trihydroxy-3-methylacetophenone in the presence of aluminium chloride [2364]. ¹H NMR [2364], MS [2364]. но СН2 ОН

BIOLOGICAL ACTIVITY: Simplified YM-26734 inhibitors of secreted phospholipase A₂ group IIA [2364].

# 1-(2-Ethyl-6-hydroxy-4-methylphenyl)-1-dodecanone

	$C_{21}H_{34}O_2$	mol. wt. 318.50
CH ₃ CO(CH ₂ ) ₁₀ CH ₃ CH ₃ CO(CH ₂ ) ₁₀ CH ₃	5-methyl-phenyl laura *without solvent at 130	rearrangement of 3-ethyl- te with aluminium chloride, )° for 2 h (75 %) [2802]; ° for 6 h (78 %) [2802].

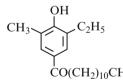
b.p.₃ 280° [2802].

#### Methyl ether C22H36O2 mol. wt. 332.53

-Obtained by methylation of the above ketone in the usual way (73 %) [2802].

b.p.23 230° [2802].

# 1-(3-Ethyl-4-hydroxy-5-methylphenyl)-1-dodecanone



 $CH_3$ CO(CH₂)₁₀CH₃

Synthesis CH₃  $C_2H_5$  C₂H₅ -Obtained by Fries rearrangement of 2-ethyl-6-methylphenyl laurate (b.p.₁₉ 218–220°) in the pre-sence of aluminium chloride (24 %) [184]. CO(CH₂)₁₀CH₃ m.p. 44.5–45.5° [184].

# 1-(4-Hydroxy-2,3,5-trimethylphenyl)-1-dodecanone

[137832-98-5]	$C_{21}H_{34}O_2$	mol. wt. 318.50
CH ₃ CH ₃	Synthesis -Refer to: [1733].	

#### 1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-dodecanone

[134081-66-6]	$C_{21}H_{34}O_5$	mol. wt. 366.50
CH ₃ O CH ₃ O CH ₃ O CH ₃ O OCH ₃	crude 2,3,4,6-tetrame	thoxydodecanophenone with a cetonitrile at $50^{\circ}$ for 1–2 h

m.p. 62–63° [1353]; ¹H NMR [1353].

**p-Toluenesulfonic ester** [134081-81-5] C₂₈H₄₀O₇S mol. wt. 520.69

-Obtained by reaction of p-toluenesulfonyl chloride with 2-hydroxy-3,4,6-trimethoxy-dodecanophenone in the presence of potassium carbonate in refluxing acetone for 6-14 h (92 %) [1353].

m.p. 85–87° [1353]; ¹H NMR [1353].

### Methyl ether $C_{22}H_{36}O_5$ mol. wt. 380.52

-Obtained by reaction of dodecanoyl chloride with 1,2,3,5-tetramethoxybenzene in the presence of aluminium chloride in ethyl ether at  $0^{\circ}$  for 30 min [1353].

# 1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-dodecanone

[134081-73-5]	$C_{21}H_{34}O_5$	mol. wt. 366.50
CH ₃ O OH CO(CH ₂ ) ₁₀ CH ₃ OCH ₃	Syntheses -Obtained by hydrogenation of 2,3,4-trimethoxydodecanopheno palladium on charcoal in ethy (1:1) until the uptake of	one over 10 % yl acetate/methanol
	(91 %) [1353].	

-Also refer to: [1351].

m.p. 43.5–44.5° [1353]; ¹H NMR [1353].

#### 12-Hydroxy-1-(2-hydroxy-3,4-dimethoxy-6-methylphenyl)-1-dodecanone

[77712-21-1]	$C_{21}H_{34}O_5$	mol. wt. 366.50
CH ₃ O CH ₂ O CH	Synthesis -Obtained by treatment of it with sodium hydroxide in m r.t. (75 %) [1147].	ts 12-acetyl ester ethanol for 2 h at

colourless needles [1147]; m.p. 82° [1147]; IR [1147], ¹H NMR [1147], MS [1147].

**12-Acetyl ester** [104966-93-0] C₂₃H₃₆O₆ mol. wt. 408.54

-Obtained by Friedel-Crafts reaction of 12-acetoxydodecanoyl chloride with 3,4,5-trimethoxy-toluene in 1,2-dichloroethane in the presence of aluminium chloride (2.1 mol) at 20° for 72 h [1147].

colourless oil [1147]; IR [1147], ¹H NMR [1147], MS [1147].

# 1-[3-(Dimethylaminomethyl)-4-hydroxyphenyl]-1-dodecanone

	$C_{21}H_{35}NO_2$	mol. wt. 333.51
OH	Synthesis	
$CH_2N(CH_3)_2$	-Refer to: [379].	
	Methyl ether [63829-15-2]	
Ý	$C_{22}H_{37}NO_2$	mol. wt. 347.54
CO(CH ₂ ) ₁₀ CH ₃	-Refer to: [2252].	

### 1-(2-Acetyl-4-hydroxy-7-benzofuranyl)-1-dodecanone

	$C_{22}H_{30}O_4$	mol. wt. 358.48
CO(CH ₂ ) ₁₀ CH ₃	Synthesis -Refer to: [682].	
OH	Methyl ether [59445-62-4] $C_{23}H_{32}O_4$	mol. wt. 372.50

-Obtained by reaction of dodecanoyl chloride with 2-acetyl-4-methoxybenzofuran with aluminium chloride (2.4 mol) in methylene chloride at r.t. for 24 h (82 %) [682].

m.p. 88° [682].

# 1-(2-Acetyl-7-hydroxy-4-benzofuranyl)-1-dodecanone

C. H. O.

	$C_{22}\Pi_{30}O_4$	mon. wt. 550.40
OH O COCH ₃	Synthesis -Refer to: [682].	
CO(CH ₂ ) ₁₀ CH ₃	Methyl ether [59445-73-7] $C_{23}H_{32}O_4$	mol. wt. 372.50

-Obtained by reaction of dodecanoyl chloride with 2-acetyl-7-methoxybenzofuran with aluminium chloride (2.4 mol) in methylene chloride at r.t. for 24 h (83 %) [682].

m.p. 83° [682].

mol wt 358.48

#### 1-(8-Hydroxy-2-methyl-5-quinolinyl)-1-dodecanone

 $\label{eq:constraint} [217815-24-2] \qquad \qquad \text{C}_{22}H_{31}NO_2 \qquad \qquad \text{mol. wt. } 341.50$ 

 $\bigcup_{\substack{I \\ CO(CH_2)_{10}CH_3}}^{OH} CH_3$ 

Syntheses -Obtained by reaction of dodecanoyl chloride with 8-hydroxyquinaldine in the presence of aluminium chloride in nitrobenzene or 1,2-dichloroethane first at 10–30°, then at 70–80° for 20 h [2261]. -Also refer to: [1336].

m.p. 52–54° [1336], 50–52° [2261]; MS [2261].

USE: Good fungicide [1336].

N-Methylcarbamate	$C_{24}H_{34}NO_3$	mol. wt. 384.54
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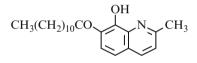
-Refer to: [1335] (36 %).

m.p. 87–90° [1335].

BIOLOGICAL ACTIVITY: Fungicide [1335].

# 1-(8-Hydroxy-2-methyl-7-quinolinyl)-1-dodecanone

[217815-27-5] C₂₂H₃₁NO₂ mol. wt. 341.50



Synthesis -Obtained by reaction of dodecanoyl chloride with 8-hydroxyquinaldine in the presence of aluminium chloride in nitrobenzene or 1,2-dichloroethane first at  $10-30^{\circ}$ , then at  $70-80^{\circ}$  for 20 h [2261].

mol. wt. 366.97

m.p. 67–68° [2261]; IR [2261], MS [2261].

# 1-[3-Chloro-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-dodecanone

C22H35ClO2

[102701-83-7] OH Cl  $\leftarrow$  CO(CH₂)₁₀C

 $\dot{C}(CH_3)_3$ 

CO(CH₂)₁₀CH₃ -Obtained by Fries rearrangement of 2-chloro-4-tertbutylphenyl laurate with aluminium chloride at 110° (68 %) [3119]. H₃)₃ b.p.₂₂ 180° [3119].

# **2,4-Dinitrophenylhydrazone** [103330-25-2] $C_{28}H_{39}ClN_4O_5$ mol. wt. 547.09

m.p. 193° [3119].

# 1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-dodecanone

 $C_{22}H_{36}O_{2}$ mol. wt. 332.53 OH CO(CH₂)₁₀CH₃ Synthesis -Refer to: [1050]. Oxime, nickel complex [81321-89-3] USE: Photog. stabilizer compns. contg. phenol derivs. and, for magenta images [1050]. 1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-dodecanone C₂₂H₃₆O₂ mol. wt. 332.53

 $\begin{array}{c} C_{22}H_{36}O_2 \\ (CH_3)_2CH \underbrace{OH}_{CH_3} \\ (CH_3)_2CH \underbrace{OH}_{C$ 

b.p.16 273° [2803].

# **2,4-Dinitrophenylhydrazone** $C_{28}H_{40}N_4O_5$ mol. wt. 512.65

m.p. 151° [2803].

#### 1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]dodecanone

(	$C_{22}H_{36}O_2$	mol. wt. 332.53
(CH ₃ ) ₂ CH CH ₃ CO(CH ₂ ) ₁₀ CH ₃	Synthesis -Obtained ( <b>XXX</b> ) by treatment 2-methyl-5-isopropyldodecanopyling ing pyridinium chloride (205) (13 %) [2660]. b.p. ₁₆ 265–267° [2660]; m.p. 59	henone with boil- $-215^{\circ}$ ) for 5 h

#### Methyl ether (XIII)

-Obtained by reaction of lauroyl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (52 %) [2660].

C₂₃H₃₈O₂

mol. wt. 346.55

#### 1-(4-Butoxy-2-hydroxyphenyl)-1-dodecanone

m.p. 42-43° [284].

**Oxime** [143286-66-2] C₂₂H₃₇NO₃ mol. wt. 363.54

-Obtained by reaction of hydroxylamine hydrochloride with 1-(4-butoxy-2-hydroxyphenyl)-1-dodecanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 58–62° [284]; ¹H NMR [284].

#### 1-(5-Butoxy-2-hydroxyphenyl)-1-dodecanone

[140943-39-1]	$C_{22}H_{36}O_{3}$	mol. wt. 348.53
ОН	Synthesis	
CO(CH ₂ ) ₁₀ CH ₃	-Refer to: [285].	
	<b>Oxime</b> [140943-25-5]	
Ý	C ₂₂ H ₃₇ NO ₃	mol. wt. 363.54
OC ₄ H ₉		

-Refer to: [285].

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

#### 1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-dodecanone

[872178-00-2]

 $C_{23}H_{36}O_4$ 

mol. wt. 376.54

 $(CH_3)_2C = CHCH_2$ HO HO HO OH Synthesis

-Obtained by reaction of prenyl bromide with 2,4,6-trihydroxydodecanophenone in the presence of potassium carbonate in refluxing acetone for 5 h (13 %) [3202].

white crystalline solid [3202]; m.p. 114° [3202]; ¹H NMR [3202], ¹³C NMR [3202], IR [3202], UV [3202], MS [3202]; TLC [3202].

BIOLOGICAL ACTIVITY: Antibiotic [3202].

#### 1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methylphenyl]-1-dodecanone

$$C_{23}H_{38}O_2$$
 mol. wt. 346.55  
OH Syntheses  
-Obtained by Fries rearrangement of 2-tert-  
butyl-5-methylphenyl laurate,  
*in the presence of aluminium chloride (1.5  
equiv.) in nitrobenzene at 25° for 6 h (72 %)  
[3118]:

*in the presence of aluminium chloride (3 equiv.) at 110° for 2 h (68 %) [3118].

b.p.17 220° [3118].

#### 1-(2-Hydroxy-5-pentylphenyl)-1-dodecanone

$$[102898-52-2] C_{23}H_{38}O_2 mol. wt. 346.55$$

# Synthesis -Obtained by reaction of lauric acid with 4-pentylphenol in the presence of boron trifluoride (66 %) [142]. b.p.₂ 189–194° [142]; $n_D^{25} = 1.500$ [142].

### 1-(4-Hydroxy-3-pentylphenyl)-1-dodecanone

C23H38O2 mol. wt. 346.55

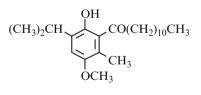


Synthesis -Obtained by reaction of lauric acid with 2-pentylphenol in the presence of boron trifluoride (66 %) [142]. b.p.2 189-194° [142].

#### 1-[2-Hydroxy-5-methoxy-6-methyl-3-(1-methylethyl)phenyl]-1-dodecanone

C23H38O3

mol. wt. 362.55

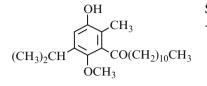


OH  $CO(CH_2)_{10}CH_3$  Synthesis Obtained by reaction of lauroyl chloride with2-methyl-5-isopropylhydroquinone dimethylether in the presence of aluminium chloridein carbox div 10 l <math>C is a fixed with the fixed set of in carbon disulfide, first at 40° for 1 h, then at  $0^{\circ}$  for 48 h and lastly on the boiling water bath for 3 h (10.5 %) [2663].

colourless crystals [2663]; b.p.₁₈ 127–129° [2663]; m.p. 91° [2663].

#### 1-[3-Hydroxy-6-methoxy-2-methyl-5-(1-methylethyl)phenyl]-1-dodecanone

$$[115211-15-9] C_{23}H_{38}O_3 mol. wt. 362.55$$



**Synthesis** 

-Obtained by reaction of lauroyl chloride with CO(CH₂)₁₀CH₃ -Obtained by reaction of lauroyl chloride with 2-methyl-5-isopropylhydroquinone dimethyl ether in the presence of aluminium chloride in carbon disulfide, first at 40° for 1 h, then at  $0^{\circ}$  for 48 h and lastly on the boiling water bath for 3 h (10.5 %) [2663].

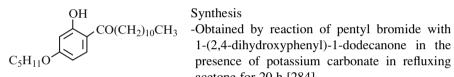
colourless crystals [2663]; b.p.₁₈ 127–129° [2663]; m.p. 91° [2663]; IR [2663].

Synthesis

#### 1-(2-Hydroxy-4-pentyloxyphenyl)-1-dodecanone

[143286-98-0]

C23H38O3 mol. wt. 362.55



m.p. 42° [284].

acetone for 20 h [284].

-Obtained by reaction of hydroxylamine hydrochloride with 1-(2-hydroxy-4-pentyloxyphenyl)-1-dodecanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 43° [284]: ¹H NMR [284].

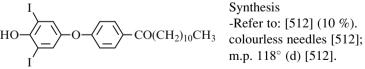
#### 4'-[4-Hydroxy-3,5-(diiodo)diphenyl]ether-4-(1-dodecanone)

4'-(4-Hydroxy-3,5-diiodophenoxy)dodecanophenone

[23951-55-5]

 $C_{24}H_{30}I_2O_3$ 

mol. wt. 619.88



Synthesis

#### 1,12-Bis(4-hydroxyphenyl)-1,12-dodecanedione

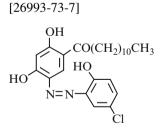
H Synthesis -Refer to: [2751]. -OH m.p. 204–208° [2751]. OH ·CO(CH₂)₁₀CO→ но

# 1,12-Bis(3,5-dihydroxyphenyl)-1,12-dodecanedione

$$\begin{array}{c} C_{24}H_{30}O_6 & \text{mol. wt. 414.50} \\ HO & OH & \text{Synthesis} \\ -CO(CH_2)_{10}CO & -CO(CH_2)_{10}CO &$$

#### 5-[[(5-Chloro-2-hydroxyphenyl)azo]-2,4-dihydroxyphenyl]-1-dodecanone

C₂₄H₃₁ClN₂O₄



Synthesis -Obtained by reaction of 2-amino-4-chlorophenol diazonium salt with 2,4-dihydroxylaurophenone in the presence of sodium hydroxide between 100 sectors 10° [577]. m.p. 247–248° [577]. Metal complexes Refer to: [577].

mol. wt. 446.97

977

# 1-(3'-Chloro-4'-hydroxy[1,1'-biphenyl]-4-yl)-1-dodecanone

$$\begin{array}{c} C_{24}H_{31}ClO_2 & \text{mol. wt. 386.66} \\ & & \\ HO \longrightarrow & \\ HO \longrightarrow & CO(CH_2)_{10}CH_3 \end{array} \\ \end{array}$$

**6-Methyloctyl ether** (S) [112780-57-1]  $C_{33}H_{49}ClO_2$  mol. wt. 513.20

-Liq.-crystal compns. contg., for display devices [2107].

# 1-(3'-Fluoro-4'-hydroxy[1,1'-biphenyl]-4-yl)-1-dodecanone

[112780-73-1] C ₂ .	$_4H_{31}FO_2$	mol. wt. 370.51
НО- <b>С</b> О(СН ₂ ) ₁₀ СН ₃	Synthesis -Refer to: [2107]. -Liqcrystal compns. devices [2107].	contg., for display

Methyl ether	[112780-72-0]	$C_{25}H_{33}FO_2$	mol. wt. 384.53
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-Liq.-crystal compns. contg., for display devices [2107].

6-Methyloctyl ether (S)	[112780-56-0]	$\mathrm{C}_{33}\mathrm{H}_{49}\mathrm{FO}_2$	mol. wt. 496.75
-Liqcrystal compns. contg.,	for display devices	[2107].	

8-Methyldecyl ether (S)	[112780-59-3]	$C_{35}H_{53}FO_2$	mol. wt. 524.80
-Liqcrystal compns. contg.	, for display devices	s [2107].	

# 5-[[(2-Hydroxyphenyl)azo]-2,4-dihydroxyphenyl]-1-dodecanone

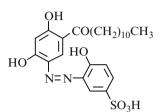
[27020-17-3]	$C_{24}H_{32}N_2O_4$	mol. wt. 412.53
HO $N=N$ $N$ $N=N$ $N=N$ $N$ $N$ $N=N$ $N$ $N=N$ $N$ $N$ $N$ $N=N$ $N$ $N$ $N$ $N$ $N$ $N$ $N$ $N$ $N$	Synthesis -Obtained by reaction of 2 salt with 2,4-dihydroxylau of sodium hydroxide betw m.p. 185–187° [577].	rophenone in the presence

# Metal complexes

Refer to: [577].

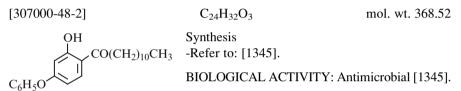
# 5-[[(2-Hydroxy-5-sulfonylphenyl)azo]-2,4-dihydroxyphenyl]-1-dodecanone

C24H32N2O7S mol. wt. 492.59



Synthesis  $CO(CH_2)_{10}CH_3$  -Obtained by reaction of 2-amino-4-sulfonylphenol diazonium salt with 2,4-dihydroxylaurophenone in the presence of sodium hydroxide between 5 and 10° [578]. Metal complexes -Refer to: [578].

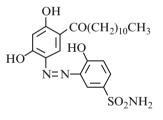
1-(2-Hydroxy-4-phenoxyphenyl)-1-dodecanone



# 5-[[(5-Aminosulfonyl-2-hydroxyphenyl)azo]-2,4-dihydroxyphenyl]-**1-dodecanone**

[26993-74-8]

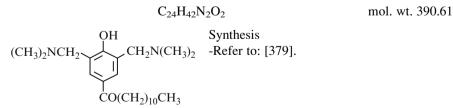
C24H33N3O6S mol. wt. 491.61



Synthesis

-Obtained by reaction of 2-amino-4-aminosulfonylphenol diazonium salt with 2,4-dihydroxylaurophenone in the presence of sodium hydroxide between 5 and  $10^{\circ}$  [577]. -Also refer to: [576]. m.p. 243-244° [576, 577]. Metal complexes -Refer to: [577].

# 1-[3,5-Bis(dimethylaminomethyl)-4-hydroxyphenyl]-1-dodecanone



### 1-[5-(4-Chlorobenzoyl)-2,4-dihydroxyphenyl]-1-dodecanone

$$\begin{array}{c} C_{25}H_{31}ClO_4 & \text{mol. wt. 430.10} \\ OH & \text{Synthesis} \\ + CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & -Preparation & \text{from} \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & -Preparation & -Preparation & -Preparation \\ + OH & -CO(CH_2)_{10}CH_3 & -Preparation & -Preparation & -Preparation & -Preparation \\ + OH & -Preparation & -P$$

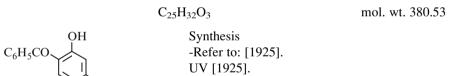
# 5-[[(2-Carboxyphenyl)azo]-2,4-dihydroxyphenyl]-1-dodecanone

Metal complexes

Refer to: [577].

# 1-(4-Benzoyl-3-hydroxyphenyl)-1-dodecanone

4'-Benzoyl-3'-hydroxydodecanophenone



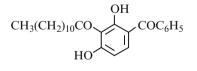
USE: Light stabilization of macromol. compds. [1925].

# 1-(3-Benzoyl-2,6-dihydroxyphenyl)-1-dodecanone

[14798-38-0]

C25H32O4

mol. wt. 396.53



 $CH_3(CH_2)_{10}CO$  HO  $COC_6H_5$   $COC_6H_5$   $Obtained by reaction of lauroyl chloride with 2,4-dihydroxybenzophenone in the presence of aluminium chloride in o-dichlorobenzene for 3 h at <math>120^{\circ}$  [134] 3 h at 120° [134].

-Also obtained by reaction of lauric acid with 2,4-dihydroxybenzophenone in the presence of para-toluenesulfonic acid in refluxing xylene for 20 h [134].

-Also refer to: [133, 1631].

m.p. 80-81° [134].

USE: For stabilizing polyolefins and vinyl halide resins against UV-light and sunlight [134]; As light stabilizer for chlorine-contg. polymers, [133]; Stabilization of vinyl halide resins and polyolefins against photodegradation [133].

#### 1-(5-Benzoyl-2,4-dihydroxyphenyl)-1-dodecanone

[14814-73-4] C25H32O4 mol. wt. 396.53 OH Syntheses -Obtained by reaction of lauroyl chloride COC₆H₅ with 2.4-dihydroxybenzophenone in the presence of alumin-HO ium chloride in o-dichlorobenzene for 3 h at 120° [134].  $CO(CH_2)_{10}CH_3$ -Also obtained by reaction of lauric acid with 2,4-dihydroxybenzophenone in the presence of paratoluenesulfonic acid in refluxing xylene for 20 h [134].

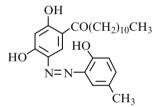
-Also refer to: [133, 1631].

m.p. 80–81° [1631], 59–61° [134].

USE: For stabilizing polyolefins and vinyl halide resins against UV-light and sunlight [134]; As light stabilizer for chlorine-contg. polymers, [133]; Stabilization of vinyl halide resins and polyolefins against photodegradation [133].

#### 5-[[(2-Hydroxy-5-methylphenyl)azo]-2,4-dihydroxyphenyl]-1-dodecanone

 $[26993-72-6] C_{25}H_{34}N_2O_4 mtext{mol. wt. } 426.56$ 

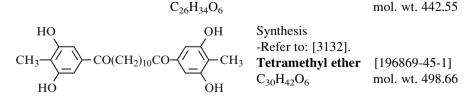


Synthesis -Obtained by reaction of 2-amino-4-methylphenol diazonium salt with 2,4-dihydroxylaurophenone in the presence of sodium hydroxide between 5 and 10° [577]. m.p. 200–201° [577]. Metal complexes Refer to: [577].

### 2-Butyl-1-(2,5-dihydroxy-3,4,6-trimethylphenyl)-1-dodecanone

$C_{25}H_{42}O_3$		mol. wt. 390.61
ОН СН ₃ СО-СН-С ₁₀ Н ₂₁	Synthesis -Refer to: [2352].	
L L L	<b>Dibenzyl ether</b> [357172-32-8]	
CH ₃ CH ₃ CH ₃	$C_{39}H_{54}O_3$	mol. wt. 570.86
ÓН	-Refer to: [2352].	

#### 1,12-Bis(3,5-dihydroxy-4-methylphenyl)-1,12-dodecanedione



-Synthesis from 3,5-dimethoxy-4-methylbenzaldehyde (70 %) [3132].

colourless crystalline solid [3132]; ¹H NMR [3132], ¹³C NMR [3132].

# 1-[5-(Acetyloxy)-2-hydroxyphenyl]-2-hexyl-1-dodecanone

# 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-dodecanone

USE: Stabilize plastics, oils, and fats against heat, light, and oxidation [951].

#### 1-[2-Hydroxy-5-(1,1,3,3-tetramethylbutyl)phenyl]-1-dodecanone

	$C_{26}H_{44}O_2$	mol. wt. 388.63
$\begin{array}{c} OH \\ CO(CH_2)_{10}CH_3 \\ CH_3 - C - CH_2 - C - CH_3 \\ CH_3 & CH_3 \end{array}$	Synthesis -Refer to: [2282]. <b>Oxime, nickel complex</b> -Refer to: [2280–2282].	[108111-24-6]

USE: Colour photog. emulsions contg. triazolotriazole deriv. magenta coupler and oil-sol. dyes and, for lightfast image formation [2282]; Photog. magenta dye image stabilizer [2284]; Silver halide photog. material contg. [2280, 2281].

#### Oxime, nickel complex [110351-70-7].

-Refer to: [1591].

-Singlet oxygen quencher, photog. photosensitive materials contg. [1591].

#### 1-[4-[(2-Ethylhexyl)oxy]-2-hydroxyphenyl]-1-dodecanone

$$\begin{array}{c} C_{26}H_{44}O_3 & \text{mol. wt. 404.63} \\ OH & Synthesis \\ C_2H_5 & CO(CH_2)_{10}CH_3 & -\text{Refer to: [2282].} \\ C_4H_9-CH-CH_2O & Oxime, nickel complex [108111-25-7] \end{array}$$

```
-Refer to: [2282, 2285, 2286].
```

USE: Colour photog. emulsions contg. triazolotriazole deriv. magenta coupler and oil-sol. dyes and, for lightfast image formation [2282]; Photog. magenta dye image stabilizer [2284]; Photog. stabilizer compns. contg., for magenta dye image stabilization [2286]; Singlet oxygen quencher, colour photog. paper contg. [2285].

#### 1-[2-Hydroxy-4-(octyloxy)phenyl]-1-dodecanone

[101002-34-0] C₂₆H₄₄O₃ mol. wt. 404.63 [143287-09-6]

Syntheses

 $C_8H_{17}O$   $CO(CH_2)_{10}CH_3$   $CO(CH_2)_{10}CH_3$ 

•CO(CH₂)₁₀CH₃ •Obtained by reaction of octyl bromide with 1-(2,4-dihydroxyphenyl)-1-dodecanone in the presence of potassium carbonate in refluxing acetone for 20 h [284].

-Refer to: [3077].

m.p. 56-59° [284].

Oxime

[101002-21-5]

C₂₆H₄₅NO₃

mol. wt. 419.65

-Obtained by reaction of hydroxylamine hydrochloride with 1-[2-hydroxy-4-(octyloxy)phenyl]-1-dodecanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

-Also refer to: [2742].

m.p. 60–63° [284, 3077]; ¹H NMR [284].

USE: Topical preparations comprising at least one aryloxime and bisabolol [483]; As lipoxygenase inhibitor [3077].

983

#### 1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-1-dodecanone

[134082-05-6]	$C_{27}H_{38}O_5$	mol. wt. 442.60
OH CH ₃ O CH ₃ O CH ₃ O CH ₂ CO(CH ₂ ) ₁₀ CH ₃ OCH ₂ C ₆ H ₅	group in 2-position b benzyloxy-3,4-dimetho	ion of one phenylmethyl y treatment of 1-(2,6-di- oxyphenyl)-1-dodecanone lrochloric acid and acetic 2 %) [1353].

m.p. 80.5–81° [1353]; ¹H NMR [1353].

# 1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]-1-dodecanone

[134081-89-3]

 $C_{27}H_{38}O_7S$ 

mol. wt. 506.66

mol. wt. 462.59

Synthesis

 $CH_{3}O \xrightarrow{OH} CO(CH_{2})_{10}CH_{3}$   $CH_{3}O \xrightarrow{O} OSO_{2}C_{6}H_{4}CH_{3}(p)$ 

m.p. 55.5–56° [1353]; ¹H NMR [1353].

#### 3-[(2-Naphthylazo)-2,4,6-trihydroxyphenyl]-1-dodecanone

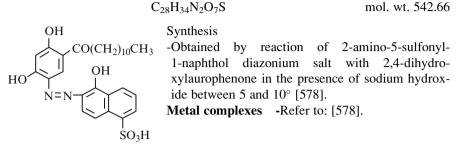
 $C_{28}H_{34}N_2O_4$ OH Syntheses OH -Obtained by 2-naphthylamine bydroxy phenyl

Syntheses -Obtained by coupling diazotized 2-naphthylamine with 2,4,6-trihydroxy-phenyl undecyl ketone [872].

```
-Also refer to: [2433].
```

USE: As water-repellent dye for wood [2433].

# 5-[[(1-Hydroxy-5-sulfonyl-2-naphthyl)azo]-2,4-dihydroxyphenyl]-1-dodecanone

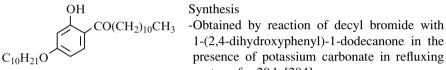


# 1-[4-(Decyloxy)-2-hydroxyphenyl]-1-dodecanone

[143286-99-1]

 $C_{28}H_{48}O_3$ **Synthesis** 

mol. wt. 432.69



1-(2,4-dihydroxyphenyl)-1-dodecanone in the presence of potassium carbonate in refluxing acetone for 20 h [284].

m.p. 52-60° [284].

Oxime $[101002-27-1]$ $C_{28}H_{49}NO_3$	mol. wt. 447.70
------------------------------------------	-----------------

-Obtained by reaction of hydroxylamine hydrochloride with 1-[4-(decyloxy)-2-hydroxyphenyl]-1-dodecanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

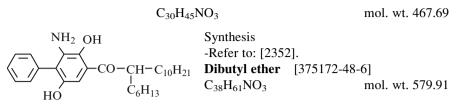
m.p. 64–69° [284, 3077]; ¹H NMR [284].

USE: Topical preparations comprising at least one aryloxime and bisabolol [483]; As lipoxygenase inhibitor [3077].

# 1-(2,5-dihydroxy-3,4,6-trimethylphenyl)-2-octyl-1-dodecanone

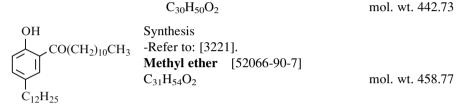
[357172-26-0]	$C_{29}H_{50}O_3$	mol. wt. 446.71
$CH_{3} \xrightarrow{OH} CO-CH-C_{10}H_{21}$ $CH_{3} \xrightarrow{I} CH_{3} \xrightarrow{C} CH_{3} \xrightarrow{C} C_{8}H_{17}$ $CH_{3} \xrightarrow{OH} CH_{3}$	Synthesis -Refer to: [2352].	

# 1-(2-Amino-3,6-dihydroxy[1,1'-biphenyl]-4-yl)-2-hexyl-1-dodecanone



-Refer to: [2352].

### 1-(5-Dodecyl-2-hydroxyphenyl)-1-dodecanone



-Refer to: [3221].

m.p. 37.5-39.5° [3221].

# 1,1'-(2,5-Dihydroxy-1,4-phenylene)bis-1-dodecanone

 $\begin{array}{c} C_{30}H_{50}O_4 & \text{mol. wt. 474.72} \\ OH & Syntheses \\ -Obtained by reaction of lauric acid with hydroquinone in the presence of zinc chloride (Nencki reaction) at 130–140° \\ OH & [156] \text{ for } 2 \text{ h} (23 \%) [1264]. \end{array}$ 

m.p. 68° [1264].

# 1,1'-(4,6-Dihydroxy-1,3-phenylene)bis-1-dodecanone

 $C_{30}H_{50}O_4$ 

mol. wt. 474.72

ОН СО(СН₂)₁₀СН₃ НО СО(СН₂)₁₀СН₃

Syntheses -Preparation by reaction of dodecanoic anhydride with resorcinol in the presence of boron trifluoride etherate [2364].

-Also obtained by reaction of dodecanoic acid with resorcinol in the presence of zinc chloride [2364].

¹H NMR [2364], MS [2364].

BIOLOGICAL ACTIVITY: Simplified YM-26734 inhibitors of secreted phospholipase A2 group IIA [2364].

#### 1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-dodecanone

[144337-28-0] C₃₀H₅₀O₅ mol. wt. 490.72 OH Syntheses

$$CH_{3}(CH_{2})_{10}CO \xrightarrow{CO(CH_{2})_{10}CH_{3}}_{HO}OH$$

-Obtained by reaction of dodecanoic anhydride with phloroglucinol in the presence of boron trifluoride etherate [2364].

-Also obtained by reaction of dodecanoic acid with phloroglucinol,

*in the presence of zinc chloride [2364];

*in the presence of boron trifluoride etherate at  $100^{\circ}$  for 2 h (reflux) (60 %) [338]. -Also refer to: [1216].

cream coloured solid [338]; ¹H NMR [338, 2364], MS [338].

BIOLOGICAL ACTIVITY: Simplified YM-26734 inhibitors of secreted phospholipase A2 group IIA [2364]; As a new class of GPR40 (FFAR1) agonists [338]; As phospholipase A2 inhibitor [1216].

#### 1-(2,5-Dihydroxy-4-dodecylphenyl)-1-dodecanone

 $[103398-77-2] C_{30}H_{52}O_3 mtext{mol. wt. 460.74} \\ OH \\ -Obtained by reaction of dodecanoic acid with 2-dodecylhydroquinone in the presence of boron trifluoride in the presence of boron tri$ 

1,2-dichloroethane at  $40-45^{\circ}$  for 1.25 h. The mixture was allowed to stand overnight (37 %) [142].

-Also refer to: [1907].

m.p. 88–90° [142, 1907].

#### 1-(2-Dodecyl-4,5-dihydroxyphenyl)-1-dodecanone

	$C_{30}H_{52}O_3$	mol. wt. 460.74
OH HO (CH ₂ ) ₁₁ CH ₃ CO(CH ₂ ) ₁₀ CH ₃	Synthesis -Refer to: [1167]. <b>Dimethyl ether</b> [930782-40-4] $C_{32}H_{56}O_3$ -Refer to: [1167].	mol. wt. 488.80

¹H NMR [1167], ¹³C NMR [1167].

# 1-(3-Dodecyl-2,4,6-trihydroxyphenyl)-1-dodecanone

[1092783-56-6] C30H52O4 mol. wt. 476.74

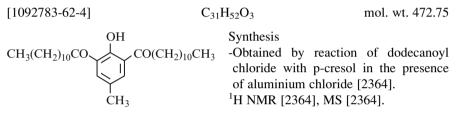
CH₃(CH₂)₁₁ HO

Synthesis  $\sim$  CO(CH₂)₁₀CH₃ -Preparation by reaction of dodecanoyl chloride with 2-dodecylphloroglucinol in the presence of aluminium chloride [2364].

¹H NMR [2364], MS [2364].

BIOLOGICAL ACTIVITY: Simplified YM-26734 inhibitors of secreted phospholipase A₂ group IIA [2364].

### 1,1'-(2-Hydroxy-5-methyl-1,3-phenylene)bis-1-dodecanone



BIOLOGICAL ACTIVITY: Simplified YM-26734 inhibitors of secreted phospholipase A₂ group IIA [2364].

# 1,1'-(2,4,6-Trihydroxy-5-methyl-1,3-phenylene)bis-1-dodecanone

[1092783-59-9]	C ₃₁ I	$H_{52}O_5$	mol. v	vt. 504.75
Ċ	CO(CH ₂ ) ₁₀ CH ₃ OH	chioride with	2-methylphlor sence of a	roglucinol
	1 10 100(1)			

¹H NMR [2364], MS [2364].

BIOLOGICAL ACTIVITY: Simplified YM-26734 inhibitors of secreted phospholipase A₂ group IIA [2364].

# 1-(3-Dodecyl-2-hydroxy-5-methylphenyl)-1-dodecanone

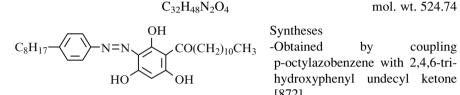
C₃₁H₅₄O₂ mol. wt. 458.77

Synthesis -Refer to: [1831]. Oxime, nickel complexes -Ultraviolet light inhibitor, spandex fibers contg., [1831].

# 1-(3-Dodecyl-2-hydroxy-5-methoxyphenyl)-1-dodecanone

[56134-31-7]	C	$_{31}H_{54}O_3$	mol. wt. 474.77
CH ₃ (CH ₂ )11	OH CO(CH ₂ ) ₁₀ CH ₃	Synthesis -Obtained by reaction of with 2-dodecyl-4-methox presence of boron trifluori for 7 h, then at r.t. for 15 h	hyphenol in the de first at 70–80°
m.p. 53–54	° [2752].		

# 3-[(4-Octylphenylazo)-2,4,6-trihydroxyphenyl]-1-dodecanone



[872].

mol. wt. 524.74

-Also refer to: [2433].

USE: As water-repellent dye for wood [2433].

# 1-[3-[(2R,4S)-3,4-Dihydro-7-hydroxy-2-(4-hydroxyphenyl)-2H-1-benzopyran-4-yl]-2,4,6-trihydroxyphenyl]-1-dodecanone (+)

(Myristinin A)

[145904-69-4]	C ₃₃	$_{3}H_{40}O_{7}$	mol. wt. 548.68	
ОН ОН НО НО	OH CO(CH ₂ ) ₁₀ CH ₃ OH	(Myristicaceae) [27 -From <i>Knema ele</i> [848]. -From <i>Horsfielddia</i>	<i>cinnamomea</i> fruits [26]. <i>gans</i> (Myristicaceae) <i>amygdaline</i> [1215]. olid [2726]; colourless	
1				

¹H NMR [848, 2726], ¹³C NMR [848, 2726], IR [2726], UV [848, 2726], MS [848, 2726];  $(\alpha)_{D}^{28} = + 39.1^{\circ}$  (methanol) [2726],  $(\alpha)_{D}^{22} = + 45^{\circ}$  (methanol) [848].

BIOLOGICAL ACTIVITY: Selective COX-2 inhibitor [2726]; Antifungal agent [2726]; Inhibit DNA Polymerase β and Cleave DNA [848]; Cytotoxicity [848].

### Pentaacetate

C43H50O12

mol. wt. 758.86

mol. wt. 548.68

-Obtained by reaction of acetic anhydride with Myristinin A in the presence of pyridine under argon at  $25^{\circ}$  for 1.5 h (86 %) [848].

C33H40O7

oily product [848]; ¹H NMR [848], MS [848].

# Mvristinin A (+)

[888489-66-5]

Isolation from natural sources

-From trunk wood of Knema elegans (Warb.) (Myristicaceae) [848]. -From fruits of Myristica cinnamomea [2726].

brown amorphous solid [2726]; ¹H NMR [848, 2726], ¹³C NMR [848, 2726], IR [2726], UV [848, 2726], MS [848]; Circular dichroism [848];  $(\alpha)_{\rm D}^{22} = +45^{\circ}$  (methanol) [848];  $(\alpha)_{\rm D}^{28} = -280^{\circ}$  (methanol) [2726].

BIOLOGICAL ACTIVITY: Cytotoxicity [848, 2726]; Genotoxicity [848]; Antifungal [2726]; Inhibition of enzyme activity [848]; Antiinflammatory [2726].

# (2R,3R,4R)-2-(4-Hydroxyphenyl)-7-hydroxy-4-(2,4,6-trihydroxy-3-(dodecanoyl)-phenyl)chromane

(Myristinin A)

$$C_{33}H_{40}O_7$$

mol. wt. 548.68

Isolation from natural sources

-From fruits of Myristica cinnamomea [2726]. -Also refer to: [1974, 1975].

¹H NMR [1974, 1975, 2726]. ¹³C NMR [1974, 1975, 2726], IR [2726], UV [2726];  $(\alpha)_{D}^{23} = +57.8^{\circ}$  (methanol) [1975],  $(\alpha)_{D}^{28} = +39.1^{\circ}$  (methanol) [2726].

BIOLOGICAL ACTIVITY: Examination of DNA [1975]; Inhibition of enzyme activity [1975]; Cytotoxicity [1975, 2726]; Antiinflammatory [2726]; Antifungal [2726].

Myristinin B and C (a mixture of these)

1-[(3R)-3-[(2R,4R)-3,4-Dihydro-7-hydroxy-2-(4-hydroxyphenyl)-2H-1-benzopyran-4-yl]-2,4,6-trihydroxyphenyl]-1-dodecanone (Myristinin B)

[457930-47-1]	$C_{33}H_{40}O_7$	mol. wt. 548.68
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# 1-[(3R)-3-[(2S,4S)-3,4-Dihydro-7-hydroxy-2-(4-hydroxyphenyl)-2H-1-benzopyran-4-yl]-2,4,6-trihydroxyphenyl]-1-dodecanone (Myristinin C)

[457926-60-2]

C33H40O7

mol. wt. 548.68

Isolation from natural sources

-From Myristica cinnamomea fruits (Myristicaceae) [2726].

brown amorphous solid [2726]; ¹H NMR [2726], ¹³C NMR [2726], IR [2726], UV [2726], MS [2726];  $(\alpha)_{\rm D}^{28} = -280^{\circ}$  (methanol) [2726].

BIOLOGICAL ACTIVITY: Selective COX-2 inhibitors [2726]; Antifungal agents [2726].

#### 1,1'-(4,4'-Dihydroxy[1,1'-biphenyl]-3,3'-diyl)bis-1-dodecanone

*in the presence of sodium chloride at 140°. The reaction is carried out by adding the ester to the melt, rapidly increasing the temperature to  $200^{\circ}$  and maintaining it there for 2 min before quick cooling [2091];

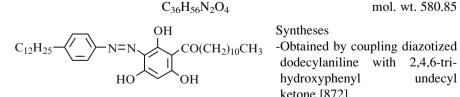
*in refluxing chlorobenzene for 24 h (85 %) [2377].

**N.B.**: The formation of ketonic products by Fries rearrangement of 4,4'-biphenyl didodecanoate with aluminium chloride (1.1 equiv.) according to [2955] could not be demonstrated. Changing the temperature and time used for the reaction also failed to give satisfactory results [2244]. Actually, the quantity of aluminium chloride used is very insufficient.

m.p. 87–88° [2377]; IR [2377].

#### 3-[(4-Dodecylphenylazo)-2,4,6-trihydroxyphenyl]-1-dodecanone

3'-(p-Dodecylphenylazo)-2',4',6'-trihydroxydodecanophenone



hydroxyphenyl undecyl ketone [872]. -Also refer to: [2433].

mol. wt. 580.85

USE: As water-repellent dye for wood [2433].

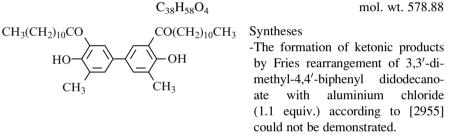
#### 1,1'-(5-Benzoyl-2,4-dihydroxy-1,3-phenylene)bis-1-dodecanone

[15041-68-6] 
$$C_{37}H_{54}O_5$$
 mol. wt. 578.83  
OH  
 $CH_3(CH_2)_{10}CO$   $CO(CH_2)_{10}CH_3$   $CO(CH_2)_{10}CH_3$   $Obtained by reaction of lauric acid with 2,4-dihydroxybenzophenone in the pre-
sence of para-toluenesulfonic acid in refluxing xylene for 20 h [134].
-Also refer to: [133].$ 

m.p. 59-61° [2252], 58-61° [134].

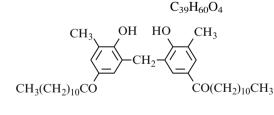
USE: For stabilizing polyolefins and vinyl halide resins against UV-light and sunlight [134]; Stabilization of vinyl halide resins and polyolefins against photodegradation [133].

# 1,1'-(4,4'-Dihydroxy-5,5'-dimethyl[1,1'-biphenyl]-3,3'-diyl)bis-1-dodecanone



Changing the temperature and time used for the reaction also failed to give satisfactory results [2244]. Actually, the quantity of aluminium chloride used is very insufficient.

#### 1,1'-[Methylenebis(2-hydroxy-3-methyl-5,1-phenylene)]bis-1-dodecanone



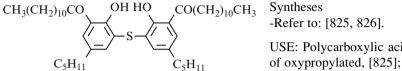
mol. wt. 592.90

Syntheses -Obtained by reaction of dodecanoyl chloride with bis(2-hydroxy-3-methylphenyl) methane according to the method described previously [2871], (46 %) [119].

m.p. 95.6–96.8° [119]; ¹H NMR [119], IR [119].

# 1,1'-[Thiobis(2-hydroxy-5-pentyl-3,1-phenylene)]bis-1-dodecanone

3',3"'-Thiobis[2'-hydroxy-5'-pentyldodecanophenone



-Refer to: [825, 826]. USE: Polycarboxylic acid esters

Polycarboxylic acid esters of oxypropylated, in breaking petroleum emulsions, [826].

#### Aromatic Hydroxyketones Derived from Various 2 **Bromododecanoic Acids**

#### 2.1 Unsubstituted Hydroxyketones

#### 2-Bromo-1-(4-hydroxyphenyl)-1-dodecanone

	$C_{18}H_{27}BrO_2$	mol. wt. 355.31
OH	Syntheses -Refer to: [2252, 2253]. <b>Methyl ether</b> [63424-84-0]	
Br CO-CH-(CH ₂ ) ₉ CH ₃	$C_{19}H_{29}BrO_2$	mol. wt. 369.34

-Obtained by reaction of bromine with 4-methoxydodecanophenone, *in nitrobenzene [2253], (70 %) [2252]; *in carbon tetrachloride at r.t. [378].

a 11 b o

m.p. 75–75.5° [378], 72–73° [574, 2252]; ¹H NMR [2252], IR [2252].

# 2-Bromo-1-(3,4-dihydroxyphenyl)-1-dodecanone

	$C_{18}H_{27}BrO_3$	mol. wt. 371.31
ОН	Synthesis	
ОН	-Refer to: [2252].	
Í Í	Dimethyl ether [63828-97-7	7]
Br	$C_{20}H_{31}BrO_3$	mol. wt. 399.37
CO-CH-(CH ₂ ) ₉ CH ₃		

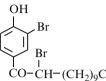
-Obtained by reaction of bromine with 3,4-dimethoxydodecanophenone in nitrobenzene (65 %) [2252]. -Also refer to: [2254].

m.p. 55° [2252]; ¹H NMR [2252], IR [2252].

#### 2.2 Substituted Hydroxyketones

# 2-Bromo-1-(3-bromo-4-hydroxyphenyl)-1-dodecanone

C₁₈H₂₆Br₂O₂ mol. wt. 434.21

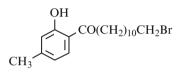


**Synthesis** -Obtained by reaction of bromine with 4-hydroxydodecanophenone in carbon tetrachloride at r.t. [378]. m.p. 77–77.5° [378].

#### CO-CH-(CH₂)_oCH₂

#### 12-Bromo-1-(2-hydroxy-4-methylphenyl)-1-dodecanone

 $C_{19}H_{29}BrO_2$ mol. wt. 369.34



# **Synthesis** -Obtained by reaction of 12-bromododecanoic acid with m-cresol in the presence of a mixture of graphite and methanesulfonic acid at 120° for 2 h (77 %) [2834].

¹H NMR [2834], ¹³C NMR [2834].

#### Aromatic Hydroxyketone Derived from 3 12-Oxododecanoic Acid

# 12-(4-Hydroxyphenyl)-12-oxo-1-dodecanoic acid

C18H26O4 mol. wt. 306.40 [23293-67-6]  $CO(CH_2)_{10}CO_2H$ Synthesis -Refer to: [1539]. m.p. 92–94° [1539]; IR [1539].

# Chapter 11 Tridecanones

# 1 Aromatic Hydroxyketones Derived from Tridecanoic Acids

# 1.1 Unsubstituted Hydroxyketones

# 1-(3-Hydroxyphenyl)-1-tridecanone

[857165-80-1] C19H30O2 mol. wt. 290.45 OH Syntheses -Obtained by treatment of its methyl ether with aluminium chloride in benzene by heating on a water bath for 2 h  $CO(CH_2)_{11}CH_3$ (80 %) [966]. -Also refer to: [967]. b.p.25 165° [966, 967];  $n_{\rm D}^{36} = 1.556$  [967]. **2,4-Dinitrophenylhydrazone**  $C_{25}H_{34}N_4O_5$ mol. wt. 470.57 m.p. 100° [967]. Methyl ether [857165-59-4] C₂₀H₃₂O₂ mol. wt. 304.47 -Obtained by condensation of dodecylmagnesium bromide with m-methoxybenzamide

-Obtained by condensation of dodecy/magnesium bromide with m-methoxybenzamide in ethyl ether. The reaction was carried out at 60° for 48 h under hydrogen atmosphere (60–70 %) [966]. -Also refer to: [967].

#### 2,4-Dinitrophenylhydrazone of the methyl ether C₂₆H₃₆N₄O₅ mol. wt. 484.60

m.p. 79° [967].

#### 1-(4-Hydroxyphenyl)-1-tridecanone

[1006710-00-4]	$C_{19}H_{30}O_2$	mol. wt. 290.45
OH	Synthesis -Obtained by reaction of tridecanoyl phenol [3457].	chloride with
$\int CO(CH_2)_{11}CH_3$	USE: Application of polyethylene glycol a metal acetate for oil recovery [3457].	alkylphenyl ether

Methyl ether 
$$[55469-24-4]$$
  $C_{20}H_{32}O_2$  mol. wt. 304.47

-Obtained by reaction of tridecanoyl chloride with anisole in the presence of graphite in refluxing 1,2-dichloroethane for 8 h (63 %) [1723].

-Also obtained by coupling 1-tridecyne with 4-bromoanisole in the presence of silicon tetrachloride and sodium iodide (Heck reaction) (30 %) [480].

-Also refer to: [152, 481, 1202, 1963].

m.p. 59° [1963]; ¹H NMR [480], IR [480], MS [480].

USE: Liq. crystal compns. contg., for display devices [152].

#### 2-Chloroethyl ether

 $C_{21}H_{33}ClO_2$ 

mol. wt. 352.94

-Obtained by reaction of tridecanoyl chloride with 2-chloroethoxybenzene in the presence of aluminium chloride in carbon disulfide for 4 h at  $50^{\circ}$  (60 %) [476].

m.p. 77–78° [476].

#### **N-Dimethylaminoethyl ether** $C_{23}H_{39}NO_2$ mol. wt. 361.57

-Obtained by reaction of 4-(2-chloroethoxy)tridecanophenone with dimethylamine in a sealed tube for 2 h at 150° [476].

free base (oil): b.p._{0.01} 204° [476]; m.p. 55–56° [476]. hydrochloride: (92.5 %) [476].

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

 $[862666-39-5] C_{19}H_{30}O_3 mol. wt. 306.45$ 

Synthesis

-Obtained by deprotection of its dimethyl ether with boron tribromide in methylene chloride at  $0^{\circ}$ . Then, the mixture was stirred overnight at r.t. (79 %) [82].

brown solid [82]; m.p. 60° [82]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82]. USE: Reactant for preparation of lithium gallium/titanium ketone complexes [82].

**Dimethyl ether** [266310-08-1] C₂₁H₃₄O₃ mol. wt. 334.50

-Obtained by treatment of 1-(2,3-dimethoxyphenyl)-1-tridecanol in acetone with potassium dichromate in dilute sulfuric acid (Jones reagent) at r.t. for 3 h (53 %) [82].

colourless oil [82]; ¹H NMR [82], ¹³C NMR [82], IR [82], MS [82].

 $C_{10}H_{20}O_2$ 

#### 1-(2,4-Dihydroxyphenyl)-1-tridecanone

	019113003	mon wit 200.12
HO CO(CH ₂ ) ₁₁ CH ₃	Synthesis -Obtained by reaction of tridecand cinol in the presence of zinc 125 and 135° [893]. b.p. ₁₁ 265–270° [893].	

C₂₁H₃₄O₃

#### **Dimethyl ether**

-To a stirred suspension of sodium hydride in dry, acid free dimethylacetamide at  $10^{\circ}$  under nitrogen was added 3,5-dimethoxyphenacylpyridinium perchlorate in dimethylacetamide. The mixture was shaken for 15 min and undecyl iodide added. After keeping overnight at r.t., the mixture was heated for 2 h at 90° and cooled at r.t. Zinc dust and acetic acid were added, the mixture stirred at r.t. for 4 h (34 %) [2603].

-Also refer to: [2627].

needles [2603]; m.p. 53° [2627], 52.5–53° [2603]; IR [2603].

#### 1-(3,4-Dihydroxyphenyl)-1-tridecanone

	$C_{19}H_{30}$	O ₃	mol. wt. 306.45
OH OH CO(CH ₂ ) ₁₁ CH ₃	Synthesis -Refer to: [256]. <b>Dimethyl ether</b> $C_{21}H_{34}O_3$	[930585-36-7]	mol. wt. 334.50

-Preparation by treatment of 1,2-dimethoxy-4-tridecylbenzene,
*with chromium trioxide in acetic acid, at r.t. for 24 h and at 40° for 16 h (57 %) [256];
*with cerium ammonium nitrate in aqueous acetonitrile for 5 h at 25° (55 %) [256].
-Also refer to: [1960, 1963].

yellow crystals; m.p. 59.5–60° [1960, 1963], 42.2–43.8° [256]; ¹H NMR [256], ¹³C NMR [256], IR [256], MS [256].

mol. wt. 306.45

mol. wt. 334.50

mol. wt. 406.65

**Dibutyl ether** [109175-98-6]

-Refer to: [1590].

USE: Image stabilizer, photog. film contg. [1590].

#### 1-(3,5-Dihydroxyphenyl)-1-tridecanone

	$C_{19}H_{30}O_3$	mol. wt. 306.45
ОН	Syntheses	
$\checkmark$	-Refer to: [3340, 3341]	
	Dimethyl ether [5259	9-08-5]
HO CO(CH ₂ ) ₁₁ CH ₃	$C_{21}H_{34}O_3$	mol. wt. 334.50

C26H46O3

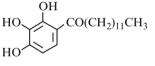
-Preparation by reaction of 3,5-dimethoxybenzoyl chloride with n-dodecylmagnesium bromide in ether and toluene mixture at  $-60^{\circ}$  for 2.5 h in the presence of ferric chloride under nitrogen atmosphere (51 %) [2627]. obtained by dodecylmagnesium -Also reaction of bromide with 3,5-dimethoxybenzamide in ether [2876, 2948]. -Refer to: [2603, 3340, 3341].

colourless plates [2627]; m.p. 53° [2627], 52.3–53° [2603];

¹H NMR [2627], IR [2627], UV [2627].

# 1-(2,3,4-Trihydroxyphenyl)-1-tridecanone

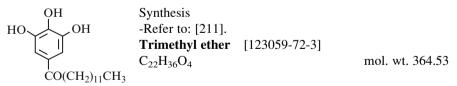
 $C_{19}H_{30}O_4$ mol. wt. 322.44



Synthesis CO(CH₂)₁₁CH₃ -Obtained by reaction of tridecanoic acid with pyrogallol in the presence of zinc chloride at 140-145° for 4 h [506]. m.p. 84–85° [506].

# 1-(3,4,5-Trihydroxyphenyl)-1-tridecanone

$$C_{19}H_{30}O_4$$
 mol. wt. 322.44

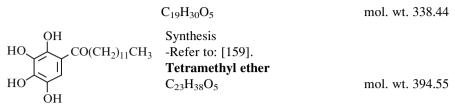


-Obtained by reaction of dodecylmagnesium bromide with 3.4.5-trimethoxybenzoyl chloride in the presence of zinc chloride in benzene at 20° for 12 h (81 %) [211].

-Also refer to: [159].

m.p. 61–62° [159], 57–59° [211]; ¹H NMR [211].

#### 1-(2,3,4,5-Tetrahydroxyphenyl)-1-tridecanone



m.p. 61-62° [159].

#### 13-Hydroxy-3-methyl-1-(2,5-dihydroxyphenyl)-1-tridecanone

[1254222-61-1] 
$$C_{20}H_{32}O_4$$
 mol. wt. 336.47  
OH CH₃ Synthesis  
COCH₂-CH-(CH₂)₁₀OH -Refer to: [720].

#### 4,8,12-Trimethyl-1-(3,4,5-trihydroxyphenyl)-1-tridecanone

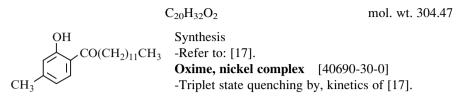
# 1.2 Substituted Hydroxyketones

# 1-(3,5-Dichloro-4-hydroxyphenyl)-1-tridecanone

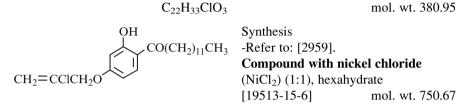
$$\begin{array}{cccc} [110581-80-1] & C_{19}H_{26}Cl_2O_2 & \text{mol. wt. 357.32} \\ & OH & Synthesis \\ Cl & Cl & -Refer to: [1463]. \\ & USE: Photog. coloration-promoting agent [1463]. \\ & CO(CH_2)_{11}CH_3 \end{array}$$

mol. wt. 360.58

#### 1-(2-Hydroxy-4-methylphenyl)-1-tridecanone



# 1-[4-(2-Chloroallyloxy)-2-hydroxyphenyl]-1-tridecanone



USE: As stabilizers (radiation) for propene polymers [2959].

#### 1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-tridecanone

0	$C_{23}H_{38}O_2$	mol. wt. 346.55
(CH ₃ ) ₂ CH CH ₃ CO(CH ₂ ) ₁₁ CH ₃	Synthesis -Obtained by treatment of 5-isopropyl-tridecanopher pyridinium chloride (2 (14 %) [2660]. b.p. ₁₁ 266–269° [2660];	none with boiling 205–215°) for 10 h

#### Methyl ether

m.p. 92-92.5° [1337].

-Preparation by reaction of tridecanoyl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (50 %) [2660].

 $C_{24}H_{40}O_{2}$ 

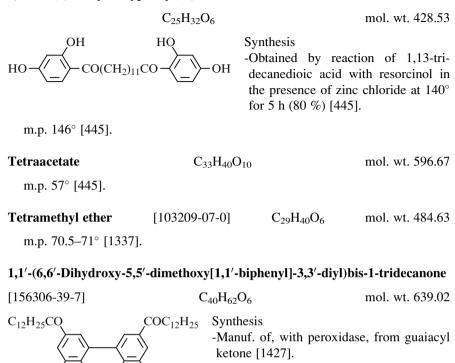
b.p.₂₁ 268–270° [2660]; m.p. 34° [2660].

#### 1,13-Bis(4-hydroxyphenyl)-1,13-tridecanedione

$$[114280-34-1] C_{25}H_{32}O_4 mol. wt. 396.53$$

$$HO \longrightarrow CO(CH_2)_{11}CO \longrightarrow OH Synthesis -Refer to: [1337]. m.p. 131-133° [1337].$$
Acetate [103161-22-4]  $C_{29}H_{36}O_6$  mol. wt. 480.60

#### 1,13-Bis(2,4-dihydroxyphenyl)-1,13-tridecanedione



BIOLOGICAL ACTIVITY: Antioxidant and fragrance-keeping effects [1427].

# 2 Aromatic Hydroxyketone Derived from 13-Oxotridecanoic Acid

OCH₃

#### 13-(2-Hydroxyphenyl)-13-oxo-1-tridecanoic acid

CH₃O

OHHC

[102166-29-0]	$C_{19}H_{28}O_4$	mol. wt. 320.43
OH CO(CH ₂ ) ₁₁ CO ₂ H	Synthesis -Refer to: [3122]. m.p. 102° [3122].	

# Chapter 12 Tetradecanones

# 1 Aromatic Hydroxyketones Derived from Tetradecanoic Acids

# 1.1 Unsubstituted Hydroxyketones

# 1-(2-Hydroxyphenyl)-1-tetradecanone

C₂₀H₃₂O₂ mol. wt. 304.47

OH  $CO(CH_2)_{12}CH_3$  Syntheses -Obtained by reaction of myristoyl chloride with phenol in the presence of aluminium chloride, *in tetrachlorethane few hours at 55° (32 %) [2548];

*in nitrobenzene for 3 h at  $70^{\circ}$  (21 %) [2549];

*in carbon disulfide for 5.5 h at  $47^{\circ}$  (36 %) [2549].

-Also obtained by Fries rearrangement of phenyl myristate with aluminium chloride in tetrachlorethane for 10 h at 70° (35 %) [2550] or in tetrachloroethane [3169]. -Also obtained by reaction of myristic acid with phenol in the presence of zinc chloride (Nencki reaction) (75 %) [2398].

m.p. 55° [2398], 53.5° [3169], 52–55° [2548].

2,4-Dinitrophenylhydrazone C ₂₆	$H_{36}N_4O_5$	mol. wt. 484.60
--------------------------------------------	----------------	-----------------

m.p. 92.5° [3169], 92–92.5° [2548].

# Methyl ether

 $C_{21}H_{34}O_2$ 

mol. wt. 318.50

-Obtained by reaction of dimethyl sulfate with 1-(2-hydroxyphenyl)-1-tetradecanone in the presence of potassium carbonate in boiling acetone for 6 h [2398].

b.p.₆₆ 180–182° [2398].

**2,3-Epoxypropyl ether** [18110-31-1] C₂₃H₃₆O₃ mol. wt. 360.54

-Obtained by reaction of epichlorohydrin (0.1 mol) with o-tetradecanoylphenol (0.1 mol) in the presence of 30 % sodium hydroxide between 40 and  $50^{\circ}$  (63 %) [2669].

b.p._{0.4} 230-240° [2669].

# 1-(4-Hydroxyphenyl)-1-tetradecanone

[2589-75-5]  $C_{20}H_{32}O_2$  mol. wt. 304.47 OH Syntheses

-Obtained by reaction of myristoyl chloride with phenol in the presence of aluminium chloride,

*in tetrachlorethane few hours at  $55^{\circ}$  (37 %) [2548];

 $CO(CH_2)_{12}CH_3$  *in nitrobenzene for 3 h at 70° (73 %) [2549];

*in carbon disulfide for 5.5 h at  $47^{\circ}$  (52 %) [2549] or first at r.t., then heated to boiling for 5 h (poor yield) [1526].

-Also obtained by Fries rearrangement of phenyl myristate with aluminium chloride in tetrachlorethane [3169], for 10 h at  $70^{\circ}$  (43 %) [2550].

-Also obtained by demethylation of the 4-myristoylanisole [2398].

-Also obtained by reaction of myristic acid with phenol in the presence of activated acid clay catalyst at 190° for 2 h [3377].

-Also refer to: [873, 1299, 2945, 3371].

m.p. 78-80° [2548, 3377], 77° [2398], 76-78° [3169], 74-74.5° [1526].

USE: Activator for peroxygen bleach in laundry detergent for mud-soiled clothing [3371]; Foaming improvement of N-acylamino alkanesulfonate detergents by, [2945]; Textile rot proofing by, [873].

2,4-Dinitrophenylhydrazone	$C_{26}H_{36}N_4O_5$	mol. wt. 484.60
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m.p. 142–143° [2548], 141.5° [3169].

-Obtained by Friedel-Crafts reaction of myristoyl chloride with anisole (90 %) [2398].

-Also refer to: [2016, 1963].

m.p. 68–68.5° [2016], 67° [1963], 63° [2398].

<b>2,4-Dinitrophenylhy</b> m.p. 103–104° [2		hyl ether	$C_{27}H_{38}N_4O_5$	mol. wt. 498.62
Oxime of the methy m.p. 66° [2398].	vl ether	C ₂₁ H ₃₅ N	O ₂	mol. wt. 333.51
Semicarbazone of t m.p. 71° [2398].	he methyl ether	C ₂₂ H	I ₃₇ N ₃ O ₂	mol. wt. 375.55
Ethyl ether	[859947-01-6]	C ₂₂	$H_{36}O_2$	mol. wt. 332.52
-Obtained by reaction of myristyl chloride with phenetole in the presence of aluminium chloride for 2 days at 60–70° (41 %) [1526]. colourless blocks [1526]; m.p. 66–67° [1526].				

-Obtained by reaction of tetradecanoyl chloride with 2-chloroethoxybenzene in the presence of aluminium chloride in carbon disulfide for 4 h at  $50^{\circ}$  (65 %) [476].

C22H35ClO2

m.p. 68–70° [476].

2-Chloroethyl ether

#### **N-Dimethylaminoethyl ether** $C_{24}H_{41}NO_2$ mol. wt. 375.60

-Obtained by reaction of 4-(2-chloroethoxy)tetradecanophenone with dimethylamine in a sealed tube for 2 h at  $150^{\circ}$  [476].

free base (oil): b.p._{0.001} 202° [476]; hydrochloride (65 %) [476]; m.p. 158° [476].

#### **2,3-Epoxypropyl ether** [18110-32-2] C₂₃H₃₆O₃ mol. wt. 360.54

-Obtained by reaction of epichlorohydrin (0.1 mol) with p-tetradecanoylphenol (0.1 mol) in the presence of 30 % sodium hydroxide between 40 and  $50^{\circ}$  (57 %) [2669].

m.p. 111° [2669].

#### **Myristate** [119531-08-7] C₃₄H₅₈O₃ mol. wt. 514.83

-Obtained by reaction of myristic acid with phenol in the presence of activated acid clay catalyst at  $190^{\circ}$  for 2 h [3377].

m.p. 83-83.5° [3377].

mol. wt. 366.97

#### 1-(2,4-Dihydroxyphenyl)-1-tetradecanone

[143286-84-4]	$C_{20}H_{32}O_3$	mol. wt. 320.47		
HO CO(CH ₂ ) ₁₂ CH	Synthesis -Obtained by reaction of tetradecanoyl chloride wit resorcinol in the presence of aluminium chloride i 1,2-dichloroethane at 65° for 5 h [284].			
1-(2,5-Dihydroxyphenyl)-1-tetradecanone				
	СНО	mal wt 220.47		

	$C_{20}H_{32}O_3$	mol. wt. 320.47
OH CO(CH ₂ ) ₁₂ CH ₃ OH	hydroquinone	reaction of tetradecanoyl chloride with dimethyl ether in the presence of alumin- [156] in carbon disulfide [159]. [156, 159].

-Refer to: [285].

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

Dimethyl ether	$C_{22}H_{36}O_{3}$	mol. wt. 348.52
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-Obtained by reaction of myristyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride in petroleum ether (quantitative yield) [1526].

plates [1526]; b.p._{0.5} 209° [714]; m.p. 51-52° [1526], 43° [714].

**Diethyl ether** 

Oxime

-Refer to: [714].

b.p._{0.3} 204° [714]; m.p. 44.5° [714].

**2,4-Dinitrophenylhydrazone of the diethyl ether**  $C_{30}H_{44}N_4O_6$  mol. wt. 556.70

 $C_{24}H_{40}O_3$ 

m.p. 78° [714].

# 1-(2,6-Dihydroxyphenyl)-1-tetradecanone

[113201-68-6] C₂₀H₃₂O₃ mol. wt. 320.47

OH OH

Syntheses -Obtaining by aromatization of 2-chloro-2-myristoyl-1,3-cyclohexanedione (70 %) [1816] according to the method [75, 1817].

-Also refer to: [2336].

[140943-08-4] C₂₀H₃₃NO₃ mol. wt. 335.49

mol. wt. 376.58

Isolation from natural sources

-From the fruits of *Knema glauca* (Myristicaceae) [2562].
-From *Myristica dactyloides* (Myristicaceae) [719, 1786, 2336].
-Of the andromeda lace bug *Stephanitis rhododendrii* [2334].
-In the wood bark of *Knema austrosiamensis* W. J. J. O. DeWilde (Myristaceae) [1133] or in Ground wood of this same plant [1134].
-From the fruit rinds of *Myristica malabarica* (Myristicaceae) (0.39 %) [2425].
-From root bark of *Myristica ceylanica* [1307, 1309].
-From the stem bark of *Myristica dactyloides* (Myristicaceae) [1308].
-In plants of the family Myristicaceae [719, 1622].
-Also refer to: [715, 1306, 1504, 3438].
yellow amorphous powder [2562]; pale yellow needles [1786]; gum [1134]; m p. 93–94° [2336].

m.p. 93–94° [2336], 91–92° [1306, 1786], 91–91.5° [719];
¹H NMR [719, 1134, 1786, 2336, 2425, 2562],
¹³C NMR [719, 1786, 2562], IR [719, 1786, 2336, 2562], UV [719, 2562],
MS [719, 1134, 1786, 2336, 2425, 2562]; GC [2425].

BIOLOGICAL ACTIVITY: Antituberculosis activity against the microbe Mycobacterium tuberculosis [2562]; Antiviral activity against herpes simplex virus type 1 [2562]; Antimycobacterial and antimalarial activities [2562]; *Myristica dactyloides* is used in native medicine in Sri Lanka and its seeds are used as adulterants to *Myristica fragrans* (nutmeg) [1504]; Elastase of human leukocytes and sputum inhibition by, Brij 35 effect on [715]; Cytotoxicity [2562].

Diacetate [114226-22-1] C₂₄H₃₆O₅ mol. wt. 404.55

-Obtained by reaction of acetic anhydride with 1-(2,6-dihydroxyphenyl)-1-tetradecanone in the presence of pyridine at  $70^{\circ}$  for 12 h [1786].

¹H NMR [1786], IR [1786], MS [1786].

**Dimethyl ether** [114226-23-2] C₂₂H₃₆O₃ mol. wt. 348.52

Syntheses

-Obtained by reaction of diazomethane with 1-(2,6-dihydroxyphenyl)-1-tetradecanone in ethyl ether [1786].

-Also obtained by methylation of 1-(2-hydroxy-6-methoxyphenyl)-1-tetradecanone [1786].

Isolation from natural sources

-From root bark of Myristica ceylanica [1307, 1309].

¹H NMR [1786], IR [1786], MS [1786].

# 1-(3,4-Dihydroxyphenyl)-1-tetradecanone

(4-Tetradecanoylcatechol)

ŌН	Syntheses
ОН	-Obtained by treatment of a pyrocatechol and tetradecanoic acid
	mixture with zinc chloride at $135-140^{\circ}$ for 2 h (20 %) [1283].
Ý	-Also refer to: [283, 1961].
$CO(CH_2)_{12}CH_3$	m.p. 103° [1961], 98–99° [1283].

Dimethyl ether	[871901-16-5]	$C_{22}H_{36}O_{3}$	mol. wt. 348.52
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-Obtained by reaction of myristyl chloride with veratrole in the presence of aluminium chloride,

*without solvent at 70° (23 %) [1960];

*in petroleum ether at  $30-40^{\circ}$  for 21-22 h (quantitative yield) [1526].

colourless needles [1526]; b.p. $_{0.3-0.5}$  200–220° [1960]; m.p. 74–75° [1960], 73–75° [1526].

# **Oxime of the dimethyl ether** C₂₂H₃₇NO₃ mol. wt. 363.54

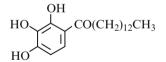
-Obtained by treatment of 1-(3,4-dimethoxyphenyl)-1-tetradecanone with hydroxylamine hydrochloride in the presence of potassium acetate in boiling ethanol for 4 h (83 %) [1960].

m.p. 54–55° [1960]

# 1-[2,3,4-Trihydroxyphenyl]-1-tetradecanone

(4-Tetradecanoylpyrogallol)

$C_{20}H_{32}O_4$	mol. wt.	336.47
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Synthesis -Obtained by reaction of tetradecanoic acid with pyrogallol in the presence of zinc chloride (Nencki reaction) at  $135-140^{\circ}$  for 2 h (25 %) [1283].

m.p. 82–84° [1283].

# 1-[2,4,6-Trihydroxyphenyl]-1-tetradecanone

(4-Tetradecanoylpyrogallol)

[147862-99-5]

 $C_{20}H_{32}O_4$ 

mol. wt. 336.47

HO OH CO(CH₂)₁₂CH₃

CO(CH₂)₁₂CH₃ -Refer to: [1133]. Isolation from natural sources -In the wood of *Knema austrosiamensis* W. J. J. O. DeWilde (Myristaceae) [1133] or in Ground wood of this same plant [1134].

waxy solid [1134]; ¹H NMR [1134], MS [1134].

# 1-[3,4,5-Trihydroxyphenyl]-1-tetradecanone

$$\begin{array}{c} C_{20}H_{32}O_4 & \text{mol. wt. 336.47} \\ \text{OH} & \text{Synthesis} \\ \text{HO} & OH & -\text{Refer to: [159].} \\ & \mathbf{Trimethyl \ ether} \\ C_{0}(CH_2)_{12}CH_3 & \text{mol. wt. 378.55} \end{array}$$

-Obtained by treatment of ethyl  $\alpha$ -(trimethoxybenzoyl)myristate (m.p. 54°) with boiling 1 % ethanolic potassium hydroxide [159].

m.p. 69° [159].

# 14-Hydroxy-1-(2,5-dihydroxyphenyl)-1-tetradecanone

[1254222-90-6] 
$$C_{20}H_{32}O_4$$
 mol. wt. 336.47  
OH Synthesis  
-Refer to: [720].  
white crystals [720]; ¹H NMR [720].

#### 1-(2,3,4,5-Tetrahydroxyphenyl)-1-tetradecanone

	$C_{20}H_{32}O_5$	mol. wt. 352.47
HO $\downarrow$ CO(CH ₂ ) ₁₂ CH ₃	Synthesis -Refer to: [159] (Japanese paper)	
НО ОН	Tetramethyl ether $C_{24}H_{40}O_5$	mol. wt. 408.57
60% [150]		

m.p. 69° [159].

# 14-Hydroxy-3-methyl-1-(2,5-dihydroxyphenyl)-1-tetradecanone

$$[1254222-62-2] C_{21}H_{34}O_4$$
mol. wt. 350.50  
OH  $CH_3$  Synthesis  
-Refer to: [720].

# 1.2 Substituted Hydroxyketones

# 1-(4,6-Dichloro-2-hydroxy-3-nitrophenyl)-1-tetradecanone

[81515-09-5]	$C_{20}H_{30}Cl_2NO_4$	mol. wt. 419.43
NO ₂ Cl	Synthesis -Refer to: [2585].	

# 1-(2,4-Dichloro-6-hydroxyphenyl)-1-tetradecanone

# 1-(5-Bromo-2-hydroxyphenyl)-1-tetradecanone

	$C_{20}H_{31}BrO_2$	mol. wt. 383.37
ОН	Synthesis	
CO(CH ₂ ) ₁₂ CH ₃	-Refer to: [468].	
	Methyl ether	
Ý	$C_{21}H_{33}BrO_2$	mol. wt. 397.39
Br		

-Obtained by reaction of myristoyl chloride with 4-bromoanisole in the presence of aluminium chloride in nitrobenzene first at  $20-25^{\circ}$  and the mixture kept 2 days at r.t. (36 %) [468].

m.p. 78.5° [468].

# 1-(4-Chloro-2-hydroxyphenyl)-1-tetradecanone

	$C_{20}H_{31}ClO_2$			mol. wt. 33	8.92
CI CO(CH ₂ ) ₁₂ CH ₃	3-chloropheny *without solve	yl myri ent at 1	state with 30° for 2	rearrangement a aluminium chlorid h (45 %) [2802]; (75 %) [2802].	of de,

 $b.p._{32} 250^{\circ}$  [2802].

2,4-Dinitrophenylhydrazone	C ₂₆ H ₃₅ ClN ₄ O ₅	mol. wt. 519.04
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m.p. 114° [2802].

# Methyl ether $C_{21}H_{33}ClO_2$ mol. wt. 352.94

-Obtained by methylation of the above ketone in the usual way (75 %) [2802].

b.p.₂₇ 127° [2802].

# 1-(5-Chloro-2-hydroxyphenyl)-1-tetradecanone

[98813-31-1]	$C_{20}H_{31}ClO_2$	mol. wt. 338.92
CO(CH ₂ ) ₁₂ CH ₃		tetradecanoyl chloride with sence of aluminium chloride

b.p.₁ 182–185° [2680]; m.p. 79–80° [2680].

# 2-Hydroxy-5-tetradecanoylbenzoic acid

 $\begin{array}{cccc} [78418-04-9] & C_{21}H_{32}O_4 & \mbox{mol. wt. 348.48} \\ & OH & Synthesis & \\ & -Obtained by saponification of the methyl ester (90 \%) [689]. \\ & \mbox{m.p. 117-118}^{\circ} [689]. \end{array}$ 

Methyl ester	[78417-98-8]	$C_{22}H_{34}O_4$	mol. wt. 362.51
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-Obtained by reaction of tetradecanoyl chloride with methyl salicylate in the presence of aluminium chloride in carbon disulfide first at  $5-10^{\circ}$ , then at r.t. for 12 h (70 %) [689].

m.p. 69–70° [689].

# 1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-tetradecanone

C	$_{21}H_{33}ClO_2$		mol. wt. 352.94
CH ₃ CH ₃ CH ₃ Cl	•	eaction of myris lphenol in the pro- 2–3 h between	esence of boron

#### 1-(2-Hydroxy-5-methylphenyl)-1-tetradecanone

[36946-08-4] C₂₁H₃₄O₂ mol. wt. 318.50 OH **Synthesis**  $CO(CH_2)_{12}CH_3$  -Refer to: [1968]. Dielectric constant [1968].

#### 1-(2,4-Dihydroxy-5-methylphenyl)-1-tetradecanone

$$[95102-16-2] C_{21}H_{34}O_3 \qquad \text{mol. wt. 334.50}$$

$$OH \qquad Syntheses \\ -Refer to: [1595, 2704].$$

$$USE: Colour developer, for thermal recording materials [1595].$$

#### 1-(2-Hydroxy-4-methoxyphenyl)-1-tetradecanone

[143287-00-7] C21H34O3 mol. wt. 334.50 OII

H₃ -Obtained by reaction of methyl bromide with 1-(2,4-dihydroxyphenyl)-1-tetradecanone in the presence of potassium carbonate in refluxing acetone for 20 h [284].

m.p. 58-60° [284].

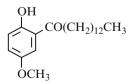
Oxime [143286-68-4] C₂₁H₃₅NO₃ mol. wt. 349.51

-Obtained by reaction of hydroxylamine hydrochloride with 1-(2-hydroxy-4-methoxyphenyl)-1-tetradecanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 83-85° [284].

#### 1-(2-Hydroxy-5-methoxyphenyl)-1-tetradecanone

$C_{21}H_{34}O_3$	mol. wt. 334.50
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Syntheses -Obtained by reaction of tetradecanoyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride [156] in carbon disulfide [159]. m.p. 51–52° [156, 159].

Synthesis

Oxime [140943-16-4]  $C_{21}H_{35}NO_{3}$ mol. wt. 349.51

-Refer to: [285].

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

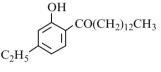
#### 1-(2-Hydroxy-6-methoxyphenyl)-1-tetradecanone

$$\begin{array}{cccc} [114226-24-3] & C_{21}H_{34}O_3 & \text{mol. wt. } 334.50 \\ \\ OH & Isolation from natural sources \\ -From Myristica dactyloides (Myristicaceae) [1308, 1786]. \\ OCH_3 & -From root bark of Myristica ceylanica [1307, 1309]. \end{array}$$

yellow solid [1786]; m.p. 51–52° [1786]; ¹H NMR [1786], ¹³C NMR [1786], IR [1786], MS [1786].

# 1-(4-Ethyl-2-hydroxyphenyl)-1-tetradecanone

[105701-24-4]	$C_{22}H_{36}O_2$	mol. wt. 332.52



Syntheses CO(CH₂)₁₂CH₃ -Obtained by Fries rearrangement of 3-ethylphenyl n-myristate (1 equiv.), *in the presence of aluminium chloride (1.3 equiv.) in nitrobenzene at 25° for 6 h (75 %) [2801];

*in the presence of aluminium chloride (2.8 M),

mol. wt. 512.64

mol. wt. 346.55

first in refluxing carbon disulfide for 2 h, then for 2 h at 130° after solvent elimination (73 %) [2801].

 $C_{28}H_{40}N_4O_5$ 

-Also refer to: [2346].

b.p.29 259° [2801].

#### 2,4-Dinitrophenylhydrazone

m.p. 48° [2801].

# Methyl ether

-Obtained by reaction of dimethyl sulfate with 1-(4-ethyl-2-hydroxyphenyl)-1-tetradecanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating on the water bath for 5 h (86 %) [2801].

C23H38O2

m.p. 54° [2801].

mol. wt. 512.64

#### 1-(5-Ethyl-2-hydroxyphenyl)-1-tetradecanone

$$\begin{array}{ccc} C_{22}H_{36}O_2 & \mbox{mol. wt. } 332.52 \\ \hline OH & Synthesis \\ -Obtained by Fries rearrangement of 4-ethylphenyl myristate with aluminium chloride at 100° for 2 h (65 %) [2800]. \\ \hline C_2H_5 & \mbox{b.p.}_{17} \ 210^\circ \ [2800]. \end{array}$$

 $C_{28}H_{40}N_4O_5$ 

2,4-Dinitrophenylhydrazone

m.p. 66° [2800].

**Oxime** [105701-23-3] C₂₂H₃₇NO₂ mol. wt. 347.54

-Refer to: [2346].

#### 1-(2-Hydroxy-4,6-dimethylphenyl)-1-tetradecanone

[874507-02-5]	$C_{22}H_{36}O_2$	mol. wt. 332.52
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ŌН	Syntheses				
CO(CH ₂ ) ₁₂ CH ₃	-Obtained	by	Fries	rearrangement	of
	3,5-dimethy	lpheny	l n-myris	tate (1 equiv.),	
CH ₃ CH ₃	*in the prese	ence of	aluminiu	m chloride (1.3 equ	iv.)
	in nitrobenzene at 25° for 6 h (56 %) [2801];				
	*in the prese	ence of	aluminiu	m chloride (2.8 M)	,

first in refluxing carbon disulfide for 2 h, then for 2 h at  $130^{\circ}$  after solvent elimination (50 %) [2801].

b.p.₂ 220° [2801].

4-Nitrophenylhydrazone

m.p. 195° [2801].

# Methyl ether

 $C_{23}H_{38}O_2$ 

C₂₈H₄₁N₃O₃

mol. wt. 346.55

mol. wt. 467.65

-Obtained by reaction of dimethyl sulfate with 1-(2-hydroxy-4,6-dimethylphenyl)-1-tetradecanone in the presence of 10 % sodium hydroxide, first at r.t., then by heating on the water bath for 5 h (74 %) [2801].

b.p.45 230° [2801].

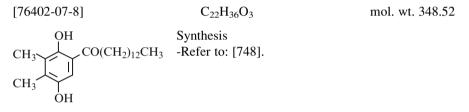
#### 1-(4-Hydroxy-3,5-dimethylphenyl)-1-tetradecanone

$$\begin{array}{cccc} C_{22}H_{36}O_2 & \text{mol. wt. } 332.52 \\ OH & Synthesis \\ -Refer to: [718]. \\ Methyl ether & [29665-49-4] \\ C_{23}H_{38}O_2 & \text{mol. wt. } 346.55 \end{array}$$

-Obtained by Fries rearrangement of 2,6-dimethylphenyl tetradecanoate in the presence of aluminium chloride in nitrobenzene [718], according to [380].

m.p. 40-41° [718].

#### 1-(2,5-Dihydroxy-3,4-dimethylphenyl)-1-tetradecanone



#### 1-(3,5-Dimethoxyphenyl)-1-tetradecanone

 $\begin{array}{c} C_{22}H_{36}O_{3} \\ CH_{3}O \underbrace{CO(CH_{2})_{12}CH_{3}}_{OCH_{3}} \\ Synthesis \\ -Refer to: [497]. \\ m.p. \ 60-61^{\circ} \ [497]. \end{array}$ 

#### 1-(5-Ethoxy-2-hydroxyphenyl)-1-tetradecanone

[140943-36-8]	$C_{22}H_{36}O_{3}$	mol. wt. 348.52
OH CO(CH ₂ ) ₁₂ CH ₃	Synthesis -Refer to: [285].	
	<b>Oxime</b> [140943-22-2] C ₂₂ H ₃₇ NO ₃	mol. wt. 363.54
OC ₂ H ₅		

-Refer to: [285].

BIOLOGICAL ACTIVITY: As lipoxygenase and cyclooxygenase inhibitor [285].

mol. wt. 560.77

mol. wt. 355.52

# 1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-tetradecanone

[134081-98-4]	$C_{22}H_{36}O_5$	mol. wt. 380.52
CH ₃ O OH CO(CH ₂ ) ₁₂ CH ₃ OH OCH ₃	methoxy-2-(4-methylp	ent of 1-[6-hydroxy-3,4-di- bhenylsulfonyloxy)phenyl]- potassium carbonate in 1-3 h (82 %) [1353].
m.p. 79–80° [1353]; ¹ H N	NMR [1353].	

C₃₆H₄₈O₅

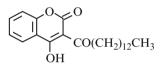
**Dibenzyl ether** 

-Refer to: [1353].

# 4-Hydroxy-3-(1-oxotetradecyl)-2H-1-benzopyran-2-one

[20924-71-4]

C23H32O4 mol. wt. 372.50



Syntheses -Obtained by reaction of tetradecanoyl chloride with CO(CH₂)₁₂CH₃ 4-hydroxycoumarin in pyridine containing one drop -Obtained by reaction of tetradecanoyl chloride with of piperidine for 12 h on a water bath (57 %) [3174].

-Also refer to: [3143].

m.p. 110.5° [3174].

# 1-(8-Hydroxy-5-quinolinyl)-1-tetradecanone

[158905-44-3]

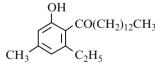
# C23H33NO2

 $CO(CH_2)_{12}CH_3$ 

Syntheses -Preparation by Fries rearrangement of 8-hydroxyquinolinyl myristate using aluminium chloride as catalyst [992]. -Also refer to: [993].

# 1-(2-Ethyl-6-hydroxy-4-methylphenyl)-1-tetradecanone

$$C_{23}H_{38}O_2$$
 mol. wt. 346.55



Syntheses CO(CH₂)₁₂CH₃ -Preparation by Fries rearrangement of 3-ethyl-5-methyl-phenyl myristate with aluminium chloride, *without solvent at 130° for 2 h (75 %) [2802]; *in nitrobenzene at 25° for 6 h (77 %) [2802].

m.p. 37° [2802].

#### Methyl ether $C_{24}H_{40}O_2$ mol. wt. 360.58

-Obtained by methylation of the above ketone in the usual way (74 %) [2802].

m.p. 58° [2802].

#### **2,4-Dinitrophenylhydrazone** $C_{29}H_{42}N_4O_5$ mol. wt. 526.68

m.p. 110° [2802].

#### 1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-tetradecanone

[134081-67-7]	$C_{23}H_{38}O_5$	mol. wt. 394.55
CH ₃ O CH ₃ O CH ₃ O OCH ₃	of crude 2,3,4,6-tetram	selective demethylation ethoxytetradecanophenone de in acetonitrile at 50° for
m.p. 66–68° [1353]; ¹ H N	MR [1353].	

**p-Toluenesulfonic ester** [134081-82-6] C₃₀H₄₄O₇S mol. wt. 548.74

-Obtained by reaction of p-toluenesulfonyl chloride with 2-hydroxy-3,4,6-trimethoxy-tetradecanophenone in the presence of potassium carbonate in refluxing acetone for 6-14 h (92 %) [1353].

m.p. 58.5–60° [1353]; ¹H NMR [1353].

# Methyl ether $C_{24}H_{40}O_5$ mol. wt. 408.57

-Obtained by reaction of tetradecanoyl chloride with 1,2,3,5-tetramethoxybenzene in the presence of aluminium chloride in ethyl ether at  $0^{\circ}$  for 30 min [1353].

#### 1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-tetradecanone

[134081-74-6]	$C_{23}H_{38}O_5$	mol. wt. 394.55
CH ₃ O OCH ₃ O OCH ₃ O OCH ₃ O	Syntheses -Obtained by hydrogenation thoxy)-2,3,4-trimethoxytetrac 10 % palladium on charco methanol (1:1) until the u ceased (90 %) [1353].	lecanophenone over al in ethyl acetate/

-Also refer to: [1351].

m.p. 52.5–54° [1353]; ¹H NMR [1353].

mol. wt. 360.58

mol. wt. 540.70

#### 1-(4,7-Dihydroxy-2,3-dimethyl-6-benzofuranyl)-1-tetradecanone

$$CH_{3}(CH_{2})_{12}CO + O CH_{3} CH_{3} CH_{3}(CH_{2})_{12}CO + O CH_{3} CH_{3} CH_{3} CH_{3} CH_{3} C_{26}H_{40}O_{4} CH_{3} C_{26}H_{40}O_{4} CH_{3} C_{26}H_{40}O_{4} CH_{3} C_{26}H_{40}O_{4} CH_{3} C_{26}H_{40}O_{4} CH_{3} C_{26}CH_{3} CH_{3} C_{26}CH_{3} CH_{3} CH_{3}$$

-Obtained by acylation of 4,7-dimethoxy-2,3-dimethylbenzofuran in the presence of stannic chloride in benzene (74 %) [1040].

m.p. 70° [1040]; LD₅₀ [1040].

USE: Prepn. and radio protective activity of, [1040].

# 1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-tetradecanone

$$(CH_3)_2CH \xrightarrow[CH_3]{OH} CO(CH_2)_{12}CH_3 \qquad Obtained by Fries rearrangement of thymyl myristate with aluminium chloride at 120° (83 %) [2803].$$

b.p.₂ 223° [2803].

**2,4-Dinitrophenylhydrazone** C₃₀H₄₄N₄O₅

m.p. 143° [2803].

# 1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-tetradecanone

	$C_{24}H_{40}O_2$	mol. wt. 360.58
(CH ₃ ) ₂ CH	Synthesis -Refer to: [2660]. <b>Methyl ether</b> ( <b>XV</b> )	)
Y CI	$H_3 C_{25}H_{42}O_2$	mol. wt. 374.61
CO(CH	· · · · · ·	on of myristyl chloride with
	thymol methyl ether in the presence of alumin chloride in carbon disulfide (44 %) [2660].	

b.p.₁ 265° [2660]; m.p. 41° [2660].

#### 1-(4-Butoxy-2-hydroxyphenyl)-1-tetradecanone

[143287-01-8]		$C_{24}H_{40}O_3$		mol. wt. 376.58
OH C ₄ H ₉ O	CO(CH ₂ ) ₁₂ CH ₃	1-(2,4-dihydr	oxyphenyl)-1 otassium carb	butyl bromide with -tetradecanone in the ponate in refluxing ace-

m.p. 45-62° [284].

**Oxime** [143286-69-5] C₂₄H₄₁NO₃ mol. wt. 391.59

-Obtained by reaction of hydroxylamine hydrochloride with 1-(4-butoxy-2-hydroxyphenyl)-1-tetradecanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 69–72° [284].

#### 1-[2,5-Dihydroxy-3,4-dimethyl-6-(2-propenyl)phenyl]-1-tetradecanone

[76402-09-0]	$C_{25}H_{40}O_3$	mol. wt. 388.58
$CH_{3}$ $CH_{3}$ $CH_{2}$ $C$	Synthesis -Obtained by allylic rearran 3,4-dimethyl-2-(2-propen- canone [748].	

#### 1-[5-Hydroxy-3,4-dimethyl-2-(2-propenyloxy)phenyl]-1-tetradecanone

[76402-08-9]	$C_{25}H_{40}O_3$	mol. wt. 388.58
ОН	Synthesis	
CH ₃	-Refer to: [748].	
CH ₃ CO(CH ₂ ) ₁₂ CH ₃		
$\dot{O}CH_2CH=CH_2$		

# 1-(2,5-Dihydroxy-3,4-dimethyl-6-propylphenyl)-1-tetradecanone

[76402-10-3]  $C_{25}H_{42}O_3$  mol. wt. 390.61 OH Synthesis CH₃ CO(CH₂)₁₂CH₃ -Refer to: [748]. CH₃ OH C₃H₇

#### 1,14-Bis(3,5-dihydroxyphenyl)-1,14-tetradecanedione

 $\begin{array}{c} C_{26}H_{34}O_6 & \text{mol. wt. } 442.55 \\ HO & OH & \text{Synthesis} \\ -CO(CH_2)_{12}CO & -CO(CH_2)_{12}CO$ 

**Tetramethyl ether** [21390-01-2] C₃₀H₄₂O₆ mol. wt. 498.66

-Preparation: To a stirred suspension of sodium hydride in dimethylacetamide (DMA) at  $10^{\circ}$  under nitrogen was added 3,5-dimethoxyphenacylpyridinium perchlorate in DMA. The mixture was stirred for 0.5 h and treated with 1,10-diiodododecane. After keeping overnight at 5°, the mixture was heated for 6 h at 80° and finally at r.t. for 4 h with zinc dust and acetic acid (26 %) [2569].

colourless plates [2569]; m.p. 88–89° [2569]; ¹H NMR [2569], IR [2569], UV [2569], MS [2569].

# 1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1-tetradecanone

 $[143287-06-3] C_{27}H_{38}O_3 mtext{mol. wt. 410.60} \\ OH \\ C_6H_5CH_2O \\ C_7H_{38}O_3 \\ C_{27}H_{38}O_3 \\ C_{27}H_{38}O_3$ 

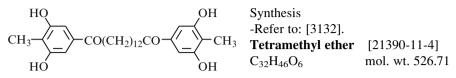
m.p. 64-68° [284].

Oxime	[143286-83-3]	C ₂₇ H ₃₉ NO ₃	mol. wt. 425.60
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-Obtained by reaction of hydroxylamine hydrochloride with 1-[2-hydroxy-4-(phenylmethoxy)-phenyl]-1-tetradecanone in the presence of potassium acetate in refluxing ethanol for 3 h [284].

m.p. 75-78° [284].

#### 1,14-Bis(3,5-dihydroxy-4-methylphenyl)-1,14-tetradecanedione



-Synthesis from 3,5-dimethoxy-4-methylbenzaldehyde (75 %) [3132]. -Also refer to: [2569].

colourless crystalline solid [3132]; m.p. 122° [2569]; ¹H NMR [2569, 3132], ¹³C NMR [3132], IR [2569], UV [2569], MS [2569].

#### 1-[4,6-Bis(butylsulfonyl)-2-hydroxy-3-nitrophenyl]-1-tetradecanone

[81515-11-9] 
$$C_{28}H_{43}NO_8S_2$$
 mol. wt. 571.75  
OH Synthesis  
 $NO_2$  CO(CH₂)₁₂CH₃ -Refer to: [2585].  
 $C_4H_9O_2S$  SO₂C₄H₉

#### 1-[4,6-Bis(butylthio)-2-hydroxy-3-nitrophenyl]-1-tetradecanone

[81515-10-8]  $C_{28}H_{47}NO_4S_2$  mol. wt. 525.82 OH Synthesis  $NO_2$  CO(CH₂)₁₂CH₃ -Refer to: [2585].  $C_4H_9S$  SC₄H₉

#### 1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-1-tetradecanone

[134082-06-7]	$C_{29}H_{42}O_5$	mol. wt. 470.65
CH ₃ O CH ₃ O CH ₃ O CH ₂ CO(CH ₂ ) ₁₂ CH ₃ OCH ₂ C ₆ H ₅	group in 2-positio 1-(2,6-dibenzyloxy-3,4-	on of one phenylmethyl n by treatment of dimethoxyphenyl)-1-tetra- trated hydrochloric acid r 2–3 h (82 %) [1353].
1		

m.p. 80.5–82.5° [1353]; ¹H NMR [1353].

mol. wt. 484.68

# Methyl ether

C₃₀H₄₄O₅

-Refer to: [1353].

# 1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]-1-tetradecanone

[134081-90-6]	$C_{29}H_{42}O_7S$	mol. wt. 534.71
CO(CH ₂ ) ₁₂ CH ₃	Synthesis -Obtained by treatment of 3,4,6-trimethoxyphenyl)-1-tet 25 % aluminium bromide i r.t. for 2–3 h (78 %) [1353]. R [1353].	radecanone with
-		

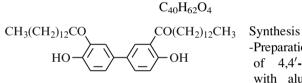
Methyl ether [134081-82-6]

C₃₀H₄₄O₇S mol. wt. 548.74

-Refer to: [1353].

m.p. 58.5–60° [1353]; ¹H NMR [1353].

# 1,1'-(4,4'-Dihydroxy[1,1'-biphenyl]-3,3'-diyl)-1-tetradecanone



-Preparation by Fries rearrangement of 4,4'-biphenyl ditetradecanoate with aluminium chloride in the refluxing chlorobenzene for 24 h (72 %) [2091].

mol. wt. 606.93

m.p. 87-88° [2091].

#### 2 Aromatic Hydroxyketones Derived from 13-Methyltetradecanoic Acids

# 13-Methyl-(2,4-dihydroxyphenyl)-1-tetradecanone

[354585-20-9]	$C_{21}H_{34}O_3$	mol. wt. 334.50
HO	Syntheses -Refer to: [3385, 3386, 3388]. m.p. 87° [3388].	

USE: Process of preparing compounds with particular structure and anticancer activity [3385]; As anticancer and immunostimulation agent [3386]; Cytotoxic [3388].

**BIOLOGICAL ACTIVITY: Cytotoxicity** [3388].

# 13-Methyl-1-(2,3,4-trihydroxyphenyl)-1-tetradecanone

[354585-22-1]	$C_{21}H_{34}O_4$	mol. wt. 350.50
OH HO CO(CH ₂ ) ₁₁ CH(CH ₃ ) ₂	Synthesis -Refer to: [3386].	
но	USE: As anticancer and agent [3386].	immunostimulation

# 13-Methyl-1-(2,4,6-trihydroxyphenyl)-1-tetradecanone

[354585-21-0]	$C_{21}H_{34}O_4$	mol. wt. 350.50
OH CO(CH ₂ ) ₁₁ CH(CH ₃ ) ₂	Synthesis -Refer to: [3386].	
ностон	USE: As anticancer and i agent [3386].	mmunostimulation

# Chapter 13 Pentadecanones

# 1 Aromatic Hydroxyketones Derived from Pentadecanoic Acids

# 1.1 Unsubstituted Hydroxyketones

# 1-(2-Hydroxyphenyl)-1-pentadecanone

 $[100486-17-7] C_{21}H_{34}O_2 mol. wt. 318.50$ OH
CO(CH₂)₁₃CH₃
Syntheses
-Obtained by reaction of pentadecanoyl chloride with
phenol in the presence of aluminium chloride,

*in ethylene dichloride for 16 h at 85° (44 %) [948];

*in nitrobenzene for 6 h at 60–70° (25 %) [1900].

-Also obtained by Fries rearrangement of phenyl pentadecanoate with aluminium chloride for 1 h at  $150^{\circ}$  (20 %) [1273].

m.p. 51.2–51.6° [1273], 51–51.5° [948], 42.5–43.5° [1900]; ¹H NMR [948], IR [948], MS [948].

#### 2,4-Dinitrophenylhydrazone

C₂₇H₃₈N₄O₅

mol. wt. 498.62

m.p. 91.4–92.8° [1273].

mol. wt. 332.53

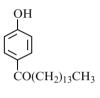
#### 1-(3-Hydroxyphenyl)-1-pentadecanone

	$C_{21}H_{34}O_2$	mol. wt. 318.50
OH	Synthesis -Refer to: [1067].	
CO(CH ₂ ) ₁₃ CH ₃	Methyl ether C ₂₂ H ₃₆ O ₂	mol. wt. 332.53

-Refer to: [1067]; m.p. 34–35° [1067].

# 1-(4-Hydroxyphenyl)-1-pentadecanone

 $[110662-32-3] \qquad \qquad C_{21}H_{34}O_2 \qquad \qquad \text{mol. wt. 318.50}$ 



Syntheses -Obtained by Fries rearrangement of phenyl pentadecanoate with aluminium chloride for 1 h at 150° [1273]; -Also obtained by reaction of pentadecanoyl chloride with phenol in the presence of aluminium chloride in nitrobenzene

for 6 h at 60–70° (68 %) [1900].

-Also refer to: [948].

b.p.₁₀ 230° [1273]; m.p. 72.8–73.5° [1900].

**2,4-Dinitrophenylhydrazone**  $C_{27}H_{38}N_4O_5$  mol. wt. 498.62

 $C_{22}H_{36}O_2$ 

m.p. 140-141° [1273].

# Methyl ether

-Refer to: [943, 1963].

m.p. 65–66° [1963];  $d_{80.5} = 0.8981$  [943];  $n_D^{80.5} = 1,47605$  [943].

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

[1346860-11-4]	$C_{21}H_{34}O_3$	mol. wt. 334.50
HO HO CO(CH ₂ ) ₁₃ CH ₃	Synthesis -Obtained by demethylation [1935]. -Also refer to: [1935].	of the dimethyl ether
	BIOLOGICAL ACTIVITY: outgrowth [1935].	Induction of neurite

**Dimethyl ether** [1346860-08-9]  $C_{23}H_{38}O_3$  mol. wt. 362.55 Refer to: [1935]

-Refer to: [1935].

BIOLOGICAL ACTIVITY: Induction of neurite outgrowth [1935].

#### 1-(2,4-Dihydroxyphenyl)-1-pentadecanone

[100486-26-8]	C ₂₁ H ₃₄ O ₃	mol. wt. 334.50	
HO CO(CH ₂ ) ₁₃ CH	resorcinol in the pre ethylene dichloride -Also obtained by r	a of pentadecanoyl chloride with esence of aluminium chloride in for 16 h at 85° (56 %) [948]. reaction of pentadecanoic acid he presence of zinc chloride for %) [1900].	
pale yellow crystals [1900]; m.p. 91.2–91.8° [1900]; ¹ H NMR [948], MS [948].			
1-(2,5-Dihydroxyphenyl)	-1-pentadecanone		
	$C_{21}H_{34}O_3$	mol. wt. 334.50	
CO(CH ₂ ) ₁₃ CH ₃ -F	ynthesis Refer to: [3274]. <b>imethyl ether</b> [855603 ₂₃ H ₃₈ O ₃	5-97-9] mol. wt. 362.55	

-Preparation by reaction of pentadecanoyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride in tetrachloroethane first at  $0^{\circ}$  for 3 h, then at  $4^{\circ}$  for 72 h (90.5 %) [3274].

white plates [3274]; b.p._{0.5} 205–207° [3274]; m.p. 37.5–38.5° [3274].

 $C_{21}H_{34}O_{3}$ 

# 1-(3,4-Dihydroxyphenyl)-1-pentadecanone

	-2154-5	
ОН	Synthesis	
ОН	-Refer to: [1960].	
	m.p. 100° [1961].	
$\mathbf{i}$	Dimethyl ether	
CO(CH ₂ ) ₁₃ CH ₃	$C_{23}H_{38}O_3$	mol. wt. 362.55

-Obtained by reaction of pentadecanoyl chloride with veratrole in the presence of aluminium chloride without solvent at  $70^{\circ}$  (22 %) [1960].

-Also obtained by condensation of veratrole with n-pentadecanoic acid in the presence of zinc chloride or aluminium chloride [1961].

b.p._{0.3} 220° [1960]; m.p. 64–65° [1960], 51–52° [1961].

mol. wt. 334.50

# 1-(3,5-Dihydroxyphenyl)-1-pentadecanone

 $\begin{array}{cccc} [124210\mathcal{-}61\mathcal{-}3] & C_{21}H_{34}O_3 & \mathcal{-}mol. \ wt. \ 334.50 \\ & OH & \\ & OH & \\ & OH & \\ & Obtained \ by \ demethylation \ of \ its \ dimethyl \ ether \\ & with \ boron \ tribromide \ in \ methylene \ chloride, \ first \\ & at \ -20^\circ, \ then \ at \ r.t. \ overnight \ (34\ \%) \ [140]. \end{array}$ 

Isolation from natural sources

-From the roots and stems of Ardisia virens Kurz (Myrsinaceae) [607].

colourless amorphous powder [140]; ¹H NMR [140], IR [140], MS [140].

BIOLOGICAL ACTIVITY: Cytotoxicity [140].

**Dimethyl ether** [124210-60-2] C₂₃H₃₈O₃ mol. wt. 362.55

-Obtained by adding PPC/Al₂O₃ to a solution of 1-(3,5-dimethoxyphenyl)-1-pentadecanol in benzene, then the mixture obtained was stirred for 2 h (92 %) [140].

colourless needles [140]; m.p. 61.5–62.5° [1068, 1069], 61–62° [140]; IR [140], UV [140], MS [140].

USE: Direct prepn. of benzylic manganese reagents from benzyl halides, sulfonates, and phosphates, their reactions and applications in org. synthesis [2978].

BIOLOGICAL ACTIVITY: Cytotoxicity [140].

# 1-(2,3,4-Trihydroxyphenyl)-1-pentadecanone

	$C_{21}H_{34}O_4$	mol. wt. 350.50
OH	Synthesis	
HO CO(CH ₂ ) ₁₃ CH ₃	-Obtained by reaction	of pentadecanoic acid with
l, lj	pyrogallol in the prese	ence of zinc chloride (Nencki
HO	reaction) at $140-145^{\circ}$	for 4 h [506].

lustrous colourless leaflets [506]; m.p. 87–88° [506].

# 15-Hydroxy-3-methyl-1-(2,5-dihydroxy)phenyl]-1-pentadecanone

[1254222-60-0]	$C_{22}H_{36}O_4$	mol. wt. 364.53
$\bigcup_{OH}^{OH} CO-CH_2-CH-(CH_2)_{12}OH$	Synthesis [ -Refer to: [720]. yellow crystals [720]; ¹ H N ¹³ C NMR [720].	MR [720],

BIOLOGICAL ACTIVITY: Inhibition of nitrite production [720]; Inhibition of tumor necrosis factor- $\alpha$  (TNF- $\alpha$ ) production [720].

Oxime [1	254222-72-4]	$C_{22}H_{37}NO_4$	mol. wt. 379.54
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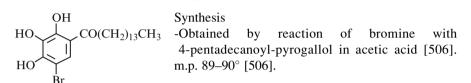
white crystals [720]; ¹H NMR [720].

BIOLOGICAL ACTIVITY: Inhibition of nitrite production [720].

## 1.2 Substituted Hydroxyketones

## 1-(5-Bromo-2,3,4-trihydroxyphenyl)-1-pentadecanone

C₂₁H₃₃BrO₄ mol. wt. 429.39



## 1-(2-Hydroxy-4-methylphenyl)-3-methyl-1-pentadecanone

 $\begin{array}{c} C_{23}H_{38}O_2 & \text{mol. wt. 346.55} \\ OH & CH_3 & Synthesis \\ -Refer to: [1903]. \\ b.p._{0.1} 162-176^{\circ} [1903]. \end{array}$ 

## 1-[2,3,4-Trihydroxy-5-(1-methylethyl)phenyl]-1-pentadecanone

[877877-96-8]	$C_{24}H_{40}O_4$	mol. wt. 392.58
HO HO CO(CH ₂ ) ₁₃ CH ₃	Synthesis -Refer to: [3267]. ¹ H NMR [3267], ¹³ C NMR [3267].	
HO' $\Upsilon$ CH(CH ₃ ) ₂	BIOLOGICAL ACTIVITY: As antiapoptotic Bcl-2 [3267].	inhibitors of

## 1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-pentadecanone

rt. 374.61
2-methyl-

-Obtained by treatment of 4-methoxy-2-methyl-5-isopropylpentadecanone with boiling pyridinium chloride (205–215°) for 7.5 h (23 %) [2660].

b.p.₁₁ 276–279° [2660]; m.p. 41° [2660].

 $CO(CH_2)_{13}CH_3$ 

OH

 $(CH_3)_2CH$ 

## Methyl ether

 $C_{26}H_{44}O_2$ 

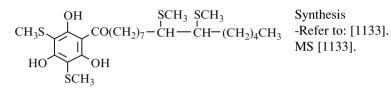
mol. wt. 388.63.

-Preparation by reaction of pentadecanoyl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (44 %) [2660].

 $b.p._{19} \ 280 - 283^\circ \ [2660]; \quad m.p. \ 44^\circ \ [2660].$ 

## 1-[3,5-(Dithiomethyl)-2,4,6-trihydroxyphenyl]-9,10-(dithiomethyl)-1-pentadecanone

$$C_{25}H_{42}O_4S_4$$
 mol. wt. 534.87



## 2 Aromatic Hydroxyketones Derived from 14-Methylpentadecanoic Acid

## 1-(2-Hydroxy-4-methylphenyl)-14-methyl-1-pentadecanone

[57080-94-1]	$C_{23}H_{38}O_2$	mol. wt. 346.55
OH	Synthesis	
CO(CH ₂ ) ₁₂ CH(CH ₃ ) ₂	-Refer to: [1903]. Oxime [57125-28-7]	
CH ₃	$C_{23}H_{39}NO_2$	mol. wt. 361.57

-Refer to: [1903].

# Chapter 14 Hexadecanones

## 1 Aromatic Hydroxyketones Derived from Hexadecanoic Acids

## 1.1 Unsubstituted Hydroxyketones

## 1-(2-Hydroxyphenyl)-1-hexadecanone

 $\begin{array}{c} [2589-84-6] & C_{22}H_{36}O_2 & \mbox{mol. wt. } 332.53 \\ OH & Syntheses \\ -Obtained by reaction of palmitoyl chloride with phenol in the presence of aluminium chloride, \\ * in tetrachlorethane few hours at 25° (26 %) [2548]; \\ * in nitrobenzene for 3 h at 70° (22 %) [2549]; \end{array}$ 

*in carbon disulfide for 5.5 h at  $47^{\circ}$  (47 %) [2549].

-Also obtained by Fries rearrangement of phenyl palmitate with aluminium chloride [1456],

*in tetrachlorethane for 10 h at  $70^{\circ}$  (15 %) [2550];

*without solvent for 1 h at 150° (20 %) [1273].

-Also obtained by reaction of palmitic acid with phenol,

*in the presence of zinc chloride (Nencki reaction) (75 %) [2398];

*in the presence of boron trifluoride first at  $60^{\circ}$ , then at  $80-90^{\circ}$  [2598]. -Also refer to: [873, 1225].

m.p. 58° [2398], 57–58° [1273], 54–56° [2548], 54–55° [2598]; TLC [1456].

USE: Textile rot proofing by, [873].

Properties, DOI 10.1007/978-3-319-14185-5_14

mol. wt. 374.56

mol. wt. 346.55

m.p. 87° [2598].

 $\label{eq:constraint} \textbf{2,4-Dinitrophenylhydrazone} \qquad C_{28}H_{40}N_4O_5 \qquad \qquad \text{mol. wt. 512.65}$ 

m.p. 96–97° [1273], 94–95° [2548], 93° [2598].

## **2,4-Dinitrophenylhydrazone, nickel complex** [116803-89-5]

C₂₄H₃₈O₃

USE: Metal complex discolouration inhibitor, silver halide photog. material contg. [2976].

## Acetate

m.p. 52° [2598].

## Methyl ether

-Obtained by reaction of dimethyl sulfate with 1-(2-hydroxyphenyl)-1-hexadecanone in the presence of potassium carbonate in boiling acetone for 6 h [2398].

C₂₃H₃₈O₂

colourless liquid [2398]; b.p.₃₀ 190° [2398]; m.p. 38° [2398].

## 1-(3-Hydroxyphenyl)-1-hexadecanone

[63442-87-5]	$C_{22}H_{36}O_2$	mol. wt. 332.53
CO(CH ₂ ) ₁₄ CH ₃	Syntheses -Refer to: [1984, 1985]. m.p. 56° [1984]; IR [1984].	

Methyl ether	[63442-84-2]	$C_{23}H_{38}O_2$	mol. wt. 346.55

-Refer to: [1984, 1985].

m.p. 42° [1984]; IR [1984].

## 1-(4-Hydroxyphenyl)-1-hexadecanone

[2589-76-6]	$C_{22}H_{36}O_{2}$	mol. wt. 332.53
OH CO(CH ₂ ) ₁₄ CH ₃	Syntheses -Obtained by reaction of palmitoyl chloride y presence of aluminium chloride, *in tetrachlorethane few hours at 25° (29 %) *in nitrobenzene for 3 h at 70° (67 %) [2549] *in carbon disulfide for 5.5 h at 47° (51 %) [2	[2548]; ];

Oxime

-Also obtained by Fries rearrangement of phenyl palmitate with aluminium chloride [1456],

*in tetrachlorethane for 10 h at  $70^{\circ}$  (20 %) [2550];

*without solvent for 1 h at 150° (30 %) [1273];

*in the presence of aluminium chloride in refluxing carbon disulfide for 5 h. Then, the mixture was kept at  $80-90^{\circ}$  for 2 h and at  $140-150^{\circ}$  for 1 h after solvent elimination [1222].

-Also obtained by demethylation of 4-palmitoylanisole with hydrobromic acid in acetic acid [2398].

-Also obtained by fusion of its methyl ether with potassium hydroxide [1757].

-Also obtained by dealkylation of its ethyl ether with aluminium chloride in carbon disulfide for 8 h at  $60-70^{\circ}$  [191].

-Also obtained by reaction of palmitic acid with phenol in the presence,

*of boron trifluoride for 2–3 h between 65 and 85° (96–98 %) [503] or first at  $60^{\circ}$ , then at 80–90° [2598];

*of activated acid clay catalyst at 190° for 2 h [3277].

-Also refer to: [873, 1057, 1116, 1225, 1299, 3424].

m.p. 85° [2398], 84.5–86° [3277], 84.5–85° [1273, 2548], 84–85° [2598], 81–82° [3424], 80° [503], 78° [191]; ¹H NMR [3424]; Cryoscopic study [182]; TLC [1456].

USE: Textile rot proofing by, [873].

## Oxime

 $C_{22}H_{37}NO_2$ 

mol. wt. 347.54

m.p. 95° [2598].

2,4-Dinitrophenylhydrazone	$C_{28}H_{40}N_4O_5$	mol. wt. 512.65
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m.p. 142.3–143.5° [1273], 141–142° [2548], 141° [2598].

iso-Nicotinylhydrazone [103159-09-7]  $C_{28}H_{41}N_3O_2$  mol. wt. 451.65

m.p. 153° [520, 521].

USE: In chemotherapy of leprosy [520, 521].

Methyl ether [102898-55-5] C₂₃H₃₈O₂ mol. wt. 346.55

-Obtained by reaction of palmitoyl chloride with anisole in the presence of aluminium chloride (90 %) [2398] at 70° on a water bath for 3 days (69 %) [1757] or for 2 h (50 %) [15].

-Also obtained by reaction of palmitoyl chloride with anisole in the presence of zinc in toluene at  $70^{\circ}$  for 5 h (91 %) [2064].

-Also obtained by reaction of palmitic acid with anisole,

*in the presence of chloracetyl anhydride (80 %) [3178];

*on the solid surface of alumina in the presence of trifluoroacetic anhydride for 180 min at r.t. (92 %) [2563].

-Also refer to: [643, 1104, 1756, 1758, 1963, 2016].

b.p.₁₅ 279–280° [1756–1758]; m.p. 75° [3178], 72.5° [2016], 72–73° [1963], 71–71.5° [15], 70.5° [1756–1758], 70° [1104, 2398]; ¹H NMR [2064, 2563], IR [2064, 2563], MS [2064];  $n_D^{80.5} = 1.47605$  [643].

## 2,4-Dinitrophenylhydrazone of the methyl ether C₂₉H₃₄N₄O₅ mol. wt. 518.61

m.p. 97–98° [2016].

Ethyl ether	[416846-26-9]	$C_{24}H_{40}O_2$	mol. wt. 360.58
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-Obtained by Friedel-Crafts reaction of palmitoyl chloride with phenetole in the presence of aluminium chloride [191, 1757]. -Also refer to: [1756, 1758].

b.p.₁₅ 288–289° [1756–1758]; m.p. 69° [1756–1758].

**2-Chloroethyl ether**  $C_{24}H_{39}ClO_2$  mol. wt. 395.03

-Obtained by reaction of hexadecanoyl chloride with 2-chloroethoxybenzene in the presence of aluminium chloride in carbon disulfide for 4 h at  $50^{\circ}$  (75 %) [476].

m.p. 70–71° [476].

**N-Dimethylaminoethyl ether** C₂₆H₄₅NO₂ mol. wt. 403.65

-Obtained by reaction of 4-(2-chloroethoxy)hexadecanophenone with dimethylamine in a sealed tube for 2 h at  $150^{\circ}$  [476].

free base: b.p._{0.3} 210° [476]; m.p. 41–42° [476]. hydrochloride (49 %) [476]; m.p. 150° [476].

**2,3-Epoxypropoxy ether** [18211-88-6] C₂₅H₄₀O₃ mol. wt. 388.58

-Obtained by reaction of epichlorohydrin (0.1 mol) with p-hexadecanoylphenol (0.1 mol) in the presence of 30 % sodium hydroxide between 40 and  $50^{\circ}$  (51 %) [2669].

m.p. 110° [2669].

#### Acetate

 $C_{24}H_{38}O_3$ 

mol. wt. 374.56

m.p. 79° [2598].

## Palmitate [120857-41-2] C₃₈H₆₆O₃ mol. wt. 570.94

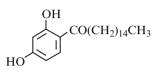
-Obtained by reaction of palmitic acid with phenol in the presence of activated acid clay catalyst at  $190^{\circ}$  for 2 h [3277].

m.p. 87-88° [3277].

## 1-(2,4-Dihydroxyphenyl)-1-hexadecanone

$$[40372-79-0] C_{22}H_{36}O_3 mol. wt. 348.53$$

Syntheses



 $CO(CH_2)_{14}CH_3$  -Obtained by reaction of palmitic acid with resorcinol,

*in the presence of boron trifluoride for 2–3 h between 65 and  $85^{\circ}$  (95–98 %) [503] or first at 60°, then at 80–90° [2598];

*in the presence of zinc chloride [16]. -Also refer to: [859, 1296, 1757, 2973].

m.p. 95–95.5° [2598], 95° [503], 94–95° [16], 89–90° [859].

USE: Protection against whole-body irradiation [1810]; Emulsifying agent, in waterproofing of leather [1296]; Protection against X-rays [1811].

Na salt	[41729-72-0]	$C_{22}H_{34}Na_2O_3$	mol. wt. 392.48
INA SAIL	[41/29-/2-0]	$C_{22}\Pi_{34}\Pi_{42}O_{3}$	11101. wt. 592.40

USE: Emulsifying agent, in waterproofing of leather [1296].

Oxime	C ₂₂ H ₃₇ NC	C ₂₂ H ₃₇ NO ₃	
m.p. 158° [2598]			
4-Nitrophenylhydr	azone C	28H41N3O4	mol. wt. 483.64
m.p. 94–95° [859	)].		
Dimethyl ether	C ₂₄ H	I ₄₀ O ₃	mol. wt. 376.58
-Obtained by Friedel-Crafts reaction of palmitoyl chloride (2 parts) with resorcinol dimethyl ether (3 parts) in the presence of aluminium chloride (2 parts) by heating the mixture from 40° to 100° [1757]. -Also refer to: [16].			
b.p. ₁₅ 289–290°	[1757]; m.p. 63.5° [	1757], 61° [16].	
Diacetate	[96968-05-7]	$C_{26}H_{40}O_5$	mol. wt. 432.60
-Refer to: [2517].			
m.p. 73–74° [259	98].		

## 1-(2,5-Dihydroxyphenyl)-1-hexadecanone

(2-Palmitoylhydroquinone)

 $\begin{array}{ccc} [95807-67-3] & C_{22}H_{36}O_3 & \mbox{mol. wt. } 348.53 \\ \\ OH & Syntheses \\ -Obtained by reaction of palmitic acid with hydroquinone in the presence of boron trifluoride, \\ & *in 1,2-dichloroethane (50 \%) [142]; \\ & *without solvent first at 60°, then at 80–90° [2598]. \end{array}$ 

-Also obtained by reaction of hexadecanoyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride [156] in carbon disulfide [159]. -Also refer to: [393, 2090, 2151].

m.p. 106–107° [142], 106° [2598], 103–104° [156, 159].

USE: Probing the redox activity of T-lymphocytes deposited at electrode surfaces with voltammetric methods (as a redox mediator) [393]; Redox Chemistry of Ca-Transporter 2-palmitoylhydroquinone in an Artificial Thin Organic Film Membrane [2090].

Oxime	C ₂₂ H ₃₇ NO ₃		mol. wt. 363.54
m.p. 102° [2598].			
Dimethyl ether	[180133-49-7]	$C_{24}H_{40}O_3$	mol. wt. 376.58
-Obtained by reaction of hexadecanoyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride [2859],			

*in carbon disulfide (78 %) [1633];

*in tetrachloroethane (69 %) [714]. -Also refer to: [1632].

b.p._{0.18} 205° [714]; m.p. 51.5° [714], 44° [1632, 1633]; ¹H NMR [2859]; MS [2859].

Phenylhydrazone of the dimethyl ether	$C_{30}H_{46}N_2O_2$	mol. wt. 466.71
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m.p. 64° [1633].

#### **Diethyl ether**

 $C_{26}H_{44}O_3$ 

mol. wt. 404.63

-Obtained by reaction of palmitoyl chloride with hydroquinone diethyl ether in the presence of aluminium chloride in tetrachloroethane [714].

## 2,4-Dinitrophenylhydrazone of the diethyl ether C32H48N4O6 mol. wt. 584.76

bright red crystals [714]; m.p. 75° [714].

## Diacetate

$$C_{26}H_{40}O_5$$

m.p. 77° [2598].

## 1-(2,6-Dihydroxyphenyl)-1-hexadecanone

$$\begin{bmatrix} 96820-25-6 \end{bmatrix} & C_{22}H_{36}O_3 & \text{mol. wt. } 348.53 \\ & \bigcirc H & \\ & \bigcirc CO(CH_2)_{14}CH_3 & \\ & \bigcirc H & \\ & \bigcirc OH & \\ & OH & \\ & \bigcirc OH & \\ & OH & \\ & \bigcirc OH & \\ & OH$$

-From fruits of Virola elongata (Myristicaceae) [1622, 1623]. -In plants of the family Myristicaceae [719].

amorphous solid [1622]; ¹H NMR [1622], ¹³C NMR [1622], IR [1622], MS [1622].

## 1-(3,4-Dihydroxyphenyl)-1-hexadecanone

(4-*Hexadecanoylcatechol*)

[54535-83-0] C22H36O3 mol. wt. 348.53 Syntheses OH -Obtained by treatment of a pyrocatechol and hexadecanoic OH. acid mixture, *with zinc chloride [1961] at 135–140° for 2 h (10 %) [1283];  $\dot{CO}(CH_2)_{14}CH_3$  *with boron trifluoride first at 60°, then at 80–90° [2598].

-Also refer to: [283, 3159].

m.p. 101–102° [2598], 99–100° [1283].

Oxime	C ₂₂ H ₃₇ NC	D ₃	mol. wt. 363.54
m.p. 103° [2598].			
2,4-Dinitrophenylhydr	azone	$C_{28}H_{40}N_4O_5$	mol. wt. 512.65
m.p. 225° [2598].			
Dimethyl ether	[855956-25-1]	$C_{24}H_{40}O_3$	mol. wt. 376.58
-Obtained by Friedel-C presence of aluminium *without solvent at 70° *in carbon disulfide [15 -Also refer to: [1960].	chloride [2574], (24 %) [1960];	palmitoyl chloride with	veratrole in the

b.p._{0.5} 230° [1960]; m.p. 79-80° [1960], 78-79° [1526], 72-72.5° [2574].

mol. wt. 432.60

mol. wt. 432.60

Diacetate

m.p. 72–73° [2598].

## 1-(2,3,4-Trihydroxyphenyl)-1-hexadecanone

(4-Hexadecanoylpyrogallol)

[40366-12-9]

 $C_{22}H_{36}O_4$ 

mol.	wt.	364.	53
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mol. wt. 544.65

OH	Syntheses
ОН	-Obtained by reaction of palmitic acid with pyrogallol,
	*in the presence of boron trifluoride for 2–3 h between 65 and
У`ОН	85° (95–98 %) [503];
$CO(CH_2)_{14}CH_3$	*in the presence of zinc chloride (Nencki reaction) at $135-140^{\circ}$
	for 2 h (30 %) [1283].

-Also refer to: [859, 1296, 2327].

m.p. 92° [503], 89–90° [1283], 84–85° [859].

USE: Colour photog. development-promoting agent, for rapid development without benzyl alc. [2327]; Emulsifying agent, in waterproofing of leather [1296].

 $C_{28}H_{40}N_4O_7$ 

2,4-Dinitrophenylhydrazone

m.p. 171–172° [859].

## 1-(2,4,6-Trihydroxyphenyl)-1-hexadecanone

 $[82461-11-8] C_{22}H_{36}O_4 mol. wt. 364.53$ 

Syntheses

HO OH CO(CH₂)₁₄CH

 $CO(CH_2)_{14}CH_3$  -Obtained by reaction of hexadecanoic (palmitic) acid with phloroglucinol in the presence of aluminium chloride and phosphorous oxychloride (16 %) [3202].

Isolation from natural sources

-From fruits of Virola elongata (Myristicaceae) [1623].

-From the related brown algae Zonaria farlowii, Zonaria diesingiana and Lobophora papenfussii (9 %) (Dictyotaceae) [1103, 3202].

white crystals [3202]; m.p. 129° [3202]; ¹H NMR [1103, 3202], ¹³C NMR [1103, 3202], IR [1103, 3202], UV [1103, 3202], MS [1103, 3202]; TLC [3202].

**Triacetate** [82460-91-1] C₂₈H₄₂O₇ mol. wt. 490.64

-Obtained by reaction of acetic anhydride with 1-(2,4,6-trihydroxyphenyl)-1-hexadecanone in the presence of pyridine at r.t. overnight (near quantitative yield) [1103].

¹H NMR [1103], IR [1103].

BIOLOGICAL ACTIVITY: Antibiotic [3202].

 $C_{26}H_{40}O_5$ 

#### 13-Hydroxy-1-(2,4,6-Trihydroxyphenyl)-1-hexadecanone

(Byssomeruliol C)

## 14-Hydroxy-1-(2,4,6-Trihydroxyphenyl)-1-hexadecanone

(Byssomeruliol D)

#### 16-Hydroxy-3-methyl-1-(2,5-dihydroxyphenyl)-1-hexadecanone

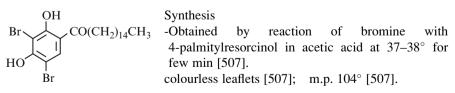
[1254222-63-3]	$C_{23}H_{38}O_4$	mol. wt. 378.55
$\bigcup_{\substack{I \\ OH \\ OH}}^{OH} COCH_2 - CH - (CH_2)_{13}OH$	Synthesis -Refer to: [720]. <b>Oxime</b> [1254222-75-7] C ₂₃ H ₃₉ NO ₄	mol. wt. 393.57

## 1.2 Substituted Hydroxyketones

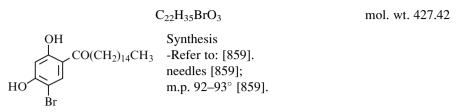
#### 1-(3,5-Dibromo-2,4-dihydroxyphenyl)-1-hexadecanone

(2,6-Dibromo-4-palmitylresorcinol)

$$C_{22}H_{34}Br_2O_3$$
 mol. wt. 506.32



## 1-(5-Bromo-2,4-dihydroxyphenyl)-1-hexadecanone



## 1-(5-Bromo-2,3,4-trihydroxyphenyl)-1-hexadecanone

C	$_{22}H_{35}BrO_4$	mol. wt. 443.42
ŎН	Syntheses	
HO CO(CH ₂ ) ₁₄ CH ₃	-Obtained by reaction of bron	nine with 4-palmitoyl-
	pyrogallol in acetic acid [506	].
НО	-Also refer to: [859].	
Br	needles [859]; m.p. 87-88° [	[859], 89–90° [506].

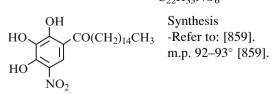
## 1-(4-Hydroxy-3-nitrophenyl)-1-hexadecanone

[70079-28-6]	$C_{22}H_{35}NO_4$	ſ	nol. wt. 377.52
OH NO ₂ CO(CH ₂ ) ₁₄ CH ₃	Syntheses -Obtained by Fries rearrangement hexadecanoate [1222]. -Also refer to: [1224, 1315]. m.p. 85–86° [1222].	of	o-nitrophenyl

## 1-(2,4-Dihydroxy-5-nitrophenyl)-1-hexadecanone

(	$C_{22}H_{35}NO_5$	mol. wt. 393.52
HO NO ₂ CO(CH ₂ ) ₁₄ CH ₃	Synthesis -Refer to: [859]. pale yellow needles [859]; m.p. 97–98° [859].	

## 1-(2,3,4-Trihydroxy-5-nitrophenyl)-1-hexadecanone



### 4-Hexadecanoylsalicylic acid

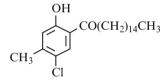
[40372-78-9] C23H36O4 mol. wt. 376.54 CO₂H **Synthesis** .OH -Refer to: [1296]. USE: Emulsifying agent, in waterproofing of leather [1296]. CO(CH₂)₁₄CH₃

## 1-(2-Chloro-6-hydroxy-4-methylphenyl)-1-hexadecanone

[24490-29-7] C23H37ClO2 mol. wt. 381.00 Synthesis  $CO(CH_2)_{14}CH_3$  -Refer to: [3138]. USE: Fluorescence of, hyperchromic shifts in, [3138].

## 1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-hexadecanone

$C_{23}H_{37}ClO_2$ mol. v	wt. 381.00
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Syntheses CO(CH₂)₁₄CH₃ -Obtained by reaction of palmitic acid with 4-chloro-3-methylphenol in the presence of boron trifluoride for 2-3 h between 65 to  $85^{\circ}$ (90 %) [503]. -Also refer to: [3138].

m.p. 86° [503]; fluorescence spectral data [3138].

## 1-(2-Hydroxy-5-methylphenyl)-1-hexadecanone

C23H38O2 mol. wt. 346.55 Syntheses OH -Obtained by reaction of palmitic acid with p-cresol in the  $CO(CH_2)_{14}CH_3$ presence of boron trifluoride for 2-3 h between 65 and 85° (90–95 %) [503]. CH₃ -Also refer to: [879].

¹H NMR [879], IR [879]. m.p. 61° [503];

**Oxime of the methyl ether** [101396-06-9] C₂₄H₄₁NO₂ mol. wt. 375.60 m.p. 51–52° [879].

USE: Palladium extn. and purifn. with, [3191].

#### 1-(4-Hydroxy-2-methylphenyl)-1-hexadecanone

$$\begin{array}{ccc} C_{23}H_{38}O_2 & \text{mol. wt. 346.55} \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\$$

-Obtained by adding a mixture of m-phenoxytoluene and hexadecanoyl chloride to a suspension of aluminium chloride in methylene chloride at  $0^{\circ}$ , then the mixture stirred for 1.5–2 h at 3–5° (31 %) [2503].

b.p.₃ 261–263° [2503]; m.p. 30–31° [2503]; ¹H NMR [2503], IR [2503], MS [2503].

## 1-(2,5-Dihydroxy-4-methylphenyl)-1-hexadecanone

-Also obtained by reaction of hexadecanoyl chloride with 2,5-dimethoxytoluene in the presence of aluminium chloride in refluxing carbon disulfide for 10 h. Demethylation was accompanied in the course of the reaction (quantitative yield) [2370]. -Also refer to: [748, 750, 3106].

yellow plates [2370]; m.p. 94–94.5° [142, 3106], 93.5° [2370]; IR [2370].

USE: As photographic antistain agent [3106]; Improvements in heat-sensitive copy materials [750].

#### 1-(2-Hydroxy-5-methoxyphenyl)-1-hexadecanone

mol. wt. 362.55 [102898-63-5] C23H38O3 **Svntheses** OH -Obtained by reaction of hexadecanoic acid with  $CO(CH_2)_{14}CH_3$ p-methoxyphenol in the presence of boron trifluoride in tetrachloroethane for 4 h. The reaction mixture was OCH₃ allowed to stand overnight at r.t. and was then heated on a steam bath for about 3 h (47 %) [142].

-Also obtained by reaction of hexadecanoyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride [156], in carbon disulfide [159].

m.p. 59-60.5° [156, 159], 57.5-59° [142].

## 1-(4-Hydroxy-2-methoxyphenyl)-1-hexadecanone

C23H38O3 mol. wt. 362.55 Syntheses -Refer to: [14, 16, 1756, 1758]. m.p. 66° [14, 16]. OCH₃ CO(CH₂)₁₄CH₃

## 1-[2-(Acetyloxy)-4-hydroxyphenyl]-1-hexadecanone

[251463-57-7]

OH

C24H38O4

mol. wt. 390.56

**Synthesis** OH -Obtained by selective deacetylation of 2,4-diacetoxyphenyl pentadecyl ketone mediated by porcine pancreatic lipase OCOCH₃ (PPL) in THF at  $42-45^{\circ}$  for 24 h in the presence of n-butanol  $CO(CH_2)_{14}CH_3$ (60 %) [2517].

white solid [2517]; m.p. 105° [2517]; ¹H NMR [2517], ¹³C NMR [2517], IR [2517], UV [2517], MS [2517].

## 1-(4-Hydroxy-2,5-dimethylphenyl)-1-hexadecanone

[95185-68-5]

 $C_{24}H_{40}O_{2}$ 

mol. wt. 360.58

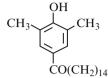
OH Synthesis -Refer to: [2704]. CH₃. CH₂ CO(CH₂)₁₄CH₃

1043

mol. wt. 376.58

#### 1-(4-Hydroxy-3,5-dimethylphenyl)-1-hexadecanone

C₂₄H₄₀O₂ mol. wt. 360.58



Syntheses

- -Obtained by reaction of hexadecanoyl chloride with 2,6-dimethylphenol in the presence of aluminium chloride [1832].
- CO(CH₂)₁₄CH₃ -Also obtained by reaction of hexadecanoic acid with 2,6-dimethylphenol in the presence of boron trifluoride [1832].

## 1-(4-Ethoxy-2-hydroxyphenyl)-1-hexadecanone

[19347-52-5]

C₂H₅C

C₂₄H₄₀O₃ Syntheses

 $CO(CH_2)_{14}CH_3$  -Obtained by reaction of hexadecanoyl chloride with m-diethoxybenzene in the presence of aluminium chloride in dichloroethane at 0°. Then, the mixture was stirred for 1 h at 10°, and then for 6 h at 20–25°.

The temperature was then raised to  $80^{\circ}$  and stirring was continued at that temperature for 5 h (78 %) [1194, 1195].

-Also obtained by treating hexadecanoyl chloride with resorcinol diethyl ether under Friedel-Crafts reaction conditions [1197, 1198].

m.p. 66–66.3° [1194, 1195]; UV [1194, 1195].

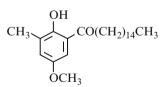
USE: UV absorber [1194]; Light-stabilizer of polymeric materials [1194].

## 1-(2-Hydroxy-5-methoxy-3-methylphenyl)-1-hexadecanone

[103168-03-2]

 $C_{24}H_{40}O_3$ 

mol. wt. 376.58



Syntheses

 $CO(CH_2)_{14}CH_3$  -Obtained by reaction of hexadecanoic acid with 4-methoxy-2-methylphenol in the presence of boron trifluoride in tetrachloroethane for 6 h. The reaction mixture was allowed to stand overnight at r.t. and was then heated on a steam bath for about 5 h (37 %) [142].

-Also refer to: [1907, 3106].

b.p.₁ 205–215° [3106]; m.p. 66–67° [142, 3106].

USE: As photographic antistain agent [3106].

#### 1-(2-Hydroxy-4,6-dimethoxyphenyl)-1-hexadecanone

[82460-89-7]	$C_{24}H_{40}O_4$	mol. wt. 392.58
CH ₃ O ^{OH} CO(CH ₂ ) ₁₄ CH ₃ OCH ₃ O ^{OCH} 3		of excess diazomethane yphenyl)-1-hexadecanone h at $25^{\circ}$ (quantitative

¹H NMR [1103], IR [1103].

Acetate [82460-90-0] C₂₆H₄₂O₅ mol. wt. 434.62

-Obtained by reaction of acetic anhydride with 1-(2-hydroxy-4,6-dimethoxy-phenyl)-1-hexadecanone in the presence of pyridine at r.t. overnight (near quantitative yield) [1103].

¹H NMR [1103], IR [1103].

## 1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-hexadecanone

ОН
CO(CH ₂ ) ₁₄ CH
СН30
OCH3

Synthesis H₃ -Preparation by treatment of 1-[6-hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy) phenyl]-1-hexadecanone with potassium carbonate in refluxing methanol for 1–3 h (89 %) [1353].

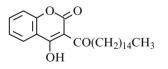
m.p. 83.5–85° [1353]; ¹H NMR [1353].

4-Hydroxy-3-(1-oxohexadecyl)-2H-1-benzopyran-2-one

4-Hydroxy-3-palmitoyl-2H-chromen-2-one

[74965-90-5] C₂₅H₃₆O₄

mol. wt. 400.56



Syntheses

CO(CH₂)₁₄CH₃ -To the PTFE reaction vessel, 4-hydroxycoumarin, a catalytic amount of piperidine, and pyridine (1 ml/ mmol) were added. After the solution was cooled to 0°, palmitoyl chloride (1.6 equiv.) was slowly added through a septum.

The mixture was sonicated (21 kHz, 40 W/cm²) for about 1.5 h under nitrogen at  $38^{\circ}$ , and monitored by TLC [746].

-Also obtained by reaction of hexadecanoyl chloride with 4-hydroxycoumarin in pyridine containing one drop of piperidine for 12 h on a water bath (61 %) [3174].

white powder [746]; m.p. 111° [3174], 103–104° [746]; ¹H NMR [746], IR [746], MS [746]; TLC [746].

BIOLOGICAL ACTIVITY: Antibacterial [746].

## 1-(8-Hydroxy-5-quinolinyl)-1-hexadecanone

C25H37NO2 [103045-61-0] mol. wt. 383.57 Syntheses

-Obtained by neutralizing an aqueous suspension of its hydrobromide with alkali [938].

-Preparation by Fries rearrangement of 8-hydroxyquinolinyl palmitate using aluminium chloride as catalyst [992].

-Also refer to: [892, 993-996, 3108].

USE: Preconcentration and speciation of chromium (III) in waters by using 5-palmitoyl-8-hydroxyquinoline immobilized on a nonpolar adsorbent [995]; Amebicidal action of, [3108].

#### C₂₅H₃₇NO₂, HBr Hydrobromide mol. wt. 464.48

-Obtained by reaction of palmitoyl chloride with 8-quinolinol in the presence of aluminium chloride in nitrobenzene first at 75° for 16 h, then at r.t. for 20 h. Then, the acetone solution was saturated with hydrogen bromide (11 %) [938].

m.p. 223° [938].

## 1-[3,6-Dihvdroxy-2-(2-propenvl)phenvl]-1-hexadecanone

1-(3,6-Dihydroxy-2-allylphenyl)-1-hexadecanone

$$[98357-90-5]$$
 C₂₅H₄₀O₃ mol. wt. 388.58

Syntheses CO(CH₂)₁₄CH₃ -Obtained by Claisen rearrangement of 1-[2-hydroxy-5-(allyloxy)phenyl]-1-hexadecanone, first at 170° and in fine at 223° under nitrogen (63 %) [142]. CH₂CH=CH₂ -Also refer to: [2151].

m.p. 77.5–78.5° [142].

## 1-[2-Hydroxy-5-(2-propenyloxy)phenyl]-1-hexadecanone

1-[2-Hydroxy-5-(allyloxy)phenyl]-1-hexadecanone

[98357-89-2]	$C_{25}H_{40}O_3$	mol. wt. 388.58
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OCH₂CH=CH₂

Syntheses

CO(CH₂)₁₄CH₃ -Obtained by reaction of allyl bromide with 2-palmitoylhydroquinone in the presence of potassium carbonate in refluxing acetone for 8 h, then at r.t. overnight (62 %) [142]. -Also refer to: [2151].

m.p. 58–59° [142].



## 1-(4-Hydroxy-2,3,5-trimethylphenyl)-1-hexadecanone

[137833-03-5]	$C_{25}H_{42}O_2$	mol. wt. 374.61
$CH_{3} \xrightarrow{CH_{3}} CH_{3}$ $CH_{3} \xrightarrow{CH_{3}} CH_{3}$ $CO(CH_{2})_{14}CH_{3}$	Synthesis -Refer to: [1733].	

## 1-(2,5-Dihydroxy-4-propylphenyl)-1-hexadecanone

[115486-55-0]	$C_{25}H_{42}O_3$	mol. wt. 390.61
$C_{3}H_{7} \xrightarrow{OH} CO(CH_{2})_{14}CH_{3}$	Synthesis -Obtained by reaction of hexa 2-propyl-hydroquinone in the trifluoride in 1,2-dichloroetha 1.25 h. The mixture was allo night (56 %) [142].	presence of boron ane at $40-45^{\circ}$ for

m.p. 76.5–77.5° [142].

## 1-(3,6-Dihydroxy-2-propylphenyl)-1-hexadecanone

C25H42O3 mol. wt. 390.61

Synthesis CO(CH₂)₁₄CH₃ -Obtained by hydrogenation of 3-allyl-2-palmitoyl-hydro-quinone in ethanol in the presence of Raney nickel under C₂H₂ 2.7 atm. of hydrogen. The reduction was complete in few 2.7 atm. of hydrogen. The reduction was complete in few min at r.t. (96 %) [142].

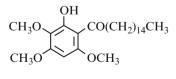
cream-coloured crystalline powder [142]; m.p. 67–68° [142].

## 1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-hexadecanone

[134081-68-8]

C₂₅H₄₂O₅

mol. wt. 422.61



H Syntheses  $CO(CH_2)_{14}CH_3$  -Obtained by partial selective demethylation of  $CO(CH_2)_{14}CH_3$  -Obtained by partial selective demethylation of CUde 2,3,4,6-tetramethoxyhexadecanophenone with aluminium chloride in acetonitrile at 50° Syntheses for 1–2 h (85 %) [1353]. -Also refer to: [1351].

m.p. 74–75° [1353]; ¹H NMR [1353].

## **p-Toluenesulfonic ester** [134081-83-7] C₃₂H₄₈O₇S mol. wt. 576.79

-Obtained by reaction of p-toluenesulfonyl chloride with 2-hydroxy-3,4,6-trimethoxy-hexadecanophenone in the presence of potassium carbonate in refluxing acetone for 6 to 14 h (96 %) [1353].

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m.p. 65.5–67.5° [1353]; <sup>1</sup>H NMR [1353].
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Methyl ether	$C_{26}H_{44}O_5$	mol. wt. 436.63
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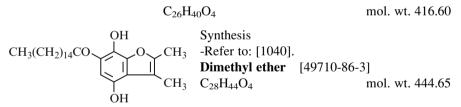
-Obtained by reaction of hexadecanoyl chloride with 1,2,3,5-tetramethoxybenzene in the presence of aluminium chloride in ethyl ether at  $0^{\circ}$  for 30 min [1353].

## 1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-hexadecanone

[134081-75-7]	$C_{25}H_{42}O_5$	mol. wt. 422.61
CH ₃ O OCH ₃ O OCH ₃ O OCH ₃ O	thoxy)-2,3,4-trimethox 10 % palladium on o	enation of (6-phenylme- yhexadecanophenone over charcoal in ethyl acetate/ the uptake of hydrogen

m.p. 59–60.5° [1353]; ¹H NMR [1353].

## 1-(4,7-Dihydroxy-2,3-dimethyl-6-benzofuranyl)-1-hexadecanone



-Obtained by acylation of 4,7-dimethoxy-2,3-dimethylbenzofuran in the presence of stannic chloride in benzene (73 %) [1040].

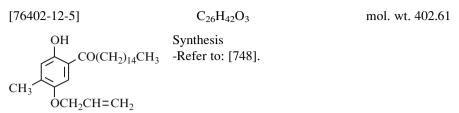
m.p. 72° [1040]; LD₅₀ [1040].

USE: Prepn. and radioprotective activity of, [1040].

## 1-[3,6-Dihydroxy-4-methyl-2-(2-propenyl)phenyl]-1-hexadecanone

 $[76402-13-6] C_{26}H_{42}O_3 \qquad \text{mol. wt. 402.61}$   $OH \qquad Synthesis \\ CO(CH_2)_{14}CH_3 \qquad -Obtained \qquad by \qquad Claisen \qquad rearrangement \qquad of \\ 1-[2-hydroxy-4-methyl-5-(2-propenyloxy)phenyl]- \\ CH_3 \qquad OH \qquad CH_2CH=CH_2 \qquad 1-hexadecanone \ [748].$ 

## 1-[2-Hydroxy-4-methyl-5-(2-propenyloxy)phenyl]-1-hexadecanone



## 1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-hexadecanone

$C_{26}H_4$	4O ₂ mol. wt. 388.63
(CH ₃ ) ₂ CH (CH ₃ ) ₂ CH (CH ₃ ) ₂ CH (CH ₃ ) ₂ CH	Synthesis -Obtained by reaction of hexadecanoic acid with thymol in the presence of aluminium chloride on heating at reflux for 12 h (81 %) [2960].

b.p.₁₃ 130–132° [2960]; m.p. 46° [2960].

## 1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-hexadecanone

(	$C_{26}H_{44}O_2$	mol. wt. 388.63
(CH ₃ ) ₂ CH	Synthesis -Refer to: [2660]. <b>Methyl ether</b> ( <b>XVII</b> ) C ₂₇ H ₄₆ O ₂	mol. wt. 402.66
$\dot{CO}(CH_2)_{14}CH_3$		

-Obtained by reaction of palmityl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (55 %) [2660].

b.p._{0.7} 235–240° [2660], b.p.₁₅ 280° [2660]; m.p. 51° [2660].

## 1-(4-Butoxy-2-hydroxyphenyl)-1-hexadecanone

[24313-92-6]	$C_{26}H_{44}O_3$	mol. wt. 404.63
CO(CH ₂ ) ₁₄ CH ₃	with m-dibutoxybenze	of hexadecanoyl chloride ne in the presence of alu- chloroethane at 0°. Then,

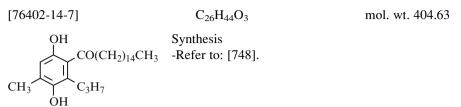
oride with m-dibutoxybenzene in the presence of aluminium chloride in dichloroethane at 0°. Then, the mixture was stirred for 1 h at 10°, and then for 6 h at 20–25°.

The temperature was then raised to 80° and stirring was continued at that temperature for 5 h (79 %) [1194, 1195].

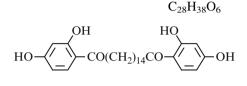
m.p. 48–49° [1194, 1195]; UV [1194, 1195].

USE: UV absorber [1194]; Light-stabilizer of polymeric materials [1194].

## 1-(3,6-Dihydroxy-4-methyl-2-propylphenyl)-1-hexadecanone



## 1,16-Bis(2,4-dihydroxyphenyl)-1,16-hexadecanedione



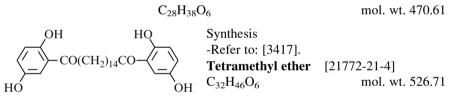
mol. wt. 470.61

Synthesis

-Obtained by reaction of 1,16-hexadecanedioic acid with resorcinol in the presence of zinc chloride at  $140^{\circ}$ for 5 h [445].

m.p. 162° [445].

## 1,16-Bis(2,5-dihydroxyphenyl)-1,16-hexadecanedione



-Obtained by reaction of thapsic acid dichloride with p-dimethoxybenzene in the presence of aluminium chloride in tetrachlorethane for 7 h at 100° (61 %) [3417].

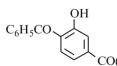
yellow needles [3417]; m.p. 78° [3417]; IR [3417].

## 1-(4-Benzoyl-3-hydroxyphenyl)-1-hexadecanone

4'-Benzoyl-3'-hydroxyhexadecanophenone

$$C_{29}H_{40}O_3$$

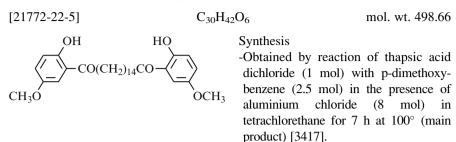
mol. wt. 436.63



Synthesis -Refer to: [1925]. UV [1925].

USE: Light stabilization of macromol. compds. [1925].

#### 1,16-Bis(2-hydroxy-5-methoxyphenyl)-1,16-hexadecanedione



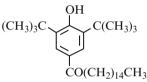
colourless needles [3417]; m.p. 157° [3417]; IR [3417].

#### 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-hexadecanone

[28441-03-4]  $C_{30}H_{52}O_{2}$ 

mol. wt. 444.74

mol. wt. 476.76



	50 52 2
	Syntheses
	-Preparation by reaction of hexadecanoyl chloride
	with 2,6-di-tert-butylphenol in the presence of alu-
	minium chloride [2145] in 1,2,2-trichloroethane at
3	$-10$ to $-20^{\circ}$ [951].
	m.p. 57–58° [951].

**O-Methyloxime** [169888-16-8]  $C_{31}H_{54}NO_2$  mol. wt. 472.78 -Refer to: [1408]; ESR [1408].

C₃₁H₅₂D₃NO₂

#### **O-d3-Methyloxime**

m.p. 37° [1408].

#### 1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-1-hexadecanone

[134082-07-8]	$C_{31}H_{46}O_5$	mol. wt. 498.70
011	0 1	

OF	
CH ₃ O	CO(CH ₂ ) ₁₄ CH
$CH_{3O}$	[∧] OCH ₂ C ₆ H ₅
5	2 0 5

Synthesis -Obtained by elimination of one phenylmethyl group in 2-position by treatment of 1-(2,6-dibenzyloxy-3,4-dimethoxyphenyl)-1-hexadecanone with concentrated hydrochloric acid and acetic acid at r.t. for 2–3 h (82 %) [1353].

m.p. 79–81° [1353]; ¹H NMR [1353].

## 1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]-1-hexadecanone

[134081-91-7]	$C_{31}H_{46}O_7S$	mol. wt. 562.77
$CH_{3}O \xrightarrow{OH} CO(CH_{2})_{14}CH_{3}$ $CH_{3}O \xrightarrow{OCH_{3}} OSO_{2}C_{6}H_{4}CH_{3}(p)$	Synthesis -Obtained by treatment 3,4,6-trimethoxyphenyl)-1- 25 % aluminium bromide r.t. for 2–3 h (90 %) [1353]	hexadecanone with e in acetonitrile at

m.p. 54–56° [1353]; ¹H NMR [1353].

## 1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-hexadecanone

[1103524-17-9]

C38H66O5

mol. wt. 602.94

mol. wt. 663.04

Synthesis OH CO(CH₂)₁₄CH₃ -Obtained by reaction of hexadecanoic CH₃(CH₂)₁₄CO acid with phloroglucinol in the presence of boron trifluoride etherate at  $100^{\circ}$  for 2 h (50-75 %) [338].

BIOLOGICAL ACTIVITY: As a new class of GPR40 (FFAR1) agonists [338].

## 1,1'-(4,4'-Dihydroxy[1,1'-biphenyl]-3,3'-diyl)-1-hexadecanone

C44H70O4 CO(CH₂)₁₄CH₃ Synthesis CH₃(CH₂)₁₄CC

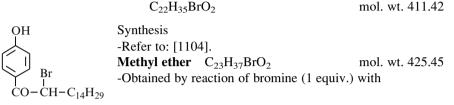
-Preparation by Fries rearrangement of 4,4'-biphenyl dihexadecanoate with aluminium chloride in refluxing chlorobenzene for 24 h (76 %) [2091].

m.p. 90-91° [2091].

#### 2 **Aromatic Hydroxyketones Derived** from Bromohexadecanoic Acids

#### Unsubstituted Hydroxyketones 2.1

## 2-Bromo-1-(4-hydroxyphenyl)-1-hexadecanone



4-methoxyhexanophenone in carbon tetrachloride [1104].

m.p. 79-80° [1104].

#### 2.2 Substituted Hydroxyketone

## 2-Bromo-1-(2-hydroxy-5-methylphenyl)-1-hexadecanone

	$C_{23}H_{37}BrO_2$	mol. wt. 425.45
$ \begin{array}{c} OH & Br \\ I \\ CO-CH-C_{14}H_{29} \end{array} $	Synthesis -Refer to: [190]. m.p. 46–47° [190].	

## **3** Aromatic Hydroxyketones Derived from 16-Oxohexadecanoic Acids

## 16-(2,4-Dihydroxyphenyl)-16-oxo-1-hexadecanoic acid

$$[720675-30-9] C_{22}H_{34}O_5$$
mol. wt. 378.51  
OH  
HO  
CO(CH₂)₁₄CO₂H  
Obtained by reaction of thapsic acid  
(tetradecamethylene-dicarbonic acid) with resor-  
cinol in the presence of zinc chloride at 140° for

(tetradecamethylene-dicarbonic acid) with resorcinol in the presence of zinc chloride at 140° for 5 h (40 %) [445].

m.p. 113–115° [445].

Methyl ester [720676-31-3] C₂₃H₃₆O₅ mol. wt. 392.54 b.p._{0.2} 260–270° [445]; m.p. 89–90° [445].

# Chapter 15 Heptadecanones

## 1 Aromatic Hydroxyketones Derived from Heptadecanoic Acids

## 1.1 Unsubstituted Hydroxyketones

## 1-(4-Hydroxyphenyl)-1-heptadecanone

[135649-79-5]	$C_{23}H_{38}O_2$	mol. wt. 346.55
ОН	Syntheses -Refer to: [3027, 3376].	
	USE: Electroluminescent device [3376].	
$CO(CH_2)_{15}CH_3$		
Methyl ether	$C_{24}H_{40}O_2$	mol. wt. 360.58
-Refer to: [1963].		
m.p. 70.5° [196	3].	
1-(2,5-Dihydroxy	phenyl)-1-heptadecanone	
[26639-19-0]	$C_{23}H_{38}O_3$	mol. wt. 362.55
OH	Syntheses	

OH Syntheses  $CO(CH_2)_{15}CH_3$  -Refer to: [1481, 1820]. USE: Diffusion of, in photographic emulsions, [1481]. OH **Dimethyl ether** [103044-77-5] C₂₅H₄₂O₃ mol. wt. 390.61

-Obtained by condensation of heptadecanoyl chloride with hydroquinone dimethyl ether [1820].

colourless prisms [1820]; m.p. 45-46° [1820]; IR [1820].

## 1-(3,4-Dihydroxyphenyl)-1-heptadecanone

	$C_{23}H_{38}O_3$	mol. wt. 362.55
OH ↓ ,OH	Syntheses -Refer to: [1175, 1962].	
<b>U</b>	m.p. 100–103° [1962].	
$\downarrow$ CO(CH ₂ ) ₁₅ CH ₃	<b>Dimethyl ether</b> [186454-86-4] C ₂₅ H ₄₂ O ₃	mol. wt. 390.61

-Obtained by reaction of n-hexadecylmagnesium bromide with 3,4-dimethoxybenzaldehyde in tetrahydrofuran under a stream of nitrogen. Then, the reaction mixture was refluxed gently for 20 h (39 %) [1175].

m.p. 67–68° [1960, 1963]; ¹H NMR [1175], ¹³C NMR [1175], MS [1175].

BIOLOGICAL ACTIVITY: Cytotoxicity [1175].

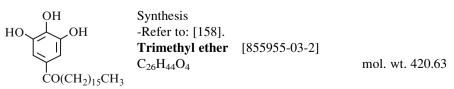
## 1-(3,5-Dihydroxyphenyl)-1-heptadecanone

 $\begin{array}{cccc} C_{23}H_{38}O_3 & \text{mol. wt. 362.55} \\ OH & Syntheses & \\ -Refer to: [3340, 3341]. \\ \textbf{Dimethyl ether} & [1186123-07-8] \\ CO(CH_2)_{15}CH_3 & C_{25}H_{42}O_3 & \text{mol. wt. 390.61} \end{array}$ 

USE: Preparation of benzoquinone natural product derivs. primin, AC-7-1, pallasone B, ardisianone A, irisquinone, dietrichequinone [3340]; Preparation of primin, AC-7, pallasone B, ardisianone A, irisquinone [3341].

## 1-(3,4,5-Trihydroxyphenyl)-1-heptadecanone

C₂₃H₃₈O₄ mol. wt. 378.55



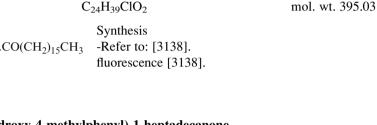
-Obtained by treatment of ethyl 2-(3,4,5-trimethoxybenzoyl)-2-pentadecylethanoate (m.p. 61.5–62°) with potassium hydroxide in boiling ethanol for 3 h (87 %) [158].

needles [158]; m.p. 72–72.5° [158].

**4-Nitrophenylhydrazone of the trimethyl ether**  $C_{32}H_{49}N_3O_5$  mol. wt. 555.76 yellow plates [158]; m.p. 111–112° [158].

## 1.2 Substituted Hydroxyketones

## 1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-heptadecanone



## 1-(3,5-Dihydroxy-4-methylphenyl)-1-heptadecanone

	$C_{24}H_{40}O_3$		mol. wt. 376.58
CO(CH ₂ ) ₁₅ CH ₃	Synthesis -Refer to: [158]. <b>Dimethyl ether</b> $C_{26}H_{44}O_3$	[855891-01-9]	mol. wt. 404.63

-Obtained by treatment of ethyl 2-(3,5-dimethoxy-4-methylbenzoyl)-2-pentadecylethanoate (m.p. 64–65°) with potassium hydroxide in boiling ethanol for 3 h (71 %) [158].

needles [158]; m.p. 76–77° [158].

HO.

4-Nitrophenylhydrazone of the dimethyl ether C₃₂H₄₉N₃O₄ mol. wt. 539.76

yellow plates [158]; m.p. 88–88.5° [158].

#### 1-(4-Ethoxy-2-hydroxyphenyl)-1-heptadecanone

$$\begin{array}{cccc} OH & Synthesis \\ -Refer to: [1834]. \\ Oxime & [33488-77-6] \\ C_{2}H_{5}O & C_{25}H_{43}NO_{3} \end{array} \quad mol. \ wt. \ 405.62 \end{array}$$

USE: Extraction agents, in copper manuf. [1834].

## 1-(2-Hydroxy-4-methoxy-3-methylphenyl)-1-heptadecanone

[855890-85-6]	$C_{25}H_{42}O_3$		mol. wt. 390.61
CH ₃ CH ₃ CH ₃ O	2,0-dimetnoxy-t	sence of alumini	adecanoyi chio-
m.p. 74.5–75.5° [158].			

4-Nitrophenylhydrazone

m.p. 95–96° [1421], 98–99° [158].

C31H47N3O4

## 1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1-heptadecanone

Synthesis

[855890-69-6]

C25H42O3

mol. wt. 390.61

mol. wt. 525.73

mol. wt. 525.73

OH	
	CO(CH ₂ ) ₁₅ CH ₂
CH ₃ 0	CH ₃

³ -Obtained Friedel-Crafts by acylation of 3,5-dimethoxy-toluene with heptadecanoyl chloride in the presence of aluminium chloride in carbon disulfide cooled in an ice bath. Then, the mixture let stand 2 h, heated 4 h at 60°, left overnight [158].

m.p. 71–72° [158].

4-Nitrophenylhydrazone C31H47N3O4

m.p. 114–115° [158], 123–124° [158].

## 1-(2-Hydroxy-4-methoxy-3,6-dimethylphenyl)-1-heptadecanone

[855954-88-0]	$C_{26}H_{44}O_3$	mol. wt. 404.63

	ОН
CH ₃	CO(CH ₂ ) ₁₅ CH ₃
CH ₃ O	[∞] CH ₃

Synthesis -Obtained by Friedel-Crafts acylation of 1,3-dimethoxy-2,5-dimethylbenzene with heptadecanoyl chloride in the presence of aluminium chloride in carbon disulfide (42 %) [158].

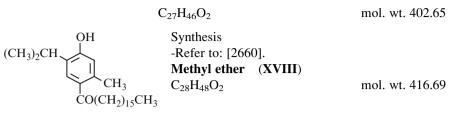
m.p. 57–58° [158].

4-Nitrophenylhydrazone m.p. 94–95° [158].

 $C_{32}H_{40}N_3O_4$ 

mol. wt. 539.76

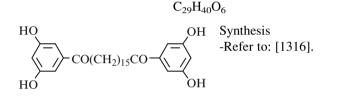
#### 1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-heptadecanone



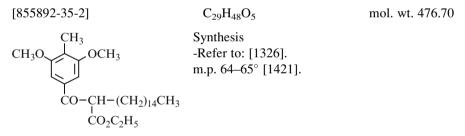
-Obtained by reaction of margaryl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (30 %) [2660].

 $b.p._{19} \ 303 - 305^{\circ} \ [2660]; \quad m.p. \ 55^{\circ} \ [2660].$ 

#### 1,17-Bis-(3,5-dihydroxyphenyl)-1,17-heptadecanedione



#### Ethyl 2-(3,5-Dimethoxy-4-methylbenzoyl)heptadecanoate



#### 1-(3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl)-1-heptadecanone

[645336-90-9]  
(CH₃)₃C 
$$\xrightarrow{OH} C(CH_3)_3 \xrightarrow{-1} C(CH_3)_3 \xrightarrow{-1} C(CH_2)_{15}CH_3 \xrightarrow{-1} CO(CH_2)_{15}CH_3 \xrightarrow{-1} CO(CH_2)_{15}CH_3 \xrightarrow{-1} CO(CH_2)_{15}CH_3 \xrightarrow{-1} CO(CH_2)_{15}CH_3 \xrightarrow{-1} C(CH_2)_{15}CH_3 \xrightarrow{-$$

 $C_{31}H_{54}O_2$ 

mol. wt. 458.77

mol. wt. 484.63

Synthesis Refer to: [1027].

USE: Stabilizer, dynamically-vulcanized blends of polyamide and silicone elastomers with high elongation and tensile strength [1027].

## 2 Aromatic Hydroxyketones Derived from 17-Bromoheptadecanoic Acid

## 17-Bromo-1-(4-hydroxy-3-methoxyphenyl)-1-heptadecanone

[13149-48-9]	$C_{24}H_{39}BrO_3$	mol. wt. 455.48
OH OCH ₃ CO(CH ₂ ) ₁₅ CH ₂ Br	Synthesis -Obtained by reaction of 17-bromohepta guaiacol in the presence of boron trifluo (73.5 %) [2750]. m.p. 77–78° [2750].	

Allyl ether	[13149-49-0]	$C_{27}H_{43}BrO_3$	mol. wt. 495.54
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-Obtained by reaction of allyl bromide with the title ketone in the presence of potassium carbonate in refluxing acetone for 9 h (84 %) [2750].

m.p. 81-84° [2750].

## 3 Aromatic Hydroxyketones Derived from 16-Methylheptadecanoic Acid

## 1-(2,4,5-Trihydroxyphenyl)-16-methyl-1-heptadecanone

1-(2,4,5-Trihydroxyphenyl)-1-isooctadecanone

 $\begin{array}{ccc} [123687-72-9] & C_{24}H_{40}O_4 & \mbox{mol. wt. } 392.58 \\ & OH & Synthesis \\ & HO & OH & \\ & OH & \\ \end{array}$ 

# Chapter 16 Octadecanones

## 1 Aromatic Hydroxyketones Derived from Octadecanoic Acids

## 1.1 Unsubstituted Hydroxyketones

## 1-(2-Hydroxyphenyl)-9-octadecyn-1-one

2'-Hydroxyphenyl heptadecyn-9, one-1

$C_{24}H_{36}$	O ₂ mol. wt. 356.55
$\bigcup_{i=1}^{OH} CO(CH_2)_7 C \equiv C - (CH_2)_7 CH_3$	Synthesis -Obtained by Fries rearrangement of phenyl stearolate with aluminium chloride at 115–120° for 2 h (43 %) [200].

 $\label{eq:2.3.4.1} \textbf{2,4-Dinitrophenylhydrazone} \quad [23803-76-1] \quad C_{30}H_{40}N_4O_5 \quad \text{mol. wt. 536.67}$ 

m.p. 185° [200].

## 1-(4-Hydroxyphenyl)-9-octadecyn-1-one

4'-Hydroxyphenyl heptadecyn-9, one-1

[23842-91-3]	$C_{24}H_{36}O_2$	mol. wt. 356.55
$\bigcup_{CO(CH_2)_7C}^{OH} \equiv C(CH_2)_7CH_3$	•	s rearrangement of phenyl minium chloride at 115–120°

**2,4-Dinitrophenylhydrazone** [23803-77-2] C₃₀H₄₀N₄O₅ mol. wt. 536.67

 $C_{24}H_{40}O_2$ 

m.p. 225° [200].

## 1-(2-Hydroxyphenyl)-1-octadecanone

[2589-85-7]

2.

mol. wt. 360.58

OH CO(CH₂)₁₆CH₃ Syntheses

-Obtained by reaction of stearoyl chloride with phenol in the presence of aluminium chloride, *in tetrachlorethane few hours at 55° (28 %) [2548]; *in nitrobenzene for 3 h at 70° (21 %) [2549];

*in carbon disulfide for 5.5 h at 47° (42 %) [2549].

-Also obtained by Fries rearrangement of phenyl stearate with aluminium chloride [1456] in tetrachlorethane for 10 h at 70° (18 %) [2550] or without solvent at 150° for 1 h [293].

-Also obtained by reaction of stearic acid with phenol in the presence of zinc chloride heated together on stand bath for 6 h [2398].

-Formation of, in stearic acid reactions with phenol, geochem. origin of alkyl aroms. in coal in relation to, [897].

-Also obtained by reaction of hydrogenated olive oil with phenol in the presence of boron trifluoride for 1.5-2 h at  $9-5^{\circ}$  [2599].

-Also refer to: [873, 2035].

b.p._{0.2} 164–182° [2035]; b.p.₁ 210–245° [2599]; m.p. 66–67° [293, 2398], 64–66° [2548], 60–64° [2550], 56–58° [2599]; TLC [1456].

Isolation from natural sources

-From karite or olive oils [2599].

USE: Textile rot proofing by, [873].

2,4-Dinitrophenylhydrazone C ₃₀ H	₄₄ N ₄ O ₅ mol. wt. 540.70
----------------------------------------------	-------------------------------------------------------------

m.p. 97.4–97.8° [293], 96–97° [2548].

## Methyl ether

 $C_{25}H_{42}O_2$ 

mol. wt. 374.61

-Obtained by reaction of dimethyl sulfate with 1-(2-hydroxyphenyl)-1-octadecanone in the presence of potassium carbonate in boiling acetone for 6 h [2398].

m.p. 42° [2398].

### 1-(3-Hydroxyphenyl)-1-octadecanone

[63442-88-6]	$C_{24}H_{40}O_2$		mol. wt. 360.58
CO(CH ₂ ) ₁₆ CH ₃	Syntheses -Refer to: [1984, 198 m.p. 62° [1984]; IF	-	
Methyl ether	[63442-85-3]	$C_{25}H_{42}O_2$	mol. wt. 374.61
-Refer to: [1984, 1985].			

m.p. 48° [1984]; IR [1984].

## 1-(4-Hydroxyphenyl)-1-octadecanone

[2589-77-7]

OH

 $C_{24}H_{40}O_{2}$ 

mol. wt. 360.58

Syntheses

-Obtained by reaction of stearoyl chloride with phenol in the presence of aluminium chloride,  $^{\circ}$  in tetrachlorethane few hours at 55° (28 %) [2548];  $^{\circ}$  in nitrobenzene for 3 h at 70° (67 %) [2549];  $^{\circ}$  in carbon disulfide for 5.5 h at 47° (54 %) [2549].

-Also obtained by Fries rearrangement of phenyl stearate,

*with aluminium chloride [1456] in nitrobenzene at  $38^{\circ}$  for 2 days (62 %) [414], in tetrachlorethane for 10 h at  $70^{\circ}$  (21 %) [2550] or without solvent at 150° for 1 h [293].

*in the presence of aluminium chloride in refluxing carbon disulfide for 5 h. Then, the mixture was kept at  $80-90^{\circ}$  for 2 h and at  $140-150^{\circ}$  for 1 h after solvent elimination [1222].

-Also obtained by reaction of stearic acid with phenol in the presence,

*of boron trifluoride for 2-3 h between 65 and  $85^{\circ}$  (70 %) [503];

*of activated acid clay catalyst at 190° for 2 h [3277].

-Also obtained by demethylation of 4-stearoylanisole with hydrobromic acid in acetic acid [2398].

-Also obtained by reaction of hydrogenated olive oil with phenol in the presence of boron trifluoride for 1.5-2 h at 5° [2599].

-Also refer to: [873, 1299, 1357, 1963, 1985, 2035, 2790, 2791].

b.p._{0.2} 209–212° [2035]; b.p.₁ 245–265° [2599]; b.p.₁₅ 320° [293]; m.p. 90–90.5° [293], 90° [2398], 89° [503], 87–89° [2548], 87–87.5° [3277], 84–87° [2550], 80–82° [2599], 76° [414]. TLC [1456].

Isolation from natural sources

-From karite or olive oils [2599].

USE: Polyamide fibers modified with, transparency of, [2790]; Nylon 6 monofilaments contg. transparency of, morphol. in relation to, [2791]; Textile rot proofing by, [873].

2,4-Dinitropheny	lhydrazone	$C_{30}H_{44}N_4O_5$	mol. wt. 540.70
m.p. 142–142.2° [293], 139.5–140° [2548].			
Semicarbazone	C ₂₅	$H_{43}N_3O_2$	mol. wt. 417.64
m.p. 133.4–134.7° [293].			
Benzoate	C ₃₁ H	44O ₃	mol. wt. 464.69
prisms [293]; m.p. 113.2–113.6° [293].			
Stearate	[122492-61-9]	$C_{42}H_{74}O_3$	mol. wt. 627.05

-Obtained by reaction of stearic acid with phenol in the presence of activated acid clay catalyst at  $190^{\circ}$  for 2 h [3277].

m.p. 90–91° [3277].

Methyl ether	[95869-30-0]	$C_{25}H_{42}O_2$	mol. wt. 374.61
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-Preparation by reaction of stearoyl chloride with anisole in the presence of aluminium chloride in nitrobenzene at r.t. overnight (90 %) [2398].

-Also obtained by treatment of anisole with octadecanoic anhydride or octadecanoyl chloride in the presence of sulfated zirconia [867].

-Synthesis of acylanisoles with Y zeolite catalyst [1941].

-Zeolite-catalysed Friedel-Crafts acylation of anisole [3264].

-Also refer to: [89, 1963, 2016, 2761 (59 %)].

m.p. 77–78° [2016], 77–77.5° [1963], 75° [2398, 2761].

## 2,4-Dinitrophenylhydrazone of the methyl ether C₃₁H₄₆N₄O₅ mol. wt. 554.73

m.p. 98–99° [2016].

USE: Films (unimol.) of, on D-cellobiose and quinol, [89].

2-Chloroethyl ether	$C_{26}H_{43}ClO_2$	mol. wt. 423.08
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-Obtained by reaction of octade canoyl chloride with 2-chloroethoxybenzene in the presence of a luminium chloride in carbon disulfide for 4 h at  $50^{\circ}$  (76 %) [476].

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m.p. 74–75° [476].
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## **N-Dimethylaminoethyl ether** $C_{28}H_{49}NO_2$ mol. wt. 431.70

-Obtained by reaction of 4-(2-chloroethoxy)octadecanophenone with dimethylamine in a sealed tube for 2 h at  $150^{\circ}$  [476].

free base: b.p._{0.3} 228° [476]; m.p. 42–43° [476]. hydrochloride: (69 %) [476]; m.p. 171° [476].

## **N-Diethylaminoethyl ether** [14392-84-8] C₃₀H₅₃NO₂ mol. wt. 459.76

-Preparation from 4-hydroxyoctadecanophenone, which is metalated and condensed with  $ClCH_2CH_2N(C_2H_5)_2$  (65 %) [414].

fumarate [414]; m.p. 71–72° [414].

#### 1-(2,4-Dihydroxyphenyl)-1-octadecanone

(4-Stearylresorcinol)

-Obtained by reaction of stearic acid with resorcinol, *in the presence of boron trifluoride for 2–3 h between 65 and 85° (95–98 %) [503]; *in the presence of zinc chloride (86–87 %) [2273], at 125–130° for 2 h [249].

-Also obtained by the Hoesch method (83 %) [2673].

-Also refer to: [16, 507, 859, 1272, 1412, 2247, 2673, 2959].

shiny colourless needles [507];

m.p. 99° [249, 503, 507], 98–99° [1412], 97° [2673], 96.5–97.5° [2247], 89–90° [859], 68–69° [2273].

**N.B.**: One of the reported melting point is obviously wrong. IR [1412], UV [1412].

USE: Protection against actinic radiations [2959].

4-Nitrophenylhydrazone	$C_{30}H_{45}N_3O_4$	mol. wt. 511.71	
m.p. 95–96° [859].			
4-(2-Propenyl) ether	$C_{27}H_{44}O_3$	mol. wt. 416.64	
USE: Protection against actinic radiations [2959].			
4-(2-Chloro-2-propenyl) ether	C ₂₇ H ₄₃ ClO ₃	mol. wt. 451.08	

USE: Protection against actinic radiations [2959].

m.p. 57.2–58.3° [2959]; UV [2959].

# 1-(2,5-Dihydroxyphenyl)-1-octadecanone

[4693-29-2]  $C_{24}H_{40}O_3$  mol. wt. 376.58 OH Syntheses -Obtained by reaction of stearic acid with hydroquinone in the presence of boron trifluoride etherate at 140° for 2 h (85 %) [142, 1992]. -Also obtained by treatment of 2-hydroxy-5-methoxy-

stearophenone with aluminium chloride [2226].

-Also obtained by reaction of octadecanoic acid with p-methoxyphenol in the presence of boron trifluoride without solvent for 5 h at  $95-100^{\circ}$  (11 %) [3184].

-Also obtained by treatment of hydroquinone and octadecanoic acid mixture in carbon tetrachloride below  $60^{\circ}$  with boron trifluoride, kept overnight at r.t., then heated 5 h at 90–95° (65 %) [3204].

-Also obtained by reaction of octadecanoyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride [156] in carbon disulfide [159].

-Also obtained by reaction of octadecanoic acid with p-methoxyphenol in the presence of boron trifluoride without solvent for 5 h at  $95-100^{\circ}$  (20 %) [3184], (11 %) [3185].

-Also refer to: [335, 394, 2251, 2349–2351, 3185].

m.p. 108–110° [142, 1992], 107–108° [3184, 3185], 106–108.5° [156, 159], 106° [3204].

USE: Photographic colour fog inhibitor [2349]; Photog. film contg., for inhibition of reaction of colour developer oxidn. product with colour-forming compds. [2350]; Colour photog. fog inhibitor, [2351]; Effect on photographic fogging in multilayered colour films [394].

**Dimethyl ether** [103048-59-5]  $C_{26}H_{44}O_3$  mol. wt. 404.63

-Obtained by reaction of octadecanoic acid chloride with hydroquinone dimethyl ether [1820] in tetrachloroethane in the presence of aluminium chloride (80 %) [1638].

-Also refer to: [714, 1637].

m.p. 56–56.5° [1820], 46° [714]; IR [1820].

# 1-(2,6-Dihydroxyphenyl)-1-octadecanone

[921758-91-0]	$C_{24}H_{40}O_3$	mol. wt. 376.58
OH CO(CH ₂ ) ₁₆ CH ₃	Synthesis -Refer to: [2320].	
ОН	USE: Black and white phototh image forming method using fl screen [2320].	C

#### 1-(3,4-Dihydroxyphenyl)-1-octadecanone

(4-Octadecanoylcatechol) (4-Stearoylpyrocatechol)

[1177-44-2]

$$C_{24}H_{40}O_3$$

mol. wt. 376.58

Syntheses -Obtained by Fries rearrangement of pyrocatechol distearate (m.p.  $83-85^{\circ}$ ) with aluminium chloride for 1 h at 110° [2646]. -Also obtained by treatment of a pyrocatechol and octadecanoic acid mixture with zinc chloride at 135–140° for 2 h (10 %) [1283].

-Also obtained by acylating pyrocatechol with octadecanoic acid at  $100^{\circ}$  for 3 h in the presence of boron trifluoride (85 %) [1605].

-Also obtained by reaction of octade canoyl chloride with pyrocatechol in the presence of aluminium chloride in nitroben zene, first at  $5-7^{\circ}$  for 2 h, then at r.t. for 2–3 days (66 %) [1726].

-Also refer to: [653, 985, 1521, 1604].

grey crystals [1605]; m.p. 100–101° [1283], 97–99° [1604, 1605], 70° [2646]. **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [1604, 1605, 1726], IR [1604, 1605], UV [985].

USE: As antioxidant for propene polymers [985].

Dimethyl ether	[501361-68-8]	$C_{26}H_{44}O_3$	mol. wt. 404.63

-Refer to: [1960, 1963].

m.p. 82–83° [1960, 1963].

USE: Formation of self-assembled monolayers on Sol-gel processed hafnium oxide as dielectric layers [3112].

#### 1-(2,3,4-Trihydroxyphenyl)-1-octadecanone

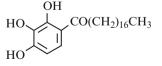
(4-Octadecanoylpyrogallol)

[103449-14-5]

 $C_{24}H_{40}O_4$ 

mol. wt. 392.58

Syntheses



.CO(CH₂)₁₆CH₃ -Obtained by reaction of stearic acid with pyrogallol, *in the presence of boron trifluoride for 2–3 h between 65 and 85° (95–98 %) [503];

*in the presence of zinc chloride (Nencki reaction) at  $135-140^{\circ}$  for 2 h (25 %) [1283].

-Also obtained by reaction of octade canoyl chloride with pyrogallol in the presence of aluminium chloride in nitroben zene, first at  $5-7^{\circ}$  for 2 h, then at r.t. for 2–3 days (40 %) [1726]. -Also refer to: [859, 1317, 2247, 2326].

m.p. 93–94° [2247], 93° [503], 91–93° [1283], 80–81° [859]. **N.B.**: One of the reported melting point is obviously wrong. ¹H NMR [1726].

USE: Colour photog. contg. antistaining agent from, [2326].

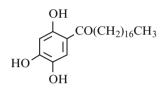
4-Nitrophenylhydrazone	C ₃₀ H ₄₅ N ₃ O ₅	mol. wt. 527.70
------------------------	---------------------------------------------------------------	-----------------

m.p. 154-155° [859].

#### 1-(2,4,5-Trihydroxyphenyl)-1-octadecanone

[57863-94-2]

C24H40O4 mol. wt. 392.58



Syntheses CO(CH₂)₁₆CH₃ -Preparation by Friedel-Crafts-type catalysts or Fries rearrangements of 1,2,4-trioctadecanoyloxybenzene with aluminium chloride in nitrobenzene [291, 292]. -Also obtained by reaction of stearonitril with phloroglucin (Hoesch reaction) [1608].

-Also refer to: [1708, 1728].

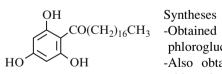
light-yellow [291]; m.p. 126–127° [1608], 118–119° [291, 292].

USE: Antioxidant [1708]; Antioxidant for fats and oils [291]; Antioxidant for fats, oils and paraffin waxes [292]; Heat stabilizers, for bicycloheptene polymers [1728]; Linear polyesters stabilization [1930].

**BIOLOGICAL ACTIVITY: Toxicity** [1708].

# 1-(2,4,6-Trihydroxyphenyl)-1-octadecanone

 $C_{24}H_{40}O_4$ 



CO(CH₂)₁₆CH₃ -Obtained by reaction of stearic nitrile with phloroglucinol (Hoesch reaction) [1608].

> -Also obtained by reaction of stearic acid with phloroglucinol in the presence of boron trifluoride for 2–3 h between 65 and 85° (95–98 %) [503].

m.p. 126–127° [1608], 126° [503].

# Monohydrate

C24H40O4, H2O

mol. wt. 410.59

mol. wt. 392.58

m.p. 118–120° [1608].

**Trimethyl ether** 

OH

HO

m.p. 67° [16].

### 1-(3,4,5-Trihydroxyphenyl)-1-octadecanone

$$\begin{array}{cccc} [180894-15-9] & C_{24}H_{40}O_4 & \mbox{mol. wt. } 392.58 \\ OH & Synthesis \\ HO & OH & -Refer to: [1639]. \\ & USE: Additive; silver halide colour photog. material [1639]. \end{array}$$

 $C_{27}H_{46}O_4$ 

_ _

CO(CH₂)₁₆CH₃

# 1-(2,4,6-Trihydroxyphenyl)-12,14-dihydroxyoctadecanone

(Byssomeruliol A and B)

.OF

[75656-31-4]  $C_{24}H_{40}O_{6}$ mol. wt. 424.59 [83212-66-2] [83213-39-2] Syntheses OH

OH OH OH Syntheses $CO(CH_2)_{10}$  CH  $CH_2$   $CH_2$  CH  $-(CH_2)_3$   $-CH_3$  -Refer to: [1930, 1931].ОН HO

¹H NMR [1930, 1931], ¹³C NMR [1930, 1931], IR [1930, 1931], UV [1930, 1931]. MS [1930, 1931].

Isolation from natural sources

- From fruit bodies and mycelia of Byssomerulius corium [1930, 1931].

#### 2,4,6-Trimethyl ether $C_{27}H_{46}O_{6}$

mol. wt. 466.66

-Refer to: [1930].

¹H NMR [1930], UV [1930], MS [1930].

# **Pentamethyl ether** [75679-83-3] [83212-56-0] C₂₉H₅₀O₆ mol. wt. 494.71

-Refer to: [1931].

MS [1931].

# 2,4,6-Triacetate

 $C_{30}H_{46}O_{9}$ 

mol. wt. 550.69

-Refer to: [1930].

¹H NMR [1930], ¹³C NMR [1930], UV [1930], MS [1930].

mol. wt. 434.66

#### 1-(2,4,6-Trihydroxyphenyl)-14-methoxy-1-octadecanone

 $C_{25}H_{42}O_5 \qquad \text{mol. wt. } 422.61$   $OH \qquad OCH_3 \qquad Synthesis \\ -Refer to: [1931]. \\ Trimethyl ether \\ C_{28}H_{48}O_5 \qquad \text{mol. wt. } 464.69$ 

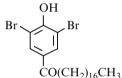
-Refer to: [1931].

MS [1326].

# 1.2 Substituted Hydroxyketones

#### 1-(3,5-Dibromo-4-hydroxyphenyl)-1-octadecanone

$$C_{24}H_{38}Br_2O_2$$
 mol. wt. 518.37

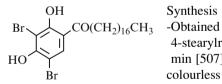


Synthesis -Obtained by reaction of bromine with 4-hydroxystearophenone in acetic acid (83 %) [501]. m.p. 80° [501].

# 1-(3,5-Dibromo-2,4-dihydroxyphenyl)-1-octadecanone

(2,6-Dibromo-4-stearylresorcinol)

 $C_{24}H_{38}Br_2O_3$  mol. wt. 534.37



Synthesis -Obtained by reaction of bromine with 4-stearylresorcinol in acetic acid at 37–38° for few min [507]. colourless leaflets [507]; m.p. 105° [507].

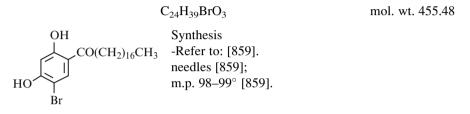
# 1-(3,5-Dichloro-2,6-dihydroxyphenyl)-1-octadecanone

 $[921758-93-2] C_{24}H_{38}Cl_2O_3 mol. wt. 445.46$  OH Synthesis -Refer to: [2320]. USE: Black and white photothermog. material and image forming method using fluorescent intensifying screen [2320].

# 1-(3-Bromo-4-hydroxyphenyl)-1-octadecanone

$$\begin{array}{ccc} C_{24}H_{39}BrO_2 & \mbox{mol. wt. 439.47} \\ OH & Synthesis \\ -Obtained by reaction of bromine with 4-hydroxy-stearophenone in acetic acid (82 %) [501]. \\ m.p. 96^{\circ} [501]. \\ CO(CH_2)_{16}CH_3 \end{array}$$

#### 1-(5-Bromo-2,4-dihydroxyphenyl)-1-octadecanone



# 1-(5-Bromo-2,3,4-trihydroxyphenyl)-1-octadecanone

$C_{24}H_{39}BrO_4$		mol. wt. 471.48
HO HO HO Br	Synthesis -Refer to: [859]. needles [859]; m.p. 86–87° [859].	

# 1-(3-Chloro-4-hydroxyphenyl)-1-octadecanone

	$C_{24}H_{39}ClO_2$	mol. wt. 395.03
ОН	Synthesis	
L CI	-Refer to: [3467].	
	Methyl ether	
$\mathbf{i}$	$C_{25}H_{41}ClO_2$	mol. wt. 409.05
CO(CH ₂ ) ₁₆ CH	I ₃	

-Obtained by reaction of stearoyl chloride with o-chloroanisole in the presence of aluminium chloride in petroleum ether at  $0^{\circ}$  for 1 h (10 %) [3467].

colourless needles [3467]; m.p. 56–57° [3467].

#### 1-(5-Chloro-2-hydroxyphenyl)-1-octadecanone

 $\begin{array}{ccc} C_{24}H_{39}ClO_2 & \text{mol. wt. 395.03} \\ & & \\ OH & & \\ CO(CH_2)_{16}CH_3 & -Refer to: [451]. \\ & & \\ Oxime, nickel complexes \\ & \\ USE: stabilizers, for propene polymers [451]. \end{array}$ 

#### 1-(3-Chloro-2,6-dihydroxyphenyl)-1-octadecanone

 $[921758-92-1] C_{24}H_{39}ClO_3 mol. wt. 411.02$   $OH CO(CH_2)_{16}CH_3 -Refer to: [2320].$  USE: Black and white photothermog. material and image forming method using fluorescent intensifying screen [2320].

#### 1-(4-Hydroxy-3-nitrophenyl)-1-octadecanone

 $\begin{array}{cccc} [70079-29-7] & C_{24}H_{39}NO_4 & \mbox{mol. wt. 405.58} \\ OH & Synthesis \\ & -Obtained by treatment of 4-octade can oylphenol with concentrated nitric acid in concentrated sulfuric acid at 0° for 1 h 15 min [1222]. \end{array}$ 

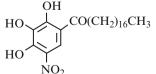
CO(CH₂)₁₆CH₃ m.p. 87.5–88.5° [1222].

#### 1-(2,4-Dihydroxy-5-nitrophenyl)-1-octadecanone

C ₂₄ H ₃₉ NO ₅		mol. wt. 421.58
HO NO ₂	Synthesis -Refer to: [859]. pale yellow prisms [859]; m.p. 97–98° [859].	

#### 1-(2,3,4-Trihydroxy-5-nitrophenyl)-1-octadecanone

 $C_{24}H_{39}NO_6$  mol. wt. 437.58



Synthesis CO(CH₂)₁₆CH₃ -Refer to: [859]. m.p. 95–96° [859].

#### 1-(2,6-Dihydroxy-4-methoxyphenyl)-6,9,12,15-tetraen-1-octadecanone

¹H NMR [1165], ¹³C NMR [1165], IR [1165], UV [1165], MS [1165].

# 1-(2-Hydroxy-3-methylphenyl)-9-octadecyn-1-one

2'-Hydroxy 3'-methyl phenyl heptadecyn-9, one-1

b.p.₆ 242° [200].

# **2,4-Dinitrophenylhydrazone** [23803-79-4] C₃₁H₄₂N₄O₅ mol. wt. 550.70 m.p. 205° [200].

#### 1-(2-Hydroxy-4-methylphenyl)-9-octadecyn-1-one

2'-Hydroxy 4'-methyl phenyl heptadecyn-9, one-1

[23803-80-7] 
$$C_{25}H_{38}O_2$$
 mol. wt. 370.58  
OH  
CO(CH₂)₇C  $\equiv$  C(CH₂)₇CH₃ Synthesis  
-Obtained by Fries rearrangement of  
m-cresyl stearolate with aluminium  
chloride at 115–120° for 2 h  
(40.5 %) [200].

b.p.3 210° [200].

# $\label{eq:2.4-Dinitrophenylhydrazone} \ensuremath{ [23803-81-8]} \ensuremath{ C_{31}H_{42}N_4O_5} \ensuremath{ mol. wt. 550.70} \ensuremath{$

m.p. 295° [200].

# 1-(2-Hydroxy-5-methylphenyl)-9-octadecyn-1-one

2'-Hydroxy 5'-methyl phenyl heptadecyn-9, one-1

[23803-82-9]	$C_{25}H_{38}O_2$	mol. wt. 370.58
$\bigcup_{\substack{I \\ CH_3}}^{OH} CO(CH_2)_7 C \equiv C(CH_2)_7 CH_3$	Synthesis -Obtained by Fries rearrange stearolate with aluminium 120° for 2 h (41 %) [200]. b.p. ₃ 220° [200].	1 .

# **2,4-Dinitrophenylhydrazone** [23803-83-0] C₃₁H₄₂N₄O₅ mol. wt. 550.70

m.p. 208° [200].

# 5-(1-Octadecanoyl)-2-hydroxybenzoic acid

[95269-86-6]	$C_{25}H_{40}O_4$	mol. wt. 404.59
COOH COOH CO(CH ₂ ) ₁₆ CH ₃	Syntheses -Obtained by reaction of stearoyl chloride w the presence of aluminium chloride in nitr *at r.t. for 12 h, then for 2 h on a water-bac *at 25° overnight (70 %) [1029].	obenzene,
-Also refer to: [21	70].	
m.p. 128–129° ¹ H NMR [1029	[1029], 117–119° [2784]; ], IR [1029].	
Methyl ether -Refer to: [2170];	C ₂₆ H ₄₂ O ₄ m.p. 194–196° [2170].	mol. wt. 418.62

Methyl ether and methyl ester	$C_{27}H_{44}O_4$	mol. wt. 432.64
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-Refer to: [2170]; m.p. 73–74° [2170].

Na salt	[95269-91-3]	C ₂₅ H ₃₉ O ₄ Na	mol. wt. 426.57
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-Obtained by treatment of the keto-acid above mentioned with 2.5 % aqueous sodium hydroxide [2784]. -Also refer to: [2170].

m.p. 240° (d) [2784].

mono-K salt	$C_{25}H_{39}O_4K$	mol. wt. 442.68
-Refer to: [2170];	m.p. 224–230° [2170].	

**N-methylmorpholine salt** [95269-97-9]  $C_{30}H_{51}NO_5$  mol. wt. 505.74 -Refer to: [2170]; m.p. 98° [2170].

Tris(2-hydroxyethyl)amine salt[95269-96-8] $C_{31}H_{55}NO_7$ mol. wt. 553.78-Refer to: [2170];m.p. 84° [2170].

Lysine salt	[95302-59-3]	$C_{31}H_{54}N_2O_6$	mol. wt. 550.78
-Refer to: [2170].			

# 1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-octadecanone

C	$C_{25}H_{41}ClO_2$	mol. wt. 409.05
CH ₃ CI	Synthesis -Obtained by reaction of steari 3-methylphenol in the presenc for 2–3 h between 65 and 85° m.p. 88° [503].	e of boron trifluoride

# 1-(2-Hydroxy-4-methylphenyl)-1-octadecanone

 $[909191-71-5] C_{25}H_{42}O_2 mtext{mol. wt. 374.61}$ OH  $CH_3$   $CO(CH_2)_{16}CH_3$   $CO(CH_2)_{16}CH_3$  Obtained by reaction of octadecanoic acid with m-cresol in the presence of graphite and methanesulfonic acid mixture at 120° for 2.5 h (85 %) [2834].-Also refer to: [451, 1111].

¹H NMR [2834], ¹³C NMR [2834], IR [2834].

USE: For nickel(2+) or cobalt(2+) extraction in kerosene. Extraction behaviour of nickel and cobalt has been carried out with LIX 84 and compared with the extractant newly synthesized [1111].

# Oxime, nickel complexes

USE: stabilizers, for propene polymers [451].

# 1-(2-Hydroxy-5-methylphenyl)-1-octadecanone

# 1-(4-Hydroxy-2-methylphenyl)-1-octadecanone

$$\begin{array}{cccc} & & & C_{25}H_{42}O_2 & & & mol. \ wt. \ 374.61 \\ & & & \\ OH & & Synthesis & & \\ & & -Refer \ to: \ [2503]. & & \\ & & & Phenyl \ ether \quad [791615-82-2] & & \\ & & & CH_3 & & C_{31}H_{46}O_2 & & & mol. \ wt. \ 450.71 \\ & & & CO(CH_2)_{16}CH_3 & & & \\ \end{array}$$

-Obtained by adding a mixture of m-phenoxytoluene and octadecanoyl chloride to a suspension of aluminium chloride in methylene chloride at  $0^{\circ}$ , then the mixture was stirred for 1.5–2 h at 3–5° (22 %) [2503].

b.p.₃ 280–282° [2503]; m.p. 49–50° [2503]; ¹H NMR [2503], IR [2503], MS [2503].

# 1-(4-Hydroxy-3-methylphenyl)-1-octadecanone

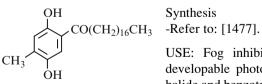
[114398-92-4]	$C_{25}H_{42}O_2$	mol. wt. 374.61
CO(CH ₂ ) ₁₆ CH ₃	Synthesis -Obtained by reaction of stearic acid with o- ence of boron trifluoride [142]. m.p. 72–73° [142].	cresol in the pres-

# 1-(2,4-Dihydroxy-5-methylphenyl)-1-octadecanone

[95185-60-7]	$C_{25}H_{42}O_3$	mol. wt. 390.61
OH CO(CH ₂ ) ₁₆ CH ₃	Synthesis -Refer to: [2704].	
HO CH ₃		

# 1-(2,5-Dihydroxy-4-methylphenyl)-1-octadecanone

[101649-67-6]	$C_{25}H_{42}O_3$	mol. wt. 390.61
ОН	Synthesis	



USE: Fog inhibitor, for diffusion-transfer heatdevelopable photothermog. materials contg. silver halide and benzotriazole deriv. silver salt and reductive developer [1477].

#### 1-(2-Hydroxy-4-methoxyphenyl)-1-octadecanone

$$[95869-36-6] C_{25}H_{42}O_3 mol. wt. 390.61$$
  
OH  
CO(CH₂)₁₆CH₃ Syntheses  
-Obtained by reaction of dimethyl sulfate with  
2,4-dihydroxystearophenone in carbon tetrachlo-  
ride in the presence of 40 % sodium hydroxide at  
70–75° [249].  
-Also prepared by reaction of stearoyl chloride with

1,3-dimethoxybenzene in the presence of aluminium chloride in chlorobenzene at  $10^{\circ}$ , and the temperature kept below  $20^{\circ}$  for 30 min [249].

m.p. 72–73° [249].

USE: Protect mineral and vegetable oils from oxidative degradation, by acting as an ultra-violet absorber [249].

#### 1-(2-Hydroxy-5-methoxyphenyl)-1-octadecanone

$$[35175-56-5]$$
  $C_{25}H_{42}O_3$  mol. wt. 390.61

**Syntheses** 

-Obtained by reaction of octadecanoic acid with p-methoxyphenol in the presence of boron trifluoride, *in tetrachloroethane for 4 h. The reaction mixture was allowed to stand overnight at r.t. and was then heated on a steam bath for about 3 h (46 %) [142];

*without solvent for 5 h at 95–100° (20 %) [3184, 3185].

-Also obtained by reaction of stearoyl chloride with 1,4-dimethoxybenzene in the presence of aluminium chloride [2226].

-Also obtained by reaction of octadecanoyl chloride with hydroquinone dimethyl ether in the presence of aluminium chloride [156] in carbon disulfide [159].

-Also obtained by acylating hydroquinone dimethyl ether with stearic acid (72 %) [3186].

b.p.₁₀ 187–191° [156]; light yellow crystals [3184, 3185]; m.p. 63–64° [156, 159], 62–63° [3184, 3185], 59.5–61.5° [142]; ¹H NMR [3184, 3185], IR [3184, 3185].

# 1-(4-Hydroxy-2,5-dimethylphenyl)-1-octadecanone

CH₃ 
$$CH_3$$
  $CH_3$   $CH_3$   $CO(CH_2)_{16}CH_3$   $CH_3$   $CO(CH_2)_{16}CH_3$ 

[95185-69-6]

 $C_{26}H_{44}O_{2}$ nthesis

mol. wt. 388.63

mol. wt. 404.63

#### 1-(4-hydroxy-3,5-dimethylphenyl)-1-octadecanone

[137832-99-6]	$C_{26}H_{44}O_2$	mol. wt. 388.63
CH ₃ CO(CH ₂ ) ₁₆ CH ₃	Syntheses -Obtained by reaction of o 2,6-dimethylphenol accordin previously [2871], (10 %) [1 -Also obtained by reaction of 2,6-dimethylphenol in the	ng to the method described 19]. octadecanoyl chloride with
	chloride [1832].	

-Also obtained by reaction of octadecanoic acid with 2,6-dimethylphenol in the presence of boron trifluoride [1832].

-Also refer to: [1733].

m.p. 62–62.5° [119]; ¹H NMR [119], IR [119].

### 1-(2-Ethoxy-4-hydroxyphenyl)-1-octadecanone

[76750-11-3]	$C_{26}H_{44}O_3$	mol. wt. 404.63
OH	Synthesis -Refer to: [2790].	
OC ₂ H ₅ CO(CH ₂ ) ₁₆ CH ₃	USE: Polyamide fibers modified with, trans	parency of, [2790].
1-5-Ethyl-2.4-dihydroxynhenyl)-1-octadecanone		

#### 1-5-Ethyl-2,4-dihydroxyphenyl)-1-octadecanone

	$C_{26}H_{44}O_3$
HO $C_2H_5$ $CO(CH_2)_{16}CH_3$	Synthesis -Refer to: [497]. m.p. 89–90° [497].

#### 1-(4-Ethoxy-2-hydroxyphenyl)-1-octadecanone

[22198-51-2]	$C_{26}H_{44}O_3$	mol. wt. 404.63
CO(CH ₂ ) ₁₆ CH ₃ C ₂ H ₅ O		

-Also obtained by reaction of octadecanoyl chloride with m-diethoxybenzene in the presence of aluminium chloride in dichloroethane at 0°. Then, the mixture was stirred for 1 h at  $10^{\circ}$ , and then for 6 h at  $20-25^{\circ}$ . The temperature was then raised to  $80^{\circ}$  and stirring was continued at that temperature for 5 h (66 %) [1194], (60–73 %) [3470].

-Also obtained by reaction of ethyl bromide with 2,4-dihydroxyoctadecanophenone in the presence of ethanolic potassium hydroxide (90–95 %) [2273]. -Also refer to: [2791].

m.p. 75–75.5° [3469, 3470], 70–70.3° [1194, 1195], 69–70° [2273]; UV [1194, 1195, 3469, 3470].

USE: UV absorber [1194]; Light-stabilizer of polymeric materials [1194]; Detn. of, by potentiometric titration in methylpyrrolidinone [1763]; Nylon 6 monofilaments contg. transparency of, morphol. in relation to, [2791].

Oxime	[33488-77-6]	$C_{26}H_{45}NO_3$	mol. wt. 419.65

-Refer to: [1569, 1834].

USE: Extn. by copper, model for, [1569, 1834].

# 1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-octadecanone

[134082-00-1]  $C_{26}H_{44}O_5$  mol. wt. 436.63

UH	Synthesis	
CO(CH ₂ ) ₁₆ CH ₃	-Preparation by treatment of 1-[6-hydroxy-3,4-di-	
	methoxy-2-(4-methylphenylsulfonyloxy)phenyl]-	
СН ₃ О ОН	1-hexadecanone with potassium carbonate in	
ÓCH ₃	refluxing methanol for 1–3 h (76 %) [1353].	

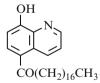
m.p. 86–87° [1353]; ¹H NMR [1353].

# 1-(8-Hydroxy-5-quinolinyl)-1-octadecanone

Syntheses

[110593-81-2]

 $C_{27}H_{41}NO_2$ 



-Preparation by Fries rearrangement of 8-hydroxyquinolinyl stearate using aluminium chloride as catalyst [992].

mol. wt. 411.63

-Also obtained by reaction of octade canoyl chloride with 8-hydroxyquinoline in the presence of a luminium chloride in nitrobenzene at  $80-85^{\circ}$  for 15–16 h (50 %) [1725].

-Also refer to: [993].

m.p. 83.2–85° [1725]; ¹H NMR [1725], IR [1725].

USE: Ion-flotation collector [1725]; Ion-flotation with, of gallium [1725].

mol. wt. 464.69

# 1-(4-Hydroxy-2,3,5-trimethylphenyl)-1-octadecanone

[137832-97-4]	$C_{27}H_{46}O_2$	mol. wt. 402.65
$CH_{3} \xrightarrow{OH} CH_{3} \xrightarrow{CH_{3}} CH_{3} \xrightarrow{CO(CH_{2})_{16}CH_{3}}$	phenol with octadecanoyl aluminium chloride	acylation of 2,3,6-trimethyl- chloride in the presence of in methylene chloride

#### 1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-octadecanone

[134081-69-9]	$C_{27}H_{46}O_5$	mol. wt. 450.66
CH ₃ O CH ₃ O CH ₃ O OCH ₃	crude 2,3,4,6-tetra	-
m.p. 72.5–74.5° [1353]; ¹ H	H NMR [1353].	

**p-Toluenesulfonic ester** [134081-84-8] C₃₄H₅₂O₇S mol. wt. 604.85

-Obtained by reaction of p-toluenesulfonyl chloride with 2-hydroxy-3,4,6-trimethoxy-octadecanophenone in the presence of potassium carbonate in refluxing acetone for 6 to 14 h (92 %) [1353].

m.p. 67.5–68.5° [1353]; ¹H NMR [1353].

# Methyl ether

-Obtained by reaction of octadecanoyl chloride with 1,2,3,5-tetramethoxybenzene in the presence of aluminium chloride in ethyl ether at  $0^{\circ}$  for 30 min [1353].

C₂₈H₄₈O₅

# 1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-octadecanone

[134081-76-8]	$C_{27}H_{46}O_5$	mol. wt. 450.66
CH ₃ O ^{OH} CO(CH ₂ ) ₁₆ CH ₃ OCH ₃ O ^{OCH} 3	Syntheses -Obtained by hydrogenatio 2,3,4-trimethoxyoctadeca palladium on charcoal in (1:1) until the uptake (92 %) [1353].	nophenone over 10 % n ethyl acetate/methanol

-Also refer to: [1351].

m.p. 64.5–66° [1353]; ¹H NMR [1353].

# 18-Hydroxy-1-(2-hydroxy-3,4-dimethoxy-6-methylphenyl)-1-octadecanone

[77712-02-8]	$C_{27}H_{46}O_5$	mol. wt. 450.66
CH ₃ O CH ₃ O CH ₃ O CH ₃ O CH ₃ O		eatment of its 18-acetyl ester droxide in methanol for 2 h at 7].
	[1147]; m.p. 101° [1147]; R [1147], MS [1147].	
18-Acetyl ester	[104988-70-7] C ₂₉ H	H ₄₈ O ₆ mol. wt. 492.97
-Obtained by Friedel-	Crafts reaction of 18-acetox	voctadecanovl chloride with

-Obtained by Friedel-Crafts reaction of 18-acetoxyoctadecanoyl chloride with 3,4,5-tri-methoxytoluene in 1,2-dichloroethane in the presence of aluminium chloride (2.1 mol) at 20° for 72 h [1147].

colourless oil [1147]; ¹H NMR [1147], IR [1147], MS [1147].

#### 1-(2-Acetyl-4-hydroxy-7-benzofuranyl)-1-octadecanone

	$C_{28}H_{42}O_4$	mol. wt. 442.64
$CO(CH_2)_{16}CH_3$		
$\sim 0$ $\sim COCH_3$	-Refer to: [682].	
	<b>Methyl ether</b> [59445-63-5]	
$\mathbf{i}$	$C_{29}H_{44}O_4$	mol. wt. 456.54
OH		

-Obtained by reaction of octade canoyl chloride with 2-acetyl-4-methoxy-benzofuran with a luminium chloride (2.4 mol) in methylene chloride at r.t. for 24 h (91 %) [682].

m.p. 94° [682].

#### 1-(2-Acetyl-7-hydroxy-4-benzofuranyl)-1-octadecanone

	$C_{28}H_{42}O_4$	mol. wt. 442.64
OH O COCH ₃	Synthesis -Refer to: [682].	
	<b>Methyl ether</b> [59445-74-8] $C_{29}H_{44}O_4$	mol. wt. 456.54
$CO(CH_2)_{16}CH_3$		

-Obtained by reaction of octade canoyl chloride with 2-acetyl-7-methoxy-benzofuran with a luminium chloride (2.4 mol) in methylene chloride at r.t. for 24 h (89 %) [682].

m.p. 96° [682].

# 1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-octadecanone

$$\begin{array}{cccc} & & & C_{28}H_{48}O_2 & & \mbox{mol. wt. 416.69} \\ & & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & &$$

USE: Palladium complex, singlet oxygen quenching by, [976].

#### Oxime, palladium complex

USE: Energy transfer to ligand-field states of, in aromatic hydrocarbon triplet state quenching [92].

# 1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-octadecanone

$C_{28}H_4$	₈ O ₂	mol. wt. 416.69
(CH ₃ ) ₂ CH (CH ₃ ) ₂ CH (CH ₃ ) ₂ CH (CH ₃ ) ₂ CH	Synthesis -Obtained by reaction of octa- thymol in the presence of al on heating at reflux for 12 h	luminium chloride
1 100 1000 [00/0]	460 [20(0]	

b.p.₁₃ 128–130° [2960]; m.p. 46° [2960].

# 1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-octadecanone

C	$C_{28}H_{48}O_2$	mol. wt. 416.69
(CH ₃ ) ₂ CH CH ₃ CO(CH ₂ ) ₁₆ CH ₃	Synthesis -Refer to: [2660]. <b>Methyl ether</b> (XIX) $C_{29}H_{50}O_2$	mol. wt. 430.71

-Obtained by reaction of stearoyl chloride with thymol methyl ether in the presence of aluminium chloride in carbon disulfide (30 %) [2660].

b.p.₂₄ 318° [2660], m.p. 59.5° [2660].

# 1-[4-(Butyloxy)-2-hydroxyphenyl]-1-octadecanone

[22198-50-1]	$C_{28}H_{48}O_3$	mol. wt. 432.69
C ₄ H ₉ O	Syntheses -Obtained by reaction of star resorcinol dibutyl ether in the ium chloride, *at 80° for 2 h (73 %) [3469 *first at 10–15°, then at 80–9	e presence of alumin-];

[41894-23-9]

-Also obtained by reaction of octadecanoyl chloride with m-dibutoxybenzene in the presence of aluminium chloride in dichloroethane at  $0^{\circ}$ . Then, the mixture was stirred for 1 h at  $10^{\circ}$ , and then for 6 h at  $20-25^{\circ}$ . The temperature was then raised to  $80^{\circ}$  and stirring was continued at that temperature for 5 h (73.5 %) [1194], (60-73 %) [3470].

m.p. 52.5-53° [1194, 1195, 3469, 3470]; UV [1194, 1195, 3469, 3470].

USE: UV absorber [1194]; Light-stabilizer of polymeric materials [1194].

# 1-(4-Hydroxy-2,3-dimethyl-5-(1-methylethyl)phenyl)-1-octadecanone

 $C_{29}H_{50}O_2$ [137833-02-4] mol. wt. 430.71 Synthesis  $(CH_3)_2CH$   $CH_3$   $CH_3$  -Refer to: [1733].

#### 1,18-Bis-(2-hydroxyphenyl)-1,18-octadecanedione

[115916-09-1]  $C_{30}H_{42}O_4$ mol. wt. 466.66 HO Synthesis -Refer to: [1188]. m.p. 112–112.5° [1188]. HO CO(CH₂)₁₆CO

**Di-2,4-dinitrophenylhydrazone** [117272-01-2] C₄₂H₅₀N₈O₁₀ mol. wt. 826.91

m.p. 150° [1188].

# 1,18-Bis-(4-hydroxyphenyl)-1,18-octadecanedione

[115915-64-5]

 $C_{30}H_{42}O_4$ 

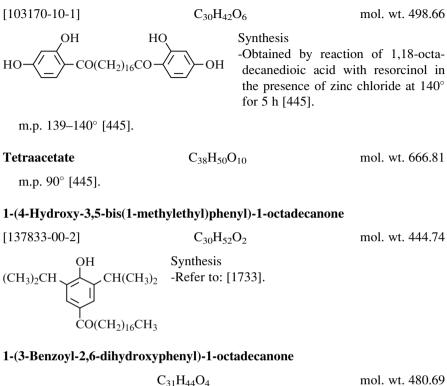
mol. wt. 466.66

HO - CO(CH₂)₁₆CO - OH Synthesis -Refer to: [1188].

m.p. 103.5° [1188].

 $\textbf{Di-2,4-dinitrophenylhydrazone} \hspace{0.2cm} [117271-96-2] \hspace{0.2cm} C_{42}H_{50}N_8O_{10} \hspace{0.2cm} \text{mol. wt. 826.91} \hspace{0.2cm}$ m.p. 170° [1188].

#### 1,18-Bis(2,4-dihydroxyphenyl)-1,18-octadecanedione



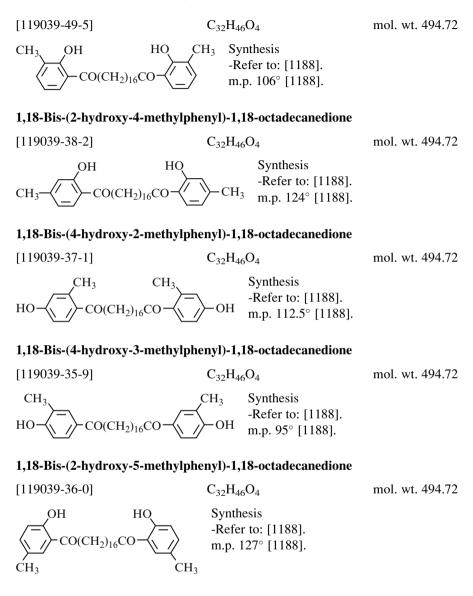
 $\begin{array}{c} \text{OH} \\ \text{CH}_3(\text{CH}_2)_{16}\text{CO} \\ \text{HO} \end{array} \begin{array}{c} \text{OH} \\ \text{I} \\ \text{HO} \end{array} \begin{array}{c} \text{Synthesis} \\ \text{-Refer to: [133].} \\ \text{USE: Stabilization of vinyl halide resins and} \\ \text{polyolefins against photodegradation [133].} \end{array}$ 

# 1-[2,3,4-Trihydroxy-6-(phenylmethoxy)phenyl]-1-octadecanone

	$C_{31}H_{46}O_5$	mol. wt. 498.70
ОН	Synthesis	
HO CO(CH ₂ ) ₁₆ CH ₃	-Refer to: [1353].	
	Trimethyl ether	
HO OCH ₂ C ₆ H ₅	$C_{34}H_{52}O_5$	mol. wt. 540.78

-Refer to: [1353].

# 1,18-Bis-(2-hydroxy-3-methylphenyl)-1,18-octadecanedione



# 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-octadecanone

[28459-33-8]	$C_{32}H_{56}O_2$	mol. wt. 472.80
(CH ₃ ) ₃ C	Syntheses -Preparation by reaction of with 2,6-di-tert-butylphene minium chloride in 1,1,2- -20° [951]. -Also refer to: [1733].	ol in the presence of alu-

m.p. 62–63° [951].

USE: Stabilize plastics, oils, and fats against heat, light, and oxidation [951].

K salt	$C_{32}H_{55}O_2K$		mol. wt. 510.89
-Refer to: [951].			
Acetate	[30392-07-5]	$C_{34}H_{58}O_3$	mol. wt. 514.83
-Refer to: [228].			
b.p. ₁₁ 305–308°	[228].		
Methyl ether	[30492-53-6]	$C_{33}H_{58}O_2$	mol. wt. 486.82
-Refer to: [228].			
b.p. ₁₁ 300–305°	[228].		

# 1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-1-octadecanone

[134082-08-9]	$C_{33}H_{50}O_5$	mol. wt. 526.76
CH ₃ O CH ₃ O CH ₃ O CH ₂ C ₆ H ₅	Synthesis -Obtained by elimination group in 2-position 1-(2,6-dibenzyloxy-3,4-dia decanone with concentrate acetic acid at r.t. for 2–3 h	by treatment of methoxyphenyl)-1-octa- ed hydrochloric acid and

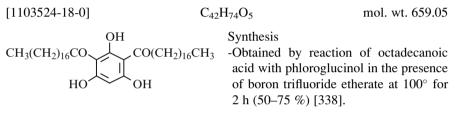
m.p. 83–84° [1353]; ¹H NMR [1353].

# 1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]-1-octadecanone

[134081-92-8]	$C_{33}H_{50}O_7S$	mol. wt. 590.82
$CH_{3}O \xrightarrow{OH} CO(CH_{2})_{16}CH_{3}$ $CH_{3}O \xrightarrow{OCH_{3}} OSO_{2}C_{6}H_{4}CH_{3}(p)$	Synthesis -Obtained by treatment 3,4,6-trimethoxyphenyl)-1-0 25 % aluminium bromide r.t. for 2–3 h (72 %) [1353].	in acetonitrile at

m.p. 58–60° [1353]; ¹H NMR [1353].

#### 1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-octadecanone



BIOLOGICAL ACTIVITY: As a new class of GPR40 (FFAR1) agonists [338].

#### 1-(2,3-Dihydroxy-5-octadecylphenyl)-1-octadecanone

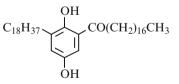
[74061-22-6]	$C_{42}H_{76}O_3$	mol. wt. 629.06
HO HO CO(CH ₂ ) ₁₆ CH ₃	Syntheses -Refer to: [1605, 2761].	
C ₁₈ H ₃₇	m.p. 59–60° [1604, 1605]; ¹ H NMR [1604, 1605], IR [1604, 1605].	

#### 1-(2,5-Dihydroxy-3-octadecylphenyl)-1-octadecanone

[72306-96-8]



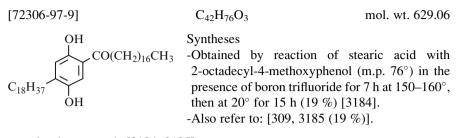
mol. wt. 629.06



OH  $C_{18}H_{37}$   $CO(CH_2)_{16}CH_3$   $CO(CH_2)_{16}CH_3$  OB  $C_{18}H_{37}$   $CO(CH_2)_{16}CH_3$  OB  $CO(CH_2)_{16}C$ 

greyish crystals [3184, 3185]; m.p. 85–86° [3184, 3185]; ¹H NMR [3184, 3185], IR [3184, 3185].

#### 1-(2,5-Dihydroxy-4-octadecylphenyl)-1-octadecanone



colourless crystals [3184, 3185]; m.p. 101° [309], 100–102° [3184, 3185]; ¹H NMR [3184, 3185], IR [3184, 3185].

### 1-(4,5-Dihydroxy-2-octadecylphenyl)-1-octadecanone

	$C_{42}H_{76}O_3$	mol. wt. 629.06
он но, 🙏	Synthesis -Refer to: [3112].	
(CH ₂ ) ₁₇ CH ₃ CO(CH ₂ ) ₁₆ CH ₃	<b>Dimethyl ether</b> [1111287-73-0]	mol. wt. 657.10

-Refer to: [3112].

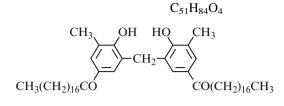
USE: Formation of self-assembled monolayers on Sol-gel processed hafnium oxide as dielectric layers [3112].

#### 1-(2-Hydroxy-5-methoxy-3-octadecylphenyl)-1-octadecanone

[72306-95-7]	$C_{43}H_{78}O_3$	mol. wt. 643.09
C ₁₈ H ₃₇ OCH ₃ OCH ₃	Syntheses -Obtained by reaction 2-octadecyl-4-methoxypl presence of boron trifluor (37 %) [3185], then (26 %) [3184].	ride for 7 h at $150-160^{\circ}$

light-yellow crystals [3184, 3185]; m.p. 74–75° [3184, 3185]; ¹H NMR [3184, 3185], IR [3184, 3185].

### 1,1'-[Methylenebis(2-hydroxy-3-methyl-5,1-phenylene)]bis-1-octadecanone

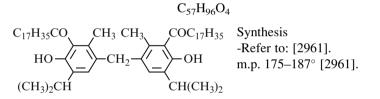


Syntheses -Obtained by reaction of octadecanoyl chloride with bis (2-hydroxy-3-methylphenyl) methane according to the method described

previously [2871], (65 %) [119].

m.p. 87.4–90° [119]; ¹H NMR [119], IR [119].

# 1,1'-[Methylenebis[4-hydroxy-5-(1-methylethyl)-2-methyl-3,1-phenylene]]bis-1-octadecanone



# 1,1'-[Thiobis(2-hydroxy-5-pentyl-3,1-phenylene)]bis-1-octadecanone

3',3"'-Thiobis[2'-hydroxy-5'-pentyl-octadecanophenone

$$C_{58}H_{98}O_4S \qquad \text{mol. wt. 891.48}$$

$$CH_3(CH_2)_{16}CO \qquad OH HO \qquad CO(CH_2)_{16}CH_3 \qquad Syntheses \\ -Refer to: [825, 826]. \qquad USE: Polycarboxylic acid esters of oxypropylated, [825];$$

Polycarboxylic acid esters of oxypropylated, in breaking petroleum emulsions [826].

# 1,1'-[Thiobis(6-hydroxy-5-pentyl-3,1-phenylene)]bis-1-octadecanone

3',3"'-Thiobis[6'-hydroxy-5'-pentyl-octadecanophenone

$$CH_{3}(CH_{2})_{16}CO \xrightarrow{CO(CH_{2})_{16}CH_{3}} Syntheses \\ -Refer to: [825, 826]. \\ USE: Polycarboxylic acid esters of oxypropylated, [825]; \\ C_{5}H_{11} OH HO C_{5}H_{11} of oxypropylated, [82]; \\ C_{5}H_{11} OH HO C_{5}H_{11} of oxypropylated, [82]; \\ C_{5}H_{11} of oxyprop$$

Polycarboxylic acid esters of oxypropylated, in breaking petroleum emulsions [826].

1089

mol. wt. 761.23

mol. 845.39

# 2 Aromatic Hydroxyketones Derived from Various Halogenooctadecanoic Acids

# 9,10,12,13,15,16-Hexabromo-1-(3,4-dihydroxyphenyl)-1-octadecanone

 $[100559-75-9] C_{24}H_{34}Br_6O_3 mol. wt. 849.96$  OH Synthesis -Refer to: [3093].  $CO(CH_2)_7CHBrCHBrCH_2CHBrCHBrCH_2CHBrCHBrCH_2CH_3$ 

Dimethyl ether	[100559-74-8]	$C_{26}H_{38}Br_6O_3$	mol. wt. 878.01
Dimentyl culci	[100339-74-0]	$C_{26} I_{38} D_{16} C_{3}$	moi. wt. 676.01

-Obtained by reaction of 9,10,12,13,15,16 hexabromooctanoyl chloride with veratrole in the presence of aluminium chloride in tetrachloroethylene at 120° (15.8 %) [3097].

-Also refer to: [3093].

USE: Useful as intermediate for urushiol [3097].

# 9,10,12,13-Tetrabromo-1-(3,4-dihydroxyphenyl)-1-octadecanone

[100559-78-2]	$C_{24}H_{36}B_{10}$	$r_4O_3$	mol. wt. 692.16
OH OH CO(CH ₂ ) ₇ CHBrCl	HBrCH ₂ CHBrCHBrC ₅ F	dimethyl ether tribromide in	treatment of its below with boron methylene chloride %) [3095].
Dimethyl ether	[100559-77-1]	$C_{26}H_{40}Br_4O_3$	mol. wt. 720.22

-Obtained by reaction of 9,10,12,13-tetrabromooctadecanoyl chloride with veratrole in the presence of aluminium chloride in refluxing carbon disulfide (42.6 %) [3095, 3096].

# 9-Chloro-1-(3,4-dihydroxyphenyl)-1-octadecanone

 $[97911-40-5] C_{24}H_{39}ClO_3 mtext{mol. wt. 411.02}$ 

and

# 10-Chloro-1-(3,4-dihydroxyphenyl)-1-octadecanone

# Chapter 17 Nonadecanones

# 1 Aromatic Hydroxyketones Derived from Nonadecanoic Acids

# 1.1 Unsubstituted Hydroxyketones

# 1-(2,5-Dihydroxyphenyl)-1-nonadecanone

 $\begin{array}{c} C_{25}H_{42}O_3 & \text{mol. wt. 390.61} \\ \\ OH & \\ CO(CH_2)_{17}CH_3 & -Refer \text{ to: } [1820]. \\ Dimethyl \ ether \ [103099-04-3] \\ C_{27}H_{46}O_3 & \text{mol. wt. 418.66} \end{array}$ 

-Obtained by condensation of nonadecanoyl chloride with hydroquinone dimethyl ether [1820].

colourless prisms [1820]; m.p. 55-56° [1820]; IR [1820].

# 2,4-Dinitrophenylhydrazone of the methyl ether

[103325-22-0]	$C_{33}H_{50}N_4O_6$	mol. wt. 598.78
red prisms [1820];	m.p. 81–82° [1820].	

#### 1-(3,5-Dihydroxyphenyl)-1-nonadecanone

$$\begin{array}{cccc} C_{25}H_{42}O_3 & \text{mol. wt. 390.61} \\ OH & Syntheses & \\ -Refer to: [2876, 2877, 2948]. \\ \textbf{Dimethyl ether } [22168-75-8] \\ HO & CO(CH_2)_{17}CH_3 & C_{27}H_{46}O_3 & \text{mol. wt. 418.66} \end{array}$$

-Obtained by reaction of octadecylmagnesium bromide with 3,5-dimethoxybenzamide in ether (56%) [2876, 2948]. -Refer to: [2877].

m.p. 69–71° [2948], 67–71° [2876].

# 1-(3,5-Dihydroxyphenyl)-2-nonadecanone

[142611-25-4]	$C_{25}H_{42}O_3$	mol. wt. 390.61
HO CH ₂ CO(CH ₂ ) ₁₆ CH ₃	Isolation from natural sources -From etiolated rice seedlings	[3003].

# 1-(2,3,4-Trihydroxyphenyl)-1-nonadecanone

[114808-66-1]	$C_{25}H_{42}O_4$	mol. wt. 406.61
OH HO CO(CH ₂ ) ₁₇ CH ₃	Syntheses -Refer to: [2714–2716].	
но	USE: Thermal recording material IR-absorbing image [2715]; Therm rials contg. higher fatty acid meta for improved image contrast and	al recording mate- l double salts and,

and optical readability [2714].

# 1-(3,4,5-Trihydroxyphenyl)-1-nonadecanone

[87667-31-0]	$C_{25}H_{42}O_4$	mol. wt. 406.61
ОН НО ОН	Syntheses -Refer to: [1327, 2229].	
CO(CH ₂ ) ₁₇ CH ₃	USE: Hydrophilic colloid layer contg., at [2229]; Colour mixing effect prevention films [1327].	

**Trimethyl ether** 

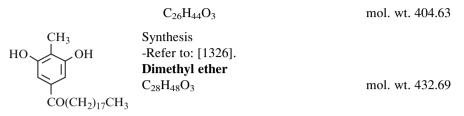
C₂₈H₄₈O₄ r

mol. wt. 448.69

m.p.  $78^{\circ}$  [1326].

#### Substituted Hydroxyketones 1.2

#### 1-(3,5-Dihydroxy-4-methylphenyl)-1-nonadecanone

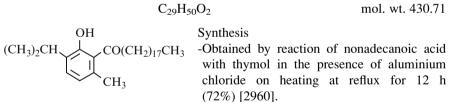


-Refer to: [1326]; m.p. 82° [1326].

#### 2-(3,5-Dihydroxy-4-methylbenzoyl)nonadecanoic acid ethyl ester

	$C_{29}H_{48}O_5$	mol. wt. 476.70
СН3 НО ОН	Synthesis -Refer to: [1326].	
	<b>Dimethyl ether</b> C ₃₁ H ₅₂ O ₅	mol. wt. 504.75
$\dot{CO}$ -CH-(CH ₂ ) ₁₆ $\dot{CO}$ -CH-(CH ₂ ) ₁₆	CH ₃ m.p. 70.5° [1326].	

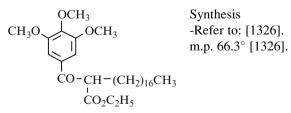
#### 1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-nonadecanone



b.p.₁₆ 131–133° [2960]; m.p. 44.5° [2960].

#### 2-(3,4,5-Trimethoxybenzoyl)nonadecanoic acid ethyl ester

$$C_{31}H_{52}O_6$$
 mol. wt. 520.75



**1-[4-Hydroxy-3-[[2-hydroxy-3-methyl-5-(1-oxooctadecyl)phenyl]methyl]-5-methyl-phenyl]-1-nonadecanone** (Chemical Abstracts)

18-[4-Hydroxy-3-(2-hydroxy-3-methyl-5-nonadecanoylbenzyl)-5-methylphenyl]-octadecanal (IUPAC)

[169553-39-3]  $C_{52}H_{86}O_4$  mol. wt. 775.24  $CH_3 \longrightarrow CH_2 \longrightarrow CH_2 \longrightarrow CH_2$  -CH₂ -

# Chapter 18 Eicosanones

# 1 Aromatic Hydroxyketones Derived from Eicosanoic Acids

# 1.1 Unsubstituted Hydroxyketones

# **1-(2,4,6-Trihydroxyphenyl)-5,8,11,14,17-eicosapentaen-1-one** (all *Z*)

 $[79553-90-5] C_{26}H_{34}O_4 mol. wt. 410.55$   $OH \\ CO(CH_2)_3(CH = CHCH_2)_5CH_3 \\HO OH \\ OH \\ OH$  Isolation from natural sources -From Zonaria tournefortii (Lamour) Mont. (Dictyotaceae) [102].

pale yellow oil [102]; ¹H NMR [102, 103], ¹³C NMR [102, 103, 1103], IR [102, 1103], UV [102, 1103], MS [102, 1103]; TLC [102].

# 1-(3,4-Dihydroxyphenyl)-1-eicosanone

[151029-60-6]

 $C_{26}H_{44}O_3$ 

mol. wt. 404.63

OH OH CO(CH₂)₁₈CH₃

Isolation from natural sources -From *Plectranthus sylvestris* [1551].

BIOLOGICAL ACTIVITY: Refer to: [1551].

#### 1-(2,3,4-Trihydroxyphenyl)-1-eicosanone

[96070-21-2]  $C_{26}H_{44}O_{4}$ mol. wt. 420.63 **Syntheses** OH CO(CH₂)₁₈CH₃ -Obtained by reaction of arachidic acid with pyrogal-HO lol in the presence of boron trifluoride and hydrogen HO fluoride in xylene on a water-bath at  $70^{\circ}$ (90 %) [505].

-Also refer to: [500].

b.p.₁₄ 308–310° [500]; m.p. 101° [500], 99° [505].

BIOLOGICAL ACTIVITY: Potential activity against lethal radiations [505].

#### 1-(2,4,6-Trihydroxyphenyl)-1-eicosanone

[79553-91-6] mol. wt. 420.63  $C_{26}H_{44}O_4$ Synthesis OH  $CO(CH_2)_{18}CH_3$  -Obtained by mild catalytic hydrogenation of 1-(2,4,6-trihydroxyphenyl)-1-eicosanone, 5,8,11,14,17-pentaene [102].

Isolation from natural sources

-From the brown alga Zonaria tournefortii [102].

m.p. 136° [102]; ¹H NMR [102], IR [102], UV [102], MS [102, 581].

#### 1-(3,4,5-Trihydroxyphenyl)-1-eicosanone

	$C_{26}H_{44}O_4$	mol. wt. 420.63
ОН НО , , ОН	Synthesis -Refer to: [1326].	
	<b>Trimethyl ether</b> $C_{29}H_{50}O_4$ -Refer to: [1326].	mol. wt. 462.71

m.p. 82° [1326].

#### 1-(2,4,6-Trihydroxyphenyl)-15-hydroxy-1-eicosanone

2-Eicosanoylphloroglucinol decahydro derivative

C26H44O5 OH CO(CH₂)₁₃ - CH - (CH₂)₄CH₃ Synthesis OH -Refer to: [103]. OH

mol. wt. 436.63

#### 2-(3,4,5-Trihydroxybenzoyl)eicosanoic acid ethyl ester

# 1-(2,5-Dihydroxyphenyl)-2-octadecyl-1-eicosanone

	$C_{44}H_{80}O_3$	mol. wt. 657.10
$\bigcup_{\substack{I \\ OH \\ C_{18}H_{37} \\ OH}}^{OH} OH$	Synthesis -Refer to: [2225]. <b>Dimethyl ether</b> [125697-50-9] $C_{46}H_{84}O_3$ -Refer to: [2225].	mol. wt. 685.17

# 1.2 Substituted Hydroxyketones

# 1-(4-Hydroxy-3-methoxyphenyl)-1-eicosanone

	$C_{27}H_{46}O_3$	mol. wt. 418.65
OH OCH ₃ CO(CH ₂ ) ₁₈ CH ₃	Synthesis -Refer to: [2369]. m.p. 70.5–71.5° [2369].	

#### 1-(2-Hydroxy-4,6-dimethoxyphenyl)-1,3-eicosanedione

ω-n-Octadecanoyl-2-hydroxy-4,6-dimethoxyacetophenone

 $\begin{array}{c} C_{28}H_{46}O_5 & \text{mol. wt. } 462.67 \\ OH & Synthesis \\ COCH_2CO(CH_2)_{16}CH_3 & -\text{Refer to: } [3167]. \\ CH_{3}O & OCH_3 & \text{m.p. } 94-96^{\circ} \ [3167]. \end{array}$ 

mol. wt. 490.72

# 20-Hydroxy-1-(2-hydroxy-3,4-dimethoxy-6-methylphenyl)-1-eicosanone

[104966-98-5]	C ₂₉ H ₅₀ O	5	mol. wt. 478.71
CH ₃ O CH ₃ O CH ₃ O CH ₃ O CH ₃ O	with so	ed by treatment of	of its 20-acetyl ester n methanol for 2 h at
colourless needles [1147]; m.p. 105° [1147]; ¹ H NMR [1147], IR [1147], MS [1147].			
20-Acetyl ester	[104966-94-1]	$C_{31}H_{52}O_6$	mol. wt. 520.75
-Obtained by Friedel-Crafts reaction of 20-acetoxyeicosanoyl chloride with 3,4,5-trimethoxy-toluene in 1,2-dichloroethane in the presence of aluminium chloride (2.1 mol) at $20^{\circ}$ for 72 h [1147].			
colourless oil [114 IR [1147], ¹ H NM	7]; R [1147], MS [1147].		

# 1-(2-Hydroxy-4, 6-dimethoxy-3, 5-dimethylphenyl)-1, 3-eicosanedione

 $C_{30}H_{50}O_{5}$   $CH_{3} \qquad OH \qquad Synthesis$   $CH_{3} \qquad COCH_{2}CO(CH_{2})_{16}CH_{3} \qquad -Refer to: [3167].$   $CH_{3}O \qquad CH_{3} \qquad CH_{3}$ 

# 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-eicosanone

[28441-04-5]	$C_{34}H_{60}O_2$	mol. wt. 500.85
$(CH_3)_3C$ $(CH_3)_3C$ $(CH_3)_3$ $(C(CH_3)_3)_3C$ $(C(CH_2)_{18}CH_3)_3$	Synthesis -Refer to: [951]. m.p. 74–76° [951].	

# 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-3-eicosanone

[54685-34-6]	$C_{34}H_{60}O_2$	mol. wt. 500.85
$(CH_3)_3C$ $C(CH_3)_3$	Synthesis -Refer to: [2758].	
CH ₂ CH ₂ COC ₁₇ H ₃₅	USE: Reaction of, with ammonium carbonate hydantoins [2758].	•

# **Chapter 19** Heneicosanones

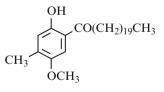
# **1** Aromatic Hydroxyketones Derived from Heneicosanoic Acids

#### 1-(2,5-Dihydroxy-4-methylphenyl)-1-heneicosanone

[13736-51-1] C₂₈H₄₈O₃ mol. wt. 432.69 Synthesis OH CO(CH₂)₁₉CH₃ -Obtained by hydrolysis of 5-heneicosanoate with a mixture (1:1) concentrated hydrochloric acid/ ethanol for 8 h (13.5 %) [3416]. CH yellow needles [3416]; OH m.p. 95–97° [3416]; IR [3416]. 5-Heneicosanoyl ether [13736-50-0] C₄₉H₈₈O₄ mol. wt. 741.24 m.p. 45–48° [3416]; IR [3416].

#### 1-(2-Hydroxy-5-methoxy-4-methylphenyl)-1-heneicosanone

Synthesis



[13736-49-7]

-Obtained by Friedel-Crafts condensation of heneicosanoyl chloride with 2,5-dimethoxytoluene in the presence of aluminium chloride in refluxing carbon disulfide for 20 h (63.2 %) [3416].

pale yellow plates [3416]; m.p. 82-83° [3416]; IR [3416].

# 1-(5-Heneicosanoyloxy-2-hydroxy-4-methylphenyl)-1-heneicosanone

2,5-Dihydroxy-4-methylheneicosanophenone 5-heneicosanoate

yellow needles [3416]; m.p. 45–48° [3416]; IR [3416].

# 2 Aromatic Hydroxyketones Derived from Bromoheneicosanoic Acid

# 9,10,12,13,15,16-Hexabromo-1-(3,4-dihydroxyphenyl)-1-heneicosanone

[100559-80-6]	$C_{27}H_{40}Br_6O_3$	mol. wt. 892.04
OH OH CO(CH ₂ ) ₇ CHBrCHBrCI	H ₂ CHBrCHBrCH ₂ CHBrCHBr	Synthesis -Refer to: [3094]. C ₅ H ₁₁

Dimethyl ether	[100568-25-0]	$\mathrm{C}_{29}\mathrm{H}_{44}\mathrm{Br}_{6}\mathrm{O}_{3}$	mol. wt. 920.09

-Refer to: [3097].

# Chapter 20 Docosanones

# 1 Aromatic Hydroxyketones Derived from Docosanoic Acids

## 1.1 Unsubstituted Hydroxyketones

### 1-(2-Hydroxyphenyl)-13-docosen-1-one

2'-Hydroxy phenyl heneicosen-13, one-1 [200]

$C_{28}H_{46}O_2$	mol. wt. 414.67
$CO(CH_2)_{11}-CH=CH(CH_2)_7CH_3$	Synthesis -Obtained by Fries rearrangement of phe- nyl erucate with aluminium chloride at 115–120° for 2 h (27 %) [200].

 $C_{34}H_{50}N_4O_5$ 

2,4-Dinitrophenylhydrazone

m.p. 140° [200].

### 1-(4-Hydroxyphenyl)-13-docosen-1-one

4'-Hydroxy phenyl heneicosen-13, one-1 [200]

$C_{28}H_{46}O_2$	mol. wt. 414.67
Synthesis	

OH  $CO(CH_2)_{11}CH = CH(CH_2)_7C$ 

-Obtained by Fries rearrangement of phenyl erucate with aluminium chloride at  $115-120^{\circ}$  for 2 h (7 %) [200].

 $CO(CH_2)_{11}CH = CH(CH_2)_7CH_3$  b.p.₁₀ 260° [200].

1103

mol. wt. 594.79

mol. wt. 432.69

2,4-Dinitrophenylhydrazone  $C_{34}H_{50}N_4O_5$ mol. wt. 594.79 m.p. 167° [200]. 1-(4-Hvdroxyphenvl)-1-docosanone mol. wt. 416.69 [47660-65-1] C28H48O2 OH Synthesis -Obtained by reaction of behenic acid with phenol in the presence of boron trifluoride for 2–3 h between 65 and  $85^{\circ}$ (75 %) [503]. CO(CH₂)₂₀CH₃ m.p. 96–97° [503].

[103396-94-7] C₃₄H₅₃N₃O₂ mol. wt. 535.81 iso-Nicotinylhydrazone m.p. 131° [520, 521].

USE: In chemotherapy of leprosy [520, 521].

#### **Polymer with formaldehyde**

USE: Point depressant for fuel oil [1344].

#### 1-(2,4-Dihydroxyphenyl)-1-docosanone

(4-Behenylresorcinol)

[856360-21-9]

HO

OH

C₂₈H₄₈O₃

**Synthesis** -Obtained by reaction of behenic acid with resorcinol

in the presence of zinc chloride at 150-160° for 4 h [507].

colourless needles [507]; m.p. 103° [507].

### 1-(3,4-Dihydroxyphenyl)-1-docosanone

 $CO(CH_2)_{20}CH_3$ 

(4-Behenylcatechol)

C28H48O3 mol. wt. 432.69

Syntheses

OH -Obtained by reaction of behenic acid with pyrocatechol, ·OH *in the presence of zinc chloride (Nencki reaction) (10 %) [507]; *in the presence of boron trifluoride at  $80^{\circ}$  (75 %) [507].  $CO(CH_2)_{20}CH_3$ colourless prisms [507]; m.p. 103° [507].

BIOLOGICAL ACTIVITY: Protection against whole-body irradiation [1810]; Protection against X-rays [1811].

[27029-47-6]

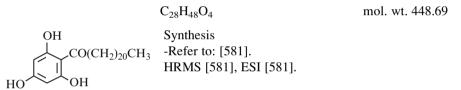
#### 1-(2,3,4-Trihydroxyphenyl)-1-docosanone

(4-Behenylpyrogallol)

[103279-18-1]	$C_{28}H_{48}O_4$	mol. wt. 448.69
OH HO HO HO	Syntheses -Obtained by reaction of be in the presence of zinc ch (70 %) [507]. -Also refer to: [1810, 1811	loride at 150–160° for 4 h
colourless needles [507]; m.p. 100–101° [507].	b.p. _{0.2} 250° [507];	

BIOLOGICAL ACTIVITY: Protection against whole-body irradiation [1810]; Protection against X-rays [1811]; Radiation damage prevention by, [2778].

#### 1-(2,4,6-Trihydroxyphenyl)-1-docosanone

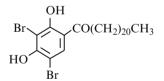


## 1.2 Substituted Hydroxyketones

#### 1-(3,5-Dibromo-2,4-dihydroxyphenyl)-1-docosanone

(2,6-Dibromo-4-behenylresorcinol)

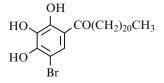
$C_{28}H_{46}Br_2O_3$	mol. wt. 590.48
-----------------------	-----------------



Synthesis -Obtained by reaction of bromine with 4-behenylresorcinol in acetic acid at 37–38° for few min (92 %) [507]. colourless needles [507]; m.p. 110° [507].

#### 1-(5-Bromo-2,3,4-trihydroxyphenyl)-1-docosanone

(6-Bromo-4-behenylpyrogallol)



Synthesis -Obtained by reaction of bromine with 4-behenylpyrogallol in acetic acid at r.t. (85 %) [507]. colourless needles [507]; m.p. 99° [507].

#### 1-(2-Hydroxy-3-methylphenyl)-13-docosen-1-one

2'-Hydroxy 3'-methyl phenyl heneicosen-13, one-1 [200]

b.p.₃ 225° [200].

 $\label{eq:constraint} \textbf{2,4-Dinitrophenylhydrazone} \qquad C_{35}H_{52}N_4O_5 \qquad \qquad \text{mol. wt. } 608.82$ 

m.p. 139° [200].

### 1-(2-Hydroxy-4-methylphenyl)-13-docosen-1-one

2'-Hydroxy 4'-methyl phenyl heneicosen-13, one-1 [200]

$C_{29}H_{48}O_2$	mol. wt. 428.70
$CO(CH_2)_{11}CH = CH(CH_2)_7CH_3$ $CH_3$	Synthesis -Obtained by Fries rearrangement of m-cresyl erucate with aluminium chloride at 115–120° for 2 h (33 %) [200].

b.p.₄ 230° [200].

 $\label{eq:constraint} \textbf{2,4-Dinitrophenylhydrazone} \qquad C_{35}H_{52}N_4O_5 \qquad \qquad \text{mol. wt. } 608.82$ 

m.p. 130° [200].

#### 1-(2-Hydroxy-5-methylphenyl)-13-docosen-1-one

2'-Hydroxy 5'-methyl phenyl heneicosen-13, one-1 [200]

$$C_{29}H_{48}O_2$$
 mol. wt. 428.70

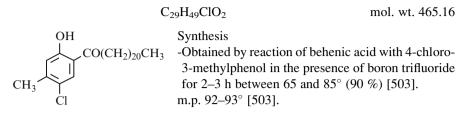
OH	Synthesis
$CO(CH_2)_{11}CH = CH(CH_2)_7CH_3$	-Obtained by Fries rearrangement of
	p-cresyl erucate with aluminium chloride at 115–120° for 2 h (39 %) [200].
ĊH ₃	b.p. ₄ 243° [200].

#### 2,4-Dinitrophenylhydrazone

C₃₅H₅₂N₄O₅ mol. wt. 608.82

m.p.  $110^{\circ}$  [200].

#### 1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-docosanone



### 1-(2-Hydroxy-5-methylphenyl)-1-docosanone

 $\begin{array}{c} C_{29}H_{50}O_2 \\ OH \\ \downarrow \\ CO(CH_2)_{20}CH_3 \\ CH_3 \end{array} \begin{array}{c} Synthesis \\ -Obtained by reaction of behenic acid with p-cresol in the presence of boron trifluoride for 2–3 h between 65 and 85° (90–95 %) [503]. \\ m.p. 80–81° [503]. \end{array}$ 

Methyl ether[103398-74-9] $C_{30}H_{52}O_2$ mol. wt. 444.74

-Obtained by refluxing a mixture of 2-hydroxy-5-methylbehenophenone and methyl iodide in the presence of potassium hydroxide for 2 h [518].

silky needles [370]; m.p. 76° [518].

### 1-(4-Hydroxy-3-methylphenyl)-1-docosanone

[95185-70-9]  $C_{29}H_{50}O_2$  mol. wt. 430.71 OH Synthesis -Refer to: [2704].  $CO(CH_2)_{20}CH_3$ 

### 1-(4-Hydroxy-2,5-dimethylphenyl)-1-docosanone

[95185-61-8]

 $C_{30}H_{52}O_2$ 

mol. wt. 444.74

CH₃ CH₃ CO(CH₂)₂₀CH₃ Synthesis -Refer to: [2704].

#### 1-(4-Hydroxy-2,3,5-trimethylphenyl)-1-docosanone

[137833-01-3]  $C_{31}H_{54}O_2$  mol. wt. 458.77 OH Synthesis  $CH_3 + CH_3 - CH_3$  -Refer to: [1733].  $CO(CH_2)_{20}CH_3$ 

#### 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-docosanone

# 2 Aromatic Hydroxyketones Derived from 22-Oxodocosanoic Acid

#### 22-(2-Hydroxy-3,4-dimethoxy-6-methylphenyl)-22-oxo-1-docosanoic acid

21-(2-Hydroxy-3,4-dimethoxy-6-methylbenzoyl)heneicosanoic acid

[79330-93-1]	$C_{31}H_{52}O_6$	mol. wt. 520.75
$CH_{3O} \xrightarrow{OH} CO(CH_{2})_{20}CO_{2}H$ $CH_{3O} \xrightarrow{CH_{3}O} CH_{3}$	Synthesis -Obtained by reaction of eicosanoate with 3,4,5 the presence of aluminium zene first at 0°, then at r.	-trimethoxytoluene in m chloride in nitroben-
m.p. 103–105° [2325]; IR [2	.325].	

Methyl ether[84978-10-9] $C_{32}H_{54}O_6$ mol. wt. 534.78

-Obtained by reaction of ethyl chloroformyl-eicosanoate with 3,4,5-trime-thoxytoluene in the presence of aluminium chloride in nitrobenzene first at  $0^{\circ}$ , then at r.t. [2325].

m.p. 88–90° [2325]; IR [2325].

Methyl ester [77711-94-5] C₃₂H₅₄O₆ mol. wt. 534.78

Methyl 21-(2-hydroxy-3,4-dimethoxy-6-methylbenzoyl)heneicosanoate

-Obtained by esterification of the title acid [2325].

m.p. 75–76° [2325]; ¹H NMR [1432], IR [2325].

# Chapter 21 Tricosanones

# 1 Aromatic Hydroxyketones Derived from Tricosanoic Acid

# 1.1 Unsubstituted Hydroxyketones

### 1-(3,4,5-Trihydroxyphenyl)-1-tricosanone

 $\begin{array}{cccc} [155084-01-8] & C_{29}H_{50}O_4 & \mbox{mol. wt. 462.71} \\ & & \\ HO & & \\ & & \\ HO & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ &$ 

USE: Blotting-resistant thermographic recording sheet containing [2083]; Thermographic recording sheet for high sensitive and plasticizer-resistant images [2084]; Thermal recording material containing electron donor from, [2082].

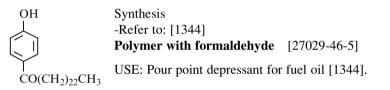
# Chapter 22 Tetracosanones

# 1 Aromatic Hydroxyketones Derived from Tetracosanoic Acids

# 1.1 Unsubstituted Hydroxyketones

### 1-(4-Hydroxyphenyl)-1-tetracosanone

C₃₀H₅₂O₂ mol. wt. 444.74



## 1-(3,4-Dihydroxyphenyl)-1-tetracosanone

[191284-02-3]

 $C_{30}H_{52}O_3$ 

mol. wt. 460.74



Synthesis -Refer to: [2215].

USE: Thermal printing material for images with good solvent resistance [2215].

#### 1-(2,4,6-Trihydroxyphenyl)-1-tetracosanone

 $\begin{array}{c} C_{30}H_{52}O_4 & \text{mol. wt. 476.74} \\ OH & Synthesis \\ + OH & CO(CH_2)_{22}CH_3 & -Refer to: [581]. \\ HRMS [581]; & ESI [581]. \end{array}$ 

### 4-(24-Hydroxy-1-oxo-5-n-propyltetracosanyl)phenol

 $C_{33}H_{58}O_{3} \text{ mol. wt. 502.82}$ OH Synthesis -Refer to: [2092].  $C_{3}H_{7} \text{ Isolation from natural sources} -From the shoots of$ *Leucas aspera*Spreng [2092].m.p. 80–81° [2092]; ¹H NMR [2092], ¹³C NMR [2092], IR [2092], UV [2092], MS [2092]; ( $\alpha$ )³⁵_D = -10.4° (ethanol) [2092].

 Diacetate
 C₃₇H₆₂O₅
 mol. wt 586.90

 m.p. 68–70° [2092];
 IR [2092], MS [2092].

# 1.2 Substituted Hydroxyketones

## 1-(4-Hydroxy-3-methylphenyl)-1-tetracosanone

 $[95102-15-1] C_{31}H_{54}O_2 mol. wt. 458.77$ 

OH Syntheses -Refer to: USE: Colo

-Refer to: [1595, 2704].

USE: Colour developer, for thermal recording materials [1595].

| CO(CH₂)₂₂CH₃

# Chapter 23 Pentacosanones

# 1 Aromatic Hydroxyketones Derived from Pentacosanoic Acid

# 1.1 Unsubstituted Hydroxyketones

### 1-(4-Hydroxyphenyl)-5-propyl-24-methoxy-1-pentacosanone

4-(24-Methoxy-24-methyl-1-oxo-5-n-propyltetracosanyl)phenol

	$C_{35}H_{62}O_3$	mol. wt. 530.88
ОН		Synthesis -Refer to: [1545].
CO(CH ₂ ) ₃	$C_{3}H_{7}$ OCH ₃ -CH - (CH ₂ ) ₁₈ - CH - CH ₃	Isolation from natural sources -From <i>Mimosa pudica</i> Linn [1545].
¹ H NMR	–113° [1545]; [1545], ¹³ C NMR [1545], II – 6.9° (ethanol) [1545].	R [1545], UV [1545], MS [1545];
Acetate	C ₃₇ H ₆₄ C	D ₄ mol. wt. 572.91

# m.p. 88–90° [1545]; IR [1545], MS [1545].

# Chapter 24 Hexacosanones

# 1 Aromatic Hydroxyketones Derived from Hexacosanoic Acid

# 1.1 Unsubstituted Hydroxyketone

### 1-(2,4,6-Trihydroxyphenyl)-1-hexacosanone

HO OH CO(CH₂)₂₄CH₃

Synthesis ,CO(CH₂)₂₄CH₃ -Refer to: [581]. HRMS [581]; ESI [581].

C32H56O4

mol. wt. 504.79

# Chapter 25 Triacontanones

# 1 Aromatic Hydroxyketones Derived from Triacontanoic Acid

## 1.1 Unsubstituted Hydroxyketone

### 1-(2,4,6-Trihydroxyphenyl)-1-[24-triacontenone

2,4,6-Trihydroxyphenyl-(24*Z*)-triacontene-1-one (*Tenulphenone C*)

C36H62O4

mol. wt. 558.89

OH Synthesis  $CO(CH_2)_{22}CH = CH(CH_2)_4CH_3$  -Refer to: [1513]. HO OH

Isolation from natural sources -From the cortexes of *Polygala tenuifolia* [1513].

m.p. 197–200° [1513]; ¹H NMR [1513], ¹³C NMR [1513], IR [1513].

# Chapter 26 Dotriacontanones

# 1 Aromatic Hydroxyketones Derived from Dotriacontanoic Acid

## 1.1 Unsubstituted Hydroxyketone

#### 1-(2,4,6-Trihydroxyphenyl)-1-[24-dotriacontenone

2,4,6-Trihydroxyphenyl-(24Z)-dotriacontene-1-one (*Tenulphenone D*)

C38H66O4

mol. wt. 586.94

OH  $CO(CH_2)_{24}CH = CH(CH_2)_4CH_3$  -Refer to: [1513]. HO OH

Isolation from natural sources. -From the cortexes of *Polygala tenuifolia* [1513].

m.p. 197–200° [1513]; ¹H NMR [1513], ¹³C NMR [1513], IR [1513].

# **Common Abbreviations**

Å	Angström units
b.p.	Boiling point (for example, b.p. _{0.1} $100^{\circ}$ means boils at $100^{\circ}$ if the
¹³ C	pressure is 0.1 mmHg) Nuclear Magnetic Resonance relative to carbon 13
(d)	Decomposition
20°	20 degrees Celsius
d	Density (for example, $d_{20}$ specific gravity at 20 °C referred to water et 4 °C)
equiv.	Equivalent
(E)	Geometric stereodescriptor used for compounds having achiral ele- ments resulting from double bonds where the groups of highest pri- ority are on the opposite side of the vertical reference plane
¹⁹ F	Nuclear Magnetic Resonance relative to fluorine 19
GC	Gaz chromatography
GC-MS	Gaz chromatography-mass spectroscopy
GLC	Gaz-liquid chromatography
h	Hour
¹ H NMR	Nuclear Magnetic Resonance relative to proton
HPLC	High Pressure Liquid Chromatography
IR	Infrared spectra
iso-	Aliphatic hydrocarbon having two methyl groups on the terminal carbon atom of the chain (for example, (CH ₃ ) ₂ CH-CH ₂ -CH ₂ -)
LD ₅₀	Median lethal dose, the quantity of a chemical that is estimated to be fatal to 50 % of the organisms tested
m-	Meta-
М	Molar (concentration)
min	Minute
Mol	Molecule
mol. wt.	Molecular weight

m.p.	Melting point
MS	Mass spectra
n-	Normal (as n-butyl)
Ν	Normal (equivalents per liter, as applied to concentration)
N.B.	Nota Bene
n _D ²⁵	Index of refraction (for 25° and sodium light)
nm	Nanometre
0-	Ortho-
p-	Para-
Pd/C	Palladium on charcoal
рК _а	Log of the reciprocal of the dissociation constant
r.t.	Room temperature
Sadtler	Sadtler Research Laboratories, Philadelphia (USA)
sec-	Secondary-
SM	Starting Material
tert-	Tertiary-
TLC	Thin Layer Chromatography
UV	Ultraviolet spectra
vol.	Volume
(Z)	Opposite of (E)

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- 5-(2,4-Dihydroxyphenyl)-5-oxo-1-pentanoic acid, 580
- 5-(2,5-Dihydroxyphenyl)-5-oxo-1-pentanoic acid, 581
- 5-(3,4-Dihydroxyphenyl)-5-oxo-1-pentanoic acid, 582
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- 4-(2,4,5-Trihydroxyphenyl)-3-methyl-4-oxo-1-butanoic acid, 418
- 4-(2,3-Dihydroxy-4-methoxyphenyl)-4-oxo-1-butanoic acid, 441
- 5-(2,3,4-Trihydroxyphenyl)-5-oxo-1-pentanoic acid, 582
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- $C_{11}H_{13}BF_2O_2$
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1-(2,3-Dichloro-4-hydroxyphenyl)-2-ethyl-1-butanone, 171 1-(2,3-Dichloro-4-methoxyphenyl)-3-methyl-1-butanone, 185 1-(2,3-Dichloro-4-hydroxyphenyl)-3,3-dimethyl-1-butanone, 252 1-(3,5-Dichloro-4-hydroxyphenyl)-3,3-dimethyl-1-butanone, 252 1-(2,3-Dichloro-4-methoxyphenyl)-1-pentanone, 479 1-(2,3-Dichloro-4-hydroxyphenyl)-4-methyl-1-pentanone, 546 5-Chloro-1-(5-chloro-2-methoxyphenyl)-1-pentanone, 572 1-(3,5-Dichloro-2-hydroxyphenyl)-1-hexanone, 621 1-(3,5-Dichloro-4-hydroxyphenyl)-1-hexanone, 621 1-(4,6-Dichloro-2-hydroxyphenyl)-1-hexanone, 622  $C_{12}H_{14}Cl_2O_3$ 1-(3,5-Dichloro-2,6-dihydroxyphenyl)-1-hexanone, 622  $C_{12}H_{14}Cl_2O_4$ 1-(3,5-Dichloro-2,4-dihydroxy-6-methoxyphenyl)-1-pentanone, 487 1-(3.5-Dichloro-2.6-dihvdroxy-4-methoxyphenyl)-1-pentanone, 487 1-(3,5-Dichloro-2,4,6-trihydroxyphenyl)-1-hexanone, 622 C12H14F2O2 1-(2,3-Difluoro-4-methoxyphenyl)-1-pentanone, 480 C₁₂H₁₄FIO₂ 1-(3-Fluoro-4-hydroxy-5-iodophenyl)-1-hexanone, 623  $C_{12}H_{14}I_2O_2$ 1-(2-Hydroxy-3,5-diiodo-4,6-dimethylphenyl)-1-butanone, 61 1-(4-Hydroxy-3,5-diiodophenyl)-1-hexanone, 623  $C_{12}H_{14}O_2$ 1-(4-Hydroxyphenyl)-2-methylene-1-pentanone, 477  $C_{12}H_{14}O_3$ 1-(2-Acetyloxyphenyl)-1-butanone, 2 1-(3-Acetyloxyphenyl)-1-butanone, 3 1-(4-Acetyloxyphenyl)-1-butanone, 5 1-(2-Ethoxyphenyl)-1,3-butanedione, 310 1-(2-Methoxy-5-methylphenyl)-1,3-butanedione, 317

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1-(2,4-Dihydroxyphenyl)-1,4-hexanedione, 597 1-(3,4-Dihydroxyphenyl)-1,4-hexanedione, 597 6-(2-Hydroxyphenyl)-6-oxo-1-hexanoic acid, 709 6-(4-Hydroxyphenyl)-6-oxo-1-hexanoic acid, 710 C12H14O4S 4-(2-Hydroxy-4-methyl-5-methylthiophenyl)-4-oxo-1-butanoic acid, 445 C12H14O5 1-(2-Acetyloxy-4,5-dihydroxyphenyl)-1-butanone, 62 1-(4,6-Dihydroxy-1,3-benzodioxol-5-yl)-2-methyl-1-butanone, 133 1-(2-Hydroxy-3,4-dimethoxyphenyl)-1,3-butanedione, 323 1-(2-Hydroxy-3,6-dimethoxyphenyl)-1,3-butanedione, 323 1-(2-Hydroxy-4,5-dimethoxyphenyl)-1,3-butanedione, 324 1-(2-Hydroxy-4,6-dimethoxyphenyl)-1,3-butanedione, 324 1-(6-Hydroxy-2,3-dimethoxyphenyl)-1,3-butanedione, 325 2,4,6-Trihydroxy-3-(3-methyl-1-oxobutyl) benzaldehyde, 387 4-(2,4-Dimethoxyphenyl)-4-oxo-1-butanoic acid, 403 Ethyl 4-(2,4-dihydroxyphenyl)-4-oxo-1-butanoate, 405 4-(2,5-Dimethoxyphenyl)-4-oxo-1-butanoic acid, 406 4-(3,4-Dimethoxyphenyl)-4-oxo-1-butanoic acid, 408 Ethyl 4-(3,4-dihydroxyphenyl)-4-oxo-1-butanoate, 410 4-(3,4-Dihydroxyphenyl)-2,3-dimethyl-4-oxo-1-butanoic acid, 419 2-Ethyl-4-(2,5-dihydroxyphenyl)-4-oxo-1-butanoic acid, 419 Methyl 4-(2-hydroxy-4-methoxyphenyl)-4-oxo-1-butanoate, 439 Methyl 4-(2-hydroxy-5-methoxyphenyl)-4-oxo-1-butanoate, 440 4-(2,4-Dihydroxy-5-ethylphenyl)-4-oxo-1-butanoic acid, 445 4-(4,5-Dihydroxy-2-ethylphenyl)-4-oxo-1-butanoic acid, 446 3,5-Dihydroxy-2-(1-oxopentyl)benzoic acid. 490

 $C_{12}H_{14}O_5$  (cont.) 3.6-Dihydroxy-2-(1-oxopentyl)benzoic acid, 490 2,4,6-Trihydroxy-3-(1-oxopentyl) benzaldehyde, 490 5-(2,4-Dihydroxy-6-methylphenyl)-5-oxo-1-pentanoic acid, 593 5-(2-Hydroxy-4-methoxyphenyl)-5-oxo-1-pentanoic acid, 593 6-(2,4-Dihydroxyphenyl)-6-oxo-1-hexanoic acid, 712 6-(2,5-Dihydroxyphenyl)-6-oxo-1-hexanoic acid, 712 6-(2,6-Dihydroxyphenyl)-6-oxo-1-hexanoic acid, 713 6-(3,4-Dihydroxyphenyl)-6-oxo-1-hexanoic acid, 713 C12H14O6 Ethyl 4-(2,3,4-trihydroxyphenyl)-4-oxo-1-butanoate, 411 1-(2-Hydroxy-3,4-dimethoxyphenyl)-4-oxo-1-butanoic acid, 446 4-(2-Hydroxy-4,5-dimethoxyphenyl)-4-oxo-1-butanoic acid, 447 C₁₂H₁₅BrO₂ 1-(3-Bromo-4-ethoxyphenyl)-1-butanone, 27 1-(3-Bromo-6-hydroxy-2,4-dimethylphenyl)-1-butanone, 62 2-Bromo-1-(2-hydroxy-4,6-dimethylphenyl)-1-butanone, 273 2-Bromo-1-(4-methoxyphenyl)-3-methyl-1-butanone, 293 1-(3-Bromo-4-methoxyphenyl)-1-pentanone, 480 2-Bromo-1-(2-methoxyphenyl)-1-pentanone, 565 2-Bromo-1-(4-methoxyphenyl)-1-pentanone, 565 2-Bromo-1-(2-hydroxy-5-methylphenyl)-1-pentanone, 573 5-Bromo-1-(2-hydroxy-5-methylphenyl)-1-pentanone, 573 2-Bromo-1-(4-hydroxyphenyl)-4-methyl-1-pentanone, 574 1-(5-Bromo-2-hydroxyphenyl)-1-hexanone, 623 2-Bromo-1-(4-hydroxyphenyl)-1-hexanone, 697 6-Bromo-1-(2-hydroxyphenyl)-1-hexanone, 697 6-Bromo-1-(3-hydroxyphenyl)-1-hexanone, 698 6-Bromo-1-(4-hydroxyphenyl)-1-hexanone, 698

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2-Bromo-1-(3,4-dimethoxyphenyl)-1-butanone, 271 2-Bromo-1-(2,5-dimethoxyphenyl)-1-butanone, 271 4-Bromo-1-(3,4-dimethoxyphenyl)-1-butanone, 275 2-Bromo-1-(3-hydroxy-4-methoxyphenyl)-3-methyl-1-butanone, 294 5-Bromo-1-(2-hydroxy-3-methoxyphenyl)-1-pentanone, 574 5-Bromo-1-(2-hydroxy-4-methoxyphenyl)-1-pentanone, 574 5-Bromo-1-(2-hydroxy-5-methoxyphenyl)-1-pentanone, 574 6-Bromo-1-(2,4-dihydroxyphenyl)-1-hexanone, 699 6-Bromo-1-(2,6-dihydroxyphenyl)-1-hexanone, 699 6-Bromo-1-(3,4-dihydroxyphenyl)-1-hexanone, 699 2-Bromo-1-(3,4-dihydroxyphenyl)-1-hexanone, 700 C₁₂H₁₅BrO₄ 1-(5-Bromo-2,3,4-trihydroxyphenyl)-1-hexanone, 623 2-Bromo-1-(2,4,5-trihydroxyphenyl)-1-hexanone, 700 C12H15BrO4S 2-Bromo-1-[4-Hydroxy-3-(methylsulfonylmethyl)phenyl] butanone, 273 C₁₂H₁₅ClFNO₂ 6-Chloro-1-(4-fluoro-2-hydroxyphenyl)-1-hexanone (Oxime), 705  $C_{12}H_{15}ClO_2$ 1-(3-Chloro-4-ethoxyphenyl)-1-butanone, 30 1-(3-Chloro-6-hydroxy-2,4-dimethylphenyl)-1-butanone, 63 1-(5-Chloro-3-ethyl-2-hydroxyphenyl)-1-butanone, 63 1-(2-Chloro-4-methoxyphenyl)-2-methyl-1-butanone, 133 1-(2-Chloro-4-hydroxyphenyl)-2-ethyl-1-butanone, 171 1-(2-Chloro-4-hydroxyphenyl)-3,3-dimethyl-1-butanone, 252 4-Chloro-1-(4-ethoxyphenyl)-1-butanone, 280 4-Chloro-1-(4-methoxy-2-methylphenyl)-1-butanone, 284 4-Chloro-1-(4-hydroxy-3,5-dimethylphenyl)-1-butanone, 286 2-Chloro-1-(2-hydroxy-5-methylphenyl)-3-methyl-1-butanone, 294

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6-Chloro-1-(3,4-dihydroxyphenyl)-1-hexanone, 701 C₁₂H₁₅ClO₄ 4-Chloro-1-(2-hydroxy-4,6-dimethoxyphenyl)-1-butanone, 287 4-Chloro-1-(4-hydroxy-2,6-dimethoxyphenyl)-1-butanone, 287 1-(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)-1-pentanone, 491 1-(3-Chloro-2,4,6-trihydroxyphenyl)-1-hexanone, 626 1-(5-chloro-2,3,4-trihydroxyphenyl)-1-hexanone, 626  $C_{12}H_{15}Cl_2NO_2$ 1-(4-Amino-5-chloro-2-methoxyphenyl)-5-chloro-1-pentanone, 572 C₁,H₁,DO₂ 1-(4-Hydroxyphenyl)-2-methyl-1-pentanone-2-d. 553 C12H15FO2 1-(4-Ethyl-5-fluoro-2-hydroxyphentl)-1-butanone, 63 1-(3-Fluoro-4-methoxyphenyl)-3-methyl-1-butanone, 186 1-(2-Fluoro-4-methoxyphenyl)-1-pentanone, 483 1-(3-Fluoro-4-methoxyphenyl)-1-pentanone, 484 1-(4-Fluoro-2-methoxyphenyl)-1-pentanone, 484 1-(3-Fluoro-4-hydroxyphenyl)-1-hexanone, 626 1-(5-Fluoro-2-hydroxyphenyl)-1-hexanone, 627  $C_{12}H_{15}IO_2$ 1-(4-Methoxy-2-iodophenyl)-1-pentanone, 484 1-(2-Methoxyphenyl)-5-iodo-1-pentanone, 569  $C_{12}H_{15}IO_3$ 1-(2,4-Diydroxy-3-iodophenyl)-3,3-dimethyl-1-butanone, 252 1-(3,4-Dimethoxyphenyl)-4-iodo-1-butanone, 291 C₁₂H₁₅NO₃ 1-(5-Amino-2-acetyloxyphenyl)-1-butanone, 40 1-[2-(Acetylamino)-4-hydroxyphenyl]-1-butanone, 64 1-[4-(Acetylamino)-2-hydroxyphenyl]-1-butanone, 64 1-[5-(Acetylamino)-2-hydroxyphenyl]-1-butanone, 64 2-Hydroxy-5-(3-methylbutyryl) benzamide, 189

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5-Hydroxy-6-(1-oxobutyl)-1,4-benzodioxane (Oxime), 62 4-(3-Amino-4-ethoxyphenyl)-4-oxo-1-butanoic acid, 428 1-(4-Methoxy-3-nitrophenyl)-1-pentanone, 485 1-(4-Hydroxyphenyl)-4-methyl-4-nitro-1-pentanone, 541 5-(3-Amino-4-methoxyphenyl)-5-oxo-1-pentanoic acid, 589 1-(4-Hydroxy-3-nitrophenyl)-1-hexanone, 627 6-(2-Hydroxyphenyl)-6-oxo-1-hexanoic acid (Oxime), 709 6-(3-Amino-4-hydroxyphenyl)-6-oxo-1-hexanoic acid, 715 C₁₂H₁₅NO₅ 1-(2,4-Dimethoxy-5-nitrophenyl)-1-butanone, 36 4-(2,4-Dimethoxyphenyl)-4-oxo-1-butanoic acid (Oxime), 404 4-(2-Amino-4,5-dimethoxyphenyl)-4-oxo-1-butanoic acid, 429 1-(4-Hydroxy-3-methoxy-2-nitrophenyl)-1-pentanone, 491 5-(2,4-Dihydroxy-6-methylphenyl)-5-oxo-1-pentanoic acid (Oxime), 593 C12H15NO5 H2O 4-(2-Amino-4,5-dimethoxyphenyl)-4-oxo-1-butanoic acid (Monohydrate), 429 C12H15NO6 1-(2,4,6-Trihydroxy-3-nitrophenyl)-1-hexanone, 628 C12H15N3O4 2,6-Dihydroxy-3-(1-oxobutyl)benzaldehyde (Semicarbazone), 387 C₁₂H₁₅O₃Na 1-(2-Hydroxy-5-methoxyphenyl)-3-methyl-1-butanone (Na salt), 193 C12H15O4Na 4-Methyl-1-(2,4,6-trihydroxyphenyl)-1-pentanone (Monosodium salt), 546  $C_{12}H_{16}BrNO_2$ 1-[3-(Aminomethyl)-5-bromo-4-hydroxyphenyl]-1-pentanone, 491  $C_{12}H_{16}BrN_3O_3$ 1-(4-Bromo-2,5-dihydroxyphenyl)-1-pentanone (Semicarbazone), 481  $C_{12}H_{16}CINO_2$ 1-(3-Chloro-2-hydroxyphenyl)-1-hexanone (Oxime), 624 1-(5-Chloro-2-hydroxyphenyl)-1-hexanone (Oxime), 625

1-(3-Amino-5-chloro-2-hydroxyphenyl)-1-hexanone, 628 C₁₂H₁₆FNO₂ 1-(3-Amino-5-fluoro-2-hydroxyphenyl)-1-hexanone, 628  $C_{12}H_{16}O_2$ 1-(4-Ethoxyphenyl)-1-butanone, 8 1-(2-Methoxy-4-methylphenyl)-1-butanone, 48 1-(2-Methoxy-5-methylphenyl)-1-butanone, 50 1-(2-Methoxy-6-methylphenyl)-1-butanone, 50 1-(4-Methoxy-2-methylphenyl)-1-butanone, 51 1-(4-Methoxy-3-methylphenyl)-1-butanone, 52 1-(3-Ethyl-2-hydroxyphenyl)-1-butanone, 65 1-(4-Ethyl-2-hydroxyphenyl)-1-butanone, 65 1-(5-Ethyl-2-hydroxyphenyl)-1-butanone, 65 1-(2-Hydroxy-3,5-dimethylphenyl)-1-butanone, 66 1-(2-Hydroxy-4,5-dimethylphenyl)-1-butanone, 66 1-(2-Hydroxy-4,6-dimethylphenyl)-1-butanone, 67 1-(4-Hydroxy-2,3-dimethylphenyl)-1-butanone, 68 1-(4-Hydroxy-2,5-dimethylphenyl)-1-butanone, 68 1-(4-Hydroxy-3,5-dimethylphenyl)-1-butanone, 68 1-(5-Hydroxy-2,4-dimethylphenyl)-1-butanone, 69 1-(2-Methoxyphenyl)-2-methyl-1-butanone, 125 1-(4-Methoxyphenyl)-2-methyl-1-butanone, 126 1-(4-Methoxyphenyl)-2-methyl-1-butanone (2S), 126 1-(4-Hydroxyphenyl)-2-methyl-1-butanone (R), 127 1-(2-Hydroxyphenyl)-2,2-dimethyl-1-butanone, 131 1-(4-Hydroxy-2-methylphenyl)-2-methyl-1-butanone (2S), 133 1-(4-Hydroxy-3-methylphenyl)-2-methyl-1-butanone, 133 2-Ethyl-1-(4-hydroxyphenyl)-1-butanone, 169 1-(2-Methoxyphenyl)-3-methyl-1-butanone, 174 1-(3-Methoxyphenyl)-3-methyl-1-butanone, 174

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C13H15BrO5 5-(5-Bromo-2,4-dimethoxyphenyl)-5-oxo-1-pentanoic acid, 586 7-(5-Bromo-2,4-dihydroxyphenyl)-7-oxo-1-heptanoic acid, 767 C13H15ClF2O3 5-Chloro-1-(2,5-dimethoxyphenyl)-2,2-difluoro-1-pentanone, 563 5-Chloro-1-(3,5-dimethoxyphenyl)-2,2-difluoro-1-pentanone, 564 C13H15ClO3 2-Chloro-1-(2-methoxyphenyl)-1,3-hexanedione, 697 C13H15ClO4 4-(4-Chlorobutyryl)-3-methylphenoxyacetic acid. 287 Methyl 5-(2-chloro-4-methoxyphenyl)-5-oxo-1-pentanoate, 586 Methyl 5-(2-chloro-5-methoxyphenyl)-5-oxo-1-pentanoate, 587 Methyl 5-(3-chloro-4-methoxyphenyl)-5-oxo-1-pentanoate, 587 7-(5-Chloro-2-hydroxyphenyl)-7-oxo-1-heptanoic acid, 768 C13H15ClO5 5-(5-Chloro-2,4-dimethoxyphenyl)-5-oxo-1-pentanoic acid, 588 7-(5-Chloro-2,4-dihydroxyphenyl)-7-oxo-1-heptanoic acid, 768 C13H15FO2 1-(3-Fluoro-4-hydroxyphenyl)-2-methylene-1-hexanone, 629 C13H15F3O3 1-(2,4-Dihydroxy-3-propylphenyl)-4,4,4-trifluoro-1-butanone, 290 C13H15NO5S 1-(4-Methoxyphenyl)-4-methyl-4-nitro-2-sulfinyl-1-pentanone, 541 C13H15NO6 5-(4-Ethoxy-3-nitrophenyl)-5-oxo-1-pentanoic acid, 588 6-(4-Methoxy-3-nitrophenyl)-6-oxo-1-hexanoic acid, 715 C₁₃H₁₅NO₇ Methyl 4-(4,5-dimethoxy-2-nitrophenyl)-4-oxo-1-butanoate, 427 C13H15N5O3 N-(3-Butanoyl-2-hydroxy-5-methylphenyl)-1H-tetrazole-5-carboxamide, 80 C₁₃H₁₅O₅Na 1-(2,4,5-Trimethoxyphenyl)-1,3-butanedione

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(Na salt), 314
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C₁₃H₁₆BrClO₂ 6-Bromo-1-(2-chloro-4-methoxyphenyl)-1-hexanone, 702 6-Bromo-1-(3-chloro-4-methoxyphenyl)-1-hexanone, 703 6-Bromo-1-(4-Chloro-2-methoxyphenyl)-1-hexanone, 703 7-Bromo-1-(5-chloro-2-hydroxyphenyl)-1-heptanone, 765 C₁₃H₁₆BrFO₂ 6-Bromo-1-(3-fluoro-4-methoxyphenyl)-1-hexanone, 703 1-(3-Bromo-5-fluoro-2-hydroxyphenyl)-1-heptanone, 731 C13H16Br2O2 2-Bromo-1-(3-bromo-4-methoxyphenyl)-1-hexanone (2S), 704 1-(3,5-Dibromo-4-hydroxyphenyl)-1-heptanone, 732 C13H16Br2O3 2-Bromo-1-(2-bromo-4,5-dimethoxyphenyl)-1-pentanone, 570 C13H16Br2O4 1-(3,5-Dibromo-2,4-dihydroxy-6-methoxyphenyl)-1-hexanone, 630 1-(3,5-Dibromo-2,6-dihydroxy-4-methoxyphenyl)-1-hexanone, 630 C₁₃H₁₆ClFO₂ 1-(3-Chloro-5-fluoro-4-methoxyphenyl)-1-hexanone, 621 6-Chloro-1-(3-fluoro-4-methoxyphenyl)-1-hexanone, 704 1-(3-Chloro-5-fluoro-2-hydroxyphenyl)-1-heptanone, 732 C13H16CINO4 1-(5-Chloro-2-hydroxy-3-nitrophenyl)-1-heptanone, 732 C₁₃H₁₆ClNO₅ 5-Chloro-1-(2,4-dimethoxy-5-nitrophenyl)-1-pentanone, 571 C13H16Cl2O2 1-(2,3-Dichloro-4-methoxyphenyl)-2-ethyl-1-butanone, 171 1-(2,3-Dichloro-4-hydroxyphenyl)-1-heptanone, 732 C13H16Cl2O3 5-Chloro-1-(3-chloro-4,5-dimethoxyphenyl)-1-pentanone, 572 1-(3,5-Dichloro-2,4-dihydroxy-6-methylphenyl)-1-hexanone, 630

C13H16Cl2O4 1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-3,3-dimethyl-1-butanone, 253 1-(3,5-Dichloro-2,4-dihydroxy-6-methoxyphenyl)-1-hexanone, 631 1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-1-hexanone, 631 C13H16I2O2 1-(4-Hydroxy-3,5-diiodophenyl)-1-heptanone, 733 C13H16I2O4 1-(2,6-Dihydroxy-3,5-diiodo-4-methoxyphenyl)-1-hexanone, 632  $C_{13}H_{16}O_2$ 1-[4-Hydroxy-3-(2-propenyl)phenyl]-1-butanone, 80 1-(4-Methoxyphenyl)-2-methylene-1-pentanone, 477 1-(2-Hydroxyphenyl)-2-methylene-1-hexanone, 615 1-(4-Hydroxyphenyl)-2-methylene-1-hexanone, 615 C13H16O3 1-[2,4-Dihydroxy-3-(2-propenyl)phenyl]-1-butanone, 80 1-[2-Hydroxy-4-(2-propenyloxy)phenyl]-1-butanone, 80 1-(3-Acetyloxyphenyl)-2-methyl-1-butanone, 125 1-(2-Acetyloxyphenyl)-3-methyl-1-butanone, 174 1-(3-Acetyloxyphenyl)-3-methyl-1-butanone, 174 1-(2-Methoxy-3,5-dimethylphenyl)-1,3-butanedione, 322 1-(3-Acetyl-2-hydroxy-5-methylphenyl)-1-butanone, 377 1-(5-Acetyl-2-hydroxyphenyl)-3-methyl-1-butanone, 378 1-[4-Hydroxy-3-(1-oxopropyl)phenyl]-1-butanone, 385 1-(2-Ethoxyphenyl)-1,3-pentanedione, 459 1-(2-Acetyloxyphenyl)-1-pentanone, 463 1-(3-Acetyloxyphenyl)-1-pentanone, 464 1-(2-Methoxyphenyl)-1,3-hexanedione, 597 1-(4-Methoxyphenyl)-1,4-hexanedione, 597 1-(2-Hydroxyphenyl)-4-methyl-1,3-hexanedione, 615 1-(2-Hydroxyphenyl)-1,6-heptanedione, 733 C12⁽¹³⁾CH16O3 1-(3,5-Dimethoxyphenyl)-1-pentanone-1-¹³C, 475

C12⁽¹⁴⁾CH18O3 1-(3,5-Dimethoxyphenyl)-1-pentanone-1-¹⁴C, 475 C₁₃H₁₆O₄ 3-Butyryl-4-ethoxybenzoic acid, 43 Ethyl 3-butyryl-4-hydroxybenzoate, 43 Methyl 5-butyryl-2-methoxybenzoate, 44 1-[2,4,6-Trihydroxy-3-(2-propenyl)phenyl]-1-butanone, 80 1-[2-(Acetyloxy)-4-hydroxyphenyl]-2-methyl-1-butanone (-), 134 5-(2-Ethylbutyryl)-2-hydroxybenzoic acid, 171 1-[2-(Acetyloxy)-4-hydroxyphenyl]-3-methyl-1-butanone, 195 5-Hydroxy-6-(2-methyl-1-oxobutyl)-1,4-benzodioxane, 195 1-(2,4-Dimethoxy-3-methylphenyl)-1,3-butanedione, 318 1-(2,4-Dimethoxy-6-methylphenyl)-1,3-butanedione, 318 Ethyl 4-(4-methoxyphenyl)-4-oxo-1-butanoate, 401 Methyl 4-(4-ethoxyphenyl)-4-oxo-1-butanoate, 402 4-(4-Methoxyphenyl)-2,2-dimethyl-4-oxo-1-butanoic acid, 418 Ethyl 4-(2-hydroxy-3-methylphenyl)-4-oxo-1-butanoate, 433 4-(2-Ethoxy-3-methylphenyl)-4-oxo-1-butanoic acid, 433 Methyl 4-(2-methoxy-5-methylphenyl)-4-oxo-1-butanoate, 434 4-(2-Ethoxy-5-methylphenyl)-4-oxo-1-butanoic acid, 435 Ethyl 4-(4-hydroxy-3-methylphenyl)-4-oxo-1-butanoate, 437 4-(3-Ethyl-4-methoxyphenyl)-4-oxo-1-butanoic acid, 442 4-(2-Methoxy-3,5-dimethylphenyl)-4-oxo-1-butanoic acid, 443 Methyl 4-(2-hydroxy-3,5-dimethylphenyl)-4-oxo-1-butanoate,444 4-(5-Methoxy-2,4-dimethylphenyl)-4-oxo-1-butanoic acid, 444 1-(4-Hydroxy-3-propylphenyl)-4-oxo-1-butanoic acid, 447 4-(2-Hydroxy-3,4,6-trimethylphenyl)-4-oxo-1-butanoic acid, 447 4-(2-Hydroxy-3,5-dimethylphenyl)-2-methyl-4-oxo-1-butanoic acid, 448 4-(2-Hydroxy-4,5-dimethylphenyl)-2-methyl-

4-oxo-1-butanoic acid, 448

 $C_{13}H_{16}O_4$  (cont.) 4-(2-Hydroxy-3,5-dimethylphenyl)-3-methyl-4-oxo-1-butanoic acid, 448 1-(2,4-Dimethoxyphenyl)-1,3-pentanedione, 461 1-(2,4-Dimethoxyphenyl)-1,4-pentanedione, 461 1-(2-Acetyl-5-hydroxyphenyl)-1-pentanone, 472 4-Methoxy-3-valeroylbenzoic acid, 489 Methyl 4-hydroxy-3-valeroylbenzoate, 489 1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1,3-pentanedione, 499 5-Hvdroxv-6-(1-oxopentvl)-1,4-benzodioxane, 499 5-Isocaproyl-2-hydroxybenzoic acid, 547 Methyl 5-(4-methoxyphenyl)-5-oxo-1-pentanoate, 579 5-(4-Ethoxyphenyl)-5-oxo-1-pentanoic acid, 580 Ethyl 5-(4-hydroxyphenyl)-5-oxo-1-pentanoate, 580 4-Ethyl-5-(2-hydroxyphenyl)-5-oxo-1-pentanoic acid, 584 5-(4-Hydroxyphenyl)-2,2-dimethyl-5-oxo-1-pentanoic acid, 584 5-(4-Hydroxyphenyl)-3,3-dimethyl-5-oxo-1-pentanoic acid, 585 Methyl 5-(2-hydroxy-4-methylphenyl)-5-oxo-1-pentanoate, 591 5-(4-Methoxy-2-methylphenyl)-5-oxo-1-pentanoic acid, 592 5-(4-Methoxy-3-methylphenyl)-5-oxo-1-pentanoic acid, 592 1-(2,4-Dihydroxy-6-methylphenyl)-1,3-hexanedione, 633 3-Hexanoyl-4-hydroxybenzoic acid, 633 5-Hexanoyl-2-hydroxybenzoic acid, 634 6-(2-Methoxyphenyl)-6-oxo-1-hexanoic acid, 710 6-(4-Methoxyphenyl)-6-oxo-1-hexanoic acid, 711 6-(2-Hydroxy-5-methylphenyl)-6-oxo-1-hexanoic acid, 715 6-(3-Hydroxy-4-methylphenyl)-6-oxo-1-hexanoic acid, 716 6-(4-Hydroxy-2-methylphenyl)-6-oxo-1-hexanoic acid, 716 6-(4-Hydroxy-3-methylphenyl)-6-oxo-1-hexanoic acid, 716 7-(2-Hydroxyphenyl)-7-oxo-1-heptanoic acid, 766 7-(4-Hydroxyphenyl)-7-oxo-1-heptanoic acid, 766

### C₁₃H₁₆O₅

1-[5-Ethyl-2,4-dihydroxy-3-(1-oxobutyl)] benzoic acid, 81 1-[2-(Acetyloxy)-4,6-dihydroxyphenyl]-3-methyl-1-butanone, 195 1-[4-(Acetyloxy)-2,6-dihydroxyphenyl]-3-methyl-1-butanone, 196 1-(2.4.5-Trimethoxyphenyl)-1,3-butanedione, 314 1-(2,4,6-Trimethoxyphenyl)-1.3-butanedione, 327 1-(2,3,4-Trimethoxyphenyl)-1,3-butanedione, 323 1-(2.3.6-Trimethoxyphenyl)-1,3-butanedione, 324 1-(2-Hydroxy-3,4-dimethoxy-6-methylphenyl)-1,3-butanedione, 326 1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-1,3-butanedione, 327 1-[2,4,6-Trihydroxy-3-(1-oxopropyl)phenyl]-1-butanone, 385 2,4,6-Trihydroxy-3-methyl-5-(3-methyl-1-oxobutyl)benzaldehyde, 389 Methyl 4-(2,4-dimethoxyphenyl)-4-oxo-1-butanoate, 404 Methyl 4-(2,5-dimethoxyphenyl)-4-oxo-1-butanoate, 406 4-(2-Ethoxy-5-methoxyphenyl)-4-oxo-1-butanoic acid, 407 4-(5-Ethoxy-2-methoxyphenyl)-4-oxo-1-butanoic acid, 407 Methyl 4-(3,4-dimethoxyphenyl)-4-oxo-1-butanoate, 409 4-[(2-Methoxymethyl)phenyl]-3-methyl-4-oxo-1-butanoic acid, 414 4-(3,4-Dimethoxyphenyl)-2-methyl-4-oxo-1-butanoic acid, 416 4-(3,4-Dimethoxyphenyl)-3-methyl-4-oxo-1-butanoic acid, 416 4-(3,5-Dimethoxyphenyl)-3-methyl-4-oxo-1-butanoic acid, 417 4-(2,5-Dimethoxy-4-methylphenyl)-4-oxo-1-butanoic acid, 437 4-(4,5-Dimethoxy-2-methylphenyl)-4-oxo-1-butanoic acid, 438 2,4,6-Trihydroxy-3-(3-methyl-1-oxopentyl) benzaldehyde (S), 558 5-(2,4-Dimethoxyphenyl)-5-oxo-1-pentanoic acid, 581 5-(2,5-Dimethoxyphenyl)-5-oxo-1-pentanoic acid, 581

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2-Bromo-1-(4-hydroxyphenyl)-1-heptanone, 764 C₁₃H₁₇BrO₃ 2-Bromo-1-(3,4-dimethoxyphenyl)-1-pentanone, 566 6-Bromo-1-(2-hydroxy-4-methoxyphenyl)-1-hexanone, 706 6-Bromo-1-(2-hydroxy-5-methoxyphenyl)-1-hexanone, 706 C₁₃H₁₇BrO₄ 2-Bromo-1-(2,4,5-trimethoxyphenyl)-1-butanone, 271 5-Bromo-1-(2-hydroxy-3,4-dimethoxyphenyl)-1-pentanone, 575 5-Bromo-1-(2-hydroxy-4,5-dimethoxyphenyl)-1-pentanone, 576 1-(3-Bromo-2,6-dihydroxy-4-methoxyphenyl)-1-hexanone, 635 C₁₃H₁₇ClO₂ 1-(2-Chloro-4-hydroxy-3-methylphenyl)-2-ethyl-1-butanone, 172 1-(3-Chloro-4-hydroxy-2-methylphenyl)-2-ethyl-1-butanone, 172 1-(2-Chloro-4-hydroxyphenyl)-2-ethyl-3-methyl-1-butanone, 196 1-(3-Chloro-4-hvdroxyphenyl)-2-ethyl-3-methyl-1-butanone, 196 3-Chloro-1-[2-hydroxy-5-(1-methylethyl) phenyl]-1-butanone, 277 4-Chloro-1-(4-propoxyphenyl)-1-butanone, 281 4-Chloro-1-[4-hydroxy-3-(1-methylethyl) phenyl]-1-butanone, 287 3-Chloro-1-(4-hydroxyphenyl)-2-(1-methylethyl)-1-butanone, 295 1-(3-Chloro-6-hydroxy-2,4-dimethylphenyl)-1-pentanone, 499 1-(5-Chloro-3-ethyl-2-hydroxyphenyl)-1-pentanone, 500 1-(4-Chloro-2-methoxyphenyl)-1-hexanone, 625 1-(2-Chloro-6-hydroxy-4-methylphenyl)-1-hexanone, 635 1-(3-Chloro-2-hydroxy-5-methylphenyl)-1-hexanone, 635 1-(5-Chloro-2-hydroxy-3-methylphenyl)-1-hexanone, 635 1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-hexanone, 636 6-Chloro-1-(2-methoxyphenyl)-1-hexanone, 700

 $C_{13}H_{17}ClO_2$  (cont.) 6-Chloro-1-(3-methoxyphenyl)-1-hexanone, 701 6-Chloro-1-(4-methoxyphenyl)-1-hexanone, 701 6-Chloro-1-(2-hydroxy-5-methylphenyl)-1-hexanone, 706 6-Chloro-1-(4-hydroxy-3-methylphenyl)-1-hexanone, 706 1-(3-Chloro-4-hydroxyphenyl)-1-heptanone, 734 1-(4-Chloro-2-hydroxyphenyl)-1-heptanone, 734 1-(5-Chloro-2-hydroxyphenyl)-1-heptanone, 734 C₁₃H₁₇ClO₃ 1-(3-Chloro-2,6-dihydroxy-5-propylphenyl)-1-butanone, 81 5-Chloro-1-(2,4-dimethoxyphenyl)-1-pentanone, 569 5-Chloro-1-(3,4-dimethoxyphenyl)-1-pentanone, 569 C₁₃H₁₇ClO₄ 1-(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)-3,3-dimethyl-1-butanone, 253 4-Chloro-1-(2,3,4-trimethoxyphenyl)-1-butanone, 283 4-Chloro-1-(2,4,6-trimethoxyphenyl)-1-butanone, 283 4-Chloro-1-(3,4,5-trimethoxyphenyl)-1-butanone, 283 1-(3-Chloro-2,4-dihydroxy-6-methoxyphenyl)-1-hexanone, 636 1-(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)-1-hexanone, 636 1-(5-Chloro-4,6-dihydroxy-2-methoxyphenyl)-1-hexanone, 637 C₁₃H₁₇DO₂ 1-(4-Methoxyphenyl)-2-methyl-1-pentanone-2-d, 553 C₁₃H₁₇FO₂ 1-(3-Fluoro-4-methoxyphenyl)-1-hexanone, 627 1-(3-Fluoro-4-hydroxyphenyl)-5-methyl-1-hexanone, 637 C₁₃H₁₇IO₄ 1-(2,6-Dihydroxy-3-iodo-4-methoxyphenyl)-1-hexanone, 638 C₁₃H₁₇NO₃ 1-(4-Ethoxy-2-hydroxyphenyl)-1-butanone (Oxime), 70 2-Hydroxy-5-(2-ethylbutyryl) benzamide, 172

C13H17NO4 1-(4-Methoxyphenyl)-4-methyl-4-nitro-1-pentanone, 541 5-(3-Amino-4-ethoxyphenyl)-5-oxo-1-pentanoic acid, 589 6-(3-Amino-4-methoxyphenyl)-6-oxo-1-hexanoic acid, 715 C13H17NO5 Methyl 4-(2-amino-4,5-dimethoxyphenyl)-4-oxo-1-butanoate, 429 C13H17NO6 1-(2,4,6-Trihydroxy-3-nitro-5-propylphenyl)-1-butanone, 82 1-(2,4,6-Trihydroxy-3-nitrophenyl)-1-heptanone, 735 C13H17N3O4 4-(2-Methoxy-5-methylphenyl)-4-oxo-1-butanoic acid (Semicarbazone), 434 6-(2-Hydroxyphenyl)-6-oxo-1-hexanoic acid (Semicarbazone), 709 C₁₃H₁₈ClNO₂ 1-(3-Amino-5-chloro-2-hydroxyphenyl)-1-heptanone, 735 C13H18CINO2, HCl 1-(3-Amino-5-chloro-2-hydroxyphenyl)-1-heptanone (Hydrochloride), 735 C₁₃H₁₈ClNO₃ 1-(5-Amino-2,4-dimethoxyphenyl)-5-chloro-1-pentanone, 572 C₁₃H₁₈FNO₂ 1-(3-Amino-5-fluoro-2-hydroxyphenyl)-1-heptanone, 735 C13H18O2 1-(2-Ethoxy-5-methylphenyl)-1-butanone, 50 1-(4-Ethoxy-3-methylphenyl)-1-butanone, 52 1-(4-Ethyl-2-methoxyphenyl)-1-butanone, 65 1-(5-Ethyl-2-methoxyphenyl)-1-butanone, 66 1-(2-Methoxy-4,5-dimethylphenyl)-1-butanone, 66 1-(2-Methoxy-4,6-dimethylphenyl)-1-butanone, 67 1-(4-Methoxy-2,3-dimethylphenyl)-1-butanone, 68 1-(4-Methoxy-3,5-dimethylphenyl)-1-butanone, 68 1-(2-Ethyl-6-hydroxy-4-methylphenyl)-1-butanone, 82 1-(3-Ethyl-4-hydroxy-5-methylphenyl)-1-butanone, 82 1-[2-Hydroxy-4-(1-methylethyl)phenyl]-1-butanone, 82 1-(2-Hydroxy-4-propylphenyl)-1-butanone, 83 1-(4-Hydroxy-3-propylphenyl)-1-butanone, 83

1-(4-Methoxy-3-methylphenyl)-2-methyl-1-butanone, 133 1-(2-Hydroxy-5-methylphenyl)-2,2-dimethyl-1-butanone, 135 2-Ethyl-1-(4-methoxyphenyl)-1-butanone, 170 2-Ethyl-1-(2-hydroxy-4-methylphenyl)-1-butanone, 172 1-(4-Ethoxyphenyl)-3-methyl-1-butanone, 176 1-(2-Methoxy-4-methylphenyl)-3-methyl-1-butanone, 190 1-(2-Methoxy-5-methylphenyl)-3-methyl-1-butanone, 190 1-(4-Methoxy-3-methylphenyl)-3-methyl-1-butanone, 191 1-(5-Ethyl-2-hydroxyphenyl)-3-methyl-1-butanone, 197 1-(4-Methoxyphenyl)-3,3-dimethyl-1-butanone, 251 1-(4-Hydroxy-3-methylphenyl)-3,3-dimethyl-1-butanone, 254 1-(2-Methoxy-4-methylphenyl)-1-pentanone, 492 1-(2-Methoxy-5-methylphenyl)-1-pentanone, 493 1-(4-Methoxy-2-methylphenyl)-1-pentanone, 494 1-(4-Methoxy-3-methylphenyl)-1-pentanone, 494 1-(5-Methoxy-2-methylphenyl)-1-pentanone, 494 1-(2-Hydroxy-4,5-dimethylphenyl)-1-pentanone, 500 1-(4-Hydroxy-2,3-dimethylphenyl)-1-pentanone, 500 1-(2-Methoxyphenyl)-4-methyl-1-pentanone, 541 1-(4-Methoxyphenyl)-4-methyl-1-pentanone, 542 1-(2-hydroxy-4-methylphenyl)-4-methyl-1-pentanone, 547 1-(2-Hydroxy-5-methylphenyl)-4-methyl-1-pentanone, 547 1-(4-Hydroxy-3-methylphenyl)-4-methyl-1-pentanone, 547 1-(4-Methoxyphenyl)-3-methyl-1-pentanone, 554 1-(4-Methoxyphenyl)-2-methyl-1-pentanone, 553 1-(2-Hydroxyphenyl)-4,4-dimethyl-1-pentanone, 555 1-(4-Hydroxyphenyl)-4,4-dimethyl-1-pentanone, 555

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4-oxo-1-butanoate, 429
C ₁₄ H ₁₉ NO ₆
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5-propylphenyl)-1-butanone, 206
1-(2,4,6-Trihydroxy-3-nitro-5-propylphenyl)-
1-pentanone, 505
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C ₁₄ H ₂₀ ClNO ₄
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5-chloro-1-pentanone, 573
$C_{14}H_{20}CIN_3O_2$
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1-pentanone (Semicarbazone), 499
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1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-
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phenyl]-1-butanone, 90
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6-methylphenyl]-1-butanone, 91
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phenyl]-1-butanone, 91
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1-butanone, 92
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1-butanone, 92
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1-butanone, 197
1-[4-Hydroxy-3-(1-methylethyl)phenyl]-

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- 1-[3-Bromo-2-hydroxy-5-(1,1-dimethylethyl) phenyl]-1-hexanone, 663 6-Bromo-1-[4-hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-hexanone, 708 C₁₆H₂₃BrO₃ 1-[4-(3-Bromopropoxy)-2-hydroxy-3-propylphenyl]-1-butanone, 108 6-Bromo-1-(3,4-dimethoxy-2,5-dimethylphenyl)-1-hexanone, 707 6-Bromo-1-[3,4-dihydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-hexanone, 708 9-Bromo-1-(2-hydroxy-5-methoxyphenyl)-1-nonanone, 863 2-Bromo-1-(3,4-dihydroxyphenyl)-1-decanone, 916 C₁₆H₂₃BrO₄ 8-Bromo-1-(2-hydroxy-4,5-dimethoxyphenyl)-1-octanone, 829 1-(5-Bromo-2,3,4-trihydroxyphenyl)-1-decanone, 878 C₁₆H₂₃ClO₂ 1-[3-Chloro-2-hydroxy-5-(1,1-dimethylethyl) phenyl]-1-hexanone, 664 1-(3-Chloro-2-hydroxyphenyl)-1-decanone, 879 1-(3-Chloro-4-hvdroxvphenvl)-1-decanone, 879 1-(4-Chloro-2-hydroxyphenyl)-1-decanone, 879 1-(5-Chloro-2-hydroxyphenyl)-1-decanone, 879 C16H23ClO3 1-(4-Chloro-2,5-dihydroxyphenyl)-1-decanone, 880 C₁₆H₂₃ClO₄ 1-(4-Butoxy-3-chloro-2,6-dihydroxyphenyl)-1-hexanone, 664 C16H23FO2 1-(2-Fluoro-4-methoxyphenyl)-2-methyl-1-octanone (+), 791 1-(3-Fluoro-4-methoxyphenyl)-2-methyl-1-octanone (+), 791 1-(3-Fluoro-4-methoxyphenyl)-1-nonanone, 842 1-(3-Fluoro-4-hydroxyphenyl)-1-decanone, 880 1-(5-Fluoro-2-hydroxyphenyl)-1-decanone, 880 C16H23NO4 1-[2-(N-Morpholinoethoxy)-4-hydroxyphenyl]-1-butanone, 108 1-[4-(N-Morpholinoethoxy)-
  - 2-hydroxyphenyl]-1-butanone, 109

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1-(3-Ethyl-4-hydroxy-5-methylphenyl)-1-heptanone, 746 1-(4-Hydroxy-3-(1-methylethyl)phenyl)-1-heptanone, 747 2-Ethyl-1-(4-hydroxyphenyl)-1-octanone (+), 784 1-(4-Methoxy-3-methylphenyl)-1-octanone, 794 1-(4-Methoxyphenyl)-2-methyl-1-octanone (+), 794 1-(4-Ethyl-2-hydroxyphenyl)-1-octanone, 798 1-(5-Ethyl-2-hydroxyphenyl)-1-octanone, 799 1-(2-Hydroxy-4,5-dimethylphenyl)-1-octanone, 799 1-(2-Hydroxy-4,6-dimethylphenyl)-1-octanone, 799 1-(4-Hydroxy-2,3-dimethylphenyl)-1-octanone, 800 1-(4-Hydroxy-2,5-dimethylphenyl)-1-octanone, 800 1-(4-Hydroxyphenyl)-3,7-dimethyl-1-octanone, 800 1-(3-Methoxyphenyl)-1-nonanone, 836 1-(4-Methoxyphenyl)-1-nonanone, 837 1-(4-Hydroxyphenyl)-2-methyl-1-nonanone (+), 8401-(4-Hydroxyphenyl)-2-methyl-1-nonanone, 840 1-(2-Hydroxy-4-methylphenyl)-1-nonanone, 843 1-(2-Hydroxy-5-methylphenyl)-1-nonanone, 844 1-(2-Hydroxy-6-methylphenyl)-1-nonanone, 844 1-(2-Hydroxyphenyl)-1-decanone, 868 1-(3-Hydroxyphenyl)-1-decanone, 869 1-(4-Hydroxyphenyl)-1-decanone, 869 C16H24O3 1-(4-Ethyl-2,5-dimethoxyphenyl)-3,3-dimethyl-1-butanone, 254 1-(2,4-Dihydroxy-5-pentylphenyl)-1-pentanone, 513 1-(2,5-Dihydroxy-4-pentylphenyl)-1-pentanone, 514 1-(3,4-Dimethoxyphenyl)-2-propyl-1-pentanone, 556 1-(3,4-Dimethoxyphenyl)-2-ethyl-1-hexanone, 618 1-(4-Butoxy-2-hydroxyphenyl)-1-hexanone, 666 1-(5-Butoxy-2-hydroxyphenyl)-1-hexanone, 666

 $C_{16}H_{24}O_3$  (cont.) 1-(3,5-Dimethoxyphenyl)-2-methyl-1-heptanone, 730 1-(2-Hydroxy-4-propoxyphenyl)-1-heptanone, 747 1-(2,3-Dimethoxyphenyl)-1-octanone, 778 1-(2,4-Dimethoxyphenyl)-1-octanone, 779 1-(2,5-Dimethoxyphenyl)-1-octanone, 780 1-(2,6-Dimethoxyphenyl)-1-octanone, 781 1-(3,4-Dimethoxyphenyl)-1-octanone, 782 1-(3,5-Dimethoxyphenyl)-1-octanone, 782 1-(4-Ethoxy-2-hydroxyphenyl)-1-octanone, 801 1-(5-Ethoxy-2-hydroxyphenyl)-1-octanone, 801 1-(5-Ethyl-2,4-dihydroxyphenyl)-1-octanone, 802 1-(2-Hydroxy-5-methoxy-4-methylphenyl)-1-octanone, 802 1-(3,5-Dihydroxy-4-methylphenyl)-1-nonanone, 844 1-(2-Hydroxy-4-methoxyphenyl)-1-nonanone, 844 1-(4-Hydroxy-3-methoxyphenyl)-1-nonanone, 845 1-(2,3-Dihydroxyphenyl)-1-decanone, 872 1-(2,4-Dihydroxyphenyl)-1-decanone, 872 1-(2.5-Dihvdroxyphenvl)-1-decanone, 873 1-(2,6-Dihydroxyphenyl)-1-decanone, 873 1-(3,4-Dihydroxyphenyl)-1-decanone, 874 C₁₆H₂₄O₃, 0.5 H₂O 1-(2,4-Dihydroxyphenyl)-1-decanone (Hemihydrate), 872  $C_{16}H_{24}O_4$ 1-(2,4,5-Triethoxyphenyl)-1-butanone, 18 2-Methyl-1-(2,4,6-trimethoxy-3,5-dimethylphenyl)-1-butanone, 137 3-Methyl-1-(2,4,6-trimethoxy-3,5-dimethylphenyl)-1-butanone, 200 1-[3-(3-Methoxypropoxy)-4-methoxyphenyl]-3-methyl-1-butanone, 207 1-[2,6-Dihydroxy-4-[(3-methylbutoxyl)] phenyl]-3-methyl-1-butanone, 216 3-Methyl-1-(2,4,6-trihydroxy-3-pentylphenyl)-1-butanone, 217 1-[2,3,4-Trihydroxy-5-(3-methylbutyl) phenyl]-3-methyl-1-butanone, 217 3-Methyl-1-[2,4,6-trihydroxy-3-(3-methylbutyl)phenyl]-1-butanone, 217 1-[2,4-Dihydroxy-3-(hydroxymethyl)-5-(2-methylpropyl)phenyl]-1-pentanone, 514

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1-(5-Butoxy-2-hydroxyphenyl)-1-hexanone (Oxime), 666 1-(5-Ethoxy-2-hydroxyphenyl)-1-octanone (Oxime), 801 C16H25NO3, HCl 1-[4-(N,N-Diethylaminoethoxy)-2-hydroxyphenyl]-1-butanone (Hydrochloride), 110 C₁₆H₂₅NO₄ 1-[3-Amino-2,4,6-trihydroxy-5-(3-methylbutyl)phenyl]-3-methyl-1-butanone, 219 C₁₆H₂₅NO₄, HCl 1-[3-Amino-2,4,6-trihydroxy-5-(3-methylbutyl)phenyl]-3-methyl-1-butanone (Hydrochloride), 219 C16H25N3O2 1-(4-Butoxy-3-methylphenyl)-1-butanone (Semicarbazone), 52 C16H25N3O3 1-(4,5-Dimethoxy-2-methylphenyl)-1-hexanone (Semicarbazone), 642 1-(3,4-Dimethoxyphenyl)-1-heptanone (Semicarbazone), 727 C₁₇H₉F₁₀NO₄S 2,2,3,3,4,4,4-Heptafluoro-1-(4-phenoxyphenyl)-1-butanone (O-[(Trifluoromethyl) sulfonvlloxime), 289 C17H14Br2O3 1-(3,5-Dibromo-2-benzoyloxyphenyl)-1-butanone, 21  $C_{17}H_{14}Br_2O_6$ 1,5-Bis(5-bromo-2,4-dihydroxyphentl)-1,5-pentanedione, 515 C₁₇H₁₄Cl₂O₄ 1,5-Bis(5-chloro-2-hydroxypheny)l-1,5-pentanedione, 516 C17H14Cl2O6 1,5-Bis(5-chloro-2,4-dihydroxypheny)l-1,5-pentanedione, 516 C₁₇H₁₄F₂O₃ 1-[5-Fluoro-3-(4-fluorobenzoyl)-2-hydroxyphenyl]-1-butanone, 386 C₁₇H₁₄O₆ 4-(2-Hydroxy-4-(phenylmethoxy)phenyl)-2,4-dioxo-1-butanoic acid, 455 C17H15BrN4O8 5-(5-Bromo-2,4-dihydroxyphenyl)-5-oxo-1-pentanoic acid (2,4-Dinitrophenylhydrazone), 586 C₁₇H₁₅ClN₄O₈ 5-(5-Chloro-2,4-dihydroxyphenyl)-5-oxo-1-pentanoic acid (2,4-Dinitrophenylhydrazone), 588

C₁₇H₁₅ClO₃ 1-(4-Chlorophenyl)-4-(4-methoxyphenyl)-1,4-butanedione, 357 C₁₇H₁₅ClO₄ 4-[4-(5-Chloro-2-hydroxyphenylmethyl) phenyl]-4-oxo-1-butanoic acid, 455 C17H16BrFN4O5 1-(3-Bromo-5-fluoro-2-hydroxyphenyl)-1-pentanone (2,4-Dinitrophenylhydrazone), 478 C17H16CIFN4O5 1-(3-Chloro-5-fluoro-2-hydroxyphenyl)-1-pentanone (2.4-Dinitrophenvlhvdrazone), 479 C17H16Cl2N2O6 1,5-Bis(5-chloro-2,4-dihydroxypheny)l-1,5-pentanedione (Dioxime), 516 C₁₇H₁₆N₄O₇ 5-(2-Hydroxyphenyl)-5-oxo-1-pentanoic acid (2,4-Dinitrophenylhydrazone), 578 5-(4-Hydroxyphenyl)-5-oxo-1-pentanoic acid (2,4-Dinitrophenylhydrazone), 579 C17H16N4O8 5-(2,4-Dihydroxyphenyl)-5-oxo-1-pentanoic acid (2,4-Dinitrophenylhydrazone), 581 C17H16O3 1-(2-Benzoyloxyphenyl)-1-butanone, 2 1-(4-Benzoyloxyphenyl)-1-butanone, 5 1-(4-Methoxyphenyl)-4-phenyl-1,3-butanedione, 354 1-(4-Methoxyphenyl)-4-phenyl-1,4-butanedione, 357 C17H16O4 1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1,3-butanedione, 332 1-(2,4-Dihydroxy-6-methylphenyl)-4-phenyl-1,3-butanedione, 355 4-(4'-Methoxybiphenyl)-4-oxo-1-butanoic acid, 452 4-(5-Methoxybiphenyl-4-yl)-4-oxo-1-butanoic acid, 452 4-(6-Methoxybiphenyl-3-yl)-4-oxo-1-butanoic acid, 452 4-[3-(Phenylmethyl)-4-hydroxyphenyl]-4-oxo-1-butanoic acid, 455 4-[5-(Phenylmethyl)-2-hydroxyphenyl]-4-oxo-1-butanoic acid, 456 1,5-Bis(2-hydroxyphenyl)-1,5-pentanedione, 517 1,5-Bis(3-hydroxyphenyl)-1,5-pentanedione, 517 1,5-Bis(4-hydroxyphenyl)-1,5-pentanedione, 518

 $C_{17}H_{16}O_4$  (cont.) 1-(2-Hydroxyphenyl)-5-(4-hydroxyphenyl)-1,5-pentanedione, 519 C17H16O5 1-[2,6-Dihydroxy-4-(phenylmethoxy)phenyl]-1,3-butanedione, 333 4-(4-Methoxy-3-phenoxyphenyl)-4-oxo-1-butanoic acid, 453 C17H16O6 1,5-Bis(2,4-dihydroxyphenyl)-1,5-pentanedione, 520 1,5-Bis(2,5-dihydroxyphenyl)-1,5-pentanedione, 520 1.5-Bis(3.4-dihvdroxvphenvl)-1,5-pentanedione, 521 1,5-Bis(3,5-dihydroxyphenyl)-1,5-pentanedione, 522 C17H16O8 1,5-Bis(2,3,4-trihydroxyphenyl)-1,5-pentanedione, 522 1,5-Bis(2,4,6-trihydroxyphenyl)-1.5-pentanedione, 522 C₁₇H₁₇BrN₄O₅ 1-(5-Bromo-2-hydroxyphenyl)-1-pentanone (2,4-Dinitrophenylhydrazone), 481 C17H17BrO2 2-Bromo-1-[(4-phenylmethoxy)phenyl]-1-butanone, 270 C17H17CIN4O5 4-Chloro-1-(4-methoxyphenyl)-1-butanone (2,4-Dinitrophenylhydrazone), 280  $C_{17}H_{17}ClO_2$ 

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5-Chloro-1-(4-hydroxy[1,1'-biphenyl]-3-yl)-1-pentanone, 576

C₁₇H₁₇FN₄O₆

1-(3-Fluoro-4-methoxyphenyl)-1-butanone (2,4-Dinitrophenylhydrazone), 33

C₁₇H₁₇FO₃

1-[4'-Fluoro-4-hydroxy-6-methoxy[1,1'biphenyl]-3-yl]-1-butanone, 263

C₁₇H₁₇NO₄

1-[(2-Phenylmethoxy)-4-nitrophenyl]-1-butanone, 35

C₁₇H₁₇NO₅

1-(3,4-Dihydroxy-5-nitrophenyl)-5-phenyl-1-pentanone, 523

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1-(2-Hydroxy-5-methylphenyl)-1-dodecanone, 959 1-(4-Hydroxy-2-methylphenyl)-1-dodecanone, 960 1-(4-Hydroxy-3-methylphenyl)-1-dodecanone, 960 1-(3-Hydroxyphenyl)-1-tridecanone, 995 1-(4-Hydroxyphenyl)-1-tridecanone, 996 C₁₈¹⁴CH₃₀O₂ 1-(2-Hydroxy-5-methylphenyl)-14C-1-dodecanone, 957 C19H30O2S 1-[3,5-Bis(1,1-dimethylethyl)-4-hvdroxyphenyl]-3-mercapto-3-methyl-1-butanone, 227 C19H30O3 1-[4-(Nonyloxy)-2-hydroxyphenyl]-1-butanone, 117 1-(2,5-Dihydroxy-4-heptyl-3-methylphenyl)-3-methyl-1-butanone, 228 1-(4-Heptyl-2-hydroxy-5-methoxyphenyl)-3-methyl-1-butanone, 228 1-(2,5-Dihydroxy-4-octylphenyl)-1-pentanone, 535 1-(4-Heptyl-2,5-dihydroxyphenyl)-4-methyl-1-pentanone, 552 1-(2,5-Dihydroxy-3,4,6-trimethylphenyl)-5-methyl-2-(1-methylethyl)-1-hexanone, 685 1-[4-(Hexyloxy)-2-hydroxyphenyl]-1-heptanone, 758 2-Butyl-1-(2,5-dihydroxy-4-methylphenyl)-1-octanone, 810 1-[2,5-Dihydroxy-4-(3-methylbutyl)phenyl]-1-octanone, 810 1-(2,5-Dihydroxy-4-pentylphenyl)-1-octanone, 810 1-(2-Hydroxy-5-pentyloxyphenyl)-1-octanone, 810 1-(4-Butoxy-2-hydroxyphenyl)-1-nonanone, 848 1-(2,4-Dihydroxy-3-propylphenyl)-1-decanone, 891 1-(2-Hydroxy-4-propoxyphenyl)-1-decanone, 891 1-(2,3-Dimethoxyphenyl)-1-undecanone, 925 1-(2,6-Dimethoxyphenyl)-1-undecanone, 926 1-(3,5-Dimethoxyphenyl)-1-undecanone, 927 12-Hydroxy-1-(2-hydroxy-5-methylphenyl)-1-dodecanone, 960 1-(2,4-Dihydroxy-5-methylphenyl)-1-dodecanone, 961

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1-(3,4-Dimethoxyphenyl)-1-decanone (Semicarbazone), 874 C20H18O5 5,7-Dihydroxy-6-(2-methyl-1-oxobutyl)-4-phenyl-2H-1-benzopyran-2-one, 149 5,7-Dihydroxy-8-(2-methyl-1-oxobutyl)-4-phenyl-2H-1-benzopyran-2-one, 150 5,7-Dihydroxy-6-(3-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one, 228 5,7-Dihydroxy-8-(3-methyl-1-oxobutyl)-4-phenyl-2H-1-benzopyran-2-one, 229  $C_{20}H_{18}O_6$ 1,6-Bis(3,4-dimethylenedioxyphenyl)-1.6-hexanedione, 676 C20H18O6Be 1-(4-Hydroxyphenyl)-1,3-butanedione (Be salt), 311 C20H18O6Cu 1-(4-Hydroxyphenyl)-1,3-butanedione (Cu salt), 311  $C_{20}H_{18}O_6Mg$ 1-(4-Hydroxyphenyl)-1,3-butanedione (Mg salt), 311  $C_{20}H_{18}O_6Zn$ 1-(4-Hydroxyphenyl)-1,3-butanedione (Zn salt), 311 C20H19ClO4 3-[2-(4-Chlorobenzoyl)acetyl]-2-hydroxy-5-methyl-1-butanone, 300 1-(3-Acetyl-2-hvdroxy-5-methylphenyl)-1-butanone (p-Chlorobenzoate), 377 C20H20Br2O4 1,6-Bis(4-methoxyphenyl)-2,5-dibromo-1,6-hexanedione, 671 C20H20Br2O6 1,8-Bis(5-bromo-2,4-dihydroxyphenyl)-1.8-octanedione, 811 C20H20Cl2O4 1,4-Bis(3-chloro-6-hydroxy-2,4-dimethylphenyl)-1.4-butanedione, 369 C20H20Cl2O5S 1,1'-[Sulfonylbis(6-chloro-4-hydroxy-3,1-phenylene)]bis-1-butanone, 300 C20H20Cl2O6 1,8-Bis(5-chloro-2,4-dihydroxyphenyl)-1,8-octanedione, 811 C20H20N2O2 1-[8-Hydroxy-4-[(2-methylphenyl)amino]-3-quinolinyl]-1-butanone, 118 C20H20N2O2S 1-[6-[2-(Aminophenyl)thio]-8-methoxy-3-quinolinyl]-1-butanone, 115 C20H20N2O2S, HCl 1-[6-[2-(Aminophenyl)thio]-8-methoxy-

3-quinolinyl]-1-butanone

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C20H22O6 (cont.) 1,4-Bis(4-hydroxy-3-methoxyphenyl)-2,3-dimethyl-1,4-butanedione (+), 370 1,4-Bis(4-hydroxy-3-methoxyphenyl)-2,3-dimethyl-1,4-butanedione (-), 370 1,4-Bis(4-hydroxy-3-methoxyphenyl)-2,3-dimethyl-1,4-butanedione (Meso), 370 1,6-Bis(2,5-dihydroxy-4-methylphenyl)-1,6-hexanedione, 687 1,6-Bis(2-hydroxy-5-methoxyphenyl)-1,6-hexanedione, 687 1,6-Bis(4-hydroxy-3-methoxyphenyl)-1.6-hexanedione, 687 1,8-Bis(2,4-dihydroxyphenyl)-1.8-octanedione, 812 1,8-Bis(3,4-dihydroxyphenyl)-1.8-octanedione, 812 1,8-Bis(3,5-dihydroxyphenyl)-1,8-octanedione, 813 C20H22O7 5,7-Diacetyloxy-6-(1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one, 104 C20H22O8 1,4-Bis(2-hydroxy-3,4-dimethoxylphenyl)-1,4-butanedione, 371 1,8-Bis(2,3,4-trihydroxyphenyl)-1.8-octanedione, 813 C20H23BrN4O5 1-[3-Bromo-2-hydroxy-5-(1,1-dimethylethyl) phenyl]-1-butanone (2,4-Dinitrophenylhydrazone), 89 C₂₀H₂₃BrO₂ 1-(3'-Bromo-4'-methoxy[1,1'-biphenyl]-4-yl)-1-heptanone, 756 1-(3'-Bromo-4'-hydroxy[1,1'-biphenyl]-4-yl)-1-octanone, 814 C₂₀H₂₃BrO₅ 4-(2-Bromo-4,5-dimethoxyphenyl)-1-(3,4-dimethoxyphenyl)-1-butanone, 118 C20H23CIN4O5 1-[3-Chloro-2-hydroxy-5-(1,1-dimethylethyl) phenyl]-1-butanone (2,4-Dinitrophenylhydrazone), 89 1-(4-Chloro-2-hydroxyphenyl)-1-octanone (2,4-Dinitrophenylhydrazone), 787  $C_{20}H_{23}ClO_2$ 3-[4-(2-Chloroethyl)phenyl]-1-(2-hydroxyphenyl)-1-hexanone, 688 1-(3'-Chloro-4'-hydroxy[1,1'-biphenyl]-4-yl)-1-octanone, 814

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## C₂₂H₂₄Cl₂O₄ 1,8-Bis(4-hydroxy-2-methylphenyl)-1,8-octanedione, 818

1,10-Bis-(5-chloro-2-hydroxyphenyl)-1,10-decanedione, 898

 $C_{22}H_{24}Cl_2O_5S$ 

1,1'-[Sulfonylbis(6-chloro-4-hydroxy-3,1-phenylene)]bis-1-pentanone, 537

C22H24Cl2O6

1,10-Bis(5-chloro-2,4-dihydroxyphenyl)-1,10-decanedione, 899

 $C_{22}H_{24}F_2O_4$ 

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C₂₂H₂₈O₇

- 2-Methyl-1-[2,4,6-tris(acetyloxy)-3-(3-methyl-2-butenyl)phenyl]-1-butanone, 142
- C22H28O7S
- 1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-hexanone (p-Toluenesulfonate), 660

 $C_{22}H_{29}NO_4$ 

- 1-[3-[[(2,5-Dihydroxyphenyl)methyl]amino]-2-hydroxyphenyl]-3,5,5-trimethyl-1-hexanone, 692
- 1-[5-[[(2,5-Dihydroxyphenyl)methyl]amino]-2-hydroxyphenyl]-3,5,5-trimethyl-1-hexanone, 692

C₂₂H₂₉N₃O₂

1-(4-Hydroxyphenyl)-1-decanone (iso-Nicotinylhydrazone), 870

C₂₂H₃₀N₂O

- 1-(4-Methoxyphenyl)-1-nonanone (Phenylhydrazone), 837
- C22H30O4
- 1-(2-Acetyl-4-hydroxy-7-benzofuranyl)-1-dodecanone, 971
- 1-(2-Acetyl-7-hydroxy-4-benzofuranyl)-1-dodecanone, 971
- C22H30O5
- 5,7-Dihydroxy-6-(3-methylbutyl)-8-(3-methyl-1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one, 239
- 5,7-Dihydroxy-8-(3-methylbutyl)-6-(3-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one, 240

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- 1-(8-Methoxy-5-quinolinyl)-1-dodecanone, 968
- 1-(8-Hydroxy-2-methyl-5-quinolinyl)-1-dodecanone, 972
- 1-(8-Hydroxy-2-methyl-7-quinolinyl)-1-dodecanone, 972
- C₂₂H₃₂O₁₂
- 1-(2,4-Dihydroxyphenyl)-1-butanone (Di-β-Dglucoside), 10

C22H32O3

1-(2-Hexyl-6-hydroxy-5-benzofuranyl)-1-octanone, 820

C22H32O4 1-(4,7-Dimethoxy-2,3-dimethyl-6-benzofuranyl)-1-decanone, 894 C22H32O5 1-(2,4-Diacetyloxyphenyl)-1-dodecanone, 945 C₂₂H₃₃ClO₃ 1-[4-(2-Chloroallyloxy)-2-hydroxyphenyl]-1-tridecanone, 1000 C22H33ClO3, NiCl2, 6 H2O 1-[4-(2-Chloroallyloxy)-2-hydroxyphenyl]-1-tridecanone (Compound with nickel chloride, hexahydrate), 1000 C22H33O13 1-[2-[(6-O-D-Apio-β-D-furanosyl-β-Dglucopyranosyl)oxy]-4,6-dihydroxyphenyl]-2-methyl-1-butanone (2S), 156 C22H34Br2O3 1-(3,5-Dibromo-2,4-dihydroxyphenyl)-1-hexadecanone, 1039  $C_{22}H_{34}Na_2O_3$ 1-(2,4-Dihydroxyphenyl)-1-hexadecanone (Na salt), 1035  $C_{22}H_{34}O_2$ 1-[4-(10-Undecenyloxy)phenyl]-1-pentanone, 469 C22H34O3 1-(4-Capryloxyphenyl)-1-octanone, 776  $C_{22}H_{34}O_4$ 1,1'-(4,6-Dihydroxy-1,3-phenylene)bis-1-octanone, 821 1-(2-Hydroxy-5-octanoyloxyphenyl)-1-octanone, 821 Methyl 2-Hydroxy-5-tetradecanoylbenzoate, 1011 C22H34O5 4-(3,4-Dihexyloxyphenyl)-4-oxo-1-butanoic acid, 410 1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-octanone, 821 1-(6,7-Dimethoxy-5-propyl-1,3-benzodioxol-4-yl)-1-decanone, 894 16-(2,4-Dihydroxyphenyl)-16-oxo-1-hexadecanoic acid, 1053 C22H34O6 4-(2,4,5-Tributoxyphenyl)-4-oxo-1-butanoic acid, 413 10-(Acetyloxy)-1-(2,3,4-trimethoxy-6-methylphenyl)-1-decanone, 893 11-Acetyloxy-1-(2-hydroxy-3,4-dimethoxy-6-methylphenyl)-1-undecanone, 931  $C_{22}H_{35}BrO_2$ 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-8-bromo-1-octanone, 830

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- 1,6-Bis(2,3,4-trimethoxyphenyl)-1,6-hexanedion, 677
- 1,8-Bis(2-hydroxy-3,4-dimethoxyphenyl)-1,8-octanedione, 825

# C24H30O12

- 1-[2-(Tetraacetyl-β-D-glucopyranosyloxy)-4-hydroxyphenyl]-1-butanone, 124
- 1-[4-(Tetraacetyl-β-D-glucopyranosyloxy)-2-hydroxyphenyl]-1-butanone, 124

#### C24H31CIN2O4

5-[[(5-Chloro-2-hydroxyphenyl)azo]-2,4-dihydroxyphenyl]–1-dodecanone, 977

# C24H31CIN4O5

- 1-[3-Chloro-2-hydroxy-5-(1,1-dimethylethyl) phenyl]-1-octanone (2,4-Dinitrophenylhydrazone), 806
- 1-(4-Chloro-2-hydroxyphenyl)-1-dodecanone (2,4-Dinitrophenylhydrazone), 955

# $C_{24}H_{31}ClO_2$

1-[5-Chloro-3-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-decanone, 910

- 1-(3'-Chloro-4'-hydroxy[1,1'-biphenyl]-4-yl)-1-dodecanone, 978  $C_{24}H_{31}FN_4O_5$ 1-(5-Fluoro-2-hydroxyphenyl)-1-dodecanone (2,4-Dinitrophenylhydrazone), 956  $C_{24}H_{31}FO_2$ (3' Eluoro 4' hydroxy[1,1' hiphenyll 4, yl)
- 1-(3'-Fluoro-4'-hydroxy[1,1'-biphenyl]-4-yl)-1-dodecanone, 978

# $C_{24}H_{32}N_2O_3\\$

- 1-[4-(4-Hexanoylphenyloxy)phenyl]-1-hexanone (Dioxime), 605
  - I-nexanone (Dioxime), b
- $C_{24}H_{32}N_2O_4$
- 1,10-Bis(2-hydroxy-3-methylphenyl)-1,10-decanedione (Dioxime), 907
- 1,10-Bis(2-hydroxy-5-methylphenyl)-1,10-decanedione (Dioxime), 908
- 5-[[(2-Hydroxyphenyl)azo]-2,4-dihydroxyphenyl]-1-dodecanone, 978

#### C24H32N2O6

- 1,6-Bis(4-ethoxy-3-methoxyphenyl)-1,6-hexanedione (Dioxime), 688
- 1,8-Bis(3,4-dimethoxyphenyl)-
  - 1,8-octanedione (Dioxime), 813
- $C_{24}H_{32}N_2O_7S$
- 5-[[(2-Hydroxy-5-sulfonylphenyl)azo]-2,4-dihydroxyphenyl]-1-dodecanone, 979
- $C_{24}H_{32}N_2O_8$
- 1,8-Bis(2-hydroxy-3,4-dimethoxyphenyl)-1,8-octanedione (Dioxime), 825
- $C_{24}H_{32}N_4O_5$
- 1-(4-Heptyl-2-hydroxyphenyl)-3-methyl-1-butanone
- (2,4-Dinitrophenylhydrazone), 225 1-[3-(1,1-Dimethylethyl)-2-hydroxy-
- 6-methylphenyl]-1-heptanone (2,4-Dinitrophenylhydrazone), 752
- 1-[3-Hydroxy-4-(3-methylbutyl) phenyl]-1-heptanone (2,4-Dinitrophenylhydrazone), 753
- 1-[2-Hydroxy-3-methyl-6-(1-methylethyl) phenyl]-1-octanone (2,4-Dinitrophenylhydrazone), 806
- 1-[2-Hydroxy-6-methyl-3-(1-methylethyl) phenyl]-1-octanone
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- (2,4-Dinitrophenylhydrazone), 886 1-(5-Ethyl-2-hydroxyphenyl)-1-decanone
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- 1-decanone (2,4-Dinitrophenylhydrazone), 887

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1-benzopyran-2-one, 238

C₂₄H₃₂O₇S 1-[2-(p-Toluenesulfonyloxy)-3,4,6-trimethoxyphenyl]-1-octanone, 804

 $\begin{array}{c} C_{24}H_{33}N_3O_3 \\ 1-(4-Heptyl-2-hydroxyphenyl)-3-methyl- \\ 1-butanone \\ (p-Nitrophenylhydrazone), 225 \\ C_{24}H_{33}N_3O_4 \\ 1-(2,4-Dihydroxyphenyl)-1-dodecanone \\ (4-Nitrophenylhydrazone), 945 \\ C_{24}H_{33}N_3O_5 \\ 1-(2,3,4-Trihydroxyphenyl)-1-dodecanone \end{array}$ 

(4-Nitrophenylhydrazone), 950

C24H33N3O6S

5-[[(5-Aminosulfonyl-2-hydroxyphenyl)azo]-2,4-dihydroxyphenyl]-1-dodecanone, 979

C₂₄H₃₄Br₆O₃

- 9,10,12,13,15,16-Hexabromo-1-(3,4-dihydroxyphenyl)-1-octadecanone, 1090
- C₂₄H₃₄NO₃
- 1-(8-Hydroxy-2-methyl-5-quinolinyl)-1-dodecanone
  - (N-Methylcarbamate), 972
- C₂₄H₃₄N₄O
- 1-[4-(4-Hexanoylphenyloxy)phenyl]-1-hexanone (Dihydrazone), 605

# C24H34O5

- 5,7-Dimethoxy-6-(3-methylbutyl)-8-(3-methyl-1-oxobutyl)-4-propyl-2H-1-benzopyran-2-one, 239
- 5,7-Dimethoxy-8-(3-methylbutyl)-6-(3-methyl-1-oxobutyl)-4-propyl-2*H*-1-benzopyran-2-one, 240
- 1-[3-Acetyl-4-[[(2E)-3,7-dimethyl-2,6-octadienyl]oxy]-2,6-dihydroxy-5-methylphenyl]-2-methyl-1-butanone, 384

# C24H34O7

- 1-(2,4,6-Triacetyloxyphenyl)-1-dodecanone, 952
- C24H35NO3
- 5-[5-Hydroxy-4-(1-oxodecyl)-2-(2-propenyl) phenoxy]pentanenitrile, 911
- C₂₄H₃₆Br₄O₃
- 9,10,12,13-Tetrabromo-1-(3,4-dihydroxyphenyl)-1-octadecanone, 1090

# C24H36O2

1-(2-Hydroxyphenyl)-9-octadecyn-1-one, 1061  $C_{24}H_{36}O_2$  (cont.) 1-(4-Hydroxyphenyl)-9-octadecyn-1-one, 1061 C24H36O4 1-(4,7-Dihydroxy-2,3-dimethyl-6-benzofuranyl)-1-tetradecanone, 1018 C24H36O5 1-(2,6-Diacetyloxyphenyl)-1-tetradecanone, 1007

#### C24H38Br2O2

1-(3,5-Dibromo-4-hydroxyphenyl)-1-octadecanone, 1070 1-(3,5-Dibromo-2,4-dihydroxyphenyl)-1-octadecanone, 1070 C24H38Cl2O3 1-(3,5-Dichloro-2,6-dihydroxyphenyl)-1-octadecanone, 1070 C24H38O3 1-[3,5-Bis(1,1-dimethylethyl)-4-acetyloxyphenyl]-1-octanone, 822 1-(2-Acetyloxyphenyl)-1-hexadecanone, 1032 1-(4-Acetyloxyphenyl)-1-hexadecanone, 1034 C24H38O4 1-[2-(Acetyloxy)-4-hydroxyphenyl]-1-hexadecanone, 1043 C24H38O5 1,1'-(2,4-Dibutoxy-6-hydroxy-1.3-phenylene)bis-3-methyl-1-butanone, 307 1,1'-(4,6-Dibutoxy-2-hydroxy-1,3-phenylene) bis-3-methyl-1-butanone, 307 1,1'-(4-Hydroxy-2,6-dimethoxy-1,3-phenylene)bis-1-octanone, 826 1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-nonanone, 862 C24H39BrO2 1-(3-Bromo-4-hydroxyphenyl)-1-octadecanone, 1071 C24H39BrO3 17-Bromo-1-(4-hydroxy-3-methoxyphenyl)-1-heptadecanone, 1060 1-(5-Bromo-2,4-dihydroxyphenyl)-1-octadecanone, 1071 C24H39BrO4 1-(5-Bromo-2,3,4-trihydroxyphenyl)-1-octadecanone, 1071 C24H39ClO2 1-[4-(2-Chloroethyloxy)phenyl]-1-hexadecanone, 1034 1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-heptadecanone, 1057 1-(3-Chloro-4-hydroxyphenyl)-1-octadecanone, 1071

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C24H40O6 1-(2,4,6-Trihydroxyphenyl)-12,14-dihydroxyoctadecanone, 1069 C24H41NO2 1-[2-Hydroxy-5-(1,1,3,3-tetramethylbutyl) phenyl]-1-decanone (Oxime), 911 1-[4-(N-Diethylaminoethyloxy)phenyl]-1-dodecanone, 944 1-[4-(N-Dimethylaminobutyloxy)phenyl]-1-dodecanone, 944 1-[4-(N-Dimethylaminoethyloxy)phenyl]-1-tetradecanone, 1005 1-(2-Methoxy-5-methylphenyl)-1-hexadecanone (Oxime), 1041 C24H41NO3 1-[4-(Dodecyloxy)-2-hydroxyphenyl]-1-hexanone (Oxime), 694 1-[2-Hydroxy-4-(octyloxy)phenyl]-1-decanone (Oxime), 912 1-(4-Butoxy-2-hydroxyphenyl)-1-tetradecanone (Oxime), 1019  $C_{24}H_{42}N_2O_2$ 1-[3,5-Bis(dimethylaminomethyl)-4-hydroxyphenyl]-1-dodecanone, 979 C25H20O4 1-(5-Methoxy-2,3-diphenyl-6-benzofuranyl)-1,3-butanedione, 334 C25H22O3 1-(6-Hydroxy-3-(4-methylphenyl)-2-phenyl-5-benzofuranyl)-1-butanone, 124  $C_{25}H_{22}O_4$ 1-(6-Hydroxy-3-(4-methoxyphenyl)-2-phenyl-5-benzofuranyl)-1-butanone, 125  $C_{25}H_{22}O_6$ Methyl 4-(2,4-di(phenylmethoxy)phenyl)-2,4-dioxo-1-butanoate, 397  $C_{25}H_{24}O_4$ 1-(2,4-Diphenylmethoxy-6-methylphenyl)-1.3-butanedione, 319 C25H24O5 5-Hydroxy-8,8-dimethyl-6-(3-methyl-1-oxobutyl)-4-phenyl-2H;8H-pyrano [2,3-f]chromen-2-one, 243  $C_{25}H_{24}O_6$ 5-Hydroxy-8-(1-hydroxy-1-methylethyl)-6-(2-methyl-1-oxobutyl)-4-phenyl-2Hfuro[2',3':5,6]benzo[1,2-b]pyran-2-one, 159 5-Hydroxy-8-(1-hydroxy-1-methylethyl)-6-(3-methyl-1-oxobutyl)-4-phenyl-2Hfuro[2',3':5,6]benzo[1,2-b]pyran-2-one, 244

#### C25H26N2O3

1-(4-Benzoyloxy-3-methoxyphenyl)-1-pentanone (Phenylhydrazone), 498

# C25H26O3

1-(4-Benzyloxy-3-methoxyphenyl)-5-phenyl-1-pentanone, 529

# C25H26O4

1-[(2,5-Diphenylmethoxy)-4-methoxyphenyl]-1-butanone, 58

3-Methyl-1-[2,4,6-trihydroxy-3,5-bis(phenylmethyl)phenyl]-1-butanone, 244

# C25H26O5

5,7-dihydroxy-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one, 160

- 5,7-dihydroxy-8-(3-methyl-2-butenyl)-6-(2-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one, 160
- 5,7-Dihydroxy-6-(3-methyl-2-butenyl)-8-(3-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one, 245
- 5,7-Dihydroxy-8-(3-methyl-2-butenyl)-6-(3-methyl-1-oxobutyl)-4-phenyl-2H-1-benzopyran-2-one, 245
- 9,10-Dihydro-8,8-dimethyl-5-hydroxy-6-(3-methyl-1-oxobutyl)-4-phenyl-8Hpyrano-[2,3-f]chromen-2-one, 247

# C25H26O6

- 9,10-Dihydro-5,9-dihydroxy-8,8-dimethyl-6-(2-methyl-1-oxobutyl)-4-phenyl-2H,8H-benzo[1,2-b:5,6-b']-dipyran-2-one, 161
- 8,9-Dihydro-5-hydroxy-8-(1-hydroxy-1-methylethyl)-6-(3-methyl-1-oxobutyl)-4-phenyl-2*H*-furo [2',3':5,6]benzo[1,2-b]pyran-2-one (Racemic), 247
- 8,9-Dihydro-5-hydroxy-8-(1-hydroxy-1-methylethyl)-6-(3-methyl-1-oxobutyl)-4-phenyl-2*H*-furo [2',3':5,6]benzo[1,2-b]pyran-2-one, 247

# $C_{25}H_{28}O_5$

5,7-Dihydroxy-8-(3-methylbutyl)-6-(3-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one, 248

# C25H28O6

1,9-Bis(4-acetyloxyphenyl)-1,9-nonanedione, 850

# C25H28O7

5,7-Dihydroxy-8-(2,3-dihydroxy-3-methylbutyl)-6-(2-methyl-1-oxobutyl)-4-phenyl-2*H*-1-benzopyran-2-one, 161

# C25H28O8

1,7-Bis(2-acetyloxy-5-methoxyphenyl)-1,7-heptanedione, 761

# C25H28O10

- 1,1'-[Methylenebis(2,4,6-trihydroxy-3-acetyl-5,1-phenylene)]bis-1-butanone, 341
- 1,1'-Methylenebis[2,4,6-trihydroxy-3-(1-oxomethyl)-5,1-phenylene]bis-3-methyl-1-butanone, 394
- C25H30Cl2O6
- 1,9-Bis(5-chloro-2,4-dimethoxyphenyl)-1,9-nonanedione, 850
- C25H30O7
- 3-[[2,3-Dihydro-4,6-dihydroxy-2-(1-methylethenyl)-5-(2-methyl-1-oxobutyl)-7-benzo-furanyl]methyl]-6-ethyl-4-hydroxy-5-methyl-2*H*-pyran-2-one, 162
- 3-[[5,7-Dihydroxy-2,2-dimethyl-6-(2-methyl-1-oxobutyl)-2*H*-1-benzopyran-8-yl]methyl]-6-ethyl-4-hydroxy-5-methyl-2*H*-pyran-2-one, 162

# $C_{25}H_{30}O_9$

- 5-[2,6-Dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl)phenyl] methyl-3-(3-methyl-1-oxobutyl)-2,4,6-trihydroxybenzaldehyde, 394
- $C_{25}H_{31}BrN_4O_5$
- 1-[3-Bromo-2-allyloxy-5-(1,1-dimethylethyl) phenyl]-1-hexanone (2,4-Dinitrophenylhydrazone), 664

# C25H31ClO4

- 1-[5-(4-Chlorobenzoyl)-2,4-dihydroxyphenyl]-1-dodecanone, 980
- C25H32Cl2N2O6
- 1,9-Bis(5-chloro-2,4-dimethoxyphenyl)-1,9-nonanedione (Dioxime), 850
- $C_{25}H_{32}N_2O_5$
- 5-[[(2-Carboxyphenyl)azo]-2,4-dihydroxyphenyl]-1-dodecanone, 980

# C25H32O3

- 1-(4-Benzoyloxyphenyl)-1-dodecanone, 942
- 1-(4-Benzoyl-3-hydroxyphenyl)-
  - 1-dodecanone, 980

# C25H32O4

- 1,5-Bis(4-butyloxyphenyl)-1,5-pentanedione, 519 1,9-Bis(4-methoxy-2-methylphenyl)-1,9-nonanedione, 859
- 1,9-Bis(4-methoxy-3-methylphenyl)-1,9-nonanedione, 860
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C27H32O8

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C27H34O9

- 1-[6-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-3,4-dihydro-3,5,7-trihydroxy-2,2-dimethyl-2*H*-1-benzopyran-8-yl]-3-methyl-1-butanone, 344
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C30H44N2O4 3-[(4-Octylphenylazo)-2,4,6-trihydroxyphenyl]-1-decanone, 915 C30H44N2O6 1,6-Bis(3,4-dipropyloxyphenyl)-1,6-hexanedione (Dioxime), 676 C30H44N4O5 1-[2-Hydroxy-6-methyl-3-(1-methylethyl) phenyl]-1-tetradecanone (2,4-Dinitrophenylhydrazone), 1018 1-(2-Hydroxyphenyl)-1-octadecanone (2,4-Dinitrophenylhydrazone), 1062 1-(4-Hvdroxvphenvl)-1-octadecanone (2,4-Dinitrophenylhydrazone), 1064 C30H44N4O6 1-(2,5-Diethyloxyphenyl)-1-tetradecanone (2,4-Dinitrophenylhydrazone), 1006 C₃₀H₄₄O₂ 1-(4'-Dodecyloxy[1,1'-biphenyl]-4-yl)-1-hexanone, 681 1-(4'-Decyloxy[1,1'-biphenyl]-4-yl)-1-octanone, 815 1-(4'-Nonyloxy[1,1'-biphenyl]-4-yl)-1-nonanone, 854 1-(4'-Octyloxy[1,1'-biphenyl]-4-yl)-1-decanone, 905 C30H44O5 1-[2,3,4-Trimethoxy-6-(phenylmethoxy) phenyl]-1-tetradecanone, 1022 C30H44O7S 1-[2-(4-Toluenesulfonyloxy)-3,4,6-trimethoxyphenyl]-1-tetradecanone, 1017 1-[3,4,6-Trimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]-1-tetradecanone, 1022 C₃₀H₄₅NO₃ 1-(2-Amino-3,6-dihydroxy[1,1'-biphenyl]-4-yl)-2-hexyl-1-dodecanone, 986 C30H45N3O4 1-(2,4-Dihydroxyphenyl)-1-octadecanone (4-Nitrophenylhydrazone), 1065 C₃₀H₄₅N₃O₅ 1-(2,3,4-Trihydroxyphenyl)-1-octadecanone (4-Nitrophenylhydrazone), 1068 C₃₀H₄₆N₂O₂ 1-(2,5-Dimethoxyphenyl)-1-hexadecanone (Phenylhydrazone), 1036 C30H46O9 1-(2,4,6-Triacetyloxyphenyl)-12,14-dihydroxyoctadecanone, 1069

C30H50O2 1-(5-Dodecyl-2-hydroxyphenyl)-1-dodecanone, 986 C30H50O3 1-(4-Lauryloxyphenyl)-1-dodecanone, 943 C₃₀H₅₀O₄ 1,1'-(2,5-Dihydroxy-1,4-phenylene)bis-1-dodecanone, 986 1,1'-(4,6-Dihydroxy-1,3-phenylene)bis-1-dodecanone, 986 C30H50O5 1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-dodecanone, 987 1-(2-Hydroxy-4,6-dimethoxy-3,5-dimethylphenyl)-1,3-eicosanedione, 1100 C30H51NO5 5-(1-Octadecanoyl)-2-hydroxybenzoic acid (N-methylmorpholine salt), 1075 C30H52O2 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-hexadecanone, 1051 1-(4-Hydroxy-3,5-bis(1-methylethyl)phenyl)-1-octadecanone, 1084 1-(2-Methoxy-5-methylphenyl)-1-docosanone, 1107 1-(4-Hvdroxy-2,5-dimethylphenyl)-1-docosanone, 1107 1-(4-Hydroxyphenyl)-1-tetracosanone, 1113 C30H52O3 1-(2,5-Dihydroxy-4-dodecylphenyl)-1-dodecanone, 987 1-(2-Dodecyl-4,5-dihydroxyphenyl)-1-dodecanone, 987 1-(3,4-Dihydroxyphenyl)-1-tetracosanone, 1113 C30H52O4 1-(3-Dodecyl-2,4,6-trihydroxyphenyl)-1-dodecanone, 988 1-(2,4,6-Trihydroxyphenyl)-1-tetracosanone, 1114 C₃₀H₅₃NO₂ 1-[4-(N-Diethylaminoethyloxy)phenyl]-1-octadecanone, 1065 C31H28N8O10 1,5-Bis(4-methoxyphenyl)-1,5-pentanedione (Di-2,4-Dinitrophenylhydrazone), 519 C31H28N8O12 1,7-Bis(2,4-dihydroxyphenyl)-1,7-heptanedione (2,4-Dinitrophenylhydrazone), 755

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C32H48N4O6 1-(2,5-Diethyloxyphenyl)-1-hexadecanone (2,4-Dinitrophenylhydrazone), 1036  $C_{32}H_{48}O_2$ 1-(4'-Dodecyloxy[1,1'-biphenyl]-4-yl)-1-octanone, 815 1-(4'-Decyloxy[1,1'-biphenyl]-4-yl)-1-decanone, 905 C32H48O7S 1-[2-(p-Toluenesulfonoxy)-3,4,6-trimethoxyphenyl]-1-hexadecanone, 1048 C32H49N3O4 1-(3,5-Dimethoxy-4-methylphenyl)-1-heptadecanone (4-Nitrophenylhydrazone), 1057 1-(2-Hydroxy-4-methoxy-3,6-dimethylphenyl)-1-heptadecanone (4-Nitrophenylhydrazone), 1058 C32H49N3O5 1-(3,4,5-Trimethoxyphenyl)-1-heptadecanone (4-Nitrophenylhydrazone), 1057 C32H54O6 Ethyl 2-(3,4,5-trimethoxybenzoyl) eicosanoate, 1099 22-(2-Methoxy-3,4-dimethoxy-6-methylphenyl)-22-oxo-1-docosanoic acid. 1108 Methyl 22-(2-Hydroxy-3,4-dimethoxy-6-methylphenyl)-22-oxo-1-docosanoate, 1109 C32H55O2K 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-octadecanone (K salt), 1086 C32H56O2 1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-octadecanone, 1086 C32H56O3 1-(2-Dodecyl-4,5-dimethoxyphenyl)-1-dodecanone, 987 C32H56O4 1-(2,4,6-Trihydroxyphenyl)-1-hexacosanone, 1117 C33H30O6 1-(2,4,6-Tribenzyloxyphenyl)-1,3,5-hexanetrione, 596 C33H32N8O10 1,9-Bis(4-hydroxyphenyl)-1,9-nonanedione (Di-2,4-dinitrophenylhydrazone), 850 C₃₃H₃₂N₈O₁₂ 1,5-Bis(3,4-dimethoxyphenyl)-1,5-pentanedione (Di-2,4-Dinitrophenylhydrazone), 521

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# C₃₆H₄₄O₁₂

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2,4,6-trihydroxyphenyl)furan-2-ylmethyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone, 349

# C₃₇H₄₆O₁₂

1-[3,5-Bis[[2,6-dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl) phenyl]methyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-butanone, 168

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- C37H52O10
- 1,1'-Methylenebis(2,4,6-trihydroxy-3,5,1-phenylene)bis-1-hexanone, 695 C₃₇H₅₄O₅
- 1,1'-(5-Benzoyl-2,4-dihydroxy-1,3-phenylene) bis-1-dodecanone, 992
- C37H62O5

4-(24-Acetyloxy-1-oxo-5-npropyltetracosanyl)phenol acetate, 1114

- C37H64O4
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- (C19H19O4)2Cu
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- C38H38O11
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- C₃₈H₄₀Cl₂N₈O₁₂
- 1,10-Bis(5-chloro-2,4-dimethoxyphenyl)-1,10-decanedione

(Di-2,4-dinitrophenylhydrazone), 899

- C38H42N8O10
- 1,10-Bis(4-methoxy-2-methylphenyl)-1,10-decanedione (Di-2,4-dinitrophenylhydrazone), 908
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  - 1,10-decanedione
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# C₃₈H₄₂N₈O₁₂

- 1,10-Bis(2,4-dimethoxyphenyl)-
  - 1,10-decanedione
    - (Di-2,4-dinitrophenylhydrazone), 902

# C38H44O15

1-[3-[(3-Acetyl-2,4-diacetyloxy-6-methoxy-5-methylphenyl)methyl]-2,4,6-tris (acetyloxy)-5-(2-acetyloxy-3-methyl-3-butenyl)phenyl]-1-butanone, 344

# C38H47NO10

1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl)pyridin-2-ylmethyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone, 349

# C₃₈H₄₈O₁₁

3"-Mergtlachyroclinopyrone (Tetraacetate), 168

# C38H48O21

1-(2,4-Dihydroxyphenyl)-1-butanone (Di-tetraacetyl-β-D-glucoside), 11

# C38H50O10

1,18-Bis(2,4-diacetyloxyphenyl)-1,18-octadecanedione, 1084

# C38H58O4

1,1'-(4,4'-Dihydroxy-5,5'-dimethyl [1,1'-biphenyl]-3,3'-diyl)bis-1-dodecanone, 992

# C38H61NO3

1-(2-Amino-3,6-dibutyloxy[1,1'-biphenyl]-4-yl)-2-hexyl-1-dodecanone, 986

# C38H66O3

1-(4-Hydroxyphenyl)-1-hexadecanone (Palmitate), 1035

# C38H66O4

1-(2,4,6-Trihydroxyphenyl)-1-[24-dotriacontenone, 1121

# C₃₈H₆₆O₅

1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-hexadecanone, 1052

# C₃₉H₃₆O₁₀

1,5-Bis(2,4-diacetyloxyphenyl)-2,4-di-(4-methylphenyl)-1,5-pentanedione, 540

# C₃₉H₃₆O₁₂

1,5-Bis(2,4-diacetyloxyphenyl)-2,4-di-(4-methoxyphenyl)-1,5-pentanedione, 540 C₃₉H₄₆O₄

1-(2,4,6-Tribenzyloxyphenyl)-1-dodecanone, 952

# C39H48O10

1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl)phenylmethyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone, 350

# C39H48O11

1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl)-(4-hydroxyphenyl)methyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone, 350 C₃₉H₅₄O₃

#### 2-Butyl-1-(2,5-dibenzyloxy-3,4,6-trimethylphenyl)-1-dodecanone, 981

# C39H60O4

1,1'-[Methylenebis(2-hydroxy-3-methyl-

5,1-phenylene)]bis-1-dodecanone, 992

# C40H48O12

1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl)-3,4-methylenedioxy-phenylmethyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone, 351

# C40H62O4

1,1'-(4,4'-Dihydroxy[1,1'-biphenyl]-3,3'-diyl)-1-tetradecanone. 1022

# C40H62O6

1,1'-(6,6'-Dihydroxy-5,5'-dimethoxy [1,1'-biphenyl]-3,3'-diyl)bis-1-tridecanone, 1001

#### C41H52O10

1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl) phenylethylmethyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone, 351

#### C42H42O7

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- 3-Methyl-1-[2,4,6-tribenzoyloxy-3,5-bis (3-methyl-2-butenyl)phenyl]-1-butanone. 232

# C42H46O7

3-Methyl-1-[2,4,6-tribenzoyloxy-3,5-bis (3-methylbutyl)phenyl]-1-butanone, 234

# $C_{42}H_{49}NO_{10}$

1-[3-Isopentanoyl-5-[(3,5-diisopentanoyl-2,4,6-trihydroxyphenyl)quinolin-4-ylmethyl]-2,4,6-trihydroxyphenyl]-3-methyl-1-butanone, 352

# C42H50N8O10

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#### $C_{72}H_{84}O_{12}$

5,11,17,23,29,35-Hexakis(2-methylbutanoyl)-37,38,39,40,41,42-hexahydroxycalix[6] arene, 353

#### C₇₈H₉₆O₁₂

5,11,17,23,29,35-Hexahexanoyl-37,38,39,40, 41,42-hexahydroxycalix[6]arene, 696

# **Chemical Abstracts Registry Numbers**

[114-42-1]	3,5-Dihydroxy-4,4-dimethyl-2-(1-oxobutyl)-6-[[2,4,6-trihydroxy-3-methyl- 5-(1-oxobutyl)phenyl]methyl]-2,5-cyclohexadien-1-one, 341
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R. Martin, J.-P. Buisson, Aromatic Hydroxyketones: Preparation & Physical Properties, DOI 10.1007/978-3-319-14185-5

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[3226-18-4]	1-(3,5-Dichloro-2-hydroxyphenyl)-1-hexanone, 621
[3226-27-5]	1-(2-Hydroxyphenyl)-1-octanone, 772
[3226-35-5]	1-(3-Chloro-4-hydroxyphenyl)-1-hexanone, 624
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[3307-04-8]	1-(4,5-Dimethoxy-2-methylphenyl)-1-hexanone, 642
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[4091-11-6]	2-Bromo-1-(2,3-dichloro-4-hydroxyphenyl)-2-ethyl-1-butanone, 293
[4101-16-0]	1-(3,4-Dimethoxyphenyl)-4-methyl-1-pentanone, 544
[4115-00-8]	1-(2,3-Dichloro-4-hydroxyphenyl)-2-methylene-1-butanone, 131
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[4609-08-9]	1,5-Bis(5-chloro-2-hydroxy-4-methylphenyl)-1,5-pentanedione, 530
[4609-09-0]	1,5-Bis(5-chloro-2-hydroxy-4-methylphenyl)-1,5-pentanedione (Dioxime), 530
[4609-10-3]	5-(4-Methoxyphenyl)-5-oxo-1-pentanoic acid, 579
[4609-11-4]	1,5-Bis(4-methoxyphenyl)-1,5-pentanedione, 518
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[4642-34-6]	1,5-Bis(4-hydroxy-2-methylphenyl)-1,5-pentanedione, 532
[4642-35-7]	5-(2-Hydroxy-4-methylphenyl)-5-oxo-1-pentanoic acid, 591
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[4642-36-8]	5-(2-Hydroxy-4-methylphenyl)-5-oxo-1-pentanoic acid (2,4-Dinitrophenylhydrazone), 591
[4642-37-9]	5-(4-Methoxy-2-methylphenyl)-5-oxo-1-pentanoic acid, 592
[4642-38-0]	1,5-Bis(4-methoxy-2-methylphenyl)-1,5-pentanedione, 532
[4642-40-4]	1,5-Bis(2-hydroxy-4-methoxyphenyl)-1,5-pentanedione, 534
[4642-41-5]	5-(2-Hydroxy-4-methoxyphenyl)-5-oxo-1-pentanoic acid, 593
[4642-42-6]	5-(2-Hydroxy-4-methoxyphenyl)-5-oxo-1-pentanoic acid (2,4-Dinitrophenylhydrazone), 594
[4642-43-7]	5-(2,4-Dihydroxyphenyl)-5-oxo-1-pentanoic acid, 580
[4642-44-8]	5-(2,4-Dihydroxyphenyl)-5-oxo-1-pentanoic acid (2,4-Dinitrophenylhydrazone), 581
[4648-94-6]	5-(4-Hydroxyphenyl)-5-oxo-1-pentanoic acid, 578
[4648-95-7]	5-(4-Hydroxyphenyl)-5-oxo-1-pentanoic acid (2,4-Dinitrophenylhydrazone), 579
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[4649-01-8]	5-(2-Hydroxy-5-methylphenyl)-5-oxo-1-pentanoic acid, 591
[4649-02-9]	5-(2-Hydroxy-5-methylphenyl)-5-oxo-1-pentanoic acid (2,4-Dinitrophenylhydrazone), 592
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[4649-04-1]	1,5-Bis(2-hydroxy-5-methylphenyl)-1,5-pentanedione (Dioxime), 532
[4650-71-9]	1,4-Bis(3,4-dimethoxyphenyl)-1,4-butanedione, 363
[4654-07-3]	5-(2,4-Dimethoxyphenyl)-5-oxo-1-pentanoic acid, 581
[4680-89-1]	5-(4-Hydroxy-3-methylphenyl)-5-oxo-1-pentanoic acid (2,4-Dinitrophenylhydrazone), 592
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[4693-17-8]	1-(2,5-Dihydroxyphenyl)-1-pentanone, 471
[4693-18-9]	1-(2,5-Dihydroxyphenyl)-1-hexanone, 608
[4693-19-0]	1-(2,5-Dihydroxyphenyl)-1-octanone, 780
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[4798-12-3]	1-(3-Chloro-4-hydroxyphenyl)-2-propyl-1-pentanone, 560
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[4807-43-6]	1,1'-(2,4,6-Trihydroxy-5-methyl-1,3-phenylene)bis-1-decanone, 915
[4808-89-3]	1-(4-Methoxyphenyl)-1,3,5-hexanetrione, 595
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[4878-81-3]	4-(4-Methoxy-7-benzo[b]thiophene)-4-oxo-1-butanoic acid, 441
[4890-45-3]	4-(2,4,6-Trihydroxyphenyl)-4-oxo-1-butanoic acid, 413
[4945-79-3]	1,5-Bis(2-hydroxyphenyl)-1,5-pentanedione, 517
[4963-67-1]	1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-pentanone (Piperazine salt), 513
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[5333-34-6]	4-(3,4-Dimethoxyphenyl)-4-oxo-1-butanoic acid, 408
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[5485-77-8]	6-(4-Methoxy-3-methylphenyl)-6-oxo-1-hexanoic acid, 717
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[5538-12-5]	6-(4-Hydroxy-2-methylphenyl)-6-oxo-1-hexanoic acid (2,4-Dinitrophenylhydrazone), 716
[5538-13-6]	1,6-Bis(4-hydroxy-2-methylphenyl)-1,6-hexanedione (Dioxime), 686
[5538-14-7]	6-(4-Methoxy-2-methylphenyl)-6-oxo-1-hexanoic acid, 716
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[5550-53-8]	1,6-Bis(4-methoxy-3-methylphenyl)-1,6-hexanedione (Dioxime), 686
[5550-54-9]	1,6-Bis(4-hydroxy-2-methylphenyl)-1,6-hexanedione, 685
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[5550-56-1]	1,6-Bis(4-methoxy-2-methylphenyl)-1,6-hexanedione, 686
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[6397-82-6]	1-(4-Methoxyphenyl)-1-hexanone, 603
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[6790-21-2]	1-(2,4,6-Trihydroxyphenyl)-1-dodecanone, 951
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[7337-50-0]	1-(2,5-Dihydroxyphenyl)-1-decanone, 873
[7356-03-8]	4-(2-Hydroxy-3,5-dimethylphenyl)-4-oxo-1-butanoic acid, 443
[7573-11-7]	1-(2,3-Dihydroxyphenyl)-1-decanone, 872
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[7658-30-2]	1,7-Bis(2,4-dihydroxyphenyl)-1,7-heptanedione, 754
[10121-26-3]	1-(2,6-Dihydroxyphenyl)-1-butanone, 12
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[10351-89-0]	1,4-Bis(2-hydroxy-3,4-dimethoxylphenyl)-1,4-butanedione, 371
[10351-90-3]	1,6-Bis(2-hydroxy-3,4-dimethoxyphenyl)-1,6-hexanedione, 691
[10351-91-4]	1,7-Bis(2-hydroxy-3,4-dimethoxyphenyl)-1,7-heptanedione, 763
[10351-92-5]	1,8-Bis(2-hydroxy-3,4-dimethoxyphenyl)-1,8-octanedione, 825
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[10365-21-6]	1,4-Bis(2,5-dimethoxyphenyl)-1,4-butanedione, 362
[10365-22-7]	1,5-Bis(2,5-dimethoxyphenyl)-1,5-pentanedione, 520
[10365-23-8]	1,6-Bis(2,5-dimethoxyphenyl)-1,6-hexanedione, 675
[10365-24-9]	1,7-Bis(2,5-dimethoxyphenyl)-1,7-heptanedione, 755
[10365-25-0]	1,9-Bis(2,5-dimethoxyphenyl)-1,9-nonanedione, 851
[10365-28-3]	1,6-Bis(2-hydroxy-5-methoxyphenyl)-1,6-hexanedione, 687
[10365-29-4]	1,7-Bis(2-hydroxy-5-methoxyphenyl)-1,7-heptanedione, 761
[10365-30-7]	1,9-Bis(2-hydroxy-5-methoxyphenyl)-1,9-nonanedione, 861
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[10365-31-8]	1,10-Bis(2-hydroxy-5-methoxyphenyl)-1,10-decanedione, 910

[10365-33-0]	1,6-Bis(2-acetyloxy-5-methoxyphenyl)-1,6-hexanedione, 687
[10365-34-1]	1,7-Bis(2-acetyloxy-5-methoxyphenyl)-1,7-heptanedione, 761
[10365-35-2]	1,8-Bis(2-acetyloxy-5-methoxyphenyl)-1,8-octanedione, 820
[10365-36-3]	1,9-Bis(2-acetyloxy-5-methoxyphenyl)-1,9-nonanedione, 861
[10365-37-4]	1,10-Bis(2-acetyloxy-5-methoxyphenyl)-1,10-decanedione, 910
[10365-53-4]	1,5-Bis(3-hydroxyphenyl)-1,5-pentanedione, 517
[10365-54-5]	1,7-Bis(3-hydroxyphenyl)-1,7-heptanedione, 754
[10365-55-6]	1,10-Bis(3-hydroxyphenyl)-1,10-decanedione, 900
[10365-60-3]	1,7-Bis(4-methoxyphenyl)-1,7-heptanedione, 754
[10365-65-8]	1,5-Bis(2-hydroxy-3-methylphenyl)-1,5-pentanedione, 531
[10365-66-9]	1,7-Bis(2-hydroxy-3-methylphenyl)-1,7-heptanedione, 760
[10365-74-9]	1,5-Bis(2-hydroxy-5-methoxyphenyl)-1,5-pentanedione, 534
[10373-31-6]	1,6-Bis(2,3,4-trimethoxyphenyl)-1,6-hexanedione, 677
[10373-32-7]	1,8-Bis(2,3,4-trimethoxyphenyl)-1,8-octanedione, 813
[10373-33-8]	1,10-Bis(2,3,4-trimethoxyphenyl)-1,10-decanedione, 904
[10388-38-2]	1,4-Bis(2,3,4-trimethoxyphenyl)-1,4-butanedione, 363
[10388-39-3]	1,4-Bis(2,3,4-trimethoxyphenyl)-1,4-butanedione
	(2,4-Dinitrophenylhydrazone), 364
[10400-43-8]	1,10-Bis(2-hydroxy-4-methylphenyl)-1,10-decanedione, 907
[10400-49-4]	1,5-Bis(2-methoxy-5-methylphenyl)-1,5-pentanedione, 532
[10400-50-7]	1,7-Bis(2-methoxy-5-methylphenyl)-1,7-heptanedione, 761
[10400-51-8]	1,10-Bis(2-methoxy-5-methylphenyl)-1,10-decanedione, 908
[10400-56-3]	1,10-Bis-(5-chloro-2-methoxyphenyl)-1,10-decanedione, 899
[10401-04-4]	1,7-Bis(2-hydroxyphenyl)-1,7-heptanedione, 753
[10401-05-5]	1,10-Bis(2-hydroxyphenyl)-1,10-decanedione, 900
[10475-16-8]	1,5-Bis(2,3,4-trimethoxyphenyl)-1,5-pentanedione, 522
[10475-17-9]	1,7-Bis(2,3,4-trimethoxyphenyl)-1,7-heptanedione, 756
[10475-18-0]	1,9-Bis(2,3,4-trimethoxyphenyl)-1,9-nonanedione, 852
[10483-67-7]	1,5-Bis(2-hydroxy-3,4-dimethoxylphenyl)-1,5-pentanedione, 536
[10483-68-8]	1,9-Bis(2-hydroxy-3,4-dimethoxyphenyl)-1,9-nonanedione, 862
[10483-69-9]	1,10-Bis(2-hydroxy-3-methylphenyl)-1,10-decanedione, 907
[10491-14-2]	1,8-Bis(2,5-dimethoxyphenyl)-1,8-octanedione, 820
[10491-15-3]	1,8-Bis(2-hydroxy-5-methoxyphenyl)-1,8-octanedione, 820
[10571-10-5]	1,5-Bis(2-hydroxy-4-methylphenyl)-1,5-pentanedione, 531
[10586-43-3]	1-(3,5-Dimethoxyphenyl)-2-methyl-1-heptanone, 730
[13149-43-4]	11-Bromo-1-(4-hydroxy-3-methoxyphenyl)-1-undecanone, 936
[13149-44-5]	11-Bromo-1-(4-allyloxy-3-methoxyphenyl)-1-undecanone, 936
[13149-48-9]	17-Bromo-1-(4-hydroxy-3-methoxyphenyl)-1-heptadecanone, 1060
[13149-49-0]	17-Bromo-1-(4-allyloxy-3-methoxyphenyl)-1-heptadecanone, 1060
[13178-17-1]	1,10-Bis(2,3,4-trihydroxyphenyl)-1,10-decanedione, 903
[13178-43-3]	1,10-Bis(4-hydroxy-3-methylphenyl)-1,10-decanedione, 909
[13210-98-5]	1-(4-Acetyloxyphenyl)-1-butanone, 5
[13221-24-4]	1,7-Bis(2-hydroxy-4-methylphenyl)-1,7-heptanedione, 760
[13282-23-0]	1,4-Bis(2-hydroxy-5-methylphenyl)-1,4-butanedione, 366
[13282-24-1]	1,6-Bis(2-hydroxy-5-methylphenyl)-1,6-hexanedione, 685

[13282-26-3]	1,8-Bis(2-hydroxy-5-methylphenyl)-1,8-octanedione, 818
[13282-28-5]	1,10-Bis(2-hydroxy-5-methylphenyl)-1,10-decanedione, 907
[13298-49-2]	1-(3,4-Dimethoxyphenyl)-1,3-butanedione, 313
[13320-65-5]	1,6-Bis(2-hydroxy-4-methylphenyl)-1,6-hexanedione, 685
[13335-54-1]	4-(2,4-Dihydroxyphenyl)-4-oxo-1-butanoic acid, 403
[13335-55-2]	Methyl 4-(2,4-dihydroxyphenyl)-4-oxo-1-butanoate, 405
[13379-59-4]	1,10-Bis(2,4-diacetyloxyphenyl)-1,10-decanedione, 902
[13404-83-6]	1-(2-Methoxyphenyl)-1-butanone, 2
[13736-49-7]	1-(2-Hydroxy-5-methoxy-4-methylphenyl)-1-heneicosanone, 1101
[13736-50-0]	1-(5-Heneicosanoyloxy-2-hydroxy-4-methylphenyl)-1-heneicosanone, 1102
[13736-51-1]	1-(2,5-Dihydroxy-4-methylphenyl)-1-heneicosanone, 1101
[13936-90-8]	1-(2,6-Dihydroxyphenyl)-3-methyl-1-butanone, 179
[13936-91-9]	1-(2,6-Dihydroxyphenyl)-1-octanone, 781
[13937-24-1]	Dimethyl [4,6-dihydroxy-5-(1-oxobutyl)phenyl]-1,3-dicarboxylate, 61
[13937-26-3]	Dimethyl [4,6-dihydroxy-5-(1-oxooctyl)phenyl]-1,3-dicarboxylate, 798
[13969-78-3]	1,10-Bis(2-hydroxy-4-methoxyphenyl)-1,10-decanedione, 910
[14035-35-9]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-butanone, 114
[14035-37-1]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-pentanone, 535
[14102-16-0]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-3-methyl-1-butanone, 227
[14248-30-7]	1-(4-Methoxyphenyl)-2-methyl-1-butanone (2,4-Dinitrophenylhydrazone), 126
[14353-76-5]	1-(2-Hydroxyphenyl)-1-decanone, 868
[14353-77-6]	1-(4-Hydroxyphenyl)-1-decanone, 869
[14353-85-6]	1-(4-Hydroxyphenyl)-1-decanone (Semicarbazone), 870
[14392-69-9]	1-(4-Hydroxyphenyl)-1-nonanone, 836
[14392-72-4]	1-(4-Hydroxyphenyl)-1-heptanone, 722
[14392-73-5]	1-(4-Hydroxyphenyl)-3,7-dimethyl-1-octanone, 800
[14392-74-6]	1-(4-Hydroxyphenyl)-3,3-dimethyl-1-butanone, 251
[14392-75-7]	1-(4-Hydroxyphenyl)-5-phenyl-1-heptanone, 731
[14392-78-0]	1-[4-(N-Diethylaminoethyloxy)phenyl]-1-octanone, 777
[14392-80-4]	1-[4-(N-Diethylaminoethyloxy)phenyl]-3,7-dimethyl-1-octanone, 801
[14392-83-7]	1-[4-(N-Diethylaminoethyloxy)phenyl]-1-dodecanone, 944
[14392-84-8]	1-[4-(N-Diethylaminoethyloxy)phenyl]-1-octadecanone, 1065
[14392-93-9]	1-[4-(N-Diethylaminoethyloxy)phenyl]-1-octanone (Fumarate), 777
[14563-40-7]	Methyl 4-(3,4-dimethoxyphenyl)-4-oxo-1-butanoate, 409
[14563-41-8]	Methyl 4-(2,4-dimethoxyphenyl)-4-oxo-1-butanoate, 404
[14617-02-8]	4-(2-Hydroxy-4-methoxyphenyl)-4-oxo-1-butanoic acid, 439
[14617-06-2]	4-(2,4-Dimethoxyphenyl)-4-oxo-1-butanoic acid, 403
[14683-92-2]	1-(4-Ethoxy-2-hydroxyphenyl)-1-heptanone, 745
[14683-93-3]	1-(2-Hydroxy-4-propoxyphenyl)-1-heptanone, 747
[14683-94-4]	1-(4-Butoxy-2-hydroxyphenyl)-1-heptanone, 751
[14683-95-5]	1-[2-Hydroxy-4-(3-methylbutoxy)phenyl]-1-heptanone, 753
[14725-80-5]	1-(2,3,4-Trihydroxyphenyl)-1-dodecanone (2,4-Dinitrophenylhydrazone), 950
[14798-38-0]	1-(3-Benzoyl-2,6-dihydroxyphenyl)-1-dodecanone, 980

[14814-73-4]	1-(5-Benzoyl-2,4-dihydroxyphenyl)-1-dodecanone, 981
[15041-68-6]	1,1'-(5-Benzoyl-2,4-dihydroxy-1,3-phenylene)bis-1-dodecanone, 992
[15116-03-7]	1-(2,3-Dimethoxyphenyl)-3-methyl-1-butanone (2,4-Dinitrophenylhydrazone), 177
[15116-04-8]	1-(2,3-Dimethoxyphenyl)-2-methyl-1-butanone (2,4-Dinitrophenylhydrazone), 127
[15116-05-9]	1-(2,3-Dimethoxyphenyl)-1-pentanone (2,4-Dinitrophenylhydrazone), 470
[15116-06-0]	1-(2-Hydroxy-3-methoxyphenyl)-3-methyl-1-butanone, 192
[15116-07-1]	1-(2-Hydroxy-3-methoxyphenyl)-2-methyl-1-butanone, 134
[15116-08-2]	1-(2-Hydroxy-3-methoxyphenyl)-1-pentanone, 496
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[22362-59-0]	1-(2-Hydroxyphenyl)-1-heptanone, 719
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[23951-55-5]	4'-[4-Hydroxy-3,5-(diiodo)diphenyl]ether-4-(1-dodecanone), 976
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	hydrazone), 850
[24336-94-5]	1,9-Bis(4-hydroxy-3-methylphenyl)-1,9-nonanedione, 860
[24336-96-7]	1,9-Bis(4-methoxy-3-methylphenyl)-1,9-nonanedione, 860
[24336-97-8]	1,9-Bis(4-hydroxy-2-methylphenyl)-1,9-nonanedione, 859
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[24339-82-0]	1,9-Bis(4-hydroxy-2-methylphenyl)-1,9-nonanedione (Di-2,4-dinitrophenyl- hydrazone), 859
[24339-87-5]	9-(5-Chloro-2-hydroxyphenyl)-9-oxo-1-nonanoic acid
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[24339-88-6]	10-(4-Hydroxy-3-methylphenyl)-10-oxo-1-decanoic acid (2,4-Dinitrophenylhydrazone), 921
[24339-89-7]	10-(4-Hydroxy-3-methylphenyl)-10-oxo-1-decanoic acid, 921
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[24339-95-5]	10-(4-Hydroxyphenyl)-10-oxo-1-decanoic acid, 918
[24339-96-6]	10-(4-Hydroxyphenyl)-10-oxo-1-decanoic acid
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[24340-00-9]	1,10-Bis(4-hydroxy-2-methylphenyl)-1,10-decanedione, 908
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[24340-01-0]	1,10-Bis(4-hydroxy-2-methylphenyl)-1,10-decanedione (Di-2,4-dinitrophenylhydrazone), 908
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[24340-03-2]	1,10-Bis(4-methoxy-2-methylphenyl)-1,10-decanedione (Di-2,4-dinitrophenylhydrazone), 908
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[24340-05-4]	10-(5-Chloro-2-hydroxyphenyl)-10-oxo-1-decanoic acid (2,4-Dinitrophenylhydrazone), 920
[24340-07-6]	1,10-Bis(2-hydroxy-5-methylphenyl)-1,10-decanedione (Di-2,4-dinitrophenylhydrazone), 908
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[25715-27-9]	1,9-Bis(5-chloro-2-hydroxy-4-methylphenyl)-1,9-nonanedione (Di-2,4-dinitrophenylhydrazone), 859
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[26086-78-2]	1,9-Bis(2-hydroxy-4-methoxyphenyl)-1,9-nonanedione, 861
[26086-80-6]	1,7-Bis(5-chloro-2,4-dihydroxyphenyl)-1,7-heptanedione, 753
[26086-81-7]	1,8-Bis(5-chloro-2,4-dihydroxyphenyl)-1,8-octanedione, 811
[26086-82-8]	1,9-Bis(5-chloro-2,4-dihydroxyphenyl)-1,9-nonanedione, 849
[26086-83-9]	1,7-Bis(5-chloro-2,4-dimethoxyphenyl)-1,7-heptanedione, 753
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[26104-02-9]	1,1'-[2,4,6-Trihydroxy-5-(3-methylbutyl)-1,3-phenylene]bis-3-methyl- 1-butanone, 306
[26104-08-5]	3-Methyl-1-[2,4,6-tribenzoyloxy-3,5-bis(3-methylbutyl)phenyl]-1-butanone, 234
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[26195-11-9]	1,10-Bis(5-chloro-2,4-dihydroxyphenyl)-1,10-decanedione, 899
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[26993-72-6]	5-[[(2-Hydroxy-5-methylphenyl)azo]-2,4-dihydroxyphenyl]- 1-dodecanone, 981
[26993-73-7]	5-[[(5-Chloro-2-hy2,3,4-Trihydroxyphenyl)enyl]-1-dodecanone, 977
[26993-74-8]	5-[[(5-Aminosulfonyl-2-hydroxyphenyl)azo]-2,4-dihydroxyphenyl]- 1-dodecanone, 979
[26993-75-9]	5-[[(2-Carboxyphenyl)azo]-2,4-dihydroxyphenyl]-1-dodecanone, 980
[27020-17-3]	5-[[(2-Hydroxyphenyl)azo]-2,4-dihydroxyphenyl]-1-dodecanone, 978
[27029-46-5]	1-(4-Hydroxyphenyl)-1-tetracosanone (Polymer with formaldehyde), 1113
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[27581-18-6]	1-(4-Chloro-2-hydroxyphenyl)-1-pentanone, 482
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[28319-38-2]	4-[1-(Acetyloxy)propyl]-6-(3,7-dimethyl-2,6-octadienyl)-5,7-dihydroxy- 8-(2-methyl-1-oxobutyl)-2 <i>H</i> -1-benzopyran-2-one, 167
[28441-00-1]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-octanone, 822
[28441-02-3]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-dodecanone, 982
[28441-03-4]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-hexadecanone, 1051
[28441-04-5]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-eicosanone, 1100
[28441-05-6]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-docosanone, 1108
[28459-33-8]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1-octadecanone, 1086
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[29207-19-0]	Methyl 5-(2-methoxy-4-methylphenyl)-5-oxo-1-pentanoate, 591
[29207-22-5]	Methyl 5-(2-methoxy-4-carbomethoxyphenyl)-5-oxo-1-pentanoate, 594
[29389-23-9]	Methyl 6-(4-methoxyphenyl)-6-oxo-1-hexanoate, 711
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[29665-52-9]	1-(4-Methoxy-3-methylphenyl)-1-butanone, 52
[29665-55-2]	1-(4-Hydroxy-3-methylphenyl)-1-dodecanone, 960
[29665-56-3]	1-(4-hydroxy-3,5-dimethylphenyl)-1-decanone, 888
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[30299-36-6]	1-(4-Ethoxyphenyl)-4-methyl-1-pentanone, 543
[30299-37-7]	1-(4-Ethoxyphenyl)-4-methyl-1-pentanone (Semicarbazone), 543
[30390-12-6]	5,7-Dihydroxy-8-(3-methyl-2-butenyl)-6-(2-methyl-1-oxobutyl)-
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[30392-06-4]	1-[3,5-Bis(1,1-dimethylethyl)-4-acetyloxyphenyl]-1-octanone, 822
[30392-07-5]	1-[3,5-Bis(1,1-dimethylethyl)-4-acetyloxyphenyl]-1-octadecanone, 1086
[30414-64-3]	7-(2,4-Dihydroxyphenyl)-7-oxo-1-heptanoic acid, 767
[30414-65-5]	1-(2,4-Dihydroxy-6-methylphenyl)-1-octanone, 795
[30414-67-6]	1-(2,6-Dihydroxy-4-methylphenyl)-1,7-octanedione, 790
[30414-68-7]	1-(2,6-Dimethoxy-4-methylphenyl)-1,7-octanedione, 790
[30492-53-6]	1-[3,5-Bis(1,1-dimethylethyl)-4-methoxyphenyl]-1-octadecanone, 1086
[30509-74-1]	1,1'-(5-Bromo-2,4,6-trihydroxy-1,3-phenylene)bis-1-pentanone (4-Trichloromethane sulfenate), 511
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[30563-62-3]	(1,3,5-Tris-trichloromethane sulfenate), 513       8,9-Dihydro-5-hydroxy-8-(1-hydroxy-1-methylethyl)-6-(3-methyl-
[30303-02-3]	1-oxobutyl)-4-phenyl-2 <i>H</i> -furo[2',3':5,6]benzo[1,2-b]pyran-2-one, 247
[30839-20-4]	1,4-Bis(4-methoxyphenyl)-2,3-bis(phenyl)-1,4-butanedione, 373
[31526-44-0]	5-(4-Methoxyphenyl)-3,3-dimethyl-5-oxo-1-pentanoic acid, 585
[31526-46-2]	Ethyl 5-(4-methoxyphenyl)-3,3-dimethyl-5-oxo-1-pentanoate, 585
[31914-19-9]	4-(2,4,5-Trimethoxyphenyl)-4-oxo-1-butanoic acid, 412
[32085-87-3]	1-(2,6-DImethoxyphenyl)-1,3-butanedione, 321
[32190-32-2]	1,1'-[Methylenebis(2,4-dihydroxy-6-methoxy-3,1-phenylene)]bis-
	1-butanone, 337
[32246-17-6]	8-(2,4-Dihydroxy-5-bromophenyl)-8-oxo-1-octanoic acid, 831
[32246-62-1]	1,7-Bis(2,4-dimethoxyphenyl)-1,7-heptanedione, 755
[32246-63-2]	1,7-Bis(2,4-dimethoxyphenyl)-1,7-heptanedione (Dioxime), 755
[32246-69-8]	1,7-Bis(3,4-dimethoxyphenyl)-1,7-heptanedione, 755
[32246-72-3]	7-(5-Bromo-2,4-dihydroxyphenyl)-7-oxo-1-heptanoic acid, 767
[32246-77-8]	8-(2,4-Dihydroxyphenyl)-8-oxo-1-octanoic acid, 830
[32246-82-5]	1,8-Bis(2,4-dimethoxyphenyl)-1,8-octanedione, 812
[32246-86-9]	8-(2,4-Dihydroxy-5-chlorophenyl)-8-oxo-1-octanoic acid, 832
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[32246-91-6]	1,8-Bis(2-hydroxy-3,4-dimethoxyphenyl)-1,8-octanedione (Dioxime), 825
[32246-93-8]	1,8-Bis(3,4-dimethoxyphenyl)-1,8-octanedione (Di-2,4-dinitrophenyl-
	hydrazone), 813
[32246-94-9]	8-(3,4-Dimethoxyphenyl)-8-oxo-1-octanoic acid, 831
[32246-95-0]	1,8-Bis(5-bromo-2,4-dihydroxyphenyl)-1,8-octanedione, 811
[32339-34-7]	1,1'-(4'-Methoxy[1,1'-biphenyl]-4-yl)-1-butanone, 261
[32339-35-8]	1,1'-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-1-butanone, 261
[32340-75-3]	7-(5-Chloro-2,4-dihydroxyphenyl)-7-oxo-1-heptanoic acid, 768
[32340-79-7]	8-(3,4-Dimethoxyphenyl)-8-oxo-1-octanoic acid
	(2,4-Dinitrophenylhydrazone), 831
[32354-10-2]	1,7-Bis(2,4-dihydroxyphenyl)-1,7-heptanedione
	(2,4-Dinitrophenylhydrazone), 755
[32435-18-0]	1,8-Bis(3,4-dimethoxyphenyl)-1,8-octanedione, 812
[33245-77-1]	5-Chloro-1-(3,4-dimethoxyphenyl)-1-pentanone, 569
[33245-78-2]	5-Chloro-1-(2,4-dimethoxyphenyl)-1-pentanone, 569
[33446-14-9]	4-(4-Methoxy-3-methylphenyl)-4-oxo-1-butanoic acid, 436
[33488-76-5]	1-(4-Ethoxy-2-hydroxyphenyl)-1-nonanone (Oxime), 845
[33488-77-6]	1-(4-Ethoxy-2-hydroxyphenyl)-1-heptadecanone (Oxime), 1057
[33488-77-6]	1-(4-Ethoxy-2-hydroxyphenyl)-1-octadecanone (Oxime), 1079
[33720-04-6]	2-Bromo-1-(4-methoxyphenyl)-4-methyl-1-pentanone, 574
[33809-55-1]	2-Bromo-1-(4-methoxyphenyl)-1-hexanone, 697
[34052-09-0]	1-(2,3-Dimethoxyphenyl)-1-butanone, 8
[34128-24-0]	1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1,3-butanedione, 332
[34190-36-8]	1-(3-Chloro-4-hydroxyphenyl)-1-pentanone, 482
[34670-10-5]	5-(4-Ethoxyphenyl)-5-oxo-1-pentanoic acid, 580
[34767-67-4]	11-Bromo-1-(3,4-diacetyloxyphenyl)-1-undecanone, 935
[34887-83-7]	1-(4-Hydroxyphenyl)-3-methyl-1-butanone, 175
[34927-48-5]	4-(2-Hydroxy-3,4,6-trimethylphenyl)-2-methyl-4-oxo-1-butanoic acid, 449
[34927-49-6]	4-(2-Hydroxy-3,4,6-trimethylphenyl)-2-methyl-4-oxo-1-butanoic acid, 449
[35031-70-0]	1-(2-Methoxyphenyl)-1-hexanone, 599
[35031-73-3]	1-(4-Ethoxyphenyl)-1-butanone, 8
[35031-74-4]	1-(4-Ethoxyphenyl)-1-hexanone, 605
[35049-65-1]	1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2 <i>H</i> -1-benzopyran-8-yl)- 1-pentanone, 511
[35081-43-3]	2-Bromo-1-[(4-phenylmethoxy)phenyl]-1-butanone, 270
[35081-44-8]	1-(4-Benzyloxyphenyl)-1-pentanone, 466
[35081-50-6]	2-Bromo-1-(4-benzyloxyphenyl)-1-pentanone, 565
[35115-14-1]	1-(2-Hydroxyphenyl)-2-methyl-1,3-butanedione, 314
[35115-15-2]	1-(2-Hydroxyphenyl)-1,3-pentanedione, 459
[35175-56-5]	1-(2-Hydroxy-5-methoxyphenyl)-1-octadecanone, 1077
[35446-28-7]	2-Bromo-1-(4-methoxyphenyl)-3-methyl-1-butanone, 293
[36287-37-3]	1,4-Bis(3,4-dimethoxyphenyl)-2,3-dimethyl-1,4-butanedione
	(meso-isomer), 368
[36287-37-3]	1,4-Bis(3,4-dimethoxyphenyl)-2,3-dimethyl-1,4-butanedione (meso), 370
[36330-86-6]	4-(4-Phenoxyphenyl)-4-oxo-1-butanoic acid, 402

[36330-87-7]	4-(4'-Methoxybiphenyl)-4-oxo-1-butanoic acid, 452
[36375-38-9]	1-(2-Hydroxy-3-methylphenyl)-1-butanone, 47
[36412-64-3]	2-Bromo-1-(4-methoxyphenyl)-1-pentanone, 565
[36478-56-5]	5,7-Dihydroxy-8-(2-methyl-1-oxobutyl)-4-pentyl-2H-
	1-benzopyran-2-one, 148
[36481-17-1]	Methyl 5-butyryl-2-hydroxybenzoate, 43
[36677-71-1]	1,1'-(4,4'-Dihydroxy[1,1'-biphenyl]-3,3'-diyl)-1-decanone, 915
[36756-42-0]	1-(4,4'-Dihydroxy[1,1'-biphenyl]-3-yl)-1-decanone, 905
[36946-07-3]	1-(2-Hydroxy-5-methylphenyl)-1-octanone, 792
[36946-08-4]	1-(2-Hydroxy-5-methylphenyl)-1-tetradecanone, 1012
[36953-87-4]	4-Hydroxy-3-(1-oxopentyl)-2H-1-benzopyran-2-one, 502
[36953-90-9]	4-Hydroxy-3-(1-oxooctyl)-2H-1-benzopyran-2-one, 803
[37166-86-2]	1,9-Bis(2,4-dihydroxyphenyl)-1,9-nonanedione (Dioxime), 851
[37166-89-5]	1,9-Bis(2,4-dimethoxyphenyl)-1,9-nonanedione, 851
[37166-91-9]	1,9-Bis(5-chloro-2,4-dihydroxyphenyl)-1,9-nonanedione (Dioxime), 850
[37166-92-0]	9-(5-Chloro-2,4-dihydroxyphenyl)-9-oxo-1-nonanoic acid, 865
[37166-94-2]	1,9-Bis(5-chloro-2,4-dimethoxyphenyl)-1,9-nonanedione (Dioxime), 850
[37166-96-4]	1,9-Bis(2-hydroxy-3,4-dimethoxyphenyl)-1,9-nonanedione (Di-2,4-dinitro-
	phenylhydrazone), 863
[37166-99-7]	1,10-Bis(2,4-dihydroxyphenyl)-1,10-decanedione (Di-2,4-dinitrophenyl-
	hydrazone), 902
[37167-01-4]	1,10-Bis(2-hydroxy-4-methoxyphenyl)-1,10-decanedione (Di-2,4-dinitro-
[271(7.02.5]	phenylhydrazone), 910
[37167-02-5]	1,10-Bis(2,4-dimethoxyphenyl)-1,10-decanedione, 902
[37174-76-8]	1,10-Bis(5-chloro-2,4-dimethoxyphenyl)-1,10-decanedione (Di-2,4-dinitro- phenylhydrazone), 899
[37174-78-0]	1,10-Bis(5-bromo-2,4-dihydroxyphenyl)-1,10-decanedione, 898
[37174-79-1]	1,10-Bis(5-bromo-2,4-dimethoxyphenyl)-1,10-decanedione, 898
[37174-80-4]	1,10-Bis(5-bromo-2,4-dimethoxyphenyl)-1,10-decanedione (Dioxime), 898
[37401-99-3]	1,10-Bis(5-chloro-2,4-dihydroxyphenyl)-1,10-decanedione (Dioxime), 899
[37402-01-0]	1,10-Bis(5-bromo-2,4-dihydroxyphenyl)-1,10-decanedione (Dioxime), 898
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[37622-68-7]	1-(2,4-Dihydroxyphenyl)-1-octanone, 778
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[37765-93-8]	1-(2-Hydroxyphenyl)-5-phenyl-1-pentanone, 523
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[38071-42-0]	1-(2,4-Dibenzyloxy-6-methylphenyl)-1,3,5-hexanetrione, 629
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[38071-44-2]	Methyl 7-(2,4-dibenzyloxy-6-methylphenyl)-3,5,7-trioxo-1-heptanoite acid, 769
[38071-45-3]	Methyl 7-(2,4-dihydroxy-6-methylphenyl)-3,5,7-trioxo-1-heptanoate, 769
[38071-49-7]	Methyl 7-(2,4-dimethoxy-6-methylphenyl)-3,5,7-trioxo-1-heptanoate, 768
[38409-24-4]	7-Methoxy-6-(2,2-dideuterio-3-methyl-1-oxobutyl)-2 <i>H</i> -1-benzopyran-2-one, 202
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[39496-86-1]	4-(3-Amino-4-methoxyphenyl)-4-oxo-1-butanoic acid, 428
[39496-87-2]	4-(3-Chloro-4-methoxyphenyl)-4-oxo-1-butanoic acid, 423
[39560-29-7]	4-(2,4-Diethoxyphenyl)-4-oxo-1-butanoic acid, 404
[39560-34-4]	4-(2-Hydroxyphenyl)-4-oxo-1-butanoic acid, 398
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[39575-48-9]	1-(4-Chloro-2-hydroxyphenyl)-3-methyl-1-pentanone (+), 557
[39575-49-0]	1-(5-Chloro-2-hydroxyphenyl)-3-methyl-1-pentanone (+), 557
[39652-80-7]	2-Methyl-1-(2,4,6-trihydroxyphenyl)-1-butanone, 129
[39652-87-4]	1-(3,4-Dihydro-5,7-dihydroxy-2,2-dimethyl-2 <i>H</i> -1-benzopyran-8-yl)-
	2-methyl-1-butanone, 141
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[40220-94-8]	1-(2,4,6-Triacetyloxyphenyl)-1-dodecanone, 952
[40336-21-8]	1-(3,4,5-Trihydroxyphenyl)-1-dodecanone, 953
[40366-12-9]	1-(2,3,4-Trihydroxyphenyl)-1-hexadecanone, 1038
[40372-78-9]	4-Hexadecanoylsalicylic acid, 1041
[40372-79-0]	1-(2,4-Dihydroxyphenyl)-1-hexadecanone, 1035
[40690-25-3]	1-(2-Hydroxy-4-methylphenyl)-1-dodecanone (Oxime, nickel complex), 958
[40690-30-0]	1-(2-Hydroxy-4-methylphenyl)-1-tridecanone (Oxime,
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[40877-17-6]	4-Chloro-1-(2-methoxyphenyl)-1-butanone, 279
[40877-19-8]	4-Chloro-1-(4-methoxyphenyl)-1-butanone, 280
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[41729-72-0]	1-(2,4-Dihydroxyphenyl)-1-hexadecanone (Na salt), 1035
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[41826-92-0]	4-(2,4,5-Triethoxyphenyl)-4-oxo-1-butanoic acid, 412
[41826-96-4]	4-(4,5-Diethoxy-2-methylphenyl)-4-oxo-1-butanoic acid, 438
[41826-97-5]	4-(5-Chloro-2,4-dimethoxyphenyl)-4-oxo-1-butanoic acid, 425
[41826-99-7]	4-(5-Ethoxy-4-methoxy-2-propylphenyl)-4-oxo-1-butanoic acid, 451
[41827-00-3]	4-(4-Methoxy-5-propoxy-2-propylphenyl)-4-oxo-1-butanoic acid, 454
[41827-02-5]	4-(5-Chloro-2,4-diethoxyphenyl)-4-oxo-1-butanoic acid, 425
[41827-04-7]	4-(2,4-Diethoxy-5-ethylphenyl)-4-oxo-1-butanoic acid, 445
[41827-05-8]	4-(4-Chloro-2,5-dimethoxyphenyl)-4-oxo-1-butanoic acid, 424
[41827-06-9]	4-(6-Methoxybenzodioxol-5-yl)-4-oxo-1-butanoic acid, 431
[41827-08-1]	4-(2-Ethoxy-4-methyl-5-methylthiophenyl)-4-oxo-1-butanoic acid, 445
[41827-09-2]	4-(2,4,5-Tributoxyphenyl)-4-oxo-1-butanoic acid, 413
[41827-10-5]	Ethyl 4-(4,5-diethoxy-2-methylphenyl)-4-oxo-1-butanoate, 439
[41827-11-6]	4-(2,5-Diethoxy-4-methylphenyl)-4-oxo-1-butanoic acid, 437
[41827-12-7]	4-(2-Hydroxy-4,5-dimethoxyphenyl)-4-oxo-1-butanoic acid, 447
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[41894-24-0]	1-(2-Hydroxy-4-methylphenyl)-1-dodecanone (Oxime, palladium complex), 958
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[42916-80-3]	Ethyl 6-(4-methoxyphenyl)-6-oxo-1-hexanoate, 711
[43043-25-0]	1-(2,3,4-Trihydroxyphenyl)-1-pentanone, 475
[43043-26-1]	1-(2,3,4-Trihydroxyphenyl)-1-hexanone, 611
[43043-27-2]	1-[2,3,4-Trihydroxyphenyl]-1-heptanone, 729
[43043-28-3]	1-(2,3,4-Trihydroxyphenyl)-1-octanone, 783
[43043-31-8]	1-[2,4,6-Trihydroxyphenyl]-1-heptanone, 729
[43043-32-9]	1-(2,4,6-Trihydroxyphenyl)-1-octanone, 783
[43221-42-7]	1-(2-Hydroxy-4-methoxyphenyl)-1-nonanone, 844
[43228-96-2]	5-Chloro-1-(2-methoxyphenyl)-1-pentanone, 567
[43228-97-3]	1-(2-Methoxyphenyl)-5-iodo-1-pentanone, 569
[47660-65-1]	1-(4-Hydroxyphenyl)-1-docosanone, 1104
[49572-23-8]	1-(4-Ethoxy-2-hydroxyphenyl)-1-dodecanone, 965
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[49582-15-2]	Trisaspidinol PBP, 348
[49583-26-8]	1-(2,4,6-Trihydroxy-3-methylphenyl)-1-pentanone, 499
[49583-27-9]	3-Methyl-1-(2,4,6-trihydroxyphenyl)-1-butanone, 194
[49710-85-2]	1-(4,7-Dimethoxy-2,3-dimethyl-6-benzofuranyl)-1-tetradecanone, 1018
[49710-86-3]	1-(4,7-Dimethoxy-2,3-dimethyl-6-benzofuranyl)-1-hexadecanone, 1048
[49710-89-6]	1-(4,7-Dihydroxy-2,3-dimethyl-6-benzofuranyl)-1-decanone, 894
[49710-91-0]	1-(2,5-Dimethoxyphenyl)-1-decanone, 873
[50113-09-5]	1-[3-[(Dimethylamino)methyl]-2,4,5-trihydroxyphenyl]-1-butanone, 85
[50342-14-8]	1-(2-hydroxy-4,5-dimethylphenyl)-1-heptanone, 743
[50444-92-3]	1-(4-Chloro-2-hydroxyphenyl)-1-hexanone, 624
[50444-95-6]	1,1'-[Sulfonylbis(6-chloro-4-hydroxy-3,1-phenylene)]bis-1-butanone, 300
[50444-96-7]	1,1'-[Sulfonylbis(6-chloro-4-hydroxy-3,1-phenylene)]bis-1-pentanone, 537
[50444-97-8]	1,1'-[Sulfonylbis(6-chloro-4-hydroxy-3,1-phenylene)]bis-1-hexanone, 693
[50652-74-9]	1-(2-Hydroxy-4-methylphenyl)-1-hexanone (Oxime), 639
[50652-75-0]	1-[2-Hydroxyphenyl]-3,5,5-trimethyl-1-hexanone (Oxime), 619
[50652-76-1]	1-(2-Hydroxy-5-methylphenyl)-1-dodecanone (Oxime), 959
[50766-16-0]	1,6-Bis(3,4-dimethoxyphenyl)-1,6-hexanedione, 675
[50766-17-1]	1,9-Bis(3,4-dimethoxyphenyl)-1,9-nonanedione, 852
[50766-18-2]	1,6-Bis(3,4-diethoxyphenyl)-1,6-hexanedione, 676
[50766-19-3]	1,6-Bis(3,4-dipropyloxyphenyl)-1,6-hexanedione, 676
[50766-20-6]	1,6-Bis(3,4-dibutyloxyphenyl)-1,6-hexanedione, 676
[50766-21-7]	1,6-Bis(4-ethoxy-3-methoxyphenyl)-1,6-hexanedione, 687
[50766-22-8]	1,6-Bis(4-benzyloxy-3-methoxyphenyl)-1,6-hexanedione, 688
[50766-25-1]	1,6-Bis(3,4-dimethoxyphenyl)-3-methyl-1,6-hexanedione, 682
[50766-27-3]	1,5-Bis(3,4-dimethoxyphenyl)-1,5-pentanedione (Dimethyloxime), 521
[50766-28-4]	1,6-Bis(3,4-dimethoxyphenyl)-1,6-hexanedione (Dioxime), 676
[50766-29-5]	1,7-Bis(3,4-dimethoxyphenyl)-1,7-heptanedione (Dioxime), 755
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[50766-30-8]	1,9-Bis(3,4-dimethoxyphenyl)-1,9-nonanedione (Dioxime), 852

[50766-33-1]	1,6-Bis(3,4-dimethylenedioxyphenyl)-1,6-hexanedione (Dimethyloxime), 677
[50766-34-2]	1,6-Bis(3,4-diethoxyphenyl)-1,6-hexanedione (Dioxime), 676
[50766-35-3]	1,6-Bis(3,4-dipropyloxyphenyl)-1,6-hexanedione (Dioxime), 676
[50766-36-4]	1,6-Bis(3,4-dibutyloxyphenyl)-1,6-hexanedione (Dioxime), 676
[50766-37-5]	1,6-Bis(4-ethoxy-3-methoxyphenyl)-1,6-hexanedione (Dioxime), 688
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[50766-42-2]	1,6-Bis(3,4-dimethoxyphenyl)-3-methyl-1,6-hexanedione (Dioxime), 682
[50874-43-6]	1-[2,4,6-Trihydroxy-3,5-bis(3-methyl-2-buten-1-yl)phenyl]-
	1-pentanone, 537
[50874-48-1]	1-[2,4,6-Trihydroxy-3,5-bis(3-methyl-2-butenyl)phenyl]-1-butanone, 119
[51067-61-9]	6-(4-Methoxyphenyl)-1-phenyl-1,6-hexanedione, 672
[51317-85-2]	4-Chloro-1-(2-hydroxy-5-methylphenyl)-1-butanone, 284
[51317-86-3]	6-Chloro-1-(2-hydroxy-5-methylphenyl)-1-hexanone, 706
[51528-14-4]	1-(2-Hydroxy-5-methylphenyl)-1-octanone (Oxime), 793
[51528-15-5]	2-Ethyl-1-(2-hydroxy-5-methylphenyl)-1-hexanone (Oxime), 657
[51528-16-6]	1-(2-Hydroxy-5-nonylphenyl)-1-octanone (Oxime), 824
[51621-21-7]	1-(2-Hydroxy[1,1'-biphenyl]-3-yl)-1-octanone, 814
[51686-50-1]	Methyl 4-(2,3,4-trimethoxyphenyl)-4-oxo-1-butanoate, 411
[51795-94-9]	6-Bromo-1-(2-methoxyphenyl)-1-hexanone, 698
[51821-14-8]	6-Bromo-1-(2-hydroxyphenyl)-1-hexanone, 697
[51830-11-6]	1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-octadecanone (Oxime), 1082
[51944-08-2]	1-(4-Hydroxyphenyl)-1,3-butanedione, 310
[51978-33-7]	1-(5-Chloro-2-hydroxyphenyl)-1-butanone, 30
[51995-88-1]	1-[5-(1-Hydroxyethyl)-2-methoxyphenyl]-3-methyl-1-butanone, 198
[51995-98-3]	1-(5-Acetyl-2-methoxyphenyl)-3-methyl-1-butanone, 379
[52016-63-4]	1,5-Bis(5-chloro-2-hydroxypheny)l-1,5-pentanedione, 516
[52016-87-2]	1-(4,6-Dichloro-2-hydroxyphenyl)-1-hexanone, 622
[52066-90-7]	1-(5-Dodecyl-2-methoxyphenyl)-1-dodecanone, 986
[52122-64-2]	1-(2-Hydroxy-4-methylphenyl)-3,5,5-trimethyl-1-hexanone (Oxime), 665
[52122-70-0]	1-(2-Hydroxy-4-methylphenyl)-1-hexanone, 639
[52122-73-3]	1-(2-Hydroxy-4-methylphenyl)-3,5,5-trimethyl-1-hexanone, 665
[52196-48-2]	1-(5-Chloro-2-hydroxyphenyl)-1-octanone, 787
[52245-99-5]	4-(4-Hydroxy-3,5-dimethylphenyl)-4-oxo-1-butanoic acid, 444
[52375-87-8]	1-(3,4-Dimethoxyphenyl)-1-hexanone, 610
[52376-23-5]	1-(4-Bromo-2,5-dihydroxyphenyl)-1-butanone, 28
[52376-24-6]	1-(4-Bromo-2,5-dihydroxyphenyl)-1-butanone (Semicarbazone), 28
[52376-25-7]	1-(4-Bromo-2,5-dihydroxyphenyl)-1-pentanone, 481
[52376-26-8]	1-(4-Bromo-2,5-dihydroxyphenyl)-1-pentanone (Semicarbazone), 481
[52672-75-0]	1-(2-Hydroxy-5-methylphenyl)-1-dodecanone (Oxime, nickel complex), 960
[52751-45-8]	1-[2,6-Dihydroxy-4-(phenylmethoxy)phenyl]-1,3-butanedione, 333
[52754-68-4]	1-(4-Methoxyphenyl)-1-nonanone, 837
[52780-68-4]	1-(4-Hydroxy-3-methylphenyl)-1-butanone, 51
[52852-89-8]	1,5-Bis(2-hydroxyphenyl)-3-methyl-1,5-pentanedione, 528
[52856-20-9]	1-(2,6-Dimethoxyphenyl)-3-methyl-1-butanone, 179
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[52856-31-2]	1-(2,6-Dimethoxyphenyl)-3-methyl-1-butanone (2,4-Dinitrophenylhydrazone), 179
[52922-74-4]	1-(2-Ethyloxyphenyl)-1-heptanone, 720
[53107-50-9]	1-(2,3-Dichloro-4-methoxyphenyl)-3-methyl-1-butanone, 185
[53107-51-0]	1-(2,3-Dichloro-4-methoxyphenyl)-3-methyl-2-methylene-1-butanone, 188
[53107-64-5]	6-Bromo-1-(2,3-dichloro-4-methoxyphenyl)-1-hexanone, 702
[53107-72-5]	7-Bromo-1-(2,3-dichloro-4-methoxyphenyl)-1-heptanone, 764
[53270-35-2]	1-(2,4-Dimethoxy-6-methylphenyl)-1,3-butanedione, 318
[53347-08-3]	1-(5-Chloro-3-ethyl-2-hydroxyphenyl)-1-butanone, 63
[53347-27-6]	1-(5-Chloro-3-ethyl-2-hydroxyphenyl)-1-pentanone, 500
[53347-28-7]	1-(5-Chloro-3-ethyl-2-hydroxyphenyl)-1-hexanone, 647
[53623-37-3]	4-(4-Ethoxyphenyl)-4-oxo-1-butanoic acid, 401
[53771-36-1]	3-Methyl-1-(3,4-dihydro-5,7-dihydroxy-2,2-dimethyl-6-(3-methyl-
	2-butenyl)-2H-1-benzopyran-8-yl)-1-butanone, 233
[53903-58-5]	2-Bromo-1-(4-hydroxyphenyl)-1-butanone, 269
[54011-26-6]	Methyl 4-(4'-methoxybiphenyl)-4-oxo-1-butanoate, 452
[54103-36-5]	1-(4-Methoxyphenyl)-1,3-pentanedione, 460
[54103-37-6]	1-(4-Methoxyphenyl)-4-phenyl-1,3-butanedione, 354
[54109-33-0]	1-(3,5-Dimethoxyphenyl)-1-pentanone-1-14C, 475
[54289-79-1]	1-(2,4-Dihydroxy-3-quinolinyl)-1-hexanone, 654
[54343-87-2]	6-Chloro-1-(2,3-dichloro-4-methoxyphenyl)-2-methylene-1-hexanone, 705
[54419-21-5]	1-(3,4-Dimethoxyphenyl)-1-butanone, 14
[54419-64-6]	1-(2,5-Dimethoxyphenyl)-1-butanone, 11
[54419-69-1]	1-(2,5-Dimethoxyphenyl)-1-butanone (2,4-Dinitrophenylhydrazone), 12
[54535-83-0]	1-(3,4-Dihydroxyphenyl)-1-hexadecanone, 1037
[54556-08-0]	1-(3,5-Dihexyl)-2,4,6-(trihydroxyphenyl)-1-hexanone, 694
[54614-64-1]	3-Methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl]- 1-butanone, 215
[54685-34-6]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-3-eicosanone, 1100
[54696-06-9]	1-(4-Methoxy-2-methylphenyl)-1-butanone, 51
[54874-25-8]	1-(2,4-Dimethoxyphenyl)-3-methyl-1-butanone, 178
[54963-60-9]	1-[5-Acetyl-2-hydroxy-3-(3-hydroxy-3-methyl-1-butenyl)phenyl]- 3-methyl-1-butanone ( <i>E</i> ), 381
[54963-61-0]	1-[5-Acetyl-2-hydroxy-3-(1-hydroxy-3-methyl-2-butenyl)phenyl]- 3-methyl-1-butanone, 381
[55007-22-2]	4-(2-Methoxy-5-methylphenyl)-4-oxo-1-butanoic acid, 434
[55049-56-4]	1-(3,5-Dimethoxyphenyl)-1-dodecanone, 949
[55382-31-5]	1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)-3-methyl-1-butanone, 199
[55382-32-6]	1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)-2-methyl-1-butanone, 135
[55469-24-4]	1-(4-Methoxyphenyl)-1-tridecanone, 996
[55507-71-6]	1-(2,3-Dichloro-4-hydroxyphenyl)-3,3-dimethyl-1-butanone, 252
[55507-79-4]	1-(2,3-Dichloro-4-hydroxyphenyl)-1-pentanone, 479
[55507-84-1]	1-(2,3-Dichloro-4-hydroxyphenyl)-1-heptanone, 732
[55576-64-2]	1-[3-[(3-Acetyl-2,6-dihydroxy-4-methoxy-5-methylphenyl)methyl]- 5-[[2,6-dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl)phenyl] methyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-butanone, 168

[55576-65-3]	1-[3,5-Bis[[2,6-dihydroxy-4-methoxy-3-methyl-5-(2-methyl-1-oxopropyl) phenyl]methyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-butanone, 168
[55576-66-4]	1-[3,5-Bis[[2,6-dihydroxy-4-methoxy-3-methyl-5-(1-oxobutyl)phenyl] methyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-butanone (2 <i>S</i> ), 169
[55576-68-6]	1-[3,5-Bis[[2,6-dihydroxy-4-methoxy-3-methyl-5-(1-acetyl)phenyl]methyl]-
[00070 00 0]	2,4,6-trihydroxyphenyl]-2-methyl-1-butanone, 346
[55785-59-6]	1,1'-[[2,4,6-Trihydroxy-5-(1-oxobutyl)-1,3-phenylene]bis[methylene-
	(2,4-dihydroxy-6-methoxy-5-methyl-3,1-phenylene)]]bis-1-butanone, 348
[55813-81-5]	1-(2-Hydroxy-5-methylphenyl)-3-methyl-1-butanone, 190
[55896-05-4]	1-(2-Hydroxy-4-methoxyphenyl)-1-undecanone, 931
[55917-79-8]	1-(2-Hydroxyphenyl)-1-dodecanone (Oxime, nickel complex), 940
[56116-77-9]	1-(4'-Methoxy[1,1'-biphenyl]-4-yl)-1-pentanone, 525
[56116-78-0]	1-(4'-Methoxy[1,1'-biphenyl]-4-yl)-1-hexanone, 680
[56116-79-1]	1-(4'-Methoxy[1,1'-biphenyl]-4-yl)-1-heptanone, 757
[56116-80-4]	1,1'-(4'-Methoxy[1,1'-biphenyl]-4-yl)-1-octanone, 815
[56116-81-5]	1-(4'-Methoxy[1,1'-biphenyl]-4-yl)-1-nonanone, 853
[56116-82-6]	1-(4'-Methoxy[1,1'-biphenyl]-4-yl)-1-decanone, 905
[56116-87-1]	1-(4'-Ethoxy[1,1'-biphenyl]-4-yl)-1-hexanone, 680
[56116-88-2]	1-(4'-Ethyloxy[1,1'-biphenyl]-4-yl)-1-heptanone, 757
[56116-89-3]	1-(4'-Ethyloxy[1,1'-biphenyl]-4-yl)-1-octanone, 815
[56116-90-6]	1-(4'-Ethyloxy[1,1'-biphenyl]-4-yl)-1-nonanone, 853
[56116-91-7]	1-(4'-Ethyloxy[1,1'-biphenyl]-4-yl)-1-decanone, 905
[56116-95-1]	1-(4'-Propyloxy[1,1'-biphenyl]-4-yl)-1-hexanone, 680
[56116-96-2]	1-(4'-Propyloxy[1,1'-biphenyl]-4-yl)-1-heptanone, 757
[56116-97-3]	1-(4'-Propyloxy[1,1'-biphenyl]-4-yl)-1-octanone, 815
[56116-98-4]	1-(4'-Propyloxy[1,1'-biphenyl]-4-yl)-1-nonanone, 853
[56116-99-5]	1-(4'-Propyloxy[1,1'-biphenyl]-4-yl)-1-decanone, 905
[56117-03-4]	1-(4'-Butyloxy[1,1'-biphenyl]-4-yl)-1-hexanone, 680
[56117-04-5]	1-(4'-Butyloxy[1,1'-biphenyl]-4-yl)-1-heptanone, 757
[56117-05-6]	1-(4'-Butyloxy[1,1'-biphenyl]-4-yl)-1-octanone, 815
[56117-06-7]	1-(4'-Butyloxy[1,1'-biphenyl]-4-yl)-1-nonanone, 853
[56117-07-8]	1-(4'-Butyloxy[1,1'-biphenyl]-4-yl)-1-decanone, 905
[56117-12-5]	1-(4'-Pentyloxy[1,1'-biphenyl]-4-yl)-1-hexanone, 680
[56117-13-6]	1-(4'-Pentyloxy[1,1'-biphenyl]-4-yl)-1-heptanone, 757
[56117-14-7]	1-(4'-Pentyloxy[1,1'-biphenyl]-4-yl)-1-octanone, 815
[56117-15-8]	1-(4'-Pentyloxy[1,1'-biphenyl]-4-yl)-1-nonanone, 853
[56117-16-9]	1-(4'-Pentyloxy[1,1'-biphenyl]-4-yl)-1-decanone, 905
[56117-21-6]	1-(4'-Hexyloxy[1,1'-biphenyl]-4-yl)-1-hexanone, 680
[56117-22-7]	1-(4'-Hexyloxy[1,1'-biphenyl]-4-yl)-1-heptanone, 757
[56117-23-8]	1-(4'-Hexyloxy[1,1'-biphenyl]-4-yl)-1-octanone, 815
[56117-24-9]	1-(4'-Hexyloxy[1,1'-biphenyl]-4-yl)-1-nonanone, 853
[56117-25-0]	1-(4'-Hexyloxy[1,1'-biphenyl]-4-yl)-1-decanone, 905
[56117-30-7]	1-(4'-Heptyloxy[1,1'-biphenyl]-4-yl)-1-hexanone, 681
[56117-31-8]	1-(4'-Heptyloxy[1,1'-biphenyl]-4-yl)-1-heptanone, 757
[56117-32-9]	1-(4'-Heptyloxy[1,1'-biphenyl]-4-yl)-1-octanone, 815

[56117-33-0]	1-(4'-Heptyloxy[1,1'-biphenyl]-4-yl)-1-decanone, 905
[56117-37-4]	$1-(4'-1)(p_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y_1(y_1)-y$
[56117-38-5]	1-(4'-Octyloxy[1,1'-biphenyl]-4-yl)-1-pentatione, 525 $1-(4'-Octyloxy[1,1'-biphenyl]-4-yl)-1-hexanone, 681$
[56117-39-6]	1-(4'-Octyloxy[1,1'-biphenyl]-4-yl)-1-heptanone, 757
[56117-40-9]	$\frac{1}{1,1'-(4'-\operatorname{Octyloxy}[1,1'-\operatorname{biphenyl}]-4-\operatorname{yl})-1-\operatorname{octanone}, 815}$
[56117-41-0]	$1.1^{-(4+Octyloxy[1,1+Ophenyl]-4-yl)-1-octatione, 015}$ 1-(4'-Octyloxy[1,1'-biphenyl]-4-yl)-1-nonanone, 853
[56117-42-1]	1-(4'-Octyloxy[1,1'-biphenyl]-4-yl)-1-decanone, 905
[56117-47-6]	1-(4'-Nonyloxy[1,1'-biphenyl]-4-yl)-1-decanone, 505 1-(4'-Nonyloxy[1,1'-biphenyl]-4-yl)-1-hexanone, 681
[56117-48-7]	1-(4'-Nonyloxy[1,1'-biphenyl]-4-yl)-1-heptanone, 757
[56117-49-8]	1-(4'-Nonyloxy[1,1'-biphenyl]-4-yl)-1-octanone, 815
[56117-50-1]	1-(4'-Nonyloxy[1,1'-bipheny]]-4-yl)-1-nonanone, 854
[56117-51-2]	1-(4'-Nonyloxy[1,1'-bipheny]]-4-yl)-1-decanone, 905
[56117-56-7]	1-(4'-Decyloxy[1,1'-bipheny]-4-yl)-1-hexanone, 681
[56117-57-8]	1-(4'-Decyloxy[1,1'-bipheny]]-4-yl)-1-heptanone, 757
[56117-58-9]	1-(4'-Decyloxy[1,1'-biphenyl]-4-yl)-1-octanone, 815
[56117-59-0]	1-(4'-Decyloxy[1,1'-biphenyl]-4-yl)-1-nonanone, 854
[56117-60-3]	1-(4'-Decyloxy[1,1'-biphenyl]-4-yl)-1-decanone, 905
[56117-65-8]	1-(4'-Dodecyloxy[1,1'-biphenyl]-4-yl)-1-hexanone, 681
[56117-66-9]	1-(4'-Dodecyloxy[1,1'-biphenyl]-4-yl)-1-heptanone, 757
[56117-67-0]	1-(4'-Dodecyloxy[1,1'-biphenyl]-4-yl)-1-octanone, 815
[56117-68-1]	1-(4'-Dodecyloxy[1,1'-biphenyl]-4-yl)-1-nonanone, 854
[56117-69-2]	1-(4'-Dodecyloxy[1,1'-biphenyl]-4-yl)-1-decanone, 905
[56134-29-3]	1-(2-Hydroxy-5-methoxyphenyl)-1-dodecanone, 962
[56134-31-7]	1-(3-Dodecyl-2-hydroxy-5-methoxyphenyl)-1-dodecanone, 989
[56134-34-0]	1-(2-Hydroxy-4,5-dimethoxyphenyl)-1-dodecanone, 966
[56134-35-1]	1-(2,4,5-Trimethoxyphenyl)-1-dodecanone, 951
[56189-90-3]	1-(4'-Heptyloxy[1,1'-biphenyl]-4-yl)-1-nonanone, 853
[56226-93-8]	1-[3-[[2,6-Dihydroxy-4-methoxy-3-methyl-5-(1-oxobutyl)phenyl]methyl]- 2,4,6-trihydroxy-5-methylphenyl]-1-butanone, 340
[56290-52-9]	1-(2-Methoxy-5-methylphenyl)-1,3-butanedione, 317
[56290-53-0]	1-(2-Methoxyphenyl)-1,3-butanedione, 309
[56397-48-9]	1-(6-Methoxy-3-methyl-2-benzofuranyl)-1-butanone, 79
[56426-10-9]	1-[4-(1,1,2,2-Tetrafluoroethyloxy)phenyl]-1-heptanone, 724
[56490-66-5]	1-[4-Hydroxy-3-(methylsulfonylmethyl)phenyl]-1-butanone, 77
[56490-73-4]	1-[3-(Methylsulfonylmethyl)-4-(phenylmethoxy)phenyl]-1-butanone, 77
[56490-82-5]	2-Bromo-1-[3-(methylsulfonylmethyl)-4-phenylmethoxy)phenyl]
	butanone, 273
[56490-86-9]	1-[(3-Chloromethyl)-4-hydroxyphenyl]-1-butanone, 45
[56686-30-7]	1-(2-Hydroxy-3-methylphenyl)-1,3-butanedione (Dioxime), 317
[56686-34-1]	1-(2-Hydroxy-5-methylphenyl)-1,3-butanedione (Dioxime), 317
[56871-93-3]	Methyl 4-(2-hydroxyphenyl)-4-oxo-1-butanoate, 399
[56872-07-2]	4-(3-Hydroxyphenyl)-4-oxo-1-butanoic acid, 399
[56872-21-0]	4-(3-Chloro-4-hydroxyphenyl)-4-oxo-1-butanoic acid, 422
[56872-34-5]	4-(3-Hydroxy-4-methylphenyl)-4-oxo-1-butanoic acid, 435
[56872-39-0]	4-(4-Hydroxyphenyl)-4-oxo-1-butanoic acid, 400

[56872-41-4]	4-(4-Hydroxy-3-nitrophenyl)-4-oxo-1-butanoic acid, 426
[56872-54-9]	1-(5-Amino-2-hydroxyphenyl)-4-oxo-1-butanoic acid, 428
[56872-60-7]	Ethyl 4-(3,4-dimethoxyphenyl)-4-oxo-1-butanoate, 409
[56872-61-8]	Ethyl 4-(3,4-dihydroxyphenyl)-4-oxo-1-butanoate, 410
[57010-67-0]	4-Chloro-1-(3,4-dimethoxyphenyl)-1-butanone, 282
[57073-43-5]	1-[2-Hydroxy-4-[(2,2,4-trimethylpentyl)oxy]phenyl]-1-nonanone (Oxime), 862
[57073-45-7]	1-[2-Hydroxy-5-(2,2,4-trimethylpentyl)phenyl]-1-nonanone (Oxime), 862
[57080-91-8]	2-Ethyl-1-[2-hydroxy-4-(1-methylethyl)phenyl]-1-hexanone (Oxime), 669
[57080-92-9]	2-Ethyl-1-[2-hydroxy-4-(1-methylethyl)phenyl]-1-hexanone, 669
[57080-94-1]	1-(2-Hydroxy-4-methylphenyl)-14-methyl-1-pentadecanone, 1030
[57080-95-2]	1-(5-Chloro-2-hydroxy-4-methylphenyl)-2-ethyl-1-hexanone (Oxime), 656
[57080-96-3]	1-(5-Chloro-2-hydroxy-4-methylphenyl)-2-ethyl-1-hexanone, 656
[57080-97-4]	2-Ethyl-1-(2-hydroxy-4-methylphenyl)-1-hexanone (Oxime), 657
[57080-99-6]	2-Ethyl-1-(2-hydroxy-4-methylphenyl)-1-hexanone, 657
[57125-28-7]	1-(2-Hydroxy-4-methylphenyl)-14-methyl-1-pentadecanone (Oxime), 1030
[57133-44-5]	1-[2-Hydroxy-3,5-di(1-methylethyl)phenyl]-1-pentanone (ion) (1-), (radical ion) (1-), 527
[57262-58-5]	7-(2-Hydroxyphenyl)-7-oxo-1-heptanoic acid, 766
[57314-80-4]	1-(2-Hydroxy-5-methoxyphenyl)-1-pentanone, 496
[57314-81-5]	1-(2-Hydroxy-5-methoxyphenyl)-1-butanone, 55
[57596-02-8]	4-(3,4-Dihydroxyphenyl)-4-oxo-1-butanoic acid, 408
[57640-96-7]	9-(3,4-Dimethoxyphenyl)-9-oxo-1-nonanoic acid, 864
[57641-18-6]	Methyl 6-(3,4-dimethoxyphenyl)-6-oxo-1-hexanoate, 714
[57641-19-7]	Ethyl 8-(3,4-dimethoxyphenyl)-8-oxo-1-octanoic acid, 831
[57641-20-0]	Ethyl 9-(3,4-dimethoxyphenyl)-9-oxo-1-nonanoate, 864
[57765-52-3]	1-(2,6-Dihydroxy-4-methoxy-3-methylphenyl)-1-pentanone, 502
[57835-34-4]	1-(2-Hydroxyphenyl)-1-octanone (Oxime), 773
[57840-53-6]	6-Bromo-1-(4-Chloro-2-methoxyphenyl)-1-hexanone, 703
[57840-61-6]	6-Bromo-1-(4-methoxyphenyl)-1-hexanone, 698
[57863-94-2]	1-(2,4,5-Trihydroxyphenyl)-1-octadecanone, 1068
[57991-55-6]	1-(2,4-Dihydroxyphenyl)-1-pentanone (Oxime), 471
[58185-73-2]	6-(2-Hydroxy-3,4,6-trimethylphenyl)-6-oxo-1-hexanoic acid, 717
[58185-75-4]	7-(2-Hydroxy-3,4,6-trimethylphenyl)-7-oxo-1-heptanoic acid, 770
[58185-77-6]	10-(2-Hydroxy-3,4,6-trimethylphenyl)-10-oxo-1-decanone, 922
[58218-16-9]	1-(2-Hydroxy-4-methylphenyl)-1,3-butanedione, 317
[58218-17-0]	1-(2-Hydroxy-3-methylphenyl)-1,3-butanedione, 317
[58530-24-8]	1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1,3-butanedione, 322
[59010-46-7]	4-(2-Hydroxy-4-methylphenyl)-4-oxo-1-butanoic acid, 433
[59010-47-8]	Methyl 4-(2-hydroxy-4-methylphenyl)-4-oxo-1-butanoate, 434
[59010-62-7]	4-(2-Hydroxyphenyl)-3-methyl-4-oxo-1-butanoic acid, 414
[59043-82-2]	1-(2,3-Dichloro-4-methoxyphenyl)-2-methylene-1-butanone, 131
[59445-62-4]	1-(2-Acetyl-4-methoxy-7-benzofuranyl)-1-dodecanone, 971
[59445-63-5]	1-(2-Acetyl-4-methoxy-7-benzofuranyl)-1-octadecanone, 1081
[59445-72-6]	1-(2-Acetyl-7-methoxy-4-benzofuranyl)-1-hexanone, 661

[59445-73-7]	1-(2-Acetyl-7-methoxy-4-benzofuranyl)-1-dodecanone, 971
[59445-74-8]	1-(2-Acetyl-7-methoxy-4-benzofuranyl)-1-octadecanone, 1081
[59445-81-7]	1-(2-Acetyl-7-hydroxy-4-benzofuranyl)-1-hexanone, 661
[59701-65-4]	4-(2,5-Dihydroxyphenyl)-4-oxo-1-butanoic acid, 405
[59701-66-5]	Methyl 4-(2-hydroxy-5-methoxyphenyl)-4-oxo-1-butanoate, 440
[60159-70-8]	1-(2-Ethoxyphenyl)-1,3-butanedione, 310
[60170-85-6]	1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-decanone, 896
[60202-03-1]	1-(2-Hydroxy-4-methylphenyl)-1-dodecanone (Oxime, cobalt alloys), 959
[60474-43-3]	1-(3-Chloro-2-hydroxyphenyl)-1-butanone, 29
[60488-53-1]	1-(2-Hydroxy-4-methoxy-5-methylphenyl)-1-dodecanone, 966
[60488-57-5]	1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1-dodecanone, 966
[60658-72-2]	1-(2-Hydroxy-4,6-dimethoxyphenyl)-1,3-hexanedione, 647
[60697-65-6]	1-(8-Hydroxy-7-quinolinyl)-1-heptanone, 745
[60755-22-8]	1-(4-Methoxyphenyl)-4-phenyl-1,4-butanedione, 357
[60831-55-2]	1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-3-methyl-1-butanone, 207
[60985-68-4]	1-[4-(2-Bromoethyloxy)phenyl]-1-heptanone, 724
[61053-78-9]	4-Chloro-1-(4-fluoro-2-hydroxyphenyl)-1-butanone, 278
[61363-13-1]	1-(3,4-Methylenedioxyphenyl)-1,4-pentanedione, 487
[61468-98-2]	5-(4-Methoxyphenyl)-2,2-dimethyl-5-oxo-1-pentanoic acid, 584
[62036-46-8]	1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1,3-pentanedione, 499
[62036-47-9]	1-(2,4-Dimethoxy-6-methylphenyl)-1,3-pentanedione, 488
[62060-62-2]	1-(2,4,5-Trihydroxyphenyl)-1-pentanone, 476
[62153-16-6]	1-(8-Hydroxy-7-quinolinyl)-1-heptanone (Copper complex), 745
[62170-25-6]	1-(4-Methoxyphenyl)-1-octanone, 775
[62189-86-0]	1-(8-Ethyloxy-7-quinolinyl)-1-heptanone, 745
[62189-88-2]	1-(8-Hydroxy-5-quinolinyl)-1-heptanone, 745
[62406-99-9]	1-(2,4,5-Trimethoxyphenyl)-1,3-butanedione, 314
[62407-00-5]	1-(2,4,5-Trimethoxyphenyl)-2-methyl-1,3-butanedione, 315
[62439-32-1]	1-(4-Hydroxyphenyl)-3-methyl-1-pentanone (+), 554
[62458-64-4]	1-(5-Acetyl-2-hydroxyphenyl)-3-methyl-1-butanone, 378
[62545-32-8]	1-[4-(Acetyloxy)-2,6-dihydroxyphenyl]-3-methyl-1-butanone, 196
[62545-33-9]	1-[2-(Acetyloxy)-4,6-dihydroxyphenyl]-3-methyl-1-butanone, 195
[62596-41-2]	1-(4-Methoxyphenyl)-1,4-hexanedione, 597
[62643-23-6]	7-(2,4-Dimethoxy-6-methylphenyl)-3,5,7-trioxo-1-heptanoic acid, 768
[62643-36-1]	1-(2,4-Dimethoxy-6-methylphenyl)-1,3,5-hexanetrione, 629
[62643-39-4]	1-(2-Benzyloxy-4-methoxy-6-methylphenyl)-1,3,5-hexanetrione, 645
[62643-41-8]	Methyl 7-(2-benzyloxy-4-methoxy-6-methylphenyl)-3,5,7-trioxo-
	1-heptanoate, 770
[62810-51-9]	1-(3-Hydroxyphenyl)-1-pentanone, 464
[62893-18-9]	4-(3-Hydroxy-4-nitrophenyl)-4-oxo-1-butanoic acid, 426
[62903-11-1]	4-(3,5-Dichloro-2-hydroxy-4-methylphenyl)-4-oxo-1-butanoic acid, 430
[62903-21-3]	4-(5-Chloro-2-hydroxy-4-methylphenyl)-4-oxo-1-butanoic acid, 432
[62903-22-4]	4-(3,5-Dichloro-2-hydroxyphenyl)-4-oxo-1-butanoic acid, 420
[62903-23-5]	4-(5-Chloro-2-hydroxyphenyl)-4-oxo-1-butanoic acid, 423
[62903-25-7]	4-(3,5-Dichloro-4-hydroxyphenyl)-4-oxo-1-butanoic acid, 420
	(continued)

[63023-50-7]	Methyl 5-(2-hydroxy-4-methylphenyl)-5-oxo-1-pentanoate, 591
[63134-27-0]	1-(2,5-Dihydroxy-4-octylphenyl)-1-octanone, 823
[63171-82-4]	Tert-Butyl 4-(2,5-dimethoxyphenyl)-4-oxo-1-butanoate, 407
[63213-25-2]	4-(2,4,6-Trimethoxyphenyl)-4-oxo-1-butanoic acid, 414
[63213-26-3]	Ethyl 4-(2,4,5-triethoxyphenyl)-4-oxo-1-butanoate, 413
[63213-28-5]	4-(2,4,5-Trihydroxyphenyl)-4-oxo-1-butanoic acid, 412
[63213-31-0]	1-(2,4,5-Triethoxyphenyl)-1-butanone, 18
[63213-32-1]	4-(2,4,5-Triethoxyphenyl)-2-methyl-4-oxo-1-butanoic acid, 418
[63213-33-2]	5-(2,4,5-Triethoxyphenyl)-5-oxo-1-pentanoic acid, 583
[63213-34-3]	4-(2,4,5-Triethoxyphenyl)-2,4-dioxo-1-butanoic acid, 398
[63213-39-8]	4-(2,4,5-Tripropoxyphenyl)-4-oxo-1-butanoic acid, 413
[63213-40-1]	4-(5-Bromo-2,4-dimethoxyphenyl)-4-oxo-1-butanoic acid, 421
[63213-41-2]	4-(2,3,4-Trimethoxyphenyl)-4-oxo-1-butanoic acid, 411
[63213-42-3]	4-(3,4-Diethoxyphenyl)-4-oxo-1-butanoic acid, 409
[63213-44-5]	Ethyl 4-(2,4,5-triethoxyphenyl)-2,4-dioxo-1-butanoate, 398
[63213-45-6]	4-(4,5-Diethoxy-2-hydroxyphenyl)-4-oxo-1-butanoic acid, 450
[63213-46-7]	4-(2,5-Diethoxyphenyl)-4-oxo-1-butanoic acid, 407
[63213-94-5]	4-(5-Chloro-2-methoxyphenyl)-4-oxo-1-butanoic acid, 424
[63335-23-9]	1-(2,6-Dihydroxyphenyl)-9-phenyl-1-nonanone, 855
[63335-24-0]	1-(2,6-Dihydroxyphenyl)-9-(4-hydroxyphenyl)-1-nonanone, 855
[63335-25-1]	1-(2,6-Dihydroxyphenyl)-9-(3,4-dihydroxyphenyl)-1-nonanone, 856
[63335-26-2]	9-(1,3-Benzodioxol-5-yl)-1-(2,6-dihydroxyphenyl)-1-nonanone, 857
[63411-80-3]	1-(2,6-Dihydroxyphenyl)-1-pentanone, 472
[63424-82-8]	3-Amino-1-(4-hydroxyphenyl)-1-dodecanone (Hydrochloride), 953
[63424-84-0]	2-Bromo-1-(4-methoxyphenyl)-1-dodecanone, 993
[63442-82-0]	1-(3-Methoxyphenyl)-1-decanone, 869
[63442-83-1]	1-(3-Methoxyphenyl)-1-dodecanone, 941
[63442-84-2]	1-(3-Methoxyphenyl)-1-hexadecanone, 1032
[63442-85-3]	1-(3-Methoxyphenyl)-1-octadecanone, 1063
[63442-86-4]	1-(3-Hydroxyphenyl)-1-dodecanone, 941
[63442-87-5]	1-(3-Hydroxyphenyl)-1-hexadecanone, 1032
[63442-88-6]	1-(3-Hydroxyphenyl)-1-octadecanone, 1063
[63467-20-9]	5-(2,5-Dimethoxyphenyl)-5-oxo-1-pentanoic acid, 581
[63471-88-5]	4-(4-Butoxyphenyl)-4-oxo-1-butanoic acid, 402
[63480-88-6]	1-(3-Hydroxyphenyl)-1-decanone, 869
[63494-45-1]	1-(2-Hydroxy-5-pentylphenyl)-1-pentanone, 513
[63828-97-7]	2-Bromo-1-(3,4-dimethoxyphenyl)-1-dodecanone, 993
[63829-15-2]	1-[3-(Dimethylaminomethyl)-4-methoxyphenyl]-1-dodecanone, 971
[63829-20-9]	1-(4-Methoxyphenyl)-1-dodecanone, 943
[63861-11-0]	2,4,6-Trihydroxy-3-methyl-5-(3-methyl-1-oxobutyl)benzaldehyde, 389
[63861-20-1]	2,4-Dihydroxy-6-methoxy-5-methyl-3-(3-methyl-1-oxobutyl) benzaldehyde, 390
[63861-21-2]	2,6-Dihydroxy-4-methoxy-5-methyl-3-(3-methyl-1-oxobutyl) benzaldehyde, 390
[64142-23-0]	1-(4-Hydroxy-3-methoxyphenyl)-1-butanone, 56

[64779-96-0]	5-Butyryl-2-methoxybenzoic acid, 44
[64808-71-5]	Methyl 5-butyryl-2-methoxybenzoate, 44
[64808-72-6]	5-(1-Oxobutyl)-2-methoxybenzoyl chloride, 41
[64957-70-6]	1-(2-Methoxyphenyl)-1-pentanone (2,4-Dinitrophenylhydrazone), 463
[65240-03-1]	2,2,3,3,4,4,4-Heptafluoro-1-(2,4-dihydroxyphenyl)-1-butanone, 289
[65240-04-2]	2,2,3,3,4,4,4-Heptafluoro-1-(2,4-dihydroxy-3-methylphenyl)- 1-butanone, 290
[65240-06-4]	1-(2,4-Dihydroxy-3-methylphenyl)-2,2,3,3,4,4,5,5,6,6,7,7,8,8,8- pentadecafluoro-1-octanone, 829
[65242-00-4]	4-Chloro-1-(2-hydroxy-4-methoxyphenyl)-1-butanone, 285
[65547-46-8]	1-(2,3,6-Trimethoxyphenyl)-1,3-butanedione, 324
[65547-48-0]	1-(2,3,4-Trimethoxyphenyl)-1,3-butanedione, 323
[65547-50-4]	1-(2,5-Dimethoxyphenyl)-1,3-butanedione, 313
[65547-52-6]	1-(2,3-Dimethoxyphenyl)-1,3-butanedione, 312
[65547-54-8]	1-(2,4-Dimethoxyphenyl)-1,3-butanedione, 312
[65547-60-6]	1-(2-Hydroxy-4,6-dimethoxyphenyl)-1,3-butanedione, 324
[65547-62-8]	1-(2-Hydroxy-3,6-dimethoxyphenyl)-1,3-butanedione, 323
[65547-63-9]	1-(2-Hydroxy-6-methoxyphenyl)-1,3-butanedione, 320
[65547-68-4]	1-(2-Hydroxy-4,5-dimethoxyphenyl)-1,3-butanedione, 324
[65547-70-8]	1-[4,5-Dimethoxy-2-(phenylmethoxy)phenyl]-1,3-butanedione, 324
[65547-71-9]	1-(6-Hydroxy-2,3-dimethoxyphenyl)-1,3-butanedione, 325
[65547-75-3]	1-(2-Hydroxy-3,4-dimethoxyphenyl)-1,3-butanedione, 323
[65547-78-6]	1-(2-Hydroxy-3-methoxyphenyl)-1,3-butanedione, 319
[65547-81-1]	1-(2-Hydroxy-5-methoxyphenyl)-1,3-butanedione, 320
[65547-83-3]	1-[5-methoxy-(2-phenylmethoxy)phenyl]-1,3-butanedione, 320
[65687-21-0]	1-(3'-Chloro-4'-methoxy[1,1'-biphenyl]-4-yl)-1-octanone, 814
[65792-31-6]	2-Methyl-1-(2,4,6-trihydroxy-3,5-dimethylphenyl)-1-butanone, 136
[65897-66-7]	1-(5-Chloro-2-hydroxyphenyl)-1,3-butanedione, 316
[66053-97-2]	1-(3,4-Dimethoxyphenyl)-1-pentanone, 473
[66123-43-1]	Ethyl 4-(4-hydroxyphenyl)-4-oxo-1-butanoate, 402
[66123-78-2]	Ethyl 5-(4-hydroxyphenyl)-5-oxo-1-pentanoate, 580
[66333-82-2]	1-(4-Methoxyphenyl)-3-methyl-1-pentanone, 554
[66346-51-8]	1-(2,4-Dimethoxy-6-methylphenyl)-1,3-hexanedione, 633
[66468-51-7]	1-(4-Ethoxy-2-hydroxyphenyl)-1-decanone (Copper complex), 888
[66475-97-6]	1-(3-Decanoyloxy-4-methoxyphenyl)-1-decanone, 885
[66476-00-4]	1-(3-Hydroxy-4-methoxyphenyl)-1-decanone, 885
[66476-01-5]	1-(4-Hydroxy-3-methoxyphenyl)-1-decanone, 885
[66711-56-6]	1-[2,4,6-Trihydroxy-3-(2-methylpropyl)phenyl]-1-hexanone, 666
[66757-68-4]	5-(2,4-Dimethoxy-6-methylphenyl)-3,5-dioxo-1-pentanoic acid, 589
[66832-64-2]	Methyl 5-(2-hydroxy-4-methoxyphenyl)-5-oxo-1-pentanoate, 594
[66832-66-4]	5-Chloro-1-(2-hydroxy-4-methoxyphenyl)-1-pentanone, 575
[67049-69-8]	1-(3,4-Dihydroxyphenyl)-2-methyl-1-butanone, 128
[67049-70-1]	1-(3,4-Dimethoxyphenyl)-2-methyl-1-butanone, 128
[67114-29-8]	1-(3,4-Dihydroxyphenyl)-2-ethyl-3-methyl-1-butanone, 184
[67188-50-5]	1-(5,8-Dimethoxy-4-methyl-2-quinolinyl)-1-nonanone, 847

[67231-33-8]	1-(2,3,4,6-Tetramethoxyphenyl)-1,3-butanedione, 328
[67231-43-0]	1-(2,3,4,5-Tetramethoxyphenyl)-1,3-butanedione, 328
[67231-44-1]	1-(2,3,4,5,6-Pentamethoxyphenyl)-1,3-butanedione, 331
[67231-46-3]	1-(2-Hydroxy-3,4,5-trimethoxyphenyl)-1,3-butanedione, 328
[67231-48-5]	1-(2-Hydroxy-3,4,5,6-tetramethoxyphenyl)-1,3-butanedione, 331
[67239-25-2]	1-(3,4-Dihydroxyphenyl)-3-methyl-1-butanone, 179
[67405-48-5]	4-(4-Methoxy-2-methylphenyl)-4-oxo-1-butanoic acid, 435
[67548-60-1]	1-(4-Chloro-2-hydroxyphenyl)-1-nonanone, 842
[67548-61-2]	1-(3-Bromo-4-hydroxyphenyl)-1-pentanone, 480
[67548-62-3]	1-(5-Bromo-2-hydroxyphenyl)-1-pentanone, 481
[67756-15-4]	1-(4-Methoxyphenyl)-1,4-decanedione, 867
[67756-16-5]	1-(4-Chlorophenyl)-4-(4-methoxyphenyl)-1,4-butanedione, 357
[67756-19-8]	1-(2,4-Dimethoxyphenyl)-1,4-pentanedione, 461
[67756-20-1]	1-(2,4-Dimethoxyphenyl)-1,4-hexanedione, 597
[67756-21-2]	1-(2,4-Dimethoxyphenyl)-1,4-decanedione, 867
[67756-23-4]	1-(3,4-Dimethoxyphenyl)-1,4-hexanedione, 597
[67756-24-5]	1-(3,4-Dimethoxyphenyl)-1,4-decanedione, 868
[67756-25-6]	1-(3,4-Dimethoxyphenyl)-4-phenyl-1,4-butanedione, 357
[68223-30-3]	1,1'-[Methylenebis(2,4,6-trihydroxy-3,5-phenylene)]bis-1-butanone, 345
[68223-33-6]	1,1',1",1"'-[Methylenebis(2,4,6-trihydroxy-5,1,3-benzenetriyl)]
	bis-3-methyl-1-butanone, 346
[68223-34-7]	1,1'-Methylenebis(2,4,6-trihydroxy-3,5,1-phenylene)bis-1-hexanone, 695
[68223-37-0]	1,1'-[Methylenebis(2,4,6-trihydroxy-3-acetyl-5,1-phenylene)] bis-1-butanone, 341
[68223-39-2]	1-[3-[(3,5-Dipropionyl)-2,4,6-trihydroxyphenylmethyl]-2,4,6-trihydroxy-
[(0000 50 5]	5-methylphenyl]-1-butanone, 339
[68223-50-7]	1-[3-(3-Acetyl-5-butyryl-2,4,6-trihydroxyphenylmethyl)-2,4,6-trihydroxy- 5-methylphenyl]-1-butanone, 338
[68436-75-9]	1-[(4-methoxy-6-methyl-2-phenylmethoxy)phenyl]-1,3-butanedione, 322
[68436-79-3]	1-[4,6-Dimethoxy-2-(phenylmethoxy)phenyl]-1,3-butanedione, 325
[68486-75-9]	1-(2-Hydroxy-3,4-dimethoxyphenyl)-4-oxo-1-butanoic acid, 446
[68754-16-5]	1-(2-Hydroxy-4,6-dimethoxyphenyl)-3-methyl-1-butanone, 199
[69271-91-6]	1-(4-Hydroxy-3,5-dimethoxyphenyl)-1-butanone, 76
[69287-13-4]	1-(4-Methoxyphenyl)-1-heptanone, 723
[69299-76-9]	Uliginosin A-iBiV, 346
[69480-05-3]	1-(2,6-Dihydroxy-4-methoxyphenyl)-1-pentanone, 498
[69480-08-6]	1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-1-butanone, 84
[69618-10-6]	1,4-Bis(2-acetyloxy-5-methylphenyl)-1,4-butanedione, 366
[69618-11-7]	1,4-Bis(2-methoxy-5-methylphenyl)-1,4-butanedione, 367
[69639-78-7]	1-(5-Bromo-2-methoxyphenyl)-4-chloro-1-butanone, 277
[69639-79-8]	1-(5-Bromo-2-hydroxyphenyl)-4-chloro-1-butanone, 277
[69657-35-8]	1-[3-(Chloromethyl)-4-methoxyphenyl]-1-undecanone, 930
[69657-36-9]	1-(4-Methoxyphenyl)-1-undecanone, 924
[69916-08-1]	1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-butanone, 97
[69916-09-2]	1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-pentanone, 512
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[69916-10-5]	1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-hexanone, 668
[69916-11-6]	1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-heptanone, 751
[69916-12-7]	1-(2,4,6-Trihydroxy-3-pentylphenyl)-1-pentanone, 515
[70079-24-2]	1-(4-Hydroxy-3-nitrophenyl)-1-dodecanone, 956
[70079-25-3]	1-(4-Hydroxy-3-nitrophenyl)-1-hexanone, 627
[70079-26-4]	1-(4-Hydroxy-3-nitrophenyl)-1-octanone, 788
[70079-27-5]	1-(4-Hydroxy-3-nitrophenyl)-1-decanone, 881
[70079-28-6]	1-(4-Hydroxy-3-nitrophenyl)-1-hexadecanone, 1040
[70079-29-7]	1-(4-Hydroxy-3-nitrophenyl)-1-octadecanone, 1072
[70206-42-7]	3-[4-(2-Chloroethyl)phenyl]-1-(2-hydroxyphenyl)-1-hexanone, 688
[70206-43-8]	3-[4-(2-Chloroethyl)phenyl]-2-hydroxy-1-(2-hydroxyphenyl)-
	1-hexanone, 688
[70219-82-8]	1-[2,6-Dihydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-2-methyl-
	1-butanone, 142
[70219-89-5]	1-[2,4-Dihydroxy-3,6-dimethoxy-5-(3-methyl-2-butenyl)phenyl]-
	1-butanone, 111
[70219-90-8]	1-[2,4-Diacetyloxy-3,6-dimethoxy-5-(3-methyl-2-butenyl)phenyl]-
[20222 25 0]	1-butanone, 111
[70233-75-9]	3,4-Dihydro-5,7-dihydroxy-8-(2-methyl-1-oxobutyl)-4-pentyl-2 <i>H</i> -
[70627 61 1]	1-benzopyran-2-one, 149         1-(3,5-Dihydroxyphenyl)-1-pentanone, 473
[70627-61-1]	
[70977-53-6]	N-(3-Butanoyl-2-hydroxy-5-methylphenyl)-1 <i>H</i> -tetrazole-5-carboxamide, 80
[70978-14-2]	N-(3-Butanoyl-2-hydroxy-5-methylphenyl)-1-phenylmethyltetrazole- 5-carboxamide, 118
[70978-45-9]	1-(2-Hydroxy-5-methyl-3-nitrophenyl)-1-butanone, 46
[70978-64-2]	1-(3-Amino-2-hydroxy-5-methylphenyl)-1-butanone, 60
[71248-64-1]	1,6-Bis(4-methoxyphenyl)-2,5-dibromo-1,6-hexanedione, 671
[71290-02-3]	1-(4,5-Dichloro-2-hydroxyphenyl)-1-butanone, 25
[71354-31-9]	5-(3-Chloro-4-methoxyphenyl)-5-oxo-1-pentanoic acid, 587
[71354-32-0]	Methyl 5-(3-chloro-4-methoxyphenyl)-5-oxo-1-pentanoate, 587
[71354-32-0]	5-(2-Chloro-5-methoxyphenyl)-5-oxo-1-pentanoite, 567
[71354-33-1]	Methyl 5-(2-chloro-5-methoxyphenyl)-5-oxo-1-pentanoate, 587
[71354-35-3]	5-(2-Chloro-4-methoxyphenyl)-5-oxo-1-pentanoate, 587
[71354-36-4]	Methyl 5-(2-chloro-4-methoxyphenyl)-5-oxo-1-pentanote acta, 586
[71394-30-4]	1-(2-Hydroxy-5-methylphenyl)-1-decanone (Oxime), 883
	2-Methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl]-
[71539-60-1]	1-butanone, 142
[71539-61-2]	2-Methyl-1-[2,4,6-tris(acetyloxy)-3-(3-methyl-2-butenyl)phenyl]-
[71007-01-2]	1-butanone, 142
[71539-62-3]	1-[2,4-Dihydroxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-methyl-
	1-butanone, 144
[71539-64-5]	1-[2,4-Diacetyloxy-6-methoxy-3-(3-methyl-2-butenyl)phenyl]-2-methyl-
	1-butanone, 145
[71539-67-8]	1-[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2,6-dihydroxyphenyl]-2-methyl-
	1-butanone ( <i>E</i> ), 151
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[71539-68-9]	1-[4-[(3,7-Dimethyl-2,6-octadienyl)oxy]-2,6-diacetyloxyphenyl]-2-methyl- 1-butanone ( <i>E</i> ), 151
[71539-70-3]	1-(3,4-Dihydro-5-hydroxy-7-methoxy-2,2-dimethyl-2 <i>H</i> -1-benzopyran-6-yl)- 2-methyl-1-butanone, 145
[71898-88-9]	1-(2-Methoxy-4-methylphenyl)-3-methyl-1-butanone, 190
[72008-04-9]	1-[3-(3,7-Dimethyl-2,6-octadienyl)-2,4,6-trihydroxyphenyl]-2-methyl- 1-butanone ( <i>E</i> ), 151
[72008-09-4]	1-[3-(3,7-Dimethyl-2,6-octadienyl)-2,4,6-tris(acetyloxy)phenyl]-2-methyl- 1-butanone, 152
[72046-93-6]	1-(2,5-Dihexadecyloxy-4-methylphenyl)-1-dodecanone, 961
[72047-09-7]	1-(2,5-Didodecyloxyphenyl)-1-dodecanone, 947
[72057-94-4]	1-(4'-Hexanoyloxy[1,1'-biphenyl]-4-yl)-1-hexanone, 681
[72236-93-2]	5-Bromo-1-[4-methoxy-2-methyl-5-(1-methylethyl)phenyl]- 1-pentanone, 576
[72236-94-3]	6-Bromo-1-[4-methoxy-2-methyl-5-(1-methylethyl)phenyl]- 1-hexanone, 708
[72236-95-4]	10-Bromo-1-[4-methoxy-2-methyl-5-(1-methylethyl)phenyl]- 1-decanone, 917
[72247-02-0]	1-(5-Ethyl-2-methoxyphenyl)-3-methyl-1-butanone, 197
[72306-95-7]	1-(2-Hydroxy-5-methoxy-3-octadecylphenyl)-1-octadecanone, 1088
[72306-96-8]	1-(2,5-Dihydroxy-3-octadecylphenyl)-1-octadecanone, 1087
[72306-97-9]	1-(2,5-Dihydroxy-4-octadecylphenyl)-1-octadecanone, 1088
[72327-93-6]	1-(2,4,6-Tribenzyloxyphenyl)-1,3,5,7-octanetetraone, 771
[72327-96-9]	1-(2,4,6-Trihydroxyphenyl)-1,3,5,7-octanetetraone, 771
[72424-10-3]	1-(3-Methoxyphenyl)-1-undecanone, 923
[72674-91-0]	Methyl 9-(3,4-methylenedioxyphenyl)-9-oxo-1-nonanoate, 864
[72724-26-6]	1-(2-Hydroxy-4-methylphenyl)-1-dodecanone, 958
[72782-46-8]	1-[2-Hydroxy-5-(1,1,3,3-tetramethylbutyl)phenyl]-1-octanone (Oxime), 822
[72793-41-0]	3-[4-(2-Chloroethyl)phenyl]-2-hydroxy-1-(2-hydroxyphenyl)- 1-hexanone, 688
[72793-42-1]	3-[4-(2-Chloroethyl)phenyl]-2-hydroxy-1-(2-hydroxyphenyl)-
	1-hexanone, 688
[72935-10-5]	2-(5,7-Dihydroxy-8-isovaleryl-2,2-dimethyl-2 <i>H</i> -chromen-6-ylmethyl)- 3,5-dihydroxy-6-isobutyryl-4,4-dimethyl-2,5-cyclohexadiene-1-one, 345
[72935-11-6]	2-(5,7-Dihydroxy-6-isovaleryl-2,2-dimethyl-2 <i>H</i> -chromen-8-ylmethyl)- 3,5-dihydroxy-6-isobutyryl-4,4-dimethyl-2,5-cyclohexadiene-1-one, 345
[73213-21-5]	1-(4,6-Dimethoxy-1,3-benzodioxol-5-yl)-2-methyl-1-butanone, 133
[73694-18-5]	1-(4,6-Dihydroxy-2,3-dimethoxyphenyl)-2-methyl-1-butanone, 137
[73694-27-6]	1-(4,6-Diacetyloxy-2,3-dimethoxyphenyl)-2-methyl-1-butanone, 137
[73991-79-4]	1-[2-Hydroxy-3,5-di(1-methylethyl)phenyl]-1-pentanone, 527
[74061-22-6]	1-(2,3-Dihydroxy-5-octadecylphenyl)-1-octadecanone, 1087
[74261-29-3]	1-[4-(4-Bromophenyloxy)phenyl]-1-nonanone, 837
[74277-78-4]	4-(4'-Hydroxybiphenyl)-4-oxo-1-butanoic acid, 451
[74362-69-9]	4-(4-Ethoxy-3-nitrophenyl)-4-oxo-1-butanoic acid, 426
[74477-96-6]	1-(2,4,6-Trihydroxy-3-pentylphenyl)-1-butanone, 100
[74477-97-7]	1-[2,4,6-Trihydroxy-3-(3-methylbutyl)phenyl]-1-butanone, 100

[74477-98-8]	1-[2,4,6-Trihydroxy-3-(3-methylbutyl)phenyl]-1-pentanone, 514
[74477-99-9]	1-[2,4,6-Trihydroxy-3-(2-propenyl)phenyl]-1-pentanone, 504
[74478-03-8]	3-Methyl-1-(2,4,6-trihydroxy-3-pentylphenyl)-1-butanone, 217
[74478-04-9]	1-(2,4,6-Trihydroxy-3-pentylphenyl)-1-hexanone, 671
[74478-05-0]	1-[2,4,6-Trihydroxy-3-(3-methylbutyl)phenyl]-1-hexanone, 670
[74478-06-1]	1-[2,4,6-Trihydroxy-3-(2-propenyl)phenyl]-1-hexanone, 655
[74478-07-2]	1-[3-(2-Butenyl)-2,4,6-trihydroxyphenyl]-1-hexanone, 662
[74478-08-3]	1-[2,4,6-Trihydroxy-3-(2-methyl-2-propenyl)phenyl]-1-hexanone, 663
[74478-09-4]	4-Methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-buten-1-yl)phenyl]-
	1-pentanone, 550
[74478-10-7]	1-[2,4,6-Trihydroxy-3-methylphenyl]-1-octanone, 797
[74478-11-8]	1-(2,4,6-Trihydroxyphenyl)-1-nonanone, 840
[74478-12-9]	1-(2,4,6-Trihydroxy-3-pentylphenyl)-1-decanone, 898
[74478-13-0]	1-[2,4,6-Trihydroxy-3-(3-methylbutyl)phenyl]-1-decanone, 898
[74478-14-1]	1-(2,4,6-Trihydroxyphenyl)-1-undecanone, 928
[74571-50-9]	1-(5-Methoxy-2-methylphenyl)-1-pentanone, 494
[74571-52-1]	1-(5-Methoxy-2-methylphenyl)-1-pentanone
	(2,4-Dinitrophenylhydrazone), 495
[74604-06-1]	1-(2-Hydroxy-5-methylphenyl)-1-heptanone (Oxime), 739
[74604-07-2]	1-(2-Hydroxy-5-methyl-3-nitrophenyl)-1-octanone (Oxime), 791
[74604-08-3]	1-(3-Chloro-2-hydroxy-5-methylphenyl)-1-octanone (Oxime), 790
[74604-11-8]	1-(2-Hydroxy-5-octylphenyl)-1-octanone (Oxime), 822
[74604-13-0]	1-(2-Hydroxy-5-methylphenyl)-1-heptanone, 738
[74604-14-1]	1-(2-Hydroxy-5-methyl-3-nitrophenyl)-1-octanone, 791
[74604-16-3]	1-(3-Chloro-2-hydroxy-5-methylphenyl)-1-octanone, 790
[74604-17-4]	2-Ethyl-1-(2-hydroxy-5-methylphenyl)-1-hexanone, 657
[74604-21-0]	1-(2-Hydroxy-5-octylphenyl)-1-octanone, 822
[74832-95-4]	1-(3,5-Dichloro-2-hydroxyphenyl)-1-octanone (Oxime), 786
[74832-96-5]	1-(3,5-Dibromo-2-hydroxyphenyl)-1-octanone (Oxime), 785
[74882-02-3]	Methyl 1-(2-hydroxy-3,4-dimethoxyphenyl)-4-oxo-1-butanoate, 446
[74882-03-4]	4-(2,3-Dihydroxy-4-methoxyphenyl)-4-oxo-1-butanoic acid, 441
[74882-04-5]	4-(2,3,4-Trihydroxyphenyl)-4-oxo-1-butanoic acid, 410
[74965-90-5]	4-Hydroxy-3-(1-oxohexadecyl)-2H-1-benzopyran-2-one, 1045
[75058-74-1]	1-(3'-Bromo-4'-methoxy[1,1'-biphenyl]-4-yl)-1-octanone, 814
[75060-45-6]	1-[5-(1,1-Dimethylethyl)-2-methoxyphenyl]-1-pentanone, 508
[75060-46-7]	1-[5-(1,1-Dimethylethyl)-2-methoxyphenyl]-1-butanone, 90
[75060-52-5]	1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-pentanone, 508
[75060-53-6]	1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-butanone, 90
[75060-70-7]	1-[(3-Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]- 1-pentanone (Hydrochloride), 515
[75060-71-8]	1-[3-(Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]- 1-butanone (Hydrochloride), 101
[75060-95-6]	1-[(3-Aminomethyl)-5-(1,1-dimethylethyl)-2-hydroxyphenyl]- 1-pentanone, 515
[75160-39-3]	1-(2-Allyloxy-4,6-dimethoxyphenyl)-1,3-butanedione, 325
	(continued)

[75160-45-1]	1-(2-Allyloxy-4-methoxy-6-methylphenyl)-1,3-butanedione, 322
[75218-94-9]	1-(2-Hydroxy-3,4-dimethoxyphenyl)-1-butanone, 74
[75343-08-7]	4-Chloro-1-(4-ethoxyphenyl)-1-butanone, 280
[75343-28-1]	4-Chloro-1-(4-ethoxyphenyl)-1-butanone
	(2,4-Dinitrophenylhydrazone), 280
[75349-76-7]	4-Chloro-1-(4-methoxyphenyl)-1-butanone
	(2,4-Dinitrophenylhydrazone), 280
[75487-43-3]	1-(2-Hydroxy-5-methylphenyl)-1-nonanone, 844
[75487-44-4]	1-(2-Hydroxy-5-methylphenyl)-1-dodecanone, 959
[75501-54-1]	4-(2-Hydroxy-5-methoxyphenyl)-4-oxo-1-butanoic acid, 440
[75656-31-4]	1-(2,4,6-Trihydroxyphenyl)-12,14-dihydroxyoctadecanone, 1069
[75679-83-3]	1-(2,4,6-Trimethoxyphenyl)-12,14-dimethoxyoctadecanone, 1069
[76092-85-8]	1-(3,5-Dibromo-2,4-dihydroxyphenyl)-1-octanone, 785
[76092-86-9]	1-(3,5-Dibromo-2,4-dihydroxyphenyl)-1-nonanone, 841
[76115-96-3]	1-(2-Hydroxy-5-methylphenyl)-1-decanone, 883
[76402-07-8]	1-(2,5-Dihydroxy-3,4-dimethylphenyl)-1-tetradecanone, 1015
[76402-08-9]	1-[5-Hydroxy-3,4-dimethyl-2-(2-propenyloxy)phenyl]- 1-tetradecanone, 1019
[76402-09-0]	1-[2,5-Dihydroxy-3,4-dimethyl-6-(2-propenyl)phenyl]-
	1-tetradecanone, 1019
[76402-10-3]	1-(2,5-Dihydroxy-3,4-dimethyl-6-propylphenyl)-1-tetradecanone, 1019
[76402-12-5]	1-[2-Hydroxy-4-methyl-5-(2-propenyloxy)phenyl]-1-hexadecanone, 1049
[76402-13-6]	1-[3,6-Dihydroxy-4-methyl-2-(2-propenyl)phenyl]-1-hexadecanone, 1048
[76402-14-7]	1-(3,6-Dihydroxy-4-methyl-2-propylphenyl)-1-hexadecanone, 1050
[76569-40-9]	1-(2,4,6-Trimethoxyphenyl)-1-butanone, 19
[76631-00-0]	1-(2,4-Dimethoxy-6-hydroxyphenyl)-1,3,5-hexanetrione, 645
[76631-01-1]	1-(2,4,6-Trimethoxyphenyl)-1,3,5-hexanetrione, 596
[76631-02-2]	1-(2,4,6-Tribenzyloxyphenyl)-1,3,5-hexanetrione, 596
[76631-04-4]	1-(2,4-Dimethoxy-6-hydroxyphenyl)-1,3,5,7-octanetetraone, 798
[76631-05-5]	1-(2,4,6-Trimethoxyphenyl)-1,3,5,7-octanetetraone, 771
[76750-11-3]	1-(2-Ethoxy-4-hydroxyphenyl)-1-octadecanone, 1078
[76752-90-4]	1-(4-Methoxy-3-nitrophenyl)-1-dodecanone, 956
[77007-22-8]	1-(3'-Bromo-4'-methoxy[1,1'-biphenyl]-4-yl)-1-heptanone, 756
[77132-64-0]	1-(4-Chloro-2-hydroxyphenyl)-1-hexanone (Phenylhydrazone), 624
[77311-66-1]	1-[5-(Acetyloxyethyl)-2-methoxyphenyl]-3-methyl-1-butanone, 208
[77346-69-1]	1-(5-Ethyl-2-hydroxyphenyl)-3-methyl-1-butanone, 197
[77346-70-4]	1-(5-Ethyl-2-hydroxyphenyl)-3-methyl-1-butanone (Isovalerate), 197
[77346-71-5]	1,1'-(5-Ethyl-2-hydroxy-1,3-phenylene)bis-3-methyl-1-butanone, 304
[77346-72-6]	1,1'-(5-Ethyl-2-methoxy-1,3-phenylene)bis-3-methyl-1-butanone, 304
[77464-71-2]	1-(2,6-Dihydroxy-4-methoxyphenyl)-6,9,12,15-tetraen- 1-octadecanone, 1073
[77711-94-5]	Methyl 22-(2-Hydroxy-3,4-dimethoxy-6-methylphenyl)-22-oxo- 1-docosanoate, 1109
[77712-02-8]	18-Hydroxy-1-(2-hydroxy-3,4-dimethoxy-6-methylphenyl)-
	1-octadecanone, 1081 (continued)

[77712-07-3]	11-Acetyloxy-1-(2-hydroxy-3,4-dimethoxy-6-methylphenyl)- 1-undecanone, 931
[77712-08-4]	11-Hydroxy-1-(2-hydroxy-3,4-dimethoxy-6-methylphenyl)-
[///12-00-4]	1-indecanone, 931
[77712-21-1]	12-Hydroxy-1-(2-hydroxy-3,4-dimethoxy-6-methylphenyl)-
	1-dodecanone, 970
[77794-62-8]	Euglobal-IIc, 393
[77942-74-6]	1-(4-Hydroxyphenyl)-2-methyl-1-butanone (R), 127
[78334-92-6]	4-(5-Methoxy-2,4-dimethylphenyl)-4-oxo-1-butanoic acid, 444
[78417-95-5]	Methyl 5-hexanoyl-2-hydroxybenzoate, 634
[78417-96-6]	Methyl 2-hydroxy-5-octanoylbenzoate, 789
[78417-97-7]	Methyl 2-hydroxy-5-decanoylbenzoate, 881
[78417-98-8]	Methyl 2-Hydroxy-5-tetradecanoylbenzoate, 1011
[78418-00-5]	5-Hexanoyl-2-hydroxybenzoic acid, 634
[78418-01-6]	2-Hydroxy-5-octanoylbenzoic acid, 789
[78418-02-7]	2-Hydroxy-5-decanoylbenzoic acid, 881
[78418-03-8]	2-Hydroxy-5-dodecanoylbenzoic acid, 956
[78418-04-9]	2-Hydroxy-5-tetradecanoylbenzoic acid, 1011
[78423-49-1]	2,4,6-Trihydroxy-3-(3-methyl-1-oxobutyl)benzaldehyde, 387
[78432-96-9]	Methyl 2-Hydroxy-5-dodecanoylbenzoate, 957
[78481-50-2]	1-(5-Methoxy-2,3-diphenyl-6-benzofuranyl)-1,3-butanedione, 334
[79111-54-9]	1-(8-Hydroxy-7-quinolinyl)-1-nonanone, 846
[79111-55-0]	1-(8-Hydroxy-5-quinolinyl)-1-nonanone (Hydrochloride), 846
[79111-56-1]	1-(8-Hydroxy-5-quinolinyl)-1-nonanone, 845
[79214-31-6]	4-Chloro-1-(5-fluoro-2-hydroxyphenyl)-1-butanone, 278
[79330-93-1]	22-(2-Hydroxy-3,4-dimethoxy-6-methylphenyl)-22-oxo-1-docosanoic acid, 1108
[79381-16-1]	6-(2,5-Dimethoxyphenyl)-6-oxo-1-hexanoic acid, 713
[79553-90-5]	1-(2,4,6-Trihydroxyphenyl)-5,8,11,14,17-eicosapentaen-1-one (all Z), 1097
[79553-91-6]	1-(2,4,6-Trihydroxyphenyl)-1-eicosanone, 1098
[79619-25-3]	1-(4-Trifluoromethyloxyphenyl)-1-pentanone, 469
[79744-63-1]	3-Methyl-1-[2,4,5-trihydroxyphenyl]-1-butanone, 181
[80081-75-0]	Ethyl 4-(2,4-dimethoxyphenyl)-2,4-dioxo-1-butanoate, 397
[80222-34-0]	1-(4-Fluoro-2-methoxyphenyl)-1-pentanone, 484
[80222-35-1]	1-(2-Fluoro-4-methoxyphenyl)-1-pentanone, 483
[80269-97-2]	4-Chloro-1-(2,4-dimethoxyphenyl)-1-butanone, 281
[80356-11-2]	1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-pentanone, 509
[80356-12-3]	1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-3-methyl- 1-butanone, 208
[80356-13-4]	1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-2-methyl- 1-butanone, 139
[80427-32-3]	1-(5-Hydroxy-2-methoxyphenyl)-1-decanone, 885
[80427-37-8]	1-(2-Hydroxy-5-methoxyphenyl)-1-decanone, 884
[80752-03-0]	5,7-Dihydroxy-6-(1-oxobutyl)-4-phenyl-2 <i>H</i> -1-benzopyran-2-one, 115
[80848-70-0]	1-[4-(2-Ethylhexyloxy-2-hydroxyphenyl]-1-hexanone (Oxime, nickel
	complex), 689

[80848-71-1]	1-[4-(2-Ethylhexyloxy)-2-hydroxyphenyl]-1-decanone (Oxime, nickel complex), 912
[80848-72-2]	1-[4-(2-Ethylhexyloxy)-2-hydroxyphenyl]-1-octanone (Oxime, nickel complex), 823
[80849-31-6]	1-(2-Hydroxy-4-methylphenyl)-1-dodecanone (Nickel complex), 958
[80851-64-5]	1-[4-(2-Ethylhexyloxy)-2-hydroxyphenyl]-1-hexanone, 689
[80856-35-5]	1-(2-Hydroxyphenyl)-1,3-hexanedione, 596
[80856-36-6]	1-(2-Hydroxyphenyl)-4-methyl-1,3-hexanedione, 615
[80904-51-4]	4-Chloro-1-(2,4,6-trimethoxyphenyl)-1-butanone, 283
[80986-13-6]	1-(2-Hydroxy-4,6-dimethoxyphenyl)-1-pentanone, 502
[81096-36-8]	1,4-Bis(4-methoxyphenyl)-2,3-dimethyl-1,4-butanedione, 367
[81141-14-2]	1-(3,5-Dichloro-2-hydroxyphenyl)-1-butanone, 24
[81321-89-3]	1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-dodecanone (Oxime, nickel complex), 973
[81515-08-4]	1-(2,4-Dichloro-6-hydroxyphenyl)-1-tetradecanone, 1010
[81515-09-5]	1-(4,6-Dichloro-2-hydroxy-3-nitrophenyl)-1-tetradecanone, 1010
[81515-10-8]	1-[4,6-Bis(butylthio)-2-hydroxy-3-nitrophenyl]-1-tetradecanone, 1021
[81515-11-9]	1-[4,6-Bis(butylsulfonyl)-2-hydroxy-3-nitrophenyl]-1-tetradecanone, 1021
[82307-87-7]	1-(2,4-Dihydroxy-5-octylphenyl)-1-hexanone (Oxime, nickel complex), 689
[82322-05-2]	1-[5-(1,1-Dimethylethyl)-2,4-dihydroxyphenyl]-1-decanone (Oxime, nickel complex), 896
[82350-83-2]	1-(4-Hydroxy-3-nitrophenyl)-1-butanone, 35
[82427-57-4]	1-(2,6-Dihydroxyphenyl)-11-phenyl-1-undecanone, 932
[82460-89-7]	1-(2-Hydroxy-4,6-dimethoxyphenyl)-1-hexadecanone, 1045
[82460-90-0]	1-(2-Acetyloxy-4,6-dimethoxyphenyl)-1-hexadecanone, 1045
[82460-91-1]	1-(2,4,6-Triacetyloxyphenyl)-1-hexadecanone, 1038
[82461-11-8]	1-(2,4,6-Trihydroxyphenyl)-1-hexadecanone, 1038
[82652-25-3]	1-(6,7-Dimethoxy-5-propyl-1,3-benzodioxol-4-yl)-1-pentanone, 507
[82652-26-4]	1-(6,7-Dimethoxy-5-propyl-1,3-benzodioxol-4-yl)-1-hexanone, 663
[82652-27-5]	1-(6,7-Dimethoxy-5-propyl-1,3-benzodioxol-4-yl)-1-heptanone, 748
[82652-28-6]	1-(6,7-Dimethoxy-5-propyl-1,3-benzodioxol-4-yl)-1-octanone, 805
[82652-29-7]	1-(6,7-Dimethoxy-5-propyl-1,3-benzodioxol-4-yl)-1-nonanone, 848
[82652-30-0]	1-(6,7-Dimethoxy-5-propyl-1,3-benzodioxol-4-yl)-1-decanone, 894
[82652-35-5]	1-(6,7-Dihydro-4-methoxy-6-methylfuro[2,3-f]-1,3-benzodioxol-8-yl)- 1-heptanone, 747
[82652-36-6]	1-(6,7-Dihydro-4-methoxy-6-methylfuro[2,3-f]-1,3-benzodioxol-8-yl)- 1-octanone, 805
[82652-37-7]	1-(6,7-Dihydro-4-methoxy-6-methylfuro[2,3-f]-1,3-benzodioxol-8-yl)- 1-decanone, 894
[82684-67-1]	1-[4-(2-Hydroxyethyloxy)phenyl]-1-decanone, 871
[82883-60-1]	1-(2,4-Diphenylmethoxy-6-methylphenyl)-1,3-butanedione, 319
[82938-20-3]	1-(4-Methoxyphenyl)-3-methyl-1-butanone, 175
[82944-57-8]	1-[4-(2-Acetoxyethyloxy)phenyl]-1-decanone, 871
[82961-09-9]	Methyl 4-(2,4,5-trimethoxyphenyl)-4-oxo-1-butanoate, 412
[83162-76-9]	13-Hydroxy-1-(2,4,6-Trihydroxyphenyl)-1-hexadecanone, 1039
[83162-77-0]	14-Hydroxy-1-(2,4,6-Trihydroxyphenyl)-1-hexadecanone, 1039

[83212-56-0]	1-(2,4,6-Trimethoxyphenyl)-12,14-dimethoxyoctadecanone, 1069
[83212-66-2]	1-(2,4,6-Trihydroxyphenyl)-12,14-dihydroxyoctadecanone, 1069
[83213-39-2]	1-(2,4,6-Trihydroxyphenyl)-12,14-dihydroxyoctadecanone, 1069
[83258-17-7]	1-(3'-Bromo-4'-methoxy[1,1'-biphenyl]-4-yl)-1-hexanone, 678
[83481-33-8]	4-(2-Hydroxy-3,4-dimethylphenyl)-4-oxo-1-butanoic acid, 443
[83671-25-4]	1-(2,4-Dihydroxyphenyl)-2-ethyl-1-hexanone, 618
[83805-59-8]	1-(3,4-Dihydro-7-hydroxy-2,2-dimethyl-2H-1-benzopyran-6-yl)-
	1,3-butanedione, 332
[83805-61-2]	1-[2-Hydroxy-6-methoxy-4-(phenylmethoxy)phenyl]-1,3-butanedione, 333
[83805-67-8]	1-(3,4-Dihydro-7-hydroxy-5-methoxy-2,2-dimethyl-2 <i>H</i> -1-benzopyran-6-yl)- 1,3-butanedione, 332
[83882-87-5]	5-Chloro-1-(4-hydroxyphenyl)-1-pentanone, 568
[83882-88-6]	5-Chloro-1-(4-difluoromethyloxyphenyl)-1-pentanone, 568
[83893-20-3]	1-(2,5-Dimethoxy-4-methylphenyl)-1-butanone, 54
[83893-22-5]	1-(2,5-Dimethoxy-4-methylphenyl)-1-pentanone, 496
[83893-24-7]	1-(2,5-Dimethoxy-4-methylphenyl)-1-hexanone, 641
[84498-20-4]	1-(2-Hydroxy-4-methylphenyl)-1-octanone (Oxime), 792
[84498-21-5]	1-(4-Hydroxyphenyl)-1-octanone (Oxime), 775
[84633-05-6]	1-[3,5-Bis[[2,4,6-trihydroxy-3-methyl-5-(1-oxobutyl)phenyl]methyl]-
	2,4,6-trihydroxyphenyl]-1-butanone, 347
[84633-06-7]	1-[3-(5-methyl-3-propionyl-2,4,6-trihydroxyphenylmethyl)-
	2,4,6-trihydroxy-5-methylphenyl]-1-butanone, 336
[84633-27-2]	1-(2,4,6-Trihydroxy-3,5-dimethylphenyl)-1-butanone, 77
[84836-32-8]	2-Ethyl-1-(4-methoxyphenyl)-1-butanone, 170
[84978-10-9]	22-(2-Methoxy-3,4-dimethoxy-6-methylphenyl)-22-oxo-1-docosanoic acid, 1108
[84978-12-1]	4-(2-Hydroxy-3,4,6-trimethylphenyl)-4-oxo-1-butanoic acid, 447
[84978-13-2]	5-(2-Hydroxy-3,4,6-trimethylphenyl)-5-oxo-1-pentanoic acid, 594
[84978-14-3]	1,5-Bis(2-hydroxy-3,4,6-trimethylphenyl)-1,5-pentanedione, 538
[84978-15-4]	1,6-Bis(2-hydroxy-3,4,6-trimethylphenyl)-1,6-hexanedione, 693
[84978-16-5]	1,10-Bis(2-hydroxy-3,4,6-trimethylphenyl)-1,10-decanedione, 915
[84978-19-8]	7-(2-Hydroxy-3,4,5-trimethylphenyl)-7-oxo-1-heptanoic acid, 770
[84978-21-2]	10-(2-Hydroxy-3,4,5-trimethylphenyl)-10-oxo-1-decanone, 922
[84978-22-3]	1,10-Bis(2-hydroxy-3,4,5-trimethylphenyl)-1,10-decanedione, 915
[85052-18-2]	1-(5-Chloro-2-hydroxyphenyl)-1-heptanone, 734
[85052-25-1]	1-(5-Chloro-2-hydroxy-3-nitrophenyl)-1-heptanone, 732
[85052-34-2]	1-[5-(1,1-Dimethylethyl)-2-hydroxyphenyl]-1-heptanone, 749
[85052-35-3]	1-[5-(1,1-Dimethylethyl)-2-hydroxy-3-nitrophenyl]-1-heptanone, 748
[85052-45-5]	1-(3-Amino-5-chloro-2-hydroxyphenyl)-1-heptanone (Hydrochloride), 735
[85052-49-9]	1-[3-Amino-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-hexanone (Hydrochloride), 667
[85052-50-2]	1-[3-Amino-5-(1,1-dimethylethyl)-2-hydroxyphenyl]-1-hexanone, 667
[85052-73-9]	1-(3-Amino-5-chloro-2-hydroxyphenyl)-1-heptanone, 735
[85157-92-2]	1-(4-Methoxyphenyl)-3,3-dimethyl-1-butanone, 251
[85298-88-0]	1-(2,6-Dihydroxyphenyl)-1-undecanone, 926
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[85298-89-1]	1-(3-Chloro-2,6-dihydroxyphenyl)-1-undecanone, 929
[85298-90-4]	1-(3,5-Dihydroxyphenyl)-1-undecanone, 927
[85298-94-8]	1-(3,5-Dichloro-2,6-dihydroxyphenyl)-1-undecanone, 929
[85298-95-9]	1-(2,6-Dimethoxyphenyl)-1-undecanone, 926
[85485-53-6]	4-Chloro-1-(4-fluoro-2-hydroxyphenyl)-1-butanone (Oxime) (1E), 278
[85602-20-6]	1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-nonanone, 849
[85602-21-7]	1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-octanone, 809
[85602-25-1]	4-Methyl-1-[2,4,6-trihydroxy-3-(2-propenyl)phenyl]-1-pentanone, 549
[85602-31-9]	4-Methyl-1-[2,4,6-trihydroxy-3-(phenylmethyl)phenyl]-1-pentanone, 551
[85602-33-1]	1-[2,4,6-Trihydroxy-3-(phenylmethyl)phenyl]-1-nonanone, 857
[85602-35-3]	4-Methyl-1-[2-hydroxy-4,6-dimethoxy-3-(3-methyl-2-buten-1-yl)phenyl]-
	1-pentanone, 551
[85602-40-0]	1-(3,4-Dihydro-5,7-dihydroxy-2H-1-benzopyran-6-yl)-1-octanone, 804
[85602-44-4]	4-Methyl-1-(2,4,6-trihydroxyphenyl)-1-pentanone (Monosodium salt), 546
[86360-63-6]	1-(2,4,6-Trimethoxyphenyl)-1-hexanone, 613
[87035-85-6]	2-Methyl-1-(2,4,6-trihydroxy-3,5-dimethylphenyl)-1-butanone, 136
[87035-88-9]	1-(4-Hydroxy-2,6-dimethoxy-3,5-dimethylphenyl)-2-methyl- 1-butanone, 140
[87338-25-8]	4-(3-Bromo-2-hydroxy-5-methoxyphenyl)-4-oxo-1-butanoic acid, 432
[87364-84-9]	4-(4,5-Dimethoxy-2-nitrophenyl)-4-oxo-1-butanoic acid, 427
[87364-85-0]	Ethyl 4-(2-amino-4,5-dimethoxyphenyl)-4-oxo-1-butanoate, 429
[87374-67-2]	Ethyl 4-(4,5-dimethoxy-2-nitrophenyl)-4-oxo-1-butanoate, 427
[87667-31-0]	1-(3,4,5-Trihydroxyphenyl)-1-nonadecanone, 1094
[87961-41-9]	5-(3,4-Methylenedioxyphenyl)-5-oxo-1-pentanoic acid, 582
[88167-05-9]	1,6-Bis(4-ethoxyphenyl)-1,6-hexanedione, 674
[88555-60-6]	1-(2-Hydroxy-4-methylphenyl)-5-methyl-1-hexanone, 650
[88555-61-7]	1-(2-Methoxy-4-methylphenyl)-5-methyl-1-hexanone, 650
[88559-37-9]	1-(8-Hydroxy-5-quinolinyl)-1-decanone, 889
[88559-38-0]	1-(8-Hydroxy-7-quinolinyl)-1-decanone, 890
[88559-39-1]	1-(5-Chloro-8-hydroxy-7-quinolinyl)-1-decanone, 889
[88559-43-7]	1-(8-Hydroxy-7-quinolinyl)-1-nonanone (Hydrazone), 846
[88559-44-8]	1-(8-Hydroxy-7-quinolinyl)-1-decanone (Hydrazone), 890
[88559-45-9]	1-(5-Chloro-8-hydroxy-7-quinolinyl)-1-decanone (Hydrazone), 889
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[88858-34-8]	1-(4-Ethoxyphenyl)-1-butanone (Semicarbazone), 8
[88924-67-8]	1-(2,4-Dihydroxy-3,5-dimethylphenyl)-1-hexanone, 651
[89647-61-0]	1-(2,6-Dihydroxy-4-methoxy-3-methylphenyl)-2-methyl-1-butanone, 135
[90033-66-2]	1-(2-Amino-5-methoxyphenyl)-1-pentanone, 486
[90269-45-7]	1-[2-Methyl-(4-phenoxyphenyl)-1-butanone, 127
[90269-46-8]	1-(4-Methoxyphenyl)-2-methyl-1-butanone, 126
[90834-05-2]	1-(2,4,5-Trimethoxyphenyl)-1-pentanone, 476
[90834-06-3]	1-(2,4,5-Trimethoxyphenyl)-1-hexanone, 612
[90834-07-4]	2-Bromo-1-(2,4,5-trimethoxyphenyl)-1-butanone, 271
[90834-08-5]	2-Bromo-1-(2,4,5-trimethoxyphenyl)-1-pentanone, 566
[90834-09-6]	2-Bromo-1-(2,4,5-trimethoxyphenyl)-1-hexanone, 700

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[90841-48-8]	2-Bromo-1-(3-hydroxyphenyl)-1-butanone, 269
[90919-46-3]	1-(5-Chloro-2,4-dihydroxyphenyl)-1-butanone, 31
[90922-78-4]	1-(2-Hydroxy-4-nitrophenyl)-1-butanone, 34
[91065-87-1]	2-Bromo-1-(4-methoxyphenyl)-1,3-butanedione, 308
[91142-94-8]	1-(4-Hydroxyphenyl)-2-methyl-1,3-butanedione, 314
[91143-26-9]	1-(4-Hydroxyphenyl)-1,3-pentanedione, 460
[91335-45-4]	2-Bromo-1-(3-methoxyphenyl)-1-butanone, 269
[91453-24-6]	1,5-Bis(2,5-dihydroxyphenyl)-1,5-pentanedione, 520
[91453-25-7]	1,5-Bis(2,5-dihydroxy-4-methylphenyl)-1,5-pentanedione, 533
[91453-26-8]	1,6-Bis(2,5-dihydroxyphenyl)-1,6-hexanedione, 675
[91453-27-9]	1,6-Bis(2,5-dihydroxy-4-methylphenyl)-1,6-hexanedione, 687
[91497-29-9]	1-[4-(Acetyloxy)-2-hydroxyphenyl]-1-butanone, 61
[91497-61-9]	4-(2-Methoxy-4-methylphenyl)-4-oxo-1-butanoic acid, 433
[91541-03-6]	4-(4-Hydroxy-3-methylphenyl)-4-oxo-1-butanoic acid, 436
[91555-33-8]	1-(2,6-Dihydroxy-4-methoxyphenyl)-3-methyl-1-butanone, 194
[91555-34-9]	1-(2-Hydroxy-4,5-dimethoxyphenyl)-1-butanone, 74
[91646-56-9]	1-(4-Methoxyphenyl)-1-butanone (Semicarbazone), 7
[91667-38-8]	1-(2-Hydroxy-4,5-dimethylphenyl)-1-butanone, 66
[91767-62-3]	4-Chloro-1-(2,5-dimethoxyphenyl)-1-butanone, 282
[91963-55-2]	1-(4-Acetyloxyphenyl)-1,3-butanedione, 311
[91964-06-6]	1-(4-Carboxy-3-methoxyphenyl)-4-oxo-1-butanoic acid, 431
[91970-63-7]	1-(2,3-Dihydroxy-5-methylphenyl)-1-butanone, 53
[91970-65-9]	1-(3-Hydroxy-4-methoxyphenyl)-1-butanone, 55
[91992-00-6]	1-(2-Hydroxy-3-nitrophenyl)-1-butanone, 34
[92016-84-7]	1-(4-Hydroxy-3-methylphenyl)-1,3-butanedione, 318
[92017-91-9]	Methyl 4-(4-methoxyphenyl)-2,3-dibromo-4-oxo-1-butanoate, 397
[92019-26-6]	1-(2-Chloro-4-hydroxy-3-methylphenyl)-2-ethyl-1-butanone, 172
[92019-28-8]	4-Chloro-1-(4-propoxyphenyl)-1-butanone, 281
[92019-50-6]	4-Chloro-1-(2,3,4-trimethoxyphenyl)-1-butanone, 283
[92035-99-9]	1-(4-Ethoxyphenyl)-3-methyl-1-butanone, 176
[92050-07-2]	1-(4-Methoxyphenyl)-4-methyl-4-nitro-1-pentanone, 541
[92050-08-3]	1-(4-Methoxyphenyl)-4-methyl-4-nitro-2-sulfinyl-1-pentanone, 541
[92120-37-1]	1-(4-Methoxyphenyl)-4-methyl-1,3,5-hexanetrione, 614
[92120-60-0]	1-(2,4-Dimethoxy-6-methylphenyl)-1-pentanone (D), 495
[92120-78-0]	1-[2,4-Dimethoxy-6-[(trimethylsilyl)methyl]phenyl]-1-pentanone, 510
[92300-78-2]	1-(4-Methoxyphenyl)-5-methyl-1-hexanone, 616
[92301-09-2]	1-(3,5-Dimethyl-4-hydroxyphenyl)-2-methyl-1-pentanone, 561
[92317-86-7]	1-(4-Butoxy-4-chlorophenyl)-1-butanone, 281
[92422-41-8]	1-(4-Amino-3-methoxyphenyl)-4-oxo-1-butanoic acid, 428
[92532-18-8]	1-(3-Benzyloxyphenyl)-1-hexanone, 601
[92655-68-0]	1-(3-Butyryloxy-4-methoxyphenyl)-1-butanone, 56
[92730-14-8]	1-(3,4-Dimethoxyphenyl)-2-ethyl-3-methyl-1-butanone, 185
[92730-24-0]	1-(2-Hydroxy-4-propoxyphenyl)-1-hexanone, 659
[92755-95-8]	1-(2-Hydroxy-4-methoxy-3-methylphenyl)-1-butanone, 71

[92755-96-9]	1-(2-Hydroxy-4-methoxy-6-methylphenyl)-1-butanone, 71
[92757-66-9]	1-(5-Acetyl-2-hydroxyphenyl)-1-butanone, 377
[92757-67-0]	1-(5-Acetyl-2-hydroxyphenyl)-1-hexanone, 646
[92844-55-4]	5,7-Dihydroxy-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxobutyl)-
	4-(1-methylpropyl)-2H-1-benzopyran-2-one $(R,R)$ , 156
[92865-60-6]	5-(2,4,5-Trimethoxyphenyl)-5-oxo-1-pentanoic acid, 583
[92907-10-3]	1-(5-Chloro-2,4-dihydroxyphenyl)-1-butanone
	(2,4-Dinitrophenylhydrazone), 31
[93041-47-5]	1-(4-Cyano-3-methoxyphenyl)-4-oxo-1-butanoic acid, 430
[93156-87-7]	1-(4-Pentyloxyphenyl)-1-pentanone, 468
[93157-10-9]	1-(3,4-Dimethoxyphenyl)-1-octanone, 782
[93175-38-3]	2-Bromo-1-(2,5-dimethoxyphenyl)-1-butanone, 271
[93249-83-3]	1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-1-decanone, 904
[93249-88-8]	1-(4'-Acetyloxy[1,1'-biphenyl]-4-yl)-1-decanone, 905
[93429-81-3]	1-(2-Hydroxy-5-methylphenyl)-4-methyl-1-pentanone, 547
[93534-71-5]	4-(3-Methoxy-4-nitrophenyl)-4-oxo-1-butanoic acid, 426
[93542-23-5]	1-(4-Hexyloxyphenyl)-1-pentanone, 468
[93542-41-7]	1-[2-Hydroxy-4-(isopentyloxy)phenyl]-1-hexanone, 670
[93650-62-5]	1-(2-Hydroxyphenyl)-3-methyl-1-butanone
	(2,4-Dinitrophenylhydrazone), 174
[93762-01-7]	1-(4-Methoxyphenyl)-5-methyl-1-hexanone (Semicarbazone), 616
[93796-34-0]	3-Methyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)-5-(3-methylbutyl)
	phenyl]-1-butanone, 233
[93970-90-2]	1-(3,4-Dihydro-7,8-dihydroxy-2,2-dimethyl-2 <i>H</i> -1-benzopyran-6-yl)- 1-butanone, 97
[93970-91-3]	1-(3,4-Dihydro-5-hydroxy-2,2-dimethyl-2H-1-benzopyran-6-yl)-
	1-butanone, 96
[93970-92-4]	1-(3,4-Dihydro-7-hydroxy-2,2-dimethyl-2 <i>H</i> -1-benzopyran-6-yl)-
	1-butanone, 96
[93970-93-5]	1-(2,4-Dihydroxy-3-methylphenyl)-1-butanone, 53
[93970-94-6]	1-(3,4-Dihydro-7-hydroxy-2,2,8-trimethyl-2 <i>H</i> -1-benzopyran-6-yl)-
[93970-95-7]	1-butanone, 106       1-(3,4-Dihydro-7-hydroxy-8-methoxy-2,2-dimethyl-2H-1-benzopyran-6-yl)-
[93970-93-7]	1-butanone, 107
[94119-33-2]	5-(2,5-Diethoxyphenyl)-5-oxo-1-pentanoic acid, 582
[94413-27-1]	1-[5-Acetyl-2-hydroxy-3-(1-hydroxy-3-methyl-2-butenyl)phenyl]-
[] [] [] [] [] [] [] [] [] [] [] [] [] [	3-methyl-1-butanone, 381
[94413-28-2]	1,1'-[5-Acetyl-2-hydroxy-1,3-phenylene]bis-3-methyl-1-butanone, 382
[94432-99-2]	1-(2,4-Dihydroxy-3-quinolinyl)-1-decanone, 890
[94613-09-9]	1-[2-Hydroxy-5-(1,1,3,3-tetramethylbutyl)phenyl]-1-decanone (Oxime), 911
[94613-10-2]	1-[2-Methoxy-5-(1,1,3,3-tetramethylbutyl)phenyl]-1-decanone
	(Oxime), 912
[94708-70-0]	1-(2-Hydroxy-4-nitrophenyl)-1-butanone (Phenylhydrazone), 35
[94711-66-7]	1-(2,4-Dihydroxyphenyl)-3-methyl-1-butanone
	(2,4-Dinitrophenylhydrazone), 178
[94759-02-1]	1-(2,4-Dihydroxyphenyl)-1-octanone (2,4-Dinitrophenylhydrazone), 779
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[94899-67-9]	1-[2-Methoxy-5-(1,1,3,3-tetramethylbutyl)phenyl]-1-decanone, 911
[94899-68-0]	1-[2-Hydroxy-5-(1,1,3,3-tetramethylbutyl)phenyl]-1-decanone, 911
[94960-09-5]	4-(4-Phenoxyphenyl)-4-oxo-1-butanoic acid (Semicarbazone), 402
[95002-59-8]	1,5-Bis(2,4-dimethoxyphenyl)-1,5-pentanedione, 520
[95102-14-0]	1-(2,4-Dihydroxy-5-methylphenyl)-1-dodecanone, 961
[95102-15-1]	1-(4-Hydroxy-3-methylphenyl)-1-tetracosanone, 1114
[95102-16-2]	1-(2,4-Dihydroxy-5-methylphenyl)-1-tetradecanone, 1012
[95102-17-3]	1-(5-Ethyl-2,4-dihydroxyphenyl)-1-hexanone, 652
[95102-18-4]	1-(4-Hydroxy-3-(1-methylethyl)phenyl)-1-heptanone, 747
[95102-20-8]	1-(4-Hydroxy-2,3-dimethylphenyl)-1-octanone, 800
[95102-21-9]	1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-octanone, 807
[95102-22-0]	1-(2-Hydroxy[1,1'-biphenyl]-5-yl)-1-octanone, 814
[95102-23-1]	1-(4-Hydroxy-2,5-dimethylphenyl)-1-decanone, 888
[95102-27-5]	1-(4-Hydroxy-3-methylphenyl)-1-heptanone, 739
[95102-30-0]	1-(4-Hydroxy-2,5-dimethylphenyl)-1-hexanone, 651
[95102-32-2]	1-[3-(1,1-Dimethylethyl)-4-hydroxyphenyl]-1-octanone, 806
[95102-33-3]	1-[1,1'-Biphenyl]-5-yl-2-hydroxy-1-pentanone, 524
[95102-34-4]	1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-hexanone, 665
[95102-35-5]	1-(6-Hydroxy[1,1'-biphenyl]-3-yl)-1-heptanone, 758
[95102-36-6]	1-[4-Hydroxy-3-(1-methylethyl)phenyl]-1-hexanone, 658
[95102-37-7]	1-[4-Hydroxy-3-(1-methylethyl)phenyl]-1-pentanone, 505
[95102-38-8]	1-(4-Hydroxy-2,5-dimethylphenyl)-1-nonanone, 845
[95102-39-9]	1-(4-Hydroxy-2,5-dimethylphenyl)-1-heptanone, 744
[95102-41-3]	1-(4-Hydroxy-3-methylphenyl)-1-octanone, 793
[95102-42-4]	1-(6-Hydroxy[1,1'-biphenyl]-3-yl)-1-hexanone, 680
[95149-08-9]	1-(2,4-Dihydroxy-3-methylphenyl)-1-octanone, 794
[95185-58-3]	1-(2,4-Dihydroxy-5-methylphenyl)-1-octanone, 795
[95185-59-4]	1-(5-Ethyl-2,4-dihydroxyphenyl)-1-octanone, 802
[95185-60-7]	1-(2,4-Dihydroxy-5-methylphenyl)-1-octadecanone, 1076
[95185-61-8]	1-(4-Hydroxy-2,5-dimethylphenyl)-1-docosanone, 1107
[95185-62-9]	1,1'-(2-Hydroxy[1,1'-biphenyl]-5-yl)-1-butanone, 260
[95185-63-0]	1-[3-(1,1-Dimethylethyl)-4-hydroxyphenyl]-1-pentanone, 508
[95185-65-2]	1-[3-(1,1-Dimethylethyl)-4-hydroxyphenyl]-1-heptanone, 749
[95185-66-3]	1-(3-Ethyl-4-hydroxyphenyl)-1-heptanone, 742
[95185-67-4]	1-(4-Hydroxy-2,5-dimethylphenyl)-1-octanone, 800
[95185-68-5]	1-(4-Hydroxy-2,5-dimethylphenyl)-1-hexadecanone, 1043
[95185-69-6]	1-(4-Hydroxy-2,5-dimethylphenyl)-1-octadecanone, 1077
[95185-70-9]	1-(4-Hydroxy-3-methylphenyl)-1-docosanone, 1107
[95185-72-1]	1-[4-Hydroxy-2-methyl-5-(1-methylethyl)phenyl]-1-butanone, 91
[95185-73-2]	1-[4-Hydroxy-3-(1-methylethyl)phenyl]-3-methyl-1-butanone, 206
[95269-86-6]	5-(1-Octadecanoyl)-2-hydroxybenzoic acid, 1074
[95269-91-3]	5-(1-Octadecanoyl)-2-hydroxybenzoic acid (Na salt), 1074
[95269-96-8]	5-(1-Octadecanoyl)-2-hydroxybenzoic acid (Tris(2-hydroxyethyl)amine salt), 1075

[95269-97-9]	5-(1-Octadecanoyl)-2-hydroxybenzoic acid (N-methylmorpholine salt), 1075
[95282-26-1]	1-(2,4-Dihydroxyphenyl)-1-nonanone (2,4-Dinitrophenylhydrazone), 838
[95302-59-3]	5-(1-Octadecanoyl)-2-hydroxybenzoic acid (Lysine salt), 1075
[95807-67-3]	1-(2,5-Dihydroxyphenyl)-1-hexadecanone, 1036
[95809-40-8]	1-(2,4-Dihydroxyphenyl)-1-decanone (2,4-Dinitrophenylhydrazone), 872
[95869-30-0]	1-(4-Methoxyphenyl)-1-octadecanone, 1064
[95869-36-6]	1-(2-Hydroxy-4-methoxyphenyl)-1-octadecanone, 1077
[95958-93-3]	1-(2,4-Dihydroxyphenyl)-1-undecanone (2,4-Dinitrophenylhydrazone), 925
[96070-21-2]	1-(2,3,4-Trihydroxyphenyl)-1-eicosanone, 1098
[96123-20-5]	1-(4'-Octyloxy[1,1-biphenyl]-4-yl)-3-methyl-1-pentanone, 563
[96251-00-2]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1,2-hexanedione, 688
[96251-01-3]	1-[3,5-(1,1-Dimethylethyl)-4-hydroxyphenyl]-3,3-dimethyl- 1,2-butanedione, 308
[96271-43-1]	1,5-Bis(5-bromo-2,4-dihydroxyphenyl)-2,4-diphenyl-1,5-pentanedione, 539
[96271-44-2]	1,5-Bis(2,4-dihydroxyphenyl)-2,4-di-(4-bromophenyl)- 1,5-pentanedione, 539
[96271-50-0]	1,5-Bis(5-chloro-2,4-dihydroxyphenyl)-2,4-diphenyl-1,5-pentanedione, 539
[96273-02-8]	1,5-Bis(2,4-dihydroxyphenyl)-1,5-pentanedione (Di-2,4-Dinitrophenyl- hydrazone), 520
[96273-22-2]	1,5-Bis(2,4-dihydroxyphenyl)-2,4-diphenyl-1,5-pentanedione, 540
[96358-74-6]	4-(2-Hydroxy-4,6-dimethylphenyl)-4-oxo-1-butanoic acid, 444
[96573-30-7]	2,4,6-Trihydroxy-3-(1-oxobutyl)benzaldehyde, 387
[96573-31-8]	2,4,6-Trihydroxy-3-(1-oxopentyl)benzaldehyde, 490
[96573-32-9]	2,4,6-Trihydroxy-3-(1-oxohexyl)benzaldehyde, 634
[96573-33-0]	2,4,6-Trihydroxy-3-(1-oxoheptyl)benzaldehyde, 737
[96573-34-1]	3-Ethyl-2,4,6-trihydroxy-5-(3-methyl-1-oxobutyl)benzaldehyde, 391
[96573-35-2]	2,4,6-Trihydroxy-3-(3-methyl-1-oxobutyl)-5-propylbenzaldehyde, 392
[96573-36-3]	3-Butyl-2,4,6-trihydroxy-5-(3-methyl-1-oxobutyl)benzaldehyde, 392
[96573-40-9]	1-[2,4,6-Trihydroxy-3-(1-oxopropyl)phenyl]-1-butanone, 385
[96573-43-2]	2,4,6-Trihydroxy-5-(3-methyl-1-oxobutyl)- 1,3-benzenedicarboxaldehyde, 388
[96590-58-8]	1,5-Bis(2,4-dihydroxyphenyl)-2,4-di-(4-methoxyphenyl)- 1,5-pentanedione, 540
[96676-42-5]	1,5-Bis(2,4-dimethoxyphenyl)-2,4-diphenyl-1,5-pentanedione, 540
[96710-34-8]	1,5-Bis(5-bromo-2,4-diacetyloxyphenyl)-2,4-diphenyl- 1,5-pentanedione, 539
[96710-35-9]	1,5-Bis(5-chloro-2,4-diacetyloxyphenyl)-2,4-diphenyl- 1,5-pentanedione, 539
[96809-09-5]	1,5-Bis(5-chloro-2,4-dihydroxypheny)l-1,5-pentanedione (Di-2,4-Dinitro- phenylhydrazone), 516
[96820-25-6]	1-(2,6-Dihydroxyphenyl)-1-hexadecanone, 1037
[96853-73-5]	3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-
_	2,4,6-trihydroxy-5-[(3,3-dimethylallyl)phenyl]-1-butanone, 343
[96966-45-9]	1-(3,4-Dimethoxyphenyl)-1-dodecanone, 949
[96968-05-7]	1-(2,4-Diacetyloxyphenyl)-1-hexadecanone, 1035

[97023-54-6]	1-[4-Hydroxy-3-(2-propenyl)phenyl]-1-butanone, 80
[97037-81-5]	1-(2-Acetyloxyphenyl)-1-pentanone, 463
[97153-67-8]	1-(3,4-Dihydroxyphenyl)-1-dodecanone (2,4-Dinitrophenylhydrazone), 948
[97271-30-2]	5-(2,4-Dibenzyloxy-6-methylphenyl)-3,5-dioxo-1-pentanoic acid, 589
[97582-34-8]	1-[4-[(Dimethylamino)methyl]-2-hydroxy-3-propylphenyl]-
	1-pentanone, 528
[97582-39-3]	1-[4-(Chloromethyl)-2-hydroxy-3-propylphenyl]-1-pentanone, 507
[97744-24-6]	2-Bromo-3,5,5-trimethyl-1-(4-pentyloxyphenyl)-1-hexanone, 707
[97744-27-9]	2-Bromo-1-(4-tert-butyloxyphenyl)-1-heptanone, 764
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[100792-79-8]	1-(3-Chloro-6-hydroxy-2,4-dimethylphenyl)-1-hexanone, 647
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[101430-19-7]	1-(3,5-Diacetyloxyphenyl)-1-hexanone, 610
[101430-33-5]	1-(3,5-Diacetyloxyphenyl)-4-methyl-1-pentanone, 545
[101499-61-0]	4-[4-Methoxy-2-methyl-5-(1-methylethyl)phenyl]-4-oxo-1-butanoic acid, 450
[101499-71-2]	Ethyl 5-(3,4-dimethoxyphenyl)-5-oxo-1-pentanoate, 582
[101577-83-7]	1-[3-Bromo-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-heptanone, 748
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[101593-70-8]	1-(3,5-Dihydroxyphenyl)-1-pentanone (2,4-Dinitrophenylhydrazone), 474
[101597-59-5]	1-(2-Hydroxyphenyl)-5-(4-hydroxyphenyl)-1,5-pentanedione, 519
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[101684-68-8]	1,5-Bis(4-propyloxyphenyl)-1,5-pentanedione, 519
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[101737-37-5]	1,4-Bis(5-methoxy-2-nitrophenyl)-2-bromo-1,4-butanedione, 355
[101741-01-9]	1-(4-Methoxyphenyl)-1-decanone, 870
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[101741-97-3]	1-(3,5-Dihydroxyphenyl)-4-methyl-1-pentanone (2,4-Dinitrophenylhydrazone), 545
[101777-85-9]	1-(4-Methoxyphenyl)-1-hexanone (Semicarbazone), 604
[101778-04-5]	1-(3,4-Dimethoxyphenyl)-3-methyl-1-butanone (Semicarbazone), 180
[101778-12-5]	1-(3,4-Dimethoxyphenyl)-1-pentanone (Semicarbazone), 473
[101784-96-7]	1-(2-Hydroxy-4-methylphenyl)-3-methyl-1-butanone (Phenylhydrazone), 190
[101789-78-0]	1-(5-Benzoyl-2-hydroxy-3-methylphenyl)-1-butanone, 386
[101790-34-5]	1-(2-Hydroxyphenyl)-6-(4-hydroxyphenyl)-1,6-hexanedione, 674
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[102003-68-9]	1-(3-Chloro-6-hydroxy-2,4-dimethylphenyl)-1-heptanone, 741
[102003-73-6]	1-[3-Chloro-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-pentanone, 507
[102011-49-4]	1-(4-Hydroxyphenyl)-1-heptanone (Nicotinylhydrazone), 722
[102020-37-1]	1-[6-Methoxy-2-methyl-3-(1-methylethyl)phenyl]-1-octanone, 807
[102020-42-8]	1-(4-Heptyl-2-hydroxy-5-methoxyphenyl)-3-methyl-1-butanone, 228
[102075-11-6]	1-(3-Chloro-6-allyloxy-2,4-dimethylphenyl)-1-pentanone, 500
[102158-27-0]	1-(3,5-Diacetyloxyphenyl)-1-butanone (2,4-Dinitrophenylhydrazone), 15
[102159-08-0]	1-(2-Acetyloxy-5-benzoyl-3-methylphenyl)-1-butanone, 386
[102161-19-3]	1-(4-Methoxyphenyl)-1-hexanone (2,4-Dinitrophenylhydrazone), 604
[102161-23-9]	1-(3,5-Dihydroxyphenyl)-1-heptanone (2,4-Dinitrophenylhydrazone), 728
[102166-29-0]	13-(2-Hydroxyphenyl)-13-oxo-1-tridecanoic acid, 1001
[102172-04-3]	1-(2,4-Dimethoxy-6-methylphenyl)-4-phenyl-1,3-butanedione, 355
[102222-55-9]	Ethyl 4-(2,3,4-trimethoxyphenyl)-4-oxo-1-butanoate, 411
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[102458-53-7]	3-Methyl-1-[2,4,5-trimethoxyphenyl]-1-butanone
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[102475-97-8]	1-[3-Chloro-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-hexanone (2,4-Dinitrophenylhydrazone), 664
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[104516-35-0]	1-(2-Hydroxy-5-methylphenyl)-1,3-pentanedione, 488
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[104967-09-1]	10-(Acetyloxy)-1-(2,3-dihydroxy-4-methoxy-6-methylphenyl)-
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[105337-84-6]	1-(2-Hydroxy-3-methylphenyl)-3-methyl-1-butanone, 189
[105401-56-7]	1-(3,5-Dihydroxyphenyl)-1-hexanone, 610
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[105475-57-8]	1-(5-Hydroxy-2,4-dimethoxyphenyl)-1-butanone, 76
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[106321-41-9]	1-[3-Chloro-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-butanone, 89
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[107259-37-0]	1,10-Bis(4-hydroxy-2,6-dimethylphenyl)-1,10-decanedione, 913
[107276-31-3]	1-(2,4-Dihydroxy-3-quinolinyl)-1-octanone, 803
[107327-65-1]	4-(5-Ethoxy-2-methoxyphenyl)-4-oxo-1-butanoic acid, 407
[107327-72-0]	4-(2-Ethoxy-5-methoxyphenyl)-4-oxo-1-butanoic acid, 407
[107522-52-1]	1,1'-(4,6-Dihydroxy-5-nitro-1,3-phenylene)bis-1-butanone, 297

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[107771-42-6]	1-(2-Hydroxy-3,6-dimethylphenyl)-1-hexanone, 649
[107772-24-7]	1-(2-Hydroxy-3,5-dimethylphenyl)-1-hexanone, 649
[107778-10-9]	1-(3,4-Dimethoxyphenyl)-1-octanone (Semicarbazone), 782
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[108111-24-6]	1-[2-Hydroxy-5-(1,1,3,3-tetramethylbutyl)phenyl]-1-dodecanone (Oxime, nickel complex), 982
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[108300-00-1]	1-[3-Chloro-2-hydroxy-5-(1,1-dimethylethyl)phenyl]-1-octanone, 806
[108401-78-1]	1-(2,3,4-Trimethoxyphenyl)-1-butanone, 16
[108515-73-7]	6-(2,5-Dihydroxyphenyl)-6-oxo-1-hexanoic acid, 712
[108623-73-0]	1-(2-Methoxy-4,5-dimethylphenyl)-1-decanone, 887
[108666-97-3]	1-(2-Hydroxy-4-methylphenyl)-1-octanone, 792
[108667-53-4]	1-(2-Hydroxy-3-methylphenyl)-1-octanone, 791
[108715-26-0]	1-(3,5-Diacetyloxyphenyl)-3-methyl-1-butanone, 180
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[116956-62-8]	1-[2,4-Dihydroxy-5-(sulfooxy)phenyl]-1-butanone (K salt), 38
[116979-51-2]	1-[6-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-
	5,7-dihydroxy-2,2-dimethyl-2 <i>H</i> -1-benzopyran-8-yl]-1-butanone, 343

[117271-96-2]	1,18-Bis-(4-hydroxyphenyl)-1,18-octadecanedione (Di-2,4-dinitrophenyl-hydrazone), 1083
[117272-01-2]	1,18-Bis-(2-hydroxyphenyl)-1,18-octadecanedione (Di-2,4-dinitrophenyl- hydrazone), 1083
[117285-75-3]	Methyl 3-(2-hydroxy-5-isovaleroyl-4-methoxyphenyl)propanoate, 216
[117285-87-7]	3,4-Dihydro-7-methoxy-6-(3-methyl-1-oxobutyl)-2 <i>H</i> -1-benzopyran- 2-one, 204
[117482-22-1]	2,2,3,3,4,4,4-Heptafluoro-1-(4-methoxyphenyl)-1-butanone, 289
[117692-92-9]	1-(4-Benzyloxyphenyl)-2,6-dimethyl-1-heptanone, 731
[117692-93-0]	1-(4-Hydroxyphenyl)-2,6-dimethyl-1-heptanone, 731
[117693-01-3]	1-(4'-Benzyloxy[1,1'-bipheny]]-4-yl)-2,6-dimethyl-1-heptanone, 762
[117693-02-4]	1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-2,6-dimethyl-1-heptanone, 762
[117706-06-6]	5-[5-Hydroxy-4-(1-oxodecyl)-2-(2-propenyl)phenoxy]pentanenitrile, 911
[118018-78-4]	6-Bromo-1-(2,4-dimethoxyphenyl)-1-hexanone, 699
[118108-79-5]	6-Bromo-1-(2-chloro-4-methoxyphenyl)-1-hexanone, 702
[118191-27-8]	1-(3,5-Dichloro-2,4-dihydroxy-6-methoxyphenyl)-1-pentanone, 487
[118191-28-9]	1-(3,5-Dichloro-2,4-dihydroxy-6-methoxyphenyl)-1-hexanone, 631
[118191-29-0]	1-(3,5-Dibromo-2,4-dihydroxy-6-methoxyphenyl)-1-hexanone, 630
[118191-30-3]	1-(3,5-Dibromo-2,6-dihydroxy-4-methoxyphenyl)-1-hexanone, 630
[118191-31-4]	1-(3,5-Dichloro-2,4-dihydroxy-6-methylphenyl)-1-hexanone, 630
[118191-32-5]	1-(3,5-Dichloro-2,4-dihydroxy-6-methoxyphenyl)-1-heptanone, 735
[118191-33-6]	1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-1-heptanone, 736
[118191-34-7]	1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-1-octanone, 789
[118191-35-8]	1-(3,5-Dichloro-2,4-dihydroxy-6-methoxyphenyl)-1-nonanone, 843
[118191-36-9]	1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-1-nonanone, 843
[118222-70-1]	1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-1-butanone, 42
[118222-71-2]	1-(3,5-Dichloro-2,4,6-trihydroxyphenyl)-1-hexanone, 622
[118222-72-3]	1-(3,5-Dichloro-2,4-dihydroxy-6-methoxyphenyl)-1-octanone, 788
[118469-84-4]	1-(2-Hydroxy-5-tetradecylphenyl)-1-hexanone, 695
[118469-92-4]	1-(2-Hydroxy-4-tetradecylphenyl)-1-hexanone, 694
[118476-18-9]	1-(2-Methoxyphenyl)-1-heptanone, 720
[118683-27-5]	4-[3-Hydroxy-4-(1-hexanoyl)-2-propylphenoxymethyl]- 3-methoxybenzoic acid, 693
[119039-35-9]	1,18-Bis-(4-hydroxy-3-methylphenyl)-1,18-octadecanedione, 1085
[119039-36-0]	1,18-Bis-(2-hydroxy-5-methylphenyl)-1,18-octadecanedione, 1085
[119039-37-1]	1,18-Bis-(4-hydroxy-2-methylphenyl)-1,18-octadecanedione, 1085
[119039-38-2]	1,18-Bis-(2-hydroxy-4-methylphenyl)-1,18-octadecanedione, 1085
[119039-49-5]	1,18-Bis-(2-hydroxy-3-methylphenyl)-1,18-octadecanedione, 1085
[119042-58-9]	5,7-Dimethoxy-6-(3-methyl-2-butenyl)-8-(3-methyl-1-oxobutyl)- 4-propyl-2 <i>H</i> -1-benzopyran-2-one, 237
[119348-65-1]	Ethyl 6-(4-hydroxyphenyl)-6-oxo-1-hexanoate, 712
[119348-66-2]	Ethyl 5-(4-hydroxyphenyl)-3,3-dimethyl-5-oxo-1-pentanoate, 585
[119531-08-7]	1-(4-Hydroxyphenyl)-1-tetradecanone (Myristate), 1005
[119691-93-9]	1-(2,4,6-Trihydroxy-3-nitrophenyl)-1-butanone, 37

[119691-95-1]	1-(2,4,6-Trihydroxy-3-nitrophenyl)-1-pentanone, 485
[119691-95-1]	1-(2,4,6-Trihydroxy-3-nitrophenyl)-1-pentanone, 483
[119691-97-3]	1-(2,4,6-Trihydroxy-3-nitrophenyl)-1-nexanone, 725
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	1-(2,4,6-Trihydroxy-3-nitro-5-propylphenyl)-1-butanone, 82
[119692-00-1]	3-Methyl-1-(2,4,6-trihydroxy-3-nitro-5-propylphenyl)-1-butanone, 206
[119692-01-2]	1-(2,4,6-Trihydroxy-3-nitro-5-propylphenyl)-1-pentanone, 505
[119692-02-3]	1-(2,4,6-Trihydroxy-3-nitro-5-propylphenyl)-1-hexanone, 657
[119748-12-8]	1-(4-Methoxyphenyl)-2-propyl-1-pentanone, 556
[119998-59-3]	1-[3-Acetyl-2,4,6-trihydroxy-5-(2-hydroxy-3-methyl-3-butenyl)phenyl]- 3-methyl-1-butanone, 383
[119998-60-6]	1-[5-Acetyl-2,3-dihydro-4,6-dihydroxy-2-(1-hydroxy-1-methylethyl)- 7-benzofuranyl]-3-methyl-1-butanone, 382
[119998-61-7]	1-[7-Acetyl-2,3-dihydro-4,6-dihydroxy-2-(1-hydroxy-1-methylethyl)- 5-benzofuranyl]-3-methyl-1-butanone, 382
[119998-63-9]	1-(8-Acetyl-3,4-dihydro-3,5,7-trihydroxy-2,2-dimethyl-2 <i>H</i> -1-benzopyran- 6-yl)-3-methyl-1-butanone, 383
[119998-64-0]	1-(6-Acetyl-3,4-dihydro-3,5,7-trihydroxy-2,2-dimethyl-2 <i>H</i> -1-benzopyran- 8-yl)-3-methyl-1-butanone, 383
[119998-65-1]	1-[7-Acetyl-2,3-dihydro-4,6-dihydroxy-2-(1-methylethenyl)- 5-benzofuranyl]-3-methyl-1-butanone, 380
[119998-66-2]	1-[5-Acetyl-2,3-dihydro-4,6-dihydroxy-2-(1-methylethenyl)- 7-benzofuranyl]-3-methyl-1-butanone, 381
[120058-71-1]	1-(3-Chloro-2,6-dihydroxy-5-propylphenyl)-1-butanone, 81
[120259-64-5]	1-(4-Fluoro-2-hydroxy-5-nitrophenyl)-1-butanone, 25
[120292-07-1]	1-(4-Fluoro-2-hydroxy-5-nitrophenyl)-3-methyl-1-butanone, 185
[120363-73-7]	5,7-Dimethoxy-6-(3-methylbutyl)-8-(3-methyl-1-oxobutyl)-4-propyl- 2 <i>H</i> -1-benzopyran-2-one, 239
[120363-74-8]	5,7-Diacetyloxy-6-(3-methylbutyl)-8-(3-methyl-1-oxobutyl)-4-propyl- 2 <i>H</i> -1-benzopyran-2-one, 239
[120363-75-9]	5,7-Dimethoxy-8-(3-methylbutyl)-6-(3-methyl-1-oxobutyl)-4-propyl- 2 <i>H</i> -1-benzopyran-2-one, 240
[120529-47-7]	1-(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)-1-pentanone, 491
[120716-96-3]	3-Methyl-1-(2,4,6-trihydroxy-3-methylphenyl)-1-pentanone (S), 559
[120716-97-4]	2,4,6-Trihydroxy-3-(3-methyl-1-oxopentyl)benzaldehyde (S), 558
[120836-88-2]	1-(4-Hydroxyphenyl)-2-methyl-1-butanone, 126
[120836-99-9]	1-(4-Hydroxyphenyl)-2-methyl-1-butanone (2S), 126
[120837-00-5]	1-(4-Hydroxyphenyl)-2-methyl-1-octanone (S), 794
[120837-01-6]	1-(4-Hydroxyphenyl)-2-methyl-1-decanone (S), 876
[120837-02-7]	1-(4-Hydroxyphenyl)-2-methyl-1-heptanone (+), 730
[120837-03-8]	1-(4-Hydroxyphenyl)-2-methyl-1-nonanone (+), 840
[120837-04-9]	2-Ethyl-1-(4-hydroxyphenyl)-1-octanone (+), 784
[120837-05-0]	1-(4-Hydroxyphenyl)-2-propyl-1-decanone (S), 876
[120837-08-3]	1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-2-methyl-1-butanone (S), 262
[120837-09-4]	1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-2-methyl-1-octanone (+), 817
[120837-13-0]	1-(4-Methoxyphenyl)-2-methyl-1-octanone (+), 794
[120837-31-2]	1-(4-Hydroxyphenyl)-2-methyl-1-nonanone, 840

[120857-41-2]	1-(4-Hydroxyphenyl)-1-hexadecanone (Palmitate), 1035
[121079-06-9]	4-Chloro-1-(2-hydroxy-4,6-dimethoxyphenyl)-1-butanone, 287
[121426-03-7]	3-Methyl-1-[2,4,6-tribenzoyloxy-3,5-bis(3-methyl-2-butenyl)phenyl]- 1-butanone, 232
[121586-48-9]	1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-1-nonanone, 853
[121586-49-0]	1-(4'-Acetyloxy[1,1'-biphenyl]-4-yl)-1-nonanone, 853
[121693-16-1]	1-[3-[(3-Acetyl-2,6-dihydroxy-4-methoxy-5-methylphenyl)methyl]- 5-[[2,6-dihydroxy-4-methoxy-3-methyl-5-(1-oxobutyl)phenyl]methyl]- 2,4,6-trihydroxyphenyl]-1-butanone, 347
[122214-67-9]	1-(4-Lauryloxyphenyl)-1-dodecanone, 943
[122427-50-3]	1,4-Bis(2-hydroxy-4-methylphenyl)-1,4-butanedione, 365
[122492-61-9]	1-(4-Stearoyloxyphenyl)-1-octadecanone, 1064
[122585-49-3]	1-[2,6-Dihydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-1-butanone, 97
[122585-50-6]	1-[2,6-Dihydroxy-4-[(4-hydroxy-3-methyl-2-butenyl)oxy]phenyl]- 1-butanone ( <i>E</i> ), 98
[122585-51-7]	1-[4-[[4-(Acetyloxy)-3-methyl-2-butenyl]oxy]-2,6-dihydroxyphenyl]- 1-butanone ( <i>E</i> ), 110
[122585-54-0]	1-[2,6-Dihydroxy-4-[(4-hydroxy-3-methyl-2-butenyl)oxy]phenyl]- 2-methyl-1-butanone ( <i>E</i> ), 143
[122585-55-1]	1-[4-[[(4-Acetyloxy)-3-methyl-2-butenyl]oxy]-2,6-dihydroxyphenyl]- 2-methyl-1-butanone ( <i>E</i> ), 146
[122585-56-2]	4-[3,5-Dihydroxy-4-(1-oxobutyl)phenoxy]-2-methyl-1-butanoic acid, 98
[122585-61-9]	1-[3-[4-(Acetyloxy)-3-methyl-2-butenyl]-2,4,6-trihydroxyphenyl]- 1-butanone ( <i>E</i> ), 110
[122585-96-0]	1-[2,6-Diacetyloxy-4-[(4-acetyloxy-3-(acetyloxymethyl)-2-butenyl)oxy] phenyl]-1-butanone, 98
[122616-67-5]	1-[2,6-Dihydroxy-4-[[4-hydroxy-3-(hydroxymethyl)-2-butenyl]oxy]phenyl]- 1-butanone, 98
[123014-46-0]	6-Bromo-1-(3,4-dimethoxyphenyl)-1-hexanone, 699
[123015-21-4]	6-Bromo-1-[3,4-dimethoxy-2-methyl-5-(1-methylethyl)phenyl]-1-hexanone, 708
[123015-22-5]	6-Bromo-1-(3,4-dimethoxy-2,5-dimethylphenyl)-1-hexanone, 707
[123015-34-9]	6-Bromo-1-(2-fluoro-4,5-dimethoxyphenyl)-1-hexanone, 704
[123015-39-4]	6-Bromo-1-(3,4-dimethoxy-2,5,6-trimethylphenyl)-1-hexanone, 708
[123020-85-9]	1-(2,4-Dihydroxyphenyl)-2-methyl-1-butanone (2S), 127
[123020-88-2]	1-(4-Hydroxyphenyl)-2-methyl-1-butanone (+), 126
[123059-72-3]	1-(3,4,5-Trimethoxyphenyl)-1-tridecanone, 998
[123127-70-8]	5,7-Dihydroxy-8-(3-methylbutyl)-6-(3-methyl-1-oxobutyl)-4-propyl- 2 <i>H</i> -1-benzopyran-2-one, 240
[123172-47-4]	1-(4-Amino-3-hydroxyphenyl)-1-butanone, 39
[123172-48-5]	1-(3-Hydroxy-4-methylaminophenyl)-1-butanone, 60
[123387-95-1]	1,5-Bis(3,4-dibutyloxyphenyl)-1,5-pentanedione, 521
[123387-96-2]	1,5-Bis(3,4-didecyloxyphenyl)-1,5-pentanedione, 521
[123471-86-3]	Methyl 4-(2-hydroxy-5-methylphenyl)-4-oxo-1-butanoate, 435
[123471-91-0]	Methyl 4-(2-hydroxy-3,5-dimethylphenyl)-4-oxo-1-butanoate, 444
[123687-72-9]	1-(2,4,5-Trihydroxyphenyl)-16-methyl-1-heptadecanone, 1060
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[124016-88-2]	6-(3-Hydroxy-4-methylphenyl)-6-oxo-1-hexanoic acid, 716
[124135-37-1]	1,4-Bis[2-hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1,4-butanedione, 373
[124141-66-8]	1,9-Bis(2,4-dihydroxyphenyl)-1,9-nonanedione (Di-2,4-dinitrophenyl- hydrazone), 851
[124210-60-2]	1-(3,5-Dimethoxyphenyl)-1-pentadecanone, 1028
[124210-61-3]	1-(3,5-Dihydroxyphenyl)-1-pentadecanone, 1028
[124259-63-8]	1-(2,4-Dimethoxy-3-methylphenyl)-1,3-butanedione, 318
[124557-51-3]	1-(2,5-Dimethoxyphenyl)-3-methyl-1-butanone, 179
[124557-52-4]	1-(2,5-Dihydroxyphenyl)-3-methyl-1-butanone, 178
[124598-07-8]	1-(2-Hydroxy-4,6-dimethoxyphenyl)-2-methyl-1-butanone (S), 136
[124598-08-9]	1-(4,6-Dihydroxy-2-methoxy-3-methylphenyl)-2-methyl- 1-butanone (S)-(+), 136
[124598-11-4]	2-Methyl-1-(2,4,6-trihydroxy-3-methylphenyl)-1-butanone (S), 134
[124598-12-5]	2-Methyl-1-(2,4,6-trimethoxyphenyl)-1-butanone (S), 130
[124598-13-6]	1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-2-methyl- 1-butanone (S), 138
[124598-14-7]	2-Methyl-1-(2,4,6-trimethoxy-3-methylphenyl)-1-butanone (S), 134
[124598-15-8]	1-[2,4-Bis(acetyloxy)-6-hydroxyphenyl]-2-methyl-1-butanone (S), 139
[124598-16-9]	2-Methyl-1-[2,4,6-tris(acetyloxy)phenyl]-1-butanone (S), 130
[124598-17-0]	1-(4,6-Diacetyloxy-2-methoxy-3-methylphenyl)-2-methyl- 1-butanone (S)-(+), 136
[124598-18-1]	2-Methyl-1-[2,4,6-tris(acetyloxy)-3-methylphenyl]-1-butanone (S), 134
[124598-19-2]	1-(2-Acetyloxy-4,6-dimethoxy-3-methylphenyl)-2-methyl-1-butanone, 138
[124960-73-2]	1-[2-(β-D-Glucopyranosyloxy)-4,6-dihydroxyphenyl]-2-methyl- 1-butanone, 145
[125009-82-7]	1-(2,6-Dihydroxyphenyl)-1-dodecanone, 947
[125074-06-8]	2-Methyl-1-(2,4,6-trihydroxyphenyl)-1-butanone, 129
[125292-98-0]	1-[3,5-Bis[[2,6-dihydroxy-4-methoxy-3-methyl-5-(1-oxobutyl)phenyl] methyl]-2,4,6-trihydroxyphenyl]-2-methyl-1-butanone, 169
[125304-88-3]	1,4-Bis(2,4-dimethoxyphenyl)-1,4-butanedione (E,E)-Di-O-methyl oxime, 362
[125500-45-0]	1-(4-Chloro-8-hydroxy-3-quinolinyl)-1-butanone, 77
[125500-46-1]	1-[8-Hydroxy-4-[(2-methylphenyl)amino]-3-quinolinyl]-1-butanone, 118
[125628-93-5]	1-(3,4-Dihydroxy-5-nitrophenyl)-1-pentanone, 485
[125628-94-6]	1-(3,4-Dihydroxy-5-nitrophenyl)-1-decanone, 881
[125697-50-9]	1-(2,5-Dimethoxyphenyl)-2-octadecyl-1-eicosanone, 1099
[126163-53-9]	1-(2',3'-Difluoro-4'-octyloxy[1,1'-biphenyl]-4-yl)-1-hexanone, 667
[126211-12-9]	1-(6-Hydroxy-2,4-dimethoxy-3-methylphenyl)-3-methyl-1-butanone, 207
[126516-15-2]	1-(4'-Acetyloxy[1,1'-biphenyl]-4-yl)-1-heptanone, 756
[127154-56-7]	1-(2-Benzyloxyphenyl)-1-pentanone, 463
[127275-13-2]	Methyl 4-(2,5-dihydroxyphenyl)-4-oxo-1-butanoate, 408
[127275-14-3]	Methyl 4-(5-chloro-2-hydroxy-4-methylphenyl)-4-oxo-1-butanoate, 432
[127313-53-5]	1-[2-Hydroxy-4-(sec-octyloxy)phenyl]-1-hexanone (Oxime), 689
[127313-65-9]	1-[2-Hydroxy-4-(sec-octyloxy)phenyl]-1-hexanone, 689
[127699-71-2]	1-(2-Hydroxy-5-methylphenyl)-1-butanone (2,4-Dinitrophenylhydrazone), 49

[127699-72-3]	1-(2-Hydroxy-5-methylphenyl)-1-octanone (2,4-Dinitrophenylhydrazone), 793
[127699-73-4]	1-(2-Hydroxy-5-methylphenyl)-1-dodecanone
[12/039-73-4]	(2,4-Dinitrophenylhydrazone), 960
[127789-29-1]	1-[2-Hydroxy-4-(1-methylheptyloxy)phenyl]-1-octanone, 823
[127789-31-5]	1-[2-Hydroxy-4-(1-methylheptyloxy)phenyl]-1-octanone (Oxime) (E), 823
[127789-34-8]	1-[2-Hydroxy-4-(1-methylheptyloxy)phenyl]-1-octanone (Oxime) (Z), 823
[127928-53-4]	1-(3-Fluoro-4-hydroxyphenyl)-2-methyl-1-octanone (+), 791
[127928-54-5]	1-(2-Fluoro-4-hydroxyphenyl)-2-methyl-1-octanone, 791
[127928-56-7]	1-(3-Fluoro-4-methoxyphenyl)-2-methyl-1-octanone (+), 791
[127928-60-3]	1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-2-methyl-1-octanone (S), 817
[127928-64-7]	1-(2-Fluoro-4-methoxyphenyl)-2-methyl-1-octanone (+), 791
[128733-94-8]	1,4-Bis(3-bromo-2-hydroxy-5-methylphenyl)-1,4-butanedione, 364
[129201-57-6]	4-(2-Ethoxy-4-methoxy-5-ethylphenyl)-4-oxo-1-butanoic acid, 445
[129218-86-6]	3-Chloro-1-(2-hydroxyphenyl)-1-butanone, 276
[129227-94-7]	1-[2,4,6-Trihydroxy-3-(1-oxopropyl)phenyl]-1-heptanone, 746
[129375-11-7]	1-[2-Hydroxy-4-methyl-5-(1-methylethyl)phenyl]-1-heptanone, 749
[129375-14-0]	1-[3-(1,1-Dimethylethyl)-2-hydroxy-6-methyl-5-(1-methylethyl)phenyl]-
	1-heptanone, 762
[129375-15-1]	1-[6-Hydroxy-2-methyl-3-(1-methylethyl)phenyl]-1-heptanone, 750
[129399-52-6]	1-[6-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-
	3,4-dihydro-3,5,7-trihydroxy-2,2-dimethyl-2 <i>H</i> -1-benzopyran-8-yl]-
[120527.00.0]	1-butanone, 343
[129527-09-9]	1-(3,5-Dichloro-4-hydroxyphenyl)-1-butanone, 25
[129527-10-2]	1-(3,5-Dichloro-4-hydroxyphenyl)-1-butanone (Na salt), 25
[129684-20-4]	1-(2,4,6-Trihydroxyphenyl)-11-phenyl-1-undecanone, 933
[130907-67-4]	6-Acetyloxy-7-(1-oxobutyl)-3-methylcoumarilic acid, 88
[131033-26-6]	1-(4-Hydroxyphenyl)-2-methyl-1-octanone (+), 794
[131252-71-6]	1,1'-(3-Hydroxy[1,1'-bipheny]]-4-yl)-1-butanone, 260
[131427-27-5]	1-(3,5-Dichloro-4-hydroxyphenyl)-1-octanone, 786
[131427-28-6]	1-(3,5-Dichloro-4-hydroxyphenyl)-1-hexanone, 621
[131427-29-7]	1-(3,5-Dichloro-2-hydroxyphenyl)-1-octanone, 786
[131699-22-4]	Methyl 5-(3,4-dimethoxyphenyl)-5-oxo-1-pentanoate, 582
[131868-27-4]	4-Methyl-1-(2,4,6-trihydroxy-3-methylphenyl)-1-pentanone, 548
[132041-56-6] [132180-62-2]	1-(4'-Methoxy[1,1'-biphenyl]-4-yl)-2-methyl-1-butanone, 2624-Cyclohexyl-1-(2-hydroxy-4-methylphenyl)-1-butanone, 257
[132180-62-2]	4-Cyclohexyl-1-(2-hydroxy-4-methylphenyl)-1-butanone, 257 4-Cyclohexyl-1-(4-hydroxy-2-methylphenyl)-1-butanone, 258
[132330-85-9]	2-Ethyl-4-(2,5-dimethoxyphenyl)-4-oxo-1-butanoic acid, 419
[132330-85-9]	2.4,6-Trihydroxy-3-methyl-5-(3-methyl-1-oxopentyl)benzaldehyde, 560
[132858-47-0]	1-(3,4-Dimethoxyphenyl)-3-methyl-1-butanone, 180
[132858-49-2]	1-(3-Hydroxyphenyl)-1-heptanone, 720
[132858-60-7]	1-(2-Hydroxy-3-methylphenyl)-1-hexanone, 638
[132858-61-8]	1-(4-Hydroxy-2-methylphenyl)-1-hexanone, 640
[132858-62-9]	1-(4-Hydroxy-2-methylphenyl)-1-hexanone, 640
[132859-07-5]	1-(2,5-Dimethoxyphenyl)-1-octanone, 780
[132037-07-5]	1-(2,3-Dimethoxyphenyi)-1-octanone, /60

[133101-50-5]	4-(2-Methoxyphenyl)-3-methyl-4-oxo-1-butanoic acid, 414
[133101-52-7]	4-[(2-Methoxymethyl)phenyl]-3-methyl-4-oxo-1-butanoic acid, 414
[133406-99-2]	1-(3-Hydroxy-4'-methoxy[1,1'-biphenyl]-4-yl)-2-methyl- 1-octanone (S), 820
[133407-00-8]	1-(3,4'-Dihydroxy[1,1'-biphenyl]-4-yl)-2-methyl-1-octanone (S), 817
[133455-19-3]	1-[4-(3-Bromopropoxy)-3-methoxyphenyl]-1-pentanone, 504
[133535-19-0]	Methyl 7-(2-hydroxyphenyl)-7-oxo-1-heptanoate, 766
[133535-20-3]	6-(2,4-Dihydroxyphenyl)-6-oxo-1-hexanoic acid, 712
[133535-21-4]	Ethyl 4-(2,4-dihydroxyphenyl)-4-oxo-1-butanoate, 405
[133559-45-2]	1-(3-Methoxyphenyl)-4-(4-methoxyphenyl)-1,4-butanedione, 359
[133831-11-5]	1-(2,5-Dihydroxy-4-methoxyphenyl)-1-octanone, 797
[133839-66-4]	3-Hydroxy-1-(2-hydroxyphenyl)-1-dodecanone, 949
[134081-63-3]	1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-hexanone, 659
[134081-64-4]	1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-octanone, 804
[134081-65-5]	1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-decanone, 892
[134081-66-6]	1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-dodecanone, 970
[134081-67-7]	1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-tetradecanone, 1017
[134081-68-8]	1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-hexadecanone, 1047
[134081-69-9]	1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-octadecanone, 1080
[134081-70-2]	1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-hexanone, 660
[134081-71-3]	1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-octanone, 805
[134081-72-4]	1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-decanone, 892
[134081-73-5]	1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-dodecanone, 970
[134081-74-6]	1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-tetradecanone, 1017
[134081-75-7]	1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-hexadecanone, 1048
[134081-76-8]	1-(6-Hydroxy-2,3,4-trimethoxyphenyl)-1-octadecanone, 1080
[134081-78-0]	1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1-hexanone (p-Toluenesulfonate), 660
[134081-79-1]	1-[2-(p-Toluenesulfonyloxy)-3,4,6-trimethoxyphenyl]-1-octanone, 804
[134081-80-4]	1-[2-(p-Toluenesulfonyloxy)-3,4,6-trimethoxyphenyl]-1-decanone, 892
[134081-81-5]	1-[2-(4-Toluenesulfonyloxy)-3,4,6-trimethoxyphenyl)-1-dodecanone, 970
[134081-82-6]	1-[2-(4-Toluenesulfonyloxy)-3,4,6-trimethoxyphenyl]- 1-tetradecanone, 1017, 1022
[134081-83-7]	1-[2-(p-Toluenesulfonoxy)-3,4,6-trimethoxyphenyl]-1-hexadecanone, 1048
[134081-84-8]	1-[2-(p-Toluenesulfonyloxy)-3,4,6-trimethoxyphenyl]- 1-octadecanone, 1080
[134081-86-0]	1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]- 1-hexanone, 690
[134081-87-1]	1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]- 1-octanone, 824
[134081-88-2]	1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]- 1-decanone, 913
[134081-89-3]	1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]- 1-dodecanone, 984
[134081-90-6]	1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]- 1-tetradecanone, 1022
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[134081-91-7]	1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]- 1-hexadecanone, 1052
[134081-92-8]	1-[6-Hydroxy-3,4-dimethoxy-2-(4-methylphenylsulfonyloxy)phenyl]- 1-octadecanone, 1087
[134081-94-0]	1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-hexanone, 653
[134081-95-1]	1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-octanone, 803
[134081-96-2]	1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-decanone, 889
[134081-97-3]	1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-dodecanone, 967
[134081-98-4]	1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-tetradecanone, 1016
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[134082-00-1]	1-(2,6-Dihydroxy-3,4-dimethoxyphenyl)-1-octadecanone, 1079
[134082-02-3]	1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-1-hexanone, 690
[134082-03-4]	1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-1-octanone, 823
[134082-04-5]	1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-1-decanone, 912
[134082-05-6]	1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-
	1-dodecanone, 984
[134082-06-7]	1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-
	1-tetradecanone, 1021
[134082-07-8]	1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]-
[134082-08-9]	1-hexadecanone, 1051
[134062-06-9]	1-[2-Hydroxy-3,4-dimethoxy-6-(phenylmethoxy)phenyl]- 1-octadecanone, 1086
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[134364-70-8]	1-(2,3-Difluoro-4-methoxyphenyl)-1-pentanone, 480
[134610-35-8]	1-(4-Hydroxy-3-methoxy-5-nitrophenyl)-1-butanone, 47
[134610-36-9]	1-(3,4-Dihydroxy-5-nitrophenyl)-1-butanone, 37
[134925-05-6]	1-(2,4-Dihydroxyphenyl)-2-methyl-1-octanone, 796
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[135649-79-5]	1-(4-Hydroxyphenyl)-1-heptadecanone, 1055
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[136116-43-3]	1-(3-Methoxyphenyl)-1-octanone, 773
[136741-47-4]	1-(5-Chloro-2-hydroxy-4-methoxyphenyl)-3-methyl-1-butanone, 188
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[136964-18-6]	1-(2-Fluoro-4-hydroxyphenyl)-1-octanone, 787
[137034-61-8]	1-(4-Hydroxyphenyl)-1-undecanone, 924
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[137832-98-5]	1-(4-Hydroxy-2,3,5-trimethylphenyl)-1-dodecanone, 969
[137832-99-6]	1-(4-hydroxy-3,5-dimethylphenyl)-1-octadecanone, 1078
[137833-00-2]	1-(4-Hydroxy-3,5-bis(1-methylethyl)phenyl)-1-octadecanone, 1084
[137833-01-3]	1-(4-Hydroxy-2,3,5-trimethylphenyl)-1-docosanone, 1108
[137833-02-4]	1-(4-Hydroxy-2,3-dimethyl-5-(1-methylethyl)phenyl)-1-octadecanone, 1083
[137833-03-5]	1-(4-Hydroxy-2,3,5-trimethylphenyl)-1-hexadecanone, 1047
[137866-03-6]	1-(3-Fluoro-4-methoxyphenyl)-1-octanone, 788

[137937-40-7]	1-(2-Hydroxy-3-methylphenyl)-2-propyl-1-pentanone, 561
[137937-41-8]	1-(2-Hydroxy-4-methylphenyl)-2-propyl-1-pentanone, 561
[137937-44-1]	1-(4-Hydroxyphenyl)-2-propyl-1-pentanone, 556
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[139409-36-2]	1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-2-methyl-1-butanone, 302
[140400-68-6]	1-[2,6-Dihydroxy-3-methyl-4-[(3-methyl-2-butenyl)oxy]phenyl]- 3-methyl-1-butanone, 222
[140400-69-7]	1-[2,6-Diacetyloxy-3-methyl-4-[(3-methyl-2-butenyl)oxy]phenyl]- 3-methyl-1-butanone, 223
[140400-70-0]	1-[2-Hydroxy-6-methoxy-3-methyl-4-[(3-methyl-2-butenyl)oxy]phenyl]-
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[140943-13-1]	1-(2-Hydroxy-5-methoxyphenyl)-1-hexanone (Oxime), 643
[140943-14-2]	1-(2-Hydroxy-5-methoxyphenyl)-1-octanone (Oxime), 796
[140943-15-3]	1-(2-Hydroxy-5-methoxyphenyl)-1-dodecanone (Oxime), 962
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[140943-19-7]	1-(5-Ethoxy-2-hydroxyphenyl)-1-hexanone (Oxime), 652
[140943-20-0]	1-(5-Ethoxy-2-hydroxyphenyl)-1-octanone (Oxime), 801
[140943-21-1]	1-(5-Ethoxy-2-hydroxyphenyl)-1-dodecanone (Oxime), 966
[140943-22-2]	1-(5-Ethoxy-2-hydroxyphenyl)-1-tetradecanone (Oxime), 1015
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[140943-31-3]	1-(5-Ethoxy-2-hydroxyphenyl)-1-pentanone, 501
[140943-32-4]	1-(5-Ethoxy-2-hydroxyphenyl)-1-hexanone, 652
[140943-33-5]	1-(5-Ethoxy-2-hydroxyphenyl)-1-octanone, 801
[140943-34-6]	1-(5-Ethoxy-2-hydroxyphenyl)-1-decanone, 888
[140943-35-7]	1-(5-Ethoxy-2-hydroxyphenyl)-1-dodecanone, 966
[140943-36-8]	1-(5-Ethoxy-2-hydroxyphenyl)-1-tetradecanone, 1015
[140943-37-9]	1-(5-Butoxy-2-hydroxyphenyl)-1-hexanone, 666
[140943-38-0]	1-(5-Butoxy-2-hydroxyphenyl)-1-octanone, 808
[140943-39-1]	1-(5-Butoxy-2-hydroxyphenyl)-1-dodecanone, 974
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[141036-68-2]	1-(4-Methoxy-3-methylphenyl)-1-hexanone, 641
[141124-94-9]	1-(4-Hydroxy-3-nitrophenyl)-1-undecanone, 930
[141124-96-1]	1-(3-Amino-4-hydroxyphenyl)-1-undecanone, 930
[141681-77-8]	1-(4'-Methoxy[1,1'-biphenyl]-4-yl)-2-methyl-1-octanone (+), 817
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[142234-79-5]	1-(2,6-Dihydroxy-4-methoxyphenyl)-1-hexanone, 644
[142611-25-4]	1-(3,5-Dihydroxyphenyl)-2-nonadecanone, 1094
[143183-56-6]	2,4,6-Tris(acetyloxy)-5-(3-methyl-1-oxobutyl)-1,3-benzenedicarbox- aldehyde, 389
[143286-57-1]	1-(2-Hydroxy-4-methoxyphenyl)-1-butanone (Oxime), 55
[143286-58-2]	1-(2-Hydroxy-4-methoxyphenyl)-1-heptanone (Oxime), 740
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[143286-60-6]	1-(2-Hydroxy-4-methoxyphenyl)-1-octanone (Oxime), 796
[143286-61-7]	1-(4-Butoxy-2-hydroxyphenyl)-1-nonanone (Oxime), 849
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[143286-63-9]	1-(2-Hydroxy-4-propoxyphenyl)-1-decanone (Oxime), 891
[143286-64-0]	1-[2-Hydroxy-4-(octyloxy)phenyl]-1-decanone (Oxime), 912
[143286-65-1]	1-(2-Hydroxy-4-methoxyphenyl)-1-dodecanone (Oxime), 962
[143286-66-2]	1-(4-Butoxy-2-hydroxyphenyl)-1-dodecanone (Oxime), 974
[143286-67-3]	1-(2-Hydroxy-4-pentyloxyphenyl)-1-dodecanone (Oxime), 976
[143286-68-4]	1-(2-Hydroxy-4-methoxyphenyl)-1-tetradecanone (Oxime), 1012
[143286-69-5]	1-(4-Butoxy-2-hydroxyphenyl)-1-tetradecanone (Oxime), 1019
[143286-81-1]	1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1-hexanone (Oxime), 683
[143286-82-2]	1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1-octanone (Oxime), 818
[143286-83-3]	1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1-tetradecanone (Oxime), 1020
[143286-84-4]	1-(2,4-Dihydroxyphenyl)-1-tetradecanone, 1006
[143286-89-9]	1-(2-Hydroxy-4-methoxyphenyl)-1-heptanone, 739
[143286-90-2]	1-[4-(Hexyloxy)-2-hydroxyphenyl]-1-heptanone, 758
[143286-91-3]	1-(2-Hydroxy-4-methoxyphenyl)-1-octanone, 795
[143286-92-4]	1-(4-Butoxy-2-hydroxyphenyl)-1-nonanone, 848
[143286-93-5]	1-(4-Hexyloxy-2-hydroxyphenyl)-1-nonanone, 856
[143286-94-6]	1-(2-Hydroxy-4-propoxyphenyl)-1-decanone, 891
[143286-95-7]	1-[2-Hydroxy-4-(octyloxy)phenyl]-1-decanone, 912
[143286-96-8]	1-(2-Hydroxy-4-methoxyphenyl)-1-dodecanone, 962
[143286-97-9]	1-(4-Butoxy-2-hydroxyphenyl)-1-dodecanone, 974
[143286-98-0]	1-(2-Hydroxy-4-pentyloxyphenyl)-1-dodecanone, 976
[143286-99-1]	1-[4-(Decyloxy)-2-hydroxyphenyl]-1-dodecanone, 985
[143287-00-7]	1-(2-Hydroxy-4-methoxyphenyl)-1-tetradecanone, 1012
[143287-01-8]	1-(4-Butoxy-2-hydroxyphenyl)-1-tetradecanone, 1019
[143287-04-1]	1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1-hexanone, 683
[143287-05-2]	1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1-octanone, 817
[143287-06-3]	1-[2-Hydroxy-4-(phenylmethoxy)phenyl]-1-tetradecanone, 1020
[143287-07-4]	1-(4-Butoxy-5-hexyl-2-hydroxyphenyl)-1-heptanone, 763
[143287-08-5]	1-(5-Hexyl-2-hydroxy-4-methoxyphenyl)-1-decanone, 906
[143287-09-6]	1-(4-Butoxy-5-hexyl-2-hydroxyphenyl)-1-decanone, 914
[143287-10-9]	1-(4-Butoxy-5-hexyl-2-hydroxyphenyl)-1-decanone (Oxime), 914
[143378-82-9]	1-(4-Hydroxy-3-methoxyphenyl)-1-nonanone, 845
[144292-57-9]	1-(2,3-Difluoro-4-benzyloxyphenyl)-1-decanone, 878
[144292-58-0]	1-(2,3-Difluoro-4-hydroxyphenyl)-1-decanone, 878
[144337-28-0]	1,1'-(2,4,6-Trihydroxy-1,3-phenylene)bis-1-dodecanone, 987

[144785-80-8]	3-Methyl-1-[2,4,6-trihydroxy-3-(3,7-dimethyl-2,6-octanediene)phenyl]- 1-butanone, 232
[144785-81-9]	1-[4,6-(Diacetyloxy)-2-hydroxy-3-(3,7-dimethyl-2,6-octanediene)phenyl]- 2-Methyl-1-butanone, 162
[144785-82-0]	1-[4,6-(Diacetyloxy)-2-hydroxy-3-(3,7-dimethyl-2,6-octanediene)phenyl]- 3-methyl-1-butanone, 248
[144785-84-2]	3-Methyl-1-[2,4,6-tris(acetyloxy)-3-(3,7-dimethyl-2,6-octanediene)phenyl]- 1-butanone, 233
[145747-19-9]	1-(2,4,5-Tris(acetyloxy)phenyl)-1-butanone, 17
[145747-22-4]	1-[2,5-Bis(acetyloxy)-4-hydroxyphenyl]-1-butanone, 89
[145747-23-5]	1-(2-Acetyloxy-4,5-dihydroxyphenyl)-1-butanone, 62
[145904-69-4]	1-[3-[(2 <i>R</i> ,4 <i>S</i> )-3,4-Dihydro-7-hydroxy-2-(4-hydroxyphenyl)-2 <i>H</i> -1- benzopyran-4-yl]-2,4,6-trihydroxyphenyl]-1-dodecanone (+), 989
[145941-31-7]	1,1'-[Methylenebis(4,5,6-trihydroxy-3,1-phenylene)]bis-1-octanone, 827
[146923-05-9]	1-[2-Methoxy-4-(2-methyl-1-propenyl)phenyl]-1-pentanone, 506
[146923-06-0]	1-[3-Methoxy-4-(2-methyl-1-propenyl)phenyl]-1-pentanone, 507
[147862-99-5]	1-[2,4,6-Trihydroxyphenyl]-1-tetradecanone, 1008
[148516-07-8]	1-(4-Hydroxy-3,5-dimethylphenyl)-1-hexanone, 651
[148707-32-8]	1-[5-Acetyl-2-hydroxy-3-(3-methyl-1,3-butadienyl)phenyl]-3-methyl- 1-butanone ( <i>E</i> ), 380
[149053-77-0]	3-Methyl-1-(2,4,6-trimethoxyphenyl)-1-butanone, 195
[149412-46-4]	5-Chloro-1-(4-fluoro-2-hydroxyphenyl)-1-pentanone, 571
[149454-86-4]	1-[4-(4-Heptanoylphenyloxy)phenyl]-1-heptanone, 724
[149696-17-3]	1-(2-Hydroxy-4,5-dimethylphenyl)-1-pentanone, 500
[150033-77-5]	1-(2-Hydroxy-5-methylphenyl)-1-pentanone, 493
[150396-49-4]	1-(3,4-Dimethoxyphenyl)-2-ethyl-1-hexanone, 618
[151029-60-6]	1-(3,4-Dihydroxyphenyl)-1-eicosanone, 1097
[152153-24-7]	1-(5-Chloro-2-hydroxy-4-methylphenyl)-1,3-butanedione, 316
[152269-19-7]	1-(4-Methoxyphenyl)-4,4-dimethyl-1-pentanone, 555
[152430-07-4]	1-(4-Acetyloxyphenyl)-2-methyl-1-decanone (S), 876
[152609-12-6]	1-[4'-Fluoro-4-hydroxy-6-methoxy[1,1'-biphenyl]-3-yl]-1-butanone, 263
[152609-14-8]	1-[4-(3-Chloropropoxy)-4'-fluoro-6-methoxy[1,1'-biphenyl]-3-yl]- 1-butanone, 263
[153756-51-5]	1-(4-Ethyl-2,5-dimethoxyphenyl)-1-butanone, 69
[153756-53-7]	1-(4-Ethyl-2,5-dimethoxyphenyl)-3,3-dimethyl-1-butanone, 254
[154736-91-1]	1-[5-[[(2,5-Dihydroxyphenyl)methyl]amino]-2-hydroxyphenyl]- 3,5,5-trimethyl-1-hexanone, 692
[154737-33-4]	1-(2-Hydroxyphenyl)-3,5,5-trimethyl-1-hexanone, 619
[154737-34-5]	1-(2-Hydroxy-5-nitrophenyl)-3,5,5-trimethyl-1-hexanone, 656
[154737-35-6]	1-(5-Amino-2-hydroxyphenyl)-3,5,5-trimethyl-1-hexanone, 660
[154737-36-7]	1-[5-[[(2,5-Dihydroxyphenyl)methylene]amino]-2-hydroxyphenyl]- 3,5,5-trimethyl-1-hexanone, 691
[154921-40-1]	1-(2,4-Dihydroxy-6-methylphenyl)-1-butanone, 53
[154921-41-2]	1-(2,4-Dihydroxy-6-methylphenyl)-1-pentanone, 495
[155084-01-8]	1-(3,4,5-Trihydroxyphenyl)-1-tricosanone, 1111

[156306-39-7]	1,1'-(6,6'-Dihydroxy-5,5'-dimethoxy[1,1'-biphenyl]-3,3'-diyl)bis- 1-tridecanone, 1001
[157687-62-2]	1,1'-(6,6'-Dihydroxy-5,5'-dimethoxy[1,1'-biphenyl]-3,3'-diyl)bis- 1-octanone, 827
[158869-45-5]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-8-bromo-1-octanone, 830
[158869-48-8]	1-[3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl]-7-bromo-1-heptanone, 766
[158905-44-3]	1-(8-Hydroxy-5-quinolinyl)-1-tetradecanone, 1016
[159211-02-6]	1-(4-Benzyloxy-3-methoxyphenyl)-4-methyl-1-pentanone, 548
[159211-07-1]	1-(3-Benzyloxy-4-methoxyphenyl)-4-methyl-1-pentanone, 548
[159457-03-1]	1-(2,4-Dihydroxyphenyl)-3-methyl-1-butanone (Oxime), 177
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[186041-40-6]	1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-2-methyl-1-heptanone (S), 758
[186041-43-0]	1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-2-methyl-1-heptanone, 758
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[186454-86-4]	1-(3,4-Dimethoxyphenyl)-1-heptadecanone, 1056
[187396-80-1]	5-Chloro-1-(5-chloro-2-methoxyphenyl)-1-pentanone, 572
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[187396-83-4]	6-Bromo-1-(4-methoxy[1,1'-biphenyl]-3-yl)-1-hexanone, 709
[187396-84-5]	5-Chloro-1-(4-methoxy[1,1'-biphenyl]-3-yl)-1-pentanone, 576
[187396-92-5]	5-Chloro-1-(2,4-dimethoxy-5-nitrophenyl)-1-pentanone, 571
[187396-94-7]	5-Chloro-1-(2-methoxy-3-nitrophenyl)-1-pentanone, 571
[187396-95-8]	5-Chloro-1-(2-methoxy-5-nitrophenyl)-1-pentanone, 571
[187396-96-9]	5-Chloro-1-(2,4,6-trimethoxy-3-nitrophenyl)-1-pentanone, 571
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[188973-66-2]	6-Bromo-1-(3-fluoro-4-methoxyphenyl)-1-hexanone, 703
[188973-67-3]	6-Bromo-1-(4-hydroxyphenyl)-1-hexanone, 698
[189568-62-5]	1-(8-Methoxy-4-propyl-3-quinolinyl)-1-butanone, 105
[189875-21-6]	1-(4-Bromo-2-hydroxyphenyl)-1-pentanone, 480
[189875-29-4]	1-(2-Hydroxy-3-methylphenyl)-1-pentanone, 492
[190248-05-6]	1,5-Bis(2-methoxyphenyl)-1,5-pentanedione, 517
[191284-02-3]	1-(3,4-Dihydroxyphenyl)-1-tetracosanone, 1113
[193687-88-6]	1-(2-Hydroxy-4-methoxyphenyl)-3-methyl-1-butanone, 192
[194359-57-4]	1-(2-Methoxy-5-methylphenyl)-1-heptanone, 739
[194792-29-5]	1-[2-Hydroxy-4-(2-propenyloxy)phenyl]-1-butanone, 80
[194792-30-8]	1-[2,4-Dihydroxy-3-(2-propenyl)phenyl]-1-butanone, 80
[194792-31-9]	1-(2,4-Dihydroxy-3-propylphenyl)-1-butanone, 83
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[194792-60-4]	1-[2,4-Dihydroxy-3-(2-propenyl)phenyl]-1-pentanone, 503
[194792-61-5]	1-(2,4-Dihydroxy-3-propylphenyl)-1-pentanone, 505
[194792-62-6]	1-[4-(3-Bromopropoxy)-2-hydroxy-3-propylphenyl]-1-pentanone, 526
[194855-33-9]	1-[2-Hydroxy-4-(phenylmethoxy)-3-propylphenyl]-3,3-dimethyl- 1-butanone, 255
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[195158-14-6]	1-[3-Methoxy-2-(phenylmethoxy)phenyl]-1-hexanone, 684
[195393-34-1]	1-(3-Acetyloxyphenyl)-1-pentanone, 464
[195393-36-3]	1-(3-Acetyloxyphenyl)-2-methyl-1-butanone, 125
[195393-39-6]	1-(3-Hydroxyphenyl)-2-methyl-1-butanone, 125
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[196813-77-1]	1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-hexanone, 636
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[196869-44-0]	1,10-Bis(3,5-dimethoxy-4-methylphenyl)-1,10-decanedione, 909
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[198878-73-8]	3,7-Dimethyl-1-(2,4,6-trihydroxyphenyl)-1-octanone, 802
[198878-75-0]	1-[3,5-Bis(3,7-dimethyl-2,6-octadienyl)-2,4,6-trihydroxyphenyl]- 3-methyl-1-butanone, 249
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[1309/0-00-/]	3,7-Dimethyl-1-[2,4,6-trihydroxy-3-(3-methyl-2-butenyl)phenyl]- 1-octanone, 818
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[198878-98-7]	1-[2-Hydroxy-6-methyl-4-[(3-methyl-2-butenyl)oxy]phenyl]-3-methyl- 1-butanone, 221
[198878-99-8]	1-[2,4-Dihydroxy-6-methyl-3-(3-methyl-2-butenyl)phenyl]-3-methyl- 1-butanone, 221
[198879-00-4]	1-[4,6-Dihydroxy-2-methyl-3-(3-methyl-2-butenyl)phenyl]-3-methyl- 1-butanone, 221
[198879-01-5]	1-[2-Hydroxy-6-methyl-3,5-bis(3-methyl-2-butenyl)]-4-[(3-methyl- 2-butenyloxy)phenyl]-3-methyl-1-butanone, 249
[198879-02-6]	1-[2-Hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-3-methyl- 1-butanone, 212
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[198879-07-1]	1-(2,4-Dihydroxy-6-methylphenyl)-3-methyl-1-butanone, 192
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[200878-66-6]	1-(3-Chloro-2,4,6-trihydroxyphenyl)-1-hexanone, 626
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[204859-45-0]	1-(2-Hydroxyphenyl)-1-hexanone (Oxime), 599
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[206051-19-6]	7-Bromo-1-(5-chloro-2-hydroxyphenyl)-1-heptanone, 765
[209122-44-1]	2-Methyl-1-(2,3,4,6-tetrahydroxyphenyl)-1-butanone, 131
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[209479-38-9]	1-[6-[2-(Aminophenyl)thio]-8-methoxy-3-quinolinyl]-1-butanone (Hydrochloride), 115
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[213387-03-2]	1-(2-Iodo-3-methoxyphenyl)-1-butanone, 33
[213622-17-4]	1-(3-Acetyl-2-hydroxy-5-methylphenyl)-1-butanone, 377
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[213622-23-2]	1-(3-Acetyl-2-hydroxy-5-methylphenyl)-1-butanone (p-Chlorobenzoate), 377
[213622-24-3]	1-(3-Acetyl-2-hydroxy-5-methylphenyl)-1-butanone (p-Methylbenzoate), 377
[213622-25-4]	1-(3-Acetyl-2-hydroxy-5-methylphenyl)-1-butanone (p-Methoxybenzoate), 378
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[213622-31-2]	3-[2-(4-Chlorobenzoyl)acetyl]-2-hydroxy-5-methyl-1-butanone, 300
[213622-32-3]	3-[2-(4-Methylbenzoyl)acetyl]-2-hydroxy-5-methyl-1-butanone, 301
[213622-33-4]	3-[2-(4-Methoxybenzoyl)acetyl]-2-hydroxy-5-methyl-1-butanone, 301
[214058-35-2]	3-Ethyl-1-(4-hydroxy-3-nitrophenyl)-1-heptanone, 742
[214534-24-4]	1-(4'-Acetyloxy[1,1'-biphenyl]-4-yl)-1-pentanone, 525
[215667-87-1]	1-(4-Methoxyphenyl)-4-iodo-1-butanone, 291
[216300-88-8]	3-Methyl-1-(3,4,5-trihydroxyphenyl)-1-butanone, 183

[216300-89-9]	1-[3,5-Dihydroxy-2-(3-methyl-2-butenyl)-4-[(3-methyl-2-butenyl)oxy] phenyl]-3 methyl-1-butanone, 231
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[216300-91-3]	3-Methyl-1-[3,4,5-trihydroxy-2-(3-methyl-2-butenyl)phenyl]- 1-butanone, 215
[216300-92-4]	1-(2,6-Dihydroxy-4-methylphenyl)-3-methyl-1-butanone, 192
[216300-95-7]	1-[2,6-Dihydroxy-4-methyl-3-(3-methyl-2-butenyl)phenyl]-3-methyl- 1-butanone, 221
[216300-96-8]	1-[2,6-Dihydroxy-4-methyl-3,5-bis(3-methyl-2-butenyl)phenyl]-3-methyl- 1-butanone, 233
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[216300-99-1]	1-[5-Chloro-2-hydroxy-4-[(3-methyl-2-butenyl)oxy]phenyl]-3-methyl- 1-butanone, 212
[216301-00-7]	1-[5-Chloro-2,4-dihydroxy-3-(3-methyl-2-butenyl)phenyl]-3-methyl- 1-butanone, 211
[216301-02-9]	1,1'-[2,4,6-Trihydroxy-5-(3-methyl-2-butenyl)-1,3-phenylene]bis- 3-methyl-1-butanone, 306
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[217815-21-9]	1-(8-Hydroxy-2-methyl-5-quinolinyl)-1-hexanone, 661
[217815-22-0]	1-(8-Hydroxy-2-methyl-5-quinolinyl)-1-nonanone, 847
[217815-23-1]	1-(8-Hydroxy-2-methyl-5-quinolinyl)-1-decanone, 893
[217815-24-2]	1-(8-Hydroxy-2-methyl-5-quinolinyl)-1-dodecanone, 972
[217815-25-3]	1-(8-Hydroxy-2-methyl-7-quinolinyl)-1-nonanone, 847
[217815-26-4]	1-(8-Hydroxy-2-methyl-7-quinolinyl)-1-decanone, 893
[217815-27-5]	1-(8-Hydroxy-2-methyl-7-quinolinyl)-1-dodecanone, 972
[217815-28-6]	1-(8-Hydroxy-7-quinolinyl)-1-dodecanone, 968
[217815-29-7]	1-(8-Hydroxy-2-methyl-7-quinolinyl)-1-hexanone, 661
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[222158-42-1]	1,4-Bis(4-methoxyphenyl)-2,3-dimethyl-1,4-butanedione (meso), 367
[224775-35-3]	8-Bromo-1-(4-methoxyphenyl)-1-octanone, 828
[227946-80-7]	1-(3-Chloro-2,4-dihydroxy-6-methoxyphenyl)-1-hexanone, 636
[227946-81-8]	1-(5-Chloro-4,6-dihydroxy-2-methoxyphenyl)-1-hexanone, 637
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[233751-78-5]	2-Hydroxy-4,6-dimethoxy-3-(3-methyl-1-oxobutyl)benzaldehyde (Tert-Butyldimethylsilyl derivative), 391
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[238074-77-6]	2-Bromo-1-(3,5-dibromo-2-hydroxyphenyl)-1-butanone, 268
[238074-78-7]	2-Bromo-1-(3,5-dibromo-2,4-dihydroxyphenyl)-1-hexanone, 702
[241133-23-3]	1-[4-(β-D-Glucopyranosyloxy)-2,6-dihydroxy-3,5-dimethylphenyl]- 2-methyl-1-butanone, 149
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[246531-45-3]	1-(8-Hydroxy-5-quinolinyl)-1-hexanone, 654
[246531-46-4]	1-(8-Hydroxy-7-quinolinyl)-1-hexanone, 654
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[251463-57-7]	1-[2-(Acetyloxy)-4-hydroxyphenyl]-1-hexadecanone, 1043
[251463-59-9]	1-[2-(Acetyloxy)-4-hydroxyphenyl]-3-methyl-1-butanone, 195
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[278619-91-3]	6-Chloro-1-(4-methoxyphenyl)-1-hexanone, 701
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[306972-97-4]	2-Bromo-1-(3-bromo-4-methoxyphenyl)-1-decanone (2S), 917
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[307000-33-5]	1-(2-Hydroxy-4-phenoxyphenyl)-3-methyl-1-butanone, 219
[307000-38-0]	1-(2-Hydroxy-4-phenoxyphenyl)-1-hexanone, 681
[307000-44-8]	1-(2-Hydroxy-4-phenoxyphenyl)-1-pentanone, 526
[307000-48-2]	1-(2-Hydroxy-4-phenoxyphenyl)-1-dodecanone, 979
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[319494-44-5]	Methyl 4-(4-ethoxyphenyl)-4-oxo-1-butanoate, 402
[326854-41-5]	1-[2-Hydroxy-4-methoxy-6-[[tris(1-methylethyl)silyl]oxy]phenyl]- 1-butanone, 119
[327023-36-9]	1-[2,3-Dichloro-4-(2-hydroxyethyl)phenyl]-2-methylene-1-butanone, 132
[331749-02-1]	1-(4-Hydroxy-3-methyl-2-quinolinyl)-1-nonanone, 846
[331749-03-2]	1-(4-Acetyloxy-3-methyl-2-quinolinyl)-1-nonanone, 847
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[342423-70-5]	1-(3-Methoxyphenyl)-1-hexanone, 600
[344408-25-9]	1-(2-Hydroxyphenyl)-2-methyl-1-butanone, 125
[344574-57-8]	1,6-Bis(2-hydroxy-5-methoxy-4-methylphenyl)-1,6-hexanedione, 690
[344578-29-6]	1,5-Bis(2-hydroxy-5-methoxy-4-methylphenyl)-1,5-pentanedione, 536
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[353499-39-5]	1-(3,4,5-Trihydroxyphenyl)-1-undecanone, 928
[354585-20-9]	13-Methyl-(2,4-dihydroxyphenyl)-1-tetradecanone, 1022
[354585-21-0]	13-Methyl-1-(2,4,6-trihydroxyphenyl)-1-tetradecanone, 1023
[354585-22-1]	13-Methyl-1-(2,3,4-trihydroxyphenyl)-1-tetradecanone, 1023
[357172-17-9]	1-[2,5-Dimethoxy-3,4-bis(methylthio)phenyl]-3,7-dimethyl-1-octanone, 808
[357172-20-4]	1-(2,5-Dihydroxy-3,4,6-trimethylphenyl)-5-methyl-2-(1-methylethyl)- 1-hexanone, 685
[357172-24-8]	1-[2,5-Dihydroxy-3,4-bis(methylthio)phenyl]-3-(2-phenylethyl)- 1-undecanone, 934
[357172-26-0]	1-(2,5-dihydroxy-3,4,6-trimethylphenyl)-2-octyl-1-dodecanone, 985
[357172-28-2]	2-Butyl-1-(2,5-dihydroxy-4-methylphenyl)-1-octanone, 810
[357172-32-8]	2-Butyl-1-(2,5-dibenzyloxy-3,4,6-trimethylphenyl)-1-dodecanone, 981
[357172-44-2]	1-(2,5-Dimethoxy-3,6-diphenoxyphenyl)-2-(1,1-dimethylethyl)- 1-octanone, 827
[357172-52-2]	1-[5-(Acetyloxy)-2-hydroxyphenyl]-2-hexyl-1-dodecanone, 982
[358369-06-9]	4-(3,4-Dimethoxyphenyl)-3-methyl-4-oxo-1-butanoic acid, 416
[358369-08-1]	4-(3,4-Dimethoxyphenyl)-2,3-dimethyl-4-oxo-1-butanoic acid, 419
[364039-57-6]	1-[7-Methoxy-2-(3,4,5-trimethoxy)-2H-1-benzopyran-3-yl]-1-butanone, 121
[371757-62-9]	1-(3-Fluoro-4-methoxyphenyl)-1-hexanone, 627
[371757-70-9]	1-(3-Chloro-5-fluoro-4-methoxyphenyl)-1-hexanone, 621
[372486-18-5]	1-(2-Hydroxy-4-methoxyphenyl)-1-pentanone, 496
[372486-19-6]	1-(2-Hydroxy-4-methoxyphenyl)-1-hexanone, 642
[374808-50-1]	Methyl 6-(2,5-dimethoxyphenyl)-6-oxo-1-hexanoate, 713
[374808-62-5]	11-Bromo-1-(2,5-dimethoxyphenyl)-1-undecanone, 935
[375172-14-6]	1-(2,5-Dihydroxyphenyl)-2-octyl-1-decanone, 877
[375172-48-6]	1-(2-Amino-3,6-dibutyloxy[1,1'-biphenyl]-4-yl)-2-hexyl-1-dodecanone, 986
[383383-01-5]	1-(3,4-Dihydroxy-2-nitrophenyl)-1-pentanone, 485
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[383383-10-6]	1-(4-Hydroxy-3-methoxy-2-nitrophenyl)-1-pentanone, 491
[390358-13-1]	1-(4'-Hydroxy[1,1'-biphenyl]-4-yl)-1-heptanone, 756
[392304-69-7]	4-(3,5-Dibromo-4-hydroxyphenyl)-4-oxo-1-butanoic acid, 419
[393519-46-5]	1-(2-Hydroxy-4-methoxyphenyl)-1-decanone, 884
[396100-57-5]	2-Bromo-1-(3-bromo-2-hydroxy-5-methylphenyl)-1-butanone, 272
[401935-06-6]	1-(5-Chloro-2-hydroxyphenyl)-1-hexanone (Oxime), 625
[404918-99-6]	1-(2-Amino-5-hydroxyphenyl)-3-methyl-1-butanone, 187
[404919-00-2]	1-(2-Amino-5-hydroxyphenyl)-1-hexanone, 628
[404919-01-3]	1-(2-Amino-5-hydroxyphenyl)-1-butanone, 38
[406174-64-9]	1-(2,4-Dihydroxyphenyl)-2-methyl-1-hexanone, 616
[406174-67-2]	1-(2,4-Diacetyloxyphenyl)-2-methyl-1-butanone (Racemic, 128
[406174-68-3]	1-(2,4-Diacetyloxyphenyl)-2-methyl-1-hexanone (Racemic), 616
[406174-71-8]	1-[2-(Acetyloxy)-4-hydroxyphenyl]-2-methyl-1-butanone (-), 134
[406174-72-9]	1-[2-(Acetyloxy)-4-hydroxyphenyl]-2-methyl-1-hexanone (-), 655
[406174-75-2]	1-(2,4-Diacetyloxyphenyl)-2-methyl-1-butanone dextrogyre (+), 128
[406174-76-3]	1-(2,4-Diacetyloxyphenyl)-2-methyl-1-hexanone (+), 617
[406174-79-6]	1-(2,4-Diacetyloxyphenyl)-2-methyl-1-butanone levogyre(-), 128
[406174-80-9]	1-(2,4-Diacetyloxyphenyl)-2-methyl-1-hexanone (-), 617
[406463-67-0]	1-[3-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-
	2,4,6-trihydroxy-5-(2-hydroxy-3-methyl-3-butenyl)phenyl]-
	1-pentanone, 538
[406463-68-1]	1-[6-[(3-Acetyl-2,4-dihydroxy-6-methoxy-5-methylphenyl)methyl]-
	3,4-dihydro-3,5,7-trihydroxy-2,2-dimethyl-2 <i>H</i> -1-benzopyran-8-yl]- 3-methyl-1-butanone, 344
[408309-74-0]	1-(5-Chloro-2-hydroxy-4-methylphenyl)-1-butanone, 45
[408310-63-4]	4-Cyclohexyl-1-(2,5-dihydroxyphenyl)-1-butanone, 256
[408336-52-7]	4-(6-Hydroxybiphenyl-3-yl)-4-oxo-1-butanoic acid, 452
[408336-68-5]	4-(6-Methoxybiphenyl-3-yl)-4-oxo-1-butanoic acid, 452
[412022-96-9]	4-(3-Cyclohexyl-4-methoxyphenyl)-4-oxo-1-butanoic acid, 453
[412033-83-1]	4-(5-Cyclohexyl-2-metoxyphenyl)-4-oxo-1-butanoic acid, 454
[412051-38-8]	2-Bromo-1-(4-hydroxyphenyl)-3-methyl-1-butanone, 293
[412340-39-7]	1,1'-(5-Ethyl-2-methoxy-1,3-phenylene)bis-2-bromo-3-methyl-
	1-butanone, 304
[416846-26-9]	1-(4-Ethyloxyphenyl)-1-hexadecanone, 1034
[423115-90-6]	1-(4-Methoxyphenyl)-1-butanone (Oxime), 7
[430425-41-5]	1-(2,5-Dimethoxyphenyl)-1-hexanone, 608
[430425-42-6]	1-(2,4-Dimethoxyphenyl)-1-decanone, 873
[434340-29-1]	1-(4-Chloro-2,5-dimethoxyphenyl)-1-decanone, 880
[434340-30-4]	2-Bromo-1-(4-chloro-2,5-dimethoxyphenyl)-1-decanone, 917
[438490-66-5]	1-(6-Hydroxy-3-(4-methoxyphenyl)-2-phenyl-5-benzofuranyl)-
	1-butanone, 125
[438490-69-8]	1-(6-Hydroxy-3-(4-methylphenyl)-2-phenyl-5-benzofuranyl)- 1-butanone, 124
[439948-78-4]	1-(2-Hydroxyphenyl)-1-nonanone (Oxime), 835
[439948-79-5]	1-(2-Hydroxyphenyl)-1-decanone (Oxime), 868
[439948-80-8]	1-(2-Hydroxyphenyl)-1-undecanone (Oxime), 923
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[441353-19-1]	1-(2,4,6-Trimethoxyphenyl)-1-octanone, 784
[447439-58-9]	1-(4-Methoxy-2-iodophenyl)-1-pentanone, 484
[457926-60-2]	1-[(3R)-3-[(2S,4S)-3,4-Dihydro-7-hydroxy-2-(4-hydroxyphenyl)-
	2H-1-benzopyran-4-yl]-2,4,6-trihydroxyphenyl]-1-dodecanone, 991
[457930-47-1]	1-[(3 <i>R</i> )-3-[(2 <i>R</i> ,4 <i>R</i> )-3,4-Dihydro-7-hydroxy-2-(4-hydroxyphenyl)-
	2H-1-benzopyran-4-yl]-2,4,6-trihydroxyphenyl]-1-dodecanone, 990
[465526-56-1]	5-Butyl-1-(4-methoxyphenyl)-4-methylene-1-nonanone, 841
[467437-62-3]	1-[2-[(6-O-D-Apio-β-D-furanosyl-β-D-glucopyranosyl)oxy]-
	4,6-dihydroxyphenyl]-2-methyl-1-butanone (2S), 156
[473835-69-7]	1-(4-Methoxyphenyl)-2-methylene-1-hexanone, 615
[474668-86-5]	1-(3,4-Dihydroxyphenyl)-1-pentanone (O-Methyloxime), 473
[474668-95-6]	1-(3,4-Dihydroxyphenyl)-1-heptanone (O-Methyloxime), 727
[479580-83-1]	1-(2-Hydroxy-3-phenoxyphenyl)-1-butanone, 101
[484676-16-6]	1-(5-Bromo-3,4,5-trimethoxyphenyl)-1-pentanone, 567
[497934-63-1]	1-(5-Amino-2-hydroxyphenyl)-1-pentanone, 486
[500109-64-8]	1-[5,7-Dihydroxy-2,2-dimethyl-6-[[2,4,6-trihydroxy-3-methyl-
	5-(2-methyl-1-oxopropyl)phenyl]methyl]-2 <i>H</i> -1-benzopyran-8-yl]-
	2-methyl-1-butanone, 166
[500109-65-9]	1-[6-[(3-Acetyl-2,4,6-trihydroxy-5-methylphenyl)methyl]-5,7-dihydroxy-
[500127 72 0]	2,2-dimethyl-2 <i>H</i> -1-benzopyran-8-yl]-2-methyl-1-butanone, 163
[500127-72-0]	1-[2-Chloro-4-(2-propynyl)phenyl]-1-butanone, 29
[500127-73-1]	1-(3-Chloro-4-hydroxyphenyl)-1-butanone, 29
[501083-60-9]	6-Chloro-1-(2-methoxyphenyl)-1-hexanone, 700
[501083-61-0]	5-Chloro-1-(2-hydroxyphenyl)-1-pentanone, 567
[501083-62-1]	6-Chloro-1-(2-hydroxyphenyl)-1-hexanone, 700
[501083-63-2]	5-Chloro-1-(3-hydroxyphenyl)-1-pentanone, 567
[501083-64-3]	6-Chloro-1-(3-hydroxyphenyl)-1-hexanone, 701
[501361-68-8]	1-(3,4-Dimethoxyphenyl)-1-octadecanone, 1067
[502139-81-3]	10-(2,5-Dihydroxyphenyl)-10-oxo-1-decanoic acid, 920
[505084-75-3]	1-(4-Hydroxy-2-methylphenyl)-2-methyl-1-butanone (2S), 133
[505084-77-5]	1-(2-Fluoro-4-hydroxyphenyl)-2-methyl-1-butanone (2S), 132
[508210-78-4]	1-[2-(Diphenylmethyl)-5-nitrophenyl]-1-butanone, 35
[526208-17-3]	1-(2-Hydroxyphenyl)-2-methyl-5-methylene-1-decanone, 876
[526208-18-4]	1-(2-Hydroxyphenyl)-6-methylene-1-undecanone, 929
[565203-85-2]	1-(6-Methoxy-3'-methoxy[1,1'-biphenyl]-3-yl)-1-pentanone, 529
[565203-88-5]	1-(6-Hydroxy-3'-methoxy[1,1'-biphenyl]-3-yl)-1-pentanone, 529
[568553-00-4]	4-(3,4-Dipropoxyphenyl)-4-oxo-1-butanoic acid, 409
[575487-37-5]	1-(4-Hydroxy-2,5-dimethylphenyl)-1-butanone, 68
[578716-69-5]	1-[3-Acetyl-2,6-dihydroxy-4-methoxy-5-(3-methyl-2-butenyl)phenyl]- 3-methyl-1-butanone, 384
[596805-35-5]	1-[(2 <i>R</i> )-2,3-Dihydro-6-hydroxy-2-(1-methylethenyl)-5-benzofuranyl]- 3-methyl-1-butanone, 211
[596805-36-6]	1-[(2S)-2,3-Dihydro-6-hydroxy-2-(1-methylethenyl)-5-benzofuranyl]- 3-methyl-1-butanone, 211
[596805-37-7]	1-[2,4-Dihydroxy-3-(2-hydroxy-3-methyl-3-butenyl)phenyl]-3-methyl- 1-butanone, 213

[596805-39-9]	1-[2,4-Dihydroxy-5-(2-hydroxy-3-methyl-3-butenyl)phenyl]-3-methyl- 1-butanone, 214
[596805-40-2]	1-[2,4-Dihydroxy-5-(3-hydroxy-3-methyl-1-butenyl)phenyl]-3-methyl- 1-butanone, 214
[596805-43-5]	1-[2,3-Dihydro-4-hydroxy-2-(1-methylethenyl)-5-benzofuranyl]-3-methyl- 1-butanone (+), 211
[596805-44-6]	1-[2,3-Dihydro-4-hydroxy-2-(1-methylethenyl)-5-benzofuranyl]-3-methyl- 1-butanone (-), 211
[645336-90-9]	1-(3,5-Bis(1,1-dimethylethyl)-4-hydroxyphenyl)-1-heptadecanone, 1059
[647008-26-2]	1-(2,5-Dihydroxy-4-methoxy-3-methylphenyl)-1-hexanone, 653
[647008-30-8]	1-(2-Hydroxy-4-methoxy-3-methylphenyl)-1-hexanone, 652
[647008-31-9]	1-(2-Acetyloxy-4-methoxy-3-methylphenyl)-1-hexanone, 652
[654643-45-5]	3-Chloro-1-(4-methoxyphenyl)-1-butanone, 277
[658702-61-5]	1-[(2 <i>R</i> ,3 <i>S</i> )-3,4-Dihydro-5,7-dihydroxy-2-methyl-3-(3-methyl- 2-butenyl)-2-(4-methyl-3-pentenyl)-2 <i>H</i> -1-benzopyran-8-yl]- 2-methyl-1-butanone (-), 164
[658702-63-7]	1-[3,4-Dihydro-5,7-dihydroxy-2-methyl-2-(4-methyl-3-pentenyl)- 2 <i>H</i> -1-benzopyran-8-yl]-2-methyl-1-butanone (+), 151
[664376-65-2]	1-(2,4-Dihydroxy-3-methylphenyl)-3-methyl-1-butanone, 191
[664376-79-8]	1-(2,4-Dihydroxy-3-propylphenyl)-3-methyl-1-butanone, 206
[664376-82-8]	1-(3-Ethyl-2,4-dihydroxyphenyl)-3-methyl-1-butanone, 198
[664376-91-4]	1-(3-Bromo-2,4-dihydroxyphenyl)-3-methyl-1-butanone, 186
[666836-96-0]	1-[4-(2-Chloroethyloxy)phenyl]-1-decanone, 871
[666836-97-1]	1-[4-(N-Dimethylaminoethyloxy)phenyl]-1-decanone, 871
[666836-99-3]	1-(4-Chloro-2-hydroxyphenyl)-1-decanone, 879
[678184-56-0]	1-(4-Hydroxy-3-methyl-6-phenyl-2-benzofuranyl)-1-pentanone, 535
[678184-68-4]	1-[(4-Methoxymethyl)-3-methyl-6-phenyl-2-benzofuranyl)-1-butanone, 116
[678184-70-8]	1-(4-Methoxymethoxy-3-methyl-6-phenyl-2-benzofuranyl)- 1-pentanone, 536
[695196-63-5]	1-(3-Fluoro-4-hydroxyphenyl)-1-hexanone, 626
[695196-65-7]	1-(4-Phenyloxyphenyl)-1-hexanone, 605
[702701-04-0]	4-(3-Cyclohexyl-4-hydroxyphenyl)-4-oxo-1-butanoic acid, 453
[709032-85-9]	4-Cyclohexyl-1-(2,4-dihydroxyphenyl)-1-butanone, 256
[717103-49-6]	1-[2-Hydroxy-5-methoxy-4-(3-methylbutyl)phenyl]-1-octanone, 816
[718608-83-4]	1-[2-(β-D-Glucopyranosyloxy)-4,6-dihydroxyphenyl]-3-methyl- 1-butanone, 223
[718608-84-5]	1-[2,4-Bis(β-D-glucopyranosyloxy)-6-hydroxyphenyl]-3-methyl- 1-butanone, 241
[719311-19-0]	2-Bromo-1-(4-trimethylsilyloxyphenyl)-4-methyl-1-pentanone, 574
[719315-63-6]	1-(2,5-Dimethoxyphenyl)-1-heptanone, 726
[719315-64-7]	1-[2-Hydroxy-5-(phenylmethoxy)phenyl]-1-heptanone, 759
[720675-30-9]	16-(2,4-Dihydroxyphenyl)-16-oxo-1-hexadecanoic acid, 1053
[720676-31-3]	Methyl 16-(2,4-dihydroxyphenyl)-16-oxo-1-hexadecanoate, 1053
[727687-84-5]	1-(3-Bromo-2-hydroxy-5-methylphenyl)-1-butanone, 44

[727687-90-3]	2-Bromo-1-(3-bromo-2-hydroxyphenyl)-1-butanone, 269
[733016-52-9]	1-(4-Methoxyphenyl)-2-methyl-1-pentanone-2-d, 553
[749924-46-7]	2,2,3,3,4,4,4-Heptafluoro-1-(4-methoxyphenyl)-1-butanone
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[749924-49-0]	2,2,3,3,4,4,4-Heptafluoro-1-(4-phenoxyphenyl)-1-butanone
	(O-[(Trifluoromethyl)sulfonyl]oxime), 289
[750646-76-5]	2-Bromo-1-(4-hydroxyphenyl)-1-pentanone, 565
[753013-71-7]	2,4-Dibromo-1-(2,6-dimethoxyphenyl)-1-butanone, 292
[757408-19-8]	3-Methyl-1-[2,3,4-trihydroxyphenyl]-1-butanone, 181
[758691-87-1]	1-(2-Hydroxy-5-nonylphenyl)-1-butanone (Oxime), 117
[760989-23-9]	1,1'-(4,6-Dihydroxy-1,3-phenylene)bis-1-octanone, 821
[778630-63-0]	1-(3-Hydroxyphenyl)-1-octanone, 773
[778637-79-9]	1-(2-Hydroxy-4,5-dimethylphenyl)-1-octanone, 799
[778641-04-6]	1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-octanone, 806
[791065-70-8]	1-(4-Hydroxyphenyl)-1-butanone (O-Ethyloxime), 5
[791137-06-9]	1-[4-(4-Hexanoylphenyloxy)phenyl]-1-hexanone, 605
[791615-78-6]	1-(4-Phenyloxy-2-methylphenyl)-1-hexanone, 641
[791615-79-7]	1-(4-Phenyloxy-2-methylphenyl)-1-octanone, 793
[791615-80-0]	1-(2-Methyl-4-phenyloxyphenyl)-1-dodecanone, 960
[791615-81-1]	1-(2-Methyl-4-phenoxyphenyl)-1-hexadecanone, 1042
[791615-82-2]	1-(4-Phenyloxy-2-methylphenyl)-1-octadecanone, 1076
[792705-86-3]	1-[1,1'-Biphenyl]-4-yl-2-hydroxy-1-hexanone, 679
[792706-16-2]	1-(1,1'-Biphenyl)-4-yl-3-hydroxy-1-pentanone, 524
[792708-48-6]	1-[1,1'-Biphenyl]-4-yl-2-hydroxy-1-hexanone (O-Methyloxime), 679
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[792708-75-9]	1-(4'-Fluoro[1,1'-biphenyl]-4-yl)-2-hydroxy-1-hexanone, 678
[792708-89-5]	1-(4'-Bromo[1,1'-biphenyl]-4-yl)-2-hydroxy-1-hexanone, 678
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[798559-82-7]	1-[2,4-Dimethoxy-5-(2-methylpropyl)phenyl]-1-pentanone, 510
[798559-83-8]	1-[2,4-Dihydroxy-3-(hydroxymethyl)-5-(2-methylpropyl)phenyl]-
	1-pentanone, 514
[798559-84-9]	1-[2,4-Dimethoxy-3-(hydroxymethyl)-5-(2-methylpropyl)phenyl]-
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[798559-89-4]	1-[4-Hydroxy-3-(hydroxymethyl)-2-methoxy-5-(2-methylpropyl)phenyl]- 1-pentanone, 527
[798559-92-9]	2,6-Dihydroxy-3-(2-methylpropyl)-5-(1-oxopentyl)benzaldehyde, 511
[798559-94-1]	1-[2,4-Dihydroxy-5-(2-methylpropyl)phenyl]-1-pentanone, 509
[798339-94-1] [808749-86-2]	1-(2,4,6-Trimethoxy-3-methylphenyl)-2-methyl-1-butanone, 139
[808751-11-3]	1-(2-Hydroxy-4,6-dimethoxy-3,5-dimethylphenyl)-2-methyl-
[000/51-11-5]	1-butanone, 139
[808751-12-4]	1-(2-Hydroxy-4,6-dimethoxy-3,5-dimethylphenyl)-3-methyl-
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[808751-13-5]	1-(6-Hydroxy-2,4-dimethoxy-3-methylphenyl)-2-methyl-1-butanone, 138
[810661-48-4]	1-(4-Methoxy-3-methylphenyl)-1-octanone, 794
[810661-49-5]	1-(3,4-Diethyloxyphenyl)-1-octanone, 781

[811801-04-4]	1-[2-Hydroxy-3-methyl-4-(4-phenoxybutoxy)phenyl]-3-methyl- 1-butanone, 235
[811801-06-6]	1-[4-[4-(2-Fluorophenoxy)butoxy]-2-hydroxy-3-methylphenyl]-3-methyl- 1-butanone, 235
[811801-08-8]	1-[4-[4-(2,3-Difluorophenoxy)butoxy]-2-hydroxy-3-methylphenyl]- 3-methyl-1-butanone, 235
[811801-17-9]	1-[3-Bromo-2-hydroxy-4-(4-phenoxybutoxy)phenyl]-3-methyl- 1-butanone, 230
[811801-19-1]	1-[3-Bromo-4-[4-(2,3-difluorophenoxy)butoxy]-2-hydroxyphenyl]- 3-methyl-1-butanone, 230
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[817630-35-6]	5-Chloro-1-(4-hydroxy-3-methoxy-5-nitrophenyl)-1-pentanone, 573
[817630-36-7]	5-Chloro-1-(4-benzyloxy-3-methoxyphenyl)-1-pentanone, 575
[817630-37-8]	5-Chloro-1-(4-hydroxy-3-methoxyphenyl)-1-pentanone, 575
[820215-94-9]	1-(3-Ethyl-2,4-dihydroxyphenyl)-2-methyl-1-hexanone, 659
[820968-19-2]	5-Chloro-1-(4-phenoxyphenyl)-1-pentanone, 568
[838822-54-1]	4-Chloro-1-(4-methoxy-2-methylphenyl)-1-butanone, 284
[848478-63-7]	3-Chloro-1-(4-hydroxyphenyl)-1-octanone, 829
[850352-39-5]	2-Bromo-1-(3,4-dimethoxyphenyl)-1-pentanone, 566
[850352-40-8]	2-Bromo-1-(2-bromo-4,5-dimethoxyphenyl)-1-pentanone, 570
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[853913-75-4]	1-[2-(β-D-Glucopyranosyloxy)-4,6-dihydroxyphenyl]-1-pentanone, 527
[854460-42-7]	1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-2-methyl-1,3-butanedione, 330
[854465-70-6]	1-[3,4-Dihydro-7-methoxy-5-(phenylmethoxy)-2,2-dimethyl- 2 <i>H</i> -1-benzopyran-8-yl]-3-methyl-1-butanone, 240
[854465-73-9]	1-[3,4-Dihydro-7-hydroxy-5-(phenylmethoxy)-2,2-dimethyl- 2 <i>H</i> -1-benzopyran-8-yl]-3-methyl-1-butanone, 240
[854659-09-9]	6-(4-Ethoxyphenyl)-6-oxo-1-hexanoic acid, 712
[854659-33-9]	1-(2,4-Dimethoxyphenyl)-1-pentanone, 471
[854659-36-2]	1-[1,1'-Biphenyl]-5-yl-2-methoxy-1-pentanone, 524
[854676-84-9]	4-(5-Bromo-2,4-dihydroxyphenyl)-4-oxo-1-butanoic acid, 421
[854677-51-3]	4-(2-Hydroxy-3-methylphenyl)-4-oxo-1-butanoic acid, 432
[854677-84-2]	4-(2-Amino-4,5-dimethoxyphenyl)-4-oxo-1-butanoic acid, 429
[854679-09-7]	4-(2,6-Dihydroxy-4-methylphenyl)-4-oxo-1-butanoic acid, 438
[854866-87-8]	4-Cyclohexyl-1-(2-hydroxy-4,6-dimethylphenyl)-1-butanone, 259
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[854870-33-0]	1-[4-Methoxy-2-methyl-5-(1-methylethyl)phenyl]-1-butanone, 91
[854870-93-2]	1,1'-(2-Methoxy[1,1'-biphenyl]-5-yl)-1-butanone, 260
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[855153-59-2]	Methyl 5-(2,5-dimethoxyphenyl)-5-oxo-1-pentanoate, 581
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[855605-97-9]	1-(2,5-Dimethoxyphenyl)-1-pentadecanone, 1027
[855620-89-2]	4-Cyclohexyl-1-(2-hydroxy-3,4-dimethylphenyl)-1-butanone, 258
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[855899-90-0]	1-(2-Hydroxy-4,6-dimethylphenyl)-1-heptanone, 744
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[855954-88-0]	1-(2-Hydroxy-4-methoxy-3,6-dimethylphenyl)-1-heptadecanone, 1058
[855955-03-2]	1-(3,4,5-Trimethoxyphenyl)-1-heptadecanone, 1056
[855956-24-0]	1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-heptanone, 750
[855956-25-1]	1-(3,4-Dimethoxyphenyl)-1-hexadecanone, 1037
[856348-12-4]	1-(2-Hydroxy-4,5-dimethoxyphenyl)-1,3,5-hexanetrione, 645
[856349-95-6]	1-(2-Hydroxy-4,5-dimethylphenyl)-1-hexanone, 649
[856349-97-8]	1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-hexanone, 665
[856360-21-9]	1-(2,4-Dihydroxyphenyl)-1-docosanone, 1104
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[856809-73-9]	4-(3,4-Dibutoxyphenyl)-4-oxo-1-butanoic acid, 410
[856809-86-4]	4-(2,5-Dimethoxy-4-methylphenyl)-4-oxo-1-butanoic acid, 437
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[857229-77-7]	4-(5-Chloro-2-methoxy-4-methylphenyl)-4-oxo-1-butanoic acid, 432
[857480-74-1]	6-(5-Chloro-2-hydroxyphenyl)-6-oxo-1-hexanoic acid, 714
[857803-59-9]	1-(2-Methoxyphenyl)-3-methyl-1-butanone, 174
[857973-71-8]	1-(4-Heptyl-2-hydroxy-5-methoxyphenyl)-4-methyl-1-pentanone, 552
[858189-64-7]	10-(2,4-Dihydroxyphenyl)-10-oxo-1-decanoic acid, 919
[858189-97-6]	Ethyl 10-(2,5-dihydroxyphenyl)-10-oxo-1-decanoate, 920
[858445-94-0]	Ethyl 4-(2,4-dimethoxyphenyl)-4-oxo-1-butanoate, 404
[859059-36-2]	4-(3,4-Diamyloxyphenyl)-4-oxo-1-butanoic acid, 410
[859080-81-2]	1-(2,4-Dimethoxyphenyl)-4-phenyl-1,3-butanedione, 354
[859310-32-0]	1-(4-Chloro-2-hydroxyphenyl)-1-dodecanone, 955
[859742-49-7]	1,10-Bis(3,4-dimethoxyphenyl)-1,10-decanedione, 903
[859786-47-3]	1-[2-Hydroxy-6-methyl-3-(1-methylethyl)phenyl]-1-pentanone, 508
[859939-56-3]	1-(2,4-Dihydroxy-5-ethylphenyl)-1-butanone, 69
[859947-01-6]	1-(4-Ethyloxyphenyl)-1-tetradecanone, 1005
[859968-25-5]	1-(3-Bromo-4-methoxyphenyl)-1-pentanone, 480
[859992-51-1]	1-(2-Hydroxy-5-methylphenyl)-1-octadecanone, 1075
[859994-77-7]	Ethyl 10-(2,4-dihydroxyphenyl)-10-oxo-1-decanoate, 919
[859995-51-0]	1-(3-Hydroxyphenyl)-1-nonanone, 836
[860189-36-2]	Ethyl 4-(2-hydroxy-4-(phenylmethoxy)phenyl)-2,4-dioxo-1-butanoate, 455
[860705-13-1]	1,4-Bis(2-hydroxy-3,4,6-trimethylphenyl)-1,4-butanedione, 372
[861310-96-5]	2-Bromo-1-(3-bromo-6-hydroxy-2,4-dimethylphenyl)-1-butanone, 273
[861349-33-9]	1-(5-Chloro-2-methoxyphenyl)-1,3-butanedione, 316
[861778-02-1]	1-(2-Hydroxy-4,6-dimethylphenyl)-1,3-butanedione, 322
[861889-69-2]	1-(3,5-Dichloro-2,4,6-trimethoxyphenyl)-1-hexanone, 622
[861889-70-5]	1-(3-Bromo-2,6-dihydroxy-4-methoxyphenyl)-1-hexanone, 635
[861889-71-6]	1-(2,6-Dihydroxy-3,5-diiodo-4-methoxyphenyl)-1-hexanone, 632

[861889-72-7]	1-(2,6-Dihydroxy-3-iodo-4-methoxyphenyl)-1-hexanone, 638
[861889-73-8]	1-(3,5-Dichloro-2,6-dihydroxy-4-phenoxyphenyl)-1-hexanone, 672
[861889-74-9]	1-(3-Chloro-2,6-dihydroxy-4-phenoxyphenyl)-1-hexanone, 678
[861889-76-1]	1-(2,6-Dihydroxy-4-methoxyphenyl)-1-heptanone, 740
[861889-77-2]	1-(2,6-Dihydroxy-4-methoxyphenyl)-1-octanone, 797
[861889-78-3]	1-(2,6-Dihydroxy-4-methoxyphenyl)-3,3-dimethyl-1-butanone, 254
[861889-80-7]	1-(4-Ethoxy-2,6-dihydroxyphenyl)-1-hexanone, 653
[861889-81-8]	1-(4-Butoxy-2,6-dihydroxyphenyl)-1-hexanone, 666
[861889-82-9]	1-[4-(Cyclopentyloxy)-2,6-dihydroxyphenyl]-1-hexanone, 668
[861889-83-0]	1-(3,5-Dichloro-2,6-dihydroxy-4-methoxyphenyl)-3,3-dimethyl- 1-butanone, 253
[861889-85-2]	1-(3,5-Dichloro-4-ethoxy-2,6-dihydroxyphenyl)-1-hexanone, 646
[861889-86-3]	1-(4-Butoxy-3,5-dichloro-2,6-dihydroxyphenyl)-1-hexanone, 662
[861889-87-4]	1-[3,5-Dichloro-4-(cyclopentyloxy)-2,6-dihydroxyphenyl]-1-hexanone, 667
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[861889-89-6]	1-(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)-1-heptanone, 737
[861889-90-9]	1-(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)-1-octanone, 790
[861889-91-0]	1-(3-Chloro-2,6-dihydroxy-4-methoxyphenyl)-3,3-dimethyl- 1-butanone, 253
[861889-93-2]	1-(3-Chloro-4-ethoxy-2,6-dihydroxyphenyl)-1-hexanone, 648
[861889-94-3]	1-(4-Butoxy-3-chloro-2,6-dihydroxyphenyl)-1-hexanone, 664
[861889-95-4]	1-[3-Chloro-4-(cyclopentyloxy)-2,6-dihydroxyphenyl]-1-hexanone, 668
[862666-31-7]	1-(2,3-Dimethoxyphenyl)-1-nonanone, 838
[862666-32-8]	1-(2,3-Dimethoxyphenyl)-1-undecanone, 925
[862666-33-9]	1-(2,3-Dihydroxyphenyl)-1-pentanone, 470
[862666-34-0]	1-(2,3-Dihydroxyphenyl)-1-hexanone, 606
[862666-35-1]	1-(2,3-Dihydroxyphenyl)-1-heptanone, 725
[862666-36-2]	1-(2,3-Dihydroxyphenyl)-1-octanone, 778
[862666-37-3]	1-(2,3-Dihydroxyphenyl)-1-nonanone, 838
[862666-38-4]	1-(2,3-Dihydroxyphenyl)-1-undecanone, 925
[862666-39-5]	1-(2,3-Dihydroxyphenyl)-1-tridecanone, 996
[864072-49-1]	1-(4-Hydroxyphenyl)-1-pentanone (Oxime), 466
[867134-00-7]	6-(4-Ethoxy-3-nitrophenyl)-6-oxo-1-hexanoic acid, 715
[867213-74-9]	11-Bromo-1-(4-methoxyphenyl)-1-undecanone, 934
[868075-01-8]	1-(3-Chloro-4-methoxyphenyl)-1-butanone, 30
[868521-08-8]	6-Bromo-1-(4-hydroxy-3-methylphenyl)-1-hexanone, 706
[870084-45-0]	5,7-dihydroxy-6-(3-methyl-2-butenyl)-8-(2-methyl-1-oxobutyl)- 4-phenyl-2 <i>H</i> -1-benzopyran-2-one, 160
[871882-61-0]	2-Chloro-1-(2-hydroxy-5-methylphenyl)-1-butanone, 276
[871882-79-0]	3-Chloro-1-(2-hydroxy-5-methylphenyl)-1-butanone, 277
[871886-71-4]	2-Chloro-1-(2-hydroxy-5-methylphenyl)-3-methyl-1-butanone, 294
[871901-16-5]	1-(3,4-Dimethoxyphenyl)-1-tetradecanone, 1008
[872178-00-2]	1-[2,4,6-Trihydroxy-3-(3-methyl-2-butenyl)phenyl]-1-dodecanone, 974
[873380-96-2]	1-(4-Hydroxy-3,5-dimethoxyphenyl)-3-methyl-1-butanone, 199
[873396-80-6]	4-(2-Ethoxy-5-methylphenyl)-4-oxo-1-butanoic acid, 435

[873416-42-3]	1-(2-Hydroxy-5-methoxy-3-pentylphenyl)-1-octanone, 815
[873989-36-7]	1-(2-Hydroxy-3,5-dimethylphenyl)-1-butanone, 66
[874487-28-2]	4-(5-Cyclopentyl-2-methoxyphenyl)-4-oxo-1-butanoic acid, 451
[874507-02-5]	1-(2-Hydroxy-4,6-dimethylphenyl)-1-tetradecanone, 1014
[875850-72-9]	1-(2-Hydroxy-3,4,6-trimethoxyphenyl)-1,3-butanedione, 328
[875854-88-9]	1,1'-(2,6-Dihydroxy-4-methyl-1,3-phenylene)bis-1-butanone, 299
[876511-19-2]	1-(2-Hydroxy-5-methoxyphenyl)-3-methyl-1-butanone, 193
[877877-92-4]	1-[2,3,4-Trihydroxy-5-(1-methylethyl)phenyl]-1-undecanone, 931
[877877-96-8]	1-[2,3,4-Trihydroxy-5-(1-methylethyl)phenyl]-1-pentadecanone, 1029
[877877-98-0]	1-[2,3,4-Trihydroxy-5-(phenylmethyl)phenyl]-1-undecanone, 933
[877878-00-7]	1-[2,3,4-Trihydroxy-5-(1-phenylundecyl)phenyl]-1-undecanone, 934
[883566-08-3]	1-(2-Hydroxy-3,5-diiodo-4-methylphenyl)-1-butanone, 42
[883566-09-4]	1-(5-Chloro-2-hydroxy-3-iodophenyl)-1-butanone, 23
[883566-10-7]	1-(5-Bromo-2-hydroxy-3-iodophenyl)-1-butanone, 20
[883566-11-8]	1-(2-Hydroxy-5-iodo-3-methylphenyl)-1-butanone, 46
[883566-12-9]	1-(2-Hydroxy-3,5-diiodo-4,6-dimethylphenyl)-1-butanone, 61
[888026-39-9]	1-(2,4,6-Trimethoxyphenyl)-1-dodecanone, 952
[888489-66-5]	Myristinin A (+), 990
[903883-85-2]	1-[3,5-Bis(1,1-dimethylethyl)-4-acetyloxyphenyl]-1-pentanone, 535
[905590-12-7]	1-(4-Ethoxy-3-propylphenyl)-4-oxo-1-butanoic acid, 447
[909191-71-5]	1-(2-Hydroxy-4-methylphenyl)-1-octadecanone, 1075
[909255-15-8]	1-(3-Amino-2,4-dihydroxyphenyl)-3-methyl-1-butanone, 187
[909255-16-9]	1-(3-Amino-5-bromo-2,4-dihydroxyphenyl)-3-methyl-1-butanone, 187
[910457-86-2]	1-(3,5-Dichloro-2,6-dihydroxyphenyl)-1-octanone, 786
[916895-75-5]	1-(3-Chloro-2,4,6-trimethoxyphenyl)-1-hexanone, 626
[918814-59-2]	2,4-Dihydroxy-3-(3-methyl-1-oxobutyl)-6-(2-propen-1-yloxy) benzaldehyde, 392
[918814-60-5]	1,1'-[2,4-Dihydroxy-6-(2-propen-1-yloxy)-1,3-phenylene]bis-3-methyl-
[710014-00-5]	1-butanone, 305
[918814-63-8]	2,4-Dihydroxy-6-(3-methylbutoxy)-3-(3-methyl-1-oxobutyl)-1-butanone,
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